AMERICAN VACUUM SOCIETY

41st National Symposium

NANO III: Third International Conference
ON NANOMETER SCALE SCIENCE & TECHNOLOGY

Colorado Convention Center
Denver, Colorado
October 24-28, 1994

FINAL PROGRAM
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## SYMPOSIUM LOCATION

<table>
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<tr>
<th>Location</th>
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<tr>
<td>Colorado Convention Center</td>
<td>700 14th Street</td>
<td>303/640-8000</td>
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<tr>
<td>Denver, CO 80202</td>
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## SYMPOSIUM REGISTRATION HOURS

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<tr>
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<tr>
<td>Sun</td>
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<tr>
<td>Mon</td>
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</tr>
<tr>
<td>Tues</td>
<td>7:30 a.m. to 6:00 p.m.</td>
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<tr>
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<td>7:30 a.m. to 5:00 p.m.</td>
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<tr>
<td>Thurs</td>
<td>7:30 a.m. to 5:00 p.m.</td>
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<tr>
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## SHORT COURSE REGISTRATION HOURS

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<td>Tu-Th</td>
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## SYMPOSIUM REGISTRATION FEES

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<tr>
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AVS tax ID Number: 04-2392373

*A bonafide full time university student must present student ID. Part-time students and postdoctoral fellows do not qualify for a student rate.

The PROCEEDINGS from this Symposium will be published in the May/June issue of JVST. Active AVS Members in 1995 will receive the PROCEEDINGS.

## EXHIBIT SHOW HOURS

<table>
<thead>
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<td>Tuesday, Oct. 25</td>
<td>11:00 a.m. to 6:00 p.m.</td>
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<td>Wednesday, Oct. 26</td>
<td>10:00 a.m. to 5:00 p.m.</td>
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<td>Thursday, Oct. 27</td>
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## OFFICE LOCATIONS AND PHONE NUMBERS

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<thead>
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<tr>
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<td>Short Course Registration</td>
<td>C 111</td>
<td>303/446-4231</td>
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<td>Message Board</td>
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<td>303/446-4231</td>
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<tr>
<td>Local Arrangements</td>
<td>A 214</td>
<td>303/446-4232</td>
</tr>
<tr>
<td>Headquarters</td>
<td>Lobby Area</td>
<td>303/446-4232</td>
</tr>
<tr>
<td>Education Center</td>
<td>Lobby Area</td>
<td>303/446-4232</td>
</tr>
<tr>
<td>Companions' Headquarters</td>
<td>Silver Room (H)</td>
<td>303/446-4232</td>
</tr>
<tr>
<td>Companions' Registration</td>
<td>Lobby Area</td>
<td>303/446-4232</td>
</tr>
<tr>
<td>(S.Mo.Tu Only)</td>
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<tr>
<td>AVS Employment Center</td>
<td>Lobby Area</td>
<td>303/446-4232</td>
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<td>JVST Editorial Office</td>
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<td>Publicity/Press Room</td>
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NANO 3: Third International Conference on Nanometer-Scale Technology

Richard Colton
Christie Marrian

American Vacuum Society
120 Wall Street
New York, New York 10005

U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

Approved for public release; distribution unlimited.

Greetings

Welcome to Denver! On behalf of the Program Committee we are pleased to present this Program for the 41st National Symposium of the American Vacuum Society. The Symposium, comprising technical sessions of the Society's eight divisions, three Topical Conferences, the short course program, and the Equipment Exhibition, is being held in the Colorado Convention Center. Many of the business functions are held in the headquarters hotel, the Radisson, located a few blocks from the Convention Center.

Both the Convention Center and the Radisson are located in the heart of downtown Denver, ideally situated to take advantage of the many cultural, historic, dining, and shopping attractions of the city. We think you will find Denver a convenient and exciting city to hold the AVS National Symposium.

Equally exciting is the technical program that we have assembled for this year. Ninety-two technical sessions and eighteen poster sessions make this the largest technical program yet offered by the AVS. Approximately one thousand papers selected from over 1300 abstracts are included in the meeting. In the tradition of the AVS, each Division offers a broad range of topical material and a selection of invited speakers representing the best current work in their fields.

There is always a large overlap of interest among division programs, and this year we are attempting a new means of coordinating these interdisciplinary interests. We have identified four Focus Areas within the program. These represent either major new topical areas that are being introduced to the program, or areas of where a common thread of interest runs through a number of divisions. We have scheduled Focus Area sessions in a sequential fashion through the week.

The four Focus Areas for this meeting are 1) Si-Based Optoelectronics; 2) Sensors, in-situ Diagnostics, and Process Control; 3) Nanostructure Fabrication and Atomic Scale Manipulation of Surfaces; 4) Surface Contamination and Control.

Focus Area sessions are highlighted in the Program at a Glance and also in the "Calendar of Events."

Three Topical Conferences are also featured at this year's meeting. NANO 3, the Third International Conference on Nanometer-Scale Science and Technology is being sponsored by the Nanometer-Scale Science and Technology Division. This program is designed to provide a forum for the science and technology of phenomena in many diverse fields which are apparent on the nanometer scale. NANO 3 also offers a special plenary session on Monday morning, and a Monday evening reception for NANO 3 attendees. Two additional topical conferences are making repeat appearances after their enthusiastic reception at last year's meeting. The Manufacturing Science and Technology Topical Conference continues our efforts to provide the research community with perspective and familiarity with manufacturing issues that present opportunities for research and to highlight AVS research that delivers or promises substantial relevance to manufacturing. The conference is again co-sponsored by the IEEE. The Biomaterials Interfaces Topical Conference provides a forum where biotechnologists and interface scientists can share their perspective views on the research and development opportunities with biomaterials.

Monday at 12:45 p.m. features the Plenary Address for this year's meeting. Our speaker is Dr. John Armstrong, Vice President, Science and Technology of IBM (retired). Dr. Armstrong's address titled, "The Role of Research in the Technological Balance of Power," examines the needs and directions of science and technology in an increasingly international economy and what contributions research can make in this new economic environment.

Wednesday evening occasions the annual AVS Awards Assembly and Reception. All Symposium registrants are welcome to attend and congratulate this year's AVS award winners.

As always, the Manufacturers Exhibit is a major part of the AVS Symposium. The Exhibit is held Tuesday through Thursday in the Convention Center. A special poster session on New Vacuum Products is scheduled for Wednesday at 5:00 p.m. in Ballroom 4 of the Convention Center.

The Convention Center is also the site of the Short Course Program. These courses run concurrently with the AVS Symposium.

The Local Arrangements Committee, chaired by Pete Sheldon will be available to assist you throughout the week. For our traveling companions, a full and interesting Companions Program is available to take advantage of the plentiful and unusual entertainment, recreational, and cultural activities available in the Denver area.

Thomas M. Mayer
Program Chair

Ellen B. Stechel
Program Vice Chair
General Information

INTRODUCTORY MEMBERSHIP OFFER

If you have paid the $295 or $335 non-member registration fee, we invite you to apply for membership through our Introductory Membership Invitation. Non-members who apply and are accepted for AVS membership during the Symposium may have $60 of their non-member registration fee transferred and credited to their 1995 membership dues. Membership application forms are available at the registration desk and the Education Center.

Completed applications must be submitted to the Education Center staff for processing during THE WEEK OF THE SYMPOSIUM ONLY.

TRANSPORTATION INFORMATION

16th STREET SHUTTLE SERVICE - Getting around Denver is easy and convenient. Once downtown, you will find the shuttles on the 16th Street Mall to be free as well as fast, running between the Lower Downtown Historic District in the northwest and Civic Center Park and the State Capitol in the southeast approximately every 90 seconds.

Most of the hotels listed above are located within one or two blocks of the 16th Street Shuttle. The stop for the Convention Center is California and 16th, which is only two blocks from the Convention Center.

FOURTEENTH AVS RUN

The Fourteenth Annual AVS Run will be held on Wednesday, October 26th. A map identifying the starting point will be provided at the race registration desk. The race site will be along the Platte River.

The run donation is $15.00 US (late fee after October 3). This includes a high-quality, commemorative tee-shirt, race numbers, awards (in many age categories) and post-race refreshments. All runners, competitive or a bit less serious, are invited to participate.

The CORPORATE DIVISION RACE will again be held. Each team needs at least three members, all working for the same organization. Contact the race director to register teams.
A) Colorado Convention Center
B) Hyatt Regency Denver
C) Holiday Inn Downtown
D) Radisson Hotel (Headquarters)
E) Westin Hotel
F) Embassy Suites Hotel
G) Executive Tower Inn
H) The Comfort Inn

CREDITS:
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Abstract Editor – Lynn Pizzo

Cover Image:
Domain structure of a two dimensional Ag-Cu alloy,
Courtesy of Bob Hwang, Sandia National Labs
Livermore, CA

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M. LIEHR, CO-CHAIR
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A. Diebold, Sematech
F.R. Dill, IBM
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J. McVittie, Stanford University
N. Peacock, HPS Div of MKS Instr
M. Kushner, Univ of Illinois
C. Sorenson, SEMI/Sematech
C. Spanos, Univ of California, Berkeley
A. Strojwas, Carnegie Mellon University
A. Testoni, Digital Equipment Corp
K. Uram, LAM Research
C. Whitman, CVC Products Inc.

Topical Conference:
Biomatieral Interfaces
J.J. HICKMAN, CO-CHAIR
SAIC
B.D. RATNER, CO-CHAIR
University of Washington
### AMERICAN VACUUM SOCIETY AWARD WINNERS

#### MEDARD W. WELCH AWARD

<table>
<thead>
<tr>
<th>Year</th>
<th>Name</th>
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<td>Gottfried K. Wehner</td>
<td>1979</td>
<td>Gert Ehrlich</td>
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<td>Kenneth C.D. Hickman</td>
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<td>Harrison E. Farnsworth</td>
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<td>1973</td>
<td>Lawrence A. Harris</td>
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<td>H.H. Wieder</td>
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<td>Homer D. Hagstrum</td>
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<td>William S. Spicer</td>
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<td>Paul A. Redhead</td>
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<td>John O’Hanlon</td>
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#### GAEDE-LANGMUIR AWARD

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#### ALBERT NERKEN AWARD

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#### PETER MARK AWARD

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#### HONORARY MEMBERSHIP

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#### JOHN THORNTON MEMORIAL AWARD

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#### NELLIE YEOH WHETTEN AWARD

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#### RUSSELL AND SIGURD VARIAN FELLOW AWARD

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<td>Anne L. Testoni (Northwestern University)</td>
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<td>Daniel Kelly (University of California, Santa Barbara)</td>
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THE AMERICAN VACUUM SOCIETY

cordially invites you
to attend
the Awards Assembly
on
Wednesday, October 26, 1994
in
Ballroom 1
Colorado Convention Center
at 6:45 p.m.
followed by the
Awards Reception
AWARDS ASSEMBLY & RECEPTION

The American Vacuum Society Awards Assembly and Reception will be held on Wednesday, October 26, 1994 at 6:45 p.m. in Ballroom I of the Colorado Convention Center. The Medard W. Welch Award will be presented to John T. Yates, Jr. The Gaede-Langmuir Award will be presented to Robert J. Celotta and Daniel T. Pierce. The Albert Nernst Award will be presented to Hajime Ishimaru. The John A. Thornton Memorial Award will be presented to David W. Hoffman, and the Peter Mark Award will be presented to Marjorie Olmstead. The newly elected Fellows as well as the Graduate Research Awards for 1994 will be presented. The winners of the award for the best Shop Notes published in the Journal of Vacuum Science and Technology will be announced. The Awards Reception will immediately follow the Awards Assembly.

MEDARD W. WELCH AWARD

The Medard W. Welch Award was established in 1969 to commemorate the pioneering efforts of M.W. Welch in founding and supporting the American Vacuum Society. It is presented to recognize and encourage outstanding research in the fields of interest to the American Vacuum Society. The award consists of a medal and a cash prize.

JOHN T. YATES, JR.

Dr. John T. Yates, Jr., University of Pittsburgh, "for the development and use of modern measurement methods to provide insights into the behavior of chemisorbed species on metal and semiconductor surfaces."

John T. Yates, Jr. received his B.S. degree from Juniata College and his Ph.D in physical chemistry from MIT. Following a three-years’ term as Assistant Professor at Amherst College, he joined the National Bureau of Standards, first as a NRC Postdoctoral Research Fellow and then, from 1965 until 1982, as a member of its scientific staff. His research is in the fields of surface chemistry and physics, including both the structure and spectroscopy of surface species, the dynamics of surface processes, and the development of new methods for research in surface chemistry.

Dr. Yates was Senior Visiting Scholar at the University of East Anglia, Norwalk, in 1970-1971, and Sherman Fairchild Distinguished Scholar at Caltech in 1977-1978.

Professor Yates joined the University of Pittsburgh in 1982 as the first R.K. Mellon Professor of Chemistry and as the Director of the new University of Pittsburgh Surface Science Center. He has established the Surface Science Center and has created a strong research program there. Here, working with students and postdoctoral staff, his influence actively extends over a wide range of research projects. He is also active in undergraduate and graduate teaching. In addition, he maintains close relationships with a number of surface science research programs in academic, government, and industrial research laboratories throughout the world, as well as serving on the editorial boards of five journals and two book series in surface science and catalysis. He is a member of the Program Selection Committee of the Gordon Research Conference. He is a coeditor of the ACS journal, Langmuir.

ROBERT J. CELOTTA

Dr. Robert J. Celotta, NIST, "for their innovative development of advanced, spin-polarized electron beam technology and their scientific contributions to atomic surface, and microstructure physics."

GAEDE-LANGMUIR AWARD

The Gaede-Langmuir Award was established in 1977 by an endowing grant from Dr. Kenneth C.D. Hickman. It is presented biennially to recognize and encourage outstanding discoveries and inventions in the sciences and technologies of interest to the American Vacuum Society. The award consists of an inscribed plaque, certificate and cash prize.
Robert Celotta received his B.S. degree in Physics from the City College of New York in 1964 and went on to get his Ph.D. in Physics at New York University in 1969. His research career actually started while he was an undergraduate working part time as a Research Assistant at IBM Watson Labs (at Columbia University) on the construction of high current cyclotron conceived by L.H. Thomas. His graduate work at NYU included atomic beam determinations of tensor polarizabilities and electron-atom collision cross sections under the supervision of Ben Bederson, currently the Editor-in-Chief at APS. Celotta then accepted a Postdoc with Jan Hall at the Joint Institute of Laboratory Astrophysics in Boulder Colorado to establish a new way to accurately determine molecular electron affinities via laser photodetachment spectrometry.

In 1971, Celotta joined the Electron Physics Group at NIST (then the National Bureau of Standards) and led an effort in monochromatic electron scattering from gases, primarily determining oscillator strengths over a very wide spectral range and exploring negative ion resonance phenomena. Then, in 1975, Dan Pierce, fresh from participating in the discovery at ETH in Zurich that GaAs could be made to emit spin polarized electrons, joined Bob Celotta on the staff of the Electron Physics Group. Thus began a period of scientific collaboration and friendship that continues to the present day. They shared the conviction that a great deal could be learned about atoms and surfaces if the electron spin parameter could be controlled and measured much more effectively than had been to date. Together with their colleagues, Pierce and Celotta set out to develop the first low energy GaAs spin polarized electron gun. This electron gun produced beams similar in current and other characteristics to conventional electron guns, yet the spin direction of the electron beam could be modulated while the current remained constant. This allows the spin dependent part of any subsequent electron interaction to be determined. The source was first used to study the spin-orbit effect in spin polarized LEED and then observed spin splittings in electron surface states. But, by 1979, they realized that surface magnetism was the most exciting application the new spin polarization techniques and they had made their first observation of a surface hysteresis curve by using polarized electron diffraction. In the years immediately following, they proposed the transverse magnetization configuration for spin-dependent photoemission, observed the effect of electron polarizaton on elastic and inelastic electron scattering from ferromagnets, observed the spin and energy of secondary electrons from ferromagnets, utilized the detection of polarized electrons in scanning electron microscopy (SEMPA) to provide images of magnetic domains, made spin polarized, inverse photoemission measurements, determined the temperature dependence of surface magnetization, and developed two new types of spin polarization detectors. Starting in the early 1980's, Celotta also led a project at NIST to measure the interactions between polarized electrons and spin polarized atom beams, in some cases making such complete scattering measurements that quantum phases and amplitudes were obtainable instead of less complete cross section information.

Currently, Celotta and his colleagues use polarization techniques in, for example, SEMPA studies of exchange coupling between magnetic layers using nanofabrication methods to produce wedge shaped crystal structures of accurate dimensions and precise structure. He is also interested in the possibility of a spin-dependent tunneling probe of magnetic structure.

Most recently, Celotta and colleagues have demonstrated that laser fields can be used to focus neutral atomic beams to form nanostructures on surfaces.

Polarized electron experiments are notoriously complex and much of the work mentioned above could not have been done without the collaborative efforts of many highly talented scientists, including former and present members of the NIST Electron Physics Group, and the highly supportive environment provided by NIST.

Robert Celotta is an author of more than 200 publications, is the Editor-in-Chief of Methods of Experimental Physics, and is a holder of three patents ranging from polarization detection to nanolithography. He has received the Distinguished Young Scientist Award from the Maryland Academy of Sciences, two IR-100 awards, the Federal Laboratory Consortium Award for Excellence in Technology Transfer, the Outstanding and Distinguished Career in Science Award of the Washington Academy of Sciences and, from NIST, the Silver Medal and Gold Medal Awards, the E.U. Condon Award, and the W.P. Slichter Award. He is a NIST Fellow, a Fellow of the American Physical Society and is the Leader of the NIST Electron Physics Group.

Daniel T. Pierce

Dr. Daniel T. Pierce, NIST. "for their innovative development of advanced, spin-polarized electron beam technology and their scientific contributions to atomic, surface, and microstructure physics".

Daniel Pierce received his B.S. degree in Physics from Stanford University in 1962. After two years teaching physics at Tri-Chandra College in Kathmandu, Nepal with the U.S. Peace Corps, he returned to Wesleyan University in Connecticut where he received an M.A. in Physics in 1966. He received his Ph.D in Applied Physics from Stanford University in 1970, for work involving photoemission measurements of the electronic structure of Ni above and below the Curie temperature and calculations of the corresponding energy distribution curves from an interpolated band structure. He remained with Prof. W.E. Spicer at Stanford for a year as a postdoctoral researcher to study amorphous semiconductors and help develop plans for the Stanford Synchrotron Radiation Laboratory.

In 1971, Dr. Pierce joined the group of
ALBERT NERKEN AWARD

The Albert Nerken Award was established in 1984 by Veeco Instruments, Inc. in recognition of its founder, Albert Nerken, as a founding member of the American Vacuum Society, his early work in the field of high vacuum and leak detection, and his contributions to the commercial development of the instrumentation. It is presented to recognize outstanding contributions to the solution of technological problems in areas of interest to the American Vacuum Society. The award consists of a cash prize and a certificate.

HAJIME ISHIMARU

Dr. Hajime Ishimaru, National Laboratory for High-Energy Physics, Japan, "for his unique contributions to the development and characterization of aluminum alloys for use in ultra-high vacuum environments."

Hajime Ishimaru was born in 1940 in Sapporo, Japan and graduated high school in Sapporo. He received a B.S. degree in physics from Hokkaido University in 1963 and an M.S. in nuclear physics from Tohoku University in 1965. In 1970, he received the SciDr in plasma physics from Nagoya University where his research was in energy-mass analysis for highly ionized steady state plasma. Dr. Ishimaru received the EngDr from Tokyo University in 1980, working in the areas of vacuum feedthroughs-coaxial, multi-pin/coaxial...
multi-pin complex vacuum feedthroughs in ultrahigh vacuum and cryogenic.

From 1969 to 1972, he worked at the Tokyo University as Research Associate on diagnosis of the plasma and molecular beam. In 1972, Dr. Ishimaru joined the National Laboratory for High Energy Physics in Tsukuba, Japan. He currently heads the TRISTAN Vacuum Group, an aluminum constructed electron-position collider beam storage ring and the B Factory under construction. He directed the construction of the beam transport line of the proton linear accelerator, beam monitoring and operation of the proton synchrotron, development of the all aluminum alloy vacuum system, and construction of TRISTAN e⁺e⁻ large storage accelerator. As Professor, he also manages the research of Ph.D Graduate students for Accelerator Science, The Graduate University for Advanced Studies in Japan.

A major contribution of Dr. Ishimaru was in aluminum vacuum technology where he pioneered the development of all aluminum ultrahigh vacuum materials, components, and systems for vacuum in the low 10⁻¹⁰ Torr region. In 1977, he discovered the residual radioactivity in aluminum decays with half-life as short as 15 hours and the first to have experimental data on the decay of the residual radioactivity of vacuum material. He then embarked upon developing techniques for surface treatment of aluminum. Initially, EX-mirrored finished was developed by enclosing the working portion of a lathe in a larger vacuum chamber under a controlled atmosphere of oxygen and argon gases. A diamond tipped tool was used to cut the inner surface of high purity aluminum producing a new oxide layer of less than 30 Å thick. Other surface treatments developed were EX-extrusion, EX-process, and EX-GBB process. Outgassing rates for these surface treatments after a 24 hour bakeout at 150°C was in the low 10⁻¹⁴ Torr L/s cm². Dr. Ishimaru developed special welding techniques for aluminum by TIG for flanges, fittings, vacuum vessels, and electron beam welding for feedthroughs and bellows for very small weld seams.

To complete this new aluminum technology, Ishimaru has developed an all aluminum conflat flange, titanium carbide coated knife-edge together with a re-usable aluminum gaskets and high strength aluminum nuts and bolts. He developed an aluminum single structure bellows for connecting to the beam line chamber for baking and adsorption of mechanical distortions and for the tolerance against "melt-down" by the particle beam. Other developments included aluminum metal seal right angle valves, double metal-seal gate valves, optical windows, feedthroughs, manipulators, non-water cooling vacuum furnace, and a portal vacuum suitcase using very light weight aluminum alloy honeycombed panels. Also, a turbomolecular pumps was developed by cooling the rotor with ceramic coating to achieve vacuum in the low 10⁻¹² Torr region. Sputter ion pumps and titanium sublimation pumps were also made from aluminum alloys. With the different aluminum components and pumps, an extremely high vacuum system of the order of 10⁻¹⁵ Torr was developed using an aluminum alloy quadrupole mass filter. A very fast pump-down system was also developed employing special moisture traps to achieve vacuum of 10⁻⁸ Torr region in less than 150 seconds.

Dr. Ishimaru is a Technical Consultant to Korea POSTECH 2 GeV synchrotron radiation source; DAONE Collider Project for INFN Frascati, Italy; Taiwan 1.5 GeV Synchrotron; Spring-8, Japan; Space Shuttle's "AKE" Project in collaboration with the Space Epitaxy Center in Houston University, and SSC Main Collider Beam Tube/HEB (now terminated). He was also instrumental in the design and development of a 40 m L-shaped aluminum alloy chamber and vacuum tank with laser beam reflecting mirror system for the National Astronomical Institute in Japan.

His work has been very extensively cited by others in the high vacuum field. He has authored and co-authored over 185 papers in vacuum journals and conference proceedings and four technical book publications. Dr. Ishimaru has 40 registered patents and over 100 patents pending. He is a Member of AVS, Japan Society of Vacuum, the Japan Society of Applied Physics, Japan Institute of Light Metals, and Member of the Editorial Advisory Board for "VACUUM," a British Vacuum Journal.

Dr. Ishimaru received numerous awards and honors for his work. In 1978, he received the Award of Excellence, Best Shop Note by the American Vacuum Society for the first bakeable metal-seal aluminum alloy flange system. Since it was the first award, he was not able to attend the award ceremony in New York due to budget constraints. In 1979, the Vacuum Society of Japan awarded Dr. Ishimaru for his contribution in the development of an 14 pin/BNC coaxial combination for cryogenic vacuum and microwave performance in the GHz region, and in 1982, for development of the first all aluminum alloy vacuum system. He received Japan's 42nd Remarkable Patent Award in 1983 for vacuum and cryogenic performance, high density, and quick connect and disconnect of the 14 pin/BNC coaxial combination feedthrough. In 1985, Japan's Remarkable Patent was awarded to Dr. Ishimaru for development of very thin and high performance thermal insulation with no particulates for vacuum systems. He received an award from the British Vacuum Society in 1985 for the first beryllium window mounted on aluminum by electron beam weld. In 1989, Physics News, from the American Institute of Physics, for the first practical 10⁻¹³ Torr XHV aluminum system and in 1991 the TAKAGI AWARD from the Institute of Japan for the first Ball Screw and Ball Bearing in UHV with long life time. Dr. Ishimaru received Taiwan's highest honor for technical contribution to vacuum technology to the SRRC Project at a dedication ceremony on October 1993, Hsinchu, Taiwan.

Dr. Ishimaru was instrumental in the establishment of ALVALAB (Aluminum Vacuum Laboratory) in 1988, an international private research consortium to develop and promote aluminum ultrahigh technology. Today, 63 member firms worldwide offer aluminum vacuum components and system technology. Dr. Ishimaru is known as the "Father" of ALVALAB.
JOHN A. THORNTON MEMORIAL AWARD AND LECTURE

To recognize outstanding research or technological innovation in the areas of interest to the American Vacuum Society, with emphasis on the fields of thin films, plasma processing, and related topics. The award is conferred annually or biannually as a suitable candidate may be identified. It consists of a cash prize, a commemorative plaque stating the nature of the award, and an honorary lectureship at a regular session of the National Symposium.

DAVID W. HOFFMAN

Dr. David W. Hoffman, Ford Motor Company, "For basic contributions to the effects of magnetron sputtering on the stress and microstructure of thin films, gas dynamics and resputtering."

David W. Hoffman studied physical metallurgy at The Ohio State University and the Massachusetts Institute of Technology, where he earned his doctorate in 1966. From 1966 until 1994 he was a member of the Scientific Research Laboratory at Ford Motor Company. Earlier this year he retired from Ford and is currently enjoying part-time employment with Advanced Modular Power Systems in Ann Arbor, Michigan. Dr. Hoffman's research interests are rooted in physical metallurgy, where his early work at Ford dealt with discrete lattice Fourier representations of ordering and clustering in binary alloys. Using this formalism he deduced a simple, elegant expression for the excess entropy of solution that greatly enhanced the accuracy of predictions of alloy ordering and clustering.

His interest in thin films was catalyzed by the automotive problem of coating plastic parts with vapor deposited decorative metals such as chromium as an alternative to electroplating without incurring failures caused by unexpectedly high residual stresses. A change meeting with John A. Thornton resulted in the discovery that chromium thin films deposited by the magnetron sputtering process on plastic parts did not exhibit cracking from residual tensile stresses. This observation launched a decade-long collaboration that uncovered the remarkable stress transition to compressive intrinsic stress at low sputtering pressures accessible through magnetron sputtering, and established the phenomenon to be one of great generality. The stress transition was found to be associated with microstructural and physical property transitions in deposited films as controlled by sputtering pressure and many other process variables. This phenomenology was extensively mapped out and linked to the presence and control of inherent energetic neutral bombarding particles during sputter deposition through a variety of unique experiments. The main impact of the stress transition phenomenology developed by Hoffman and Thornton was to establish that the properties and growth structures of sputter-deposited materials are produced by the flux of energetic particles streaming from target to substrate, as controlled by the degree of gas-scatter dissipation and dis-alignment in transit and by the configuration of the target, substrates, and their relative deployment.

To simulate the observed sputter deposition phenomena, Dr. Hoffman pioneered the technique now widely known as ion peening or ion assisted deposition and published a seminal paper in this area. He also pioneered in the conceptualization and fabrication of a rotating magnets magnetron, specializing in a version with a helical rotating discharge. He was the first to observe and report gas dynamic effects caused by magnetron sputtering. He observed and named the phenomenon of intrinsic resputtering and deduced the theoretical explanation of its unusual universal curve. He quantified the flux of backscattered neutralized ions from sputtering targets and demonstrated the utility of the analytic expressions by quantitative predictions of embedded inert gas in thin films. His work has appeared in over 50 publications and is extensively cited.

Dr. Hoffman was elected a Fellow of the American Vacuum Society with the first class of AVS Fellows in 1993. Professionally, Dr. Hoffman has been active in the American Vacuum Society, where he has served as Associate Editor of the Journal of Vacuum Science and Technology A and as Program Chairman of the Thin Films Division. He has lectured on applications of thin films in AVS Short Courses. In 1985 he was elected to the AVS National Board of Directors, where he initiated the AVS Investment Advisory Committee and was instrumental in establishing the John A. Thornton Memorial Award and Lecture. In 1988 he was elected President of the AVS for 1990. Since then he has served on the Investment Advisory Committee, the Long Range Planning Committee, and as AVS representative to the Governing Board of the American Institute of Physics, where he currently also serves on the Executive Committee of the Governing Board.

PETER MARK MEMORIAL AWARD

The Peter Mark Memorial Award was established in 1979 in memory of Dr. Peter Mark who served as Editor of the Journal of Vacuum Science and Technology from 1975 to 1979. The award is presented to a young scientist or engineer (35 years of age or under) for outstanding theoretical or experimental work, at least some of which must have been published in JVST. The award consists of a cash prize and a certificate.

MARJORIE OLMSTEAD

Dr. Marjorie Olmstead, University of Washington, "For elucidating the nature of semiconductor surfaces and the heteroepitaxial growth of insulating materials on these surfaces."
Marjorie Olmstead received her B.A. (Highest Honors) from Swarthmore College in Pennsylvania in 1979, majoring in physics with minors in applied mathematics and physical chemistry. She attended graduate school in physics at the University of California, Berkeley, where she received her M.A. in 1982 and her Ph.D in 1985. At Berkeley, she developed the technique of photothermal displacement spectroscopy to measure very small optical absorption signals, and used the technique to measure the optical adsorption of dangling bond states on cleaved Si and Ge. Her thesis work exploited these measurements to probe the symmetry, reconstructions, phase transitions and electron-phonon interaction on these surfaces.

Dr. Olmstead joined the Xerox Palo Alto Research Center as a Member of the Research Staff in 1985. She returned to Berkeley in 1986 as an Assistant Professor of Physics, and held a joint appointment as a Faculty Scientist at the Lawrence Berkeley Laboratory's Center for Advanced Materials from 1988 through 1993. She joined the faculty at the University of Washington, Seattle, in 1991, where she is currently an Associate Professor of Physics and Adjunct Associate Professor of Chemistry.

Beginning with her work at Xerox, Dr. Olmstead has pursued an interest in the chemical, structural and energetic constraints controlling the heteroepitaxy of strongly disparate materials. Using in-situ core-level and valence-band photoemission spectroscopy and X-ray standing wave fluorescence, Dr. Olmstead and her collaborators have shown how interface reactions, and the distinct surface energies and reaction tendencies of the resultant interface compounds, can control the structure and electronic properties of interfaces between strongly dissimilar materials, as well as the morphology of the resultant overlayer film.

Prof. Olmstead's current research centers on the kinetics of heteroepitaxy and on the structural and electronic properties of ultra-thin ionic materials. She and her students are among the first in the world to apply the technique of component-resolved X-ray photoelectron diffraction to study the kinetics of heteroepitaxial growth. Their site-specific diffraction studies of calcium fluoride growth on silicon have yielded in situ kinetic information on the heteroepitaxy of strongly dissimilar systems, and have demonstrated the complex variety of growth modes accessible. Recently, her group has made both experimental and theoretical advances in understanding the role of thin film geometry on core-level energies in insulators.

Prof. Olmstead received a National Science Foundation Presidential Young Investigator Award and an IBM Faculty Development Award in 1986, and was the spokesperson for a multi-investigator project funded through the 1989 Department of Energy "2X Initiative" Competition.

Prof. Olmstead enjoys teaching at both the graduate and undergraduate level. She is active in the American Vacuum Society, and currently holds a member of the Electronic Materials and Processing Division program committee and the Puget Sound Representative to the Pacific Northwest Chapter of the AVS.

**JVST SHOP NOTE AWARD**

The JVST Shop Note Award was established in 1970 by the AVS Vacuum Technology Division. It is presented to encourage the publication of novel solutions to instrumental or experimental problems as JVST Shop Notes in the previous year. The award consists of a certificate and cash prize. U. Bischler and E. Bertel have been selected as the 1994 winners of the JVST Shop Note Award for their contribution to "Simple Source of Atomic Hydrogen for Ultrahigh Vacuum Applications," JVST A11, 458 (1993).

**NELLIE YEOH WHETTEN AWARD**

The Nellie Yeoh Whetten Award was established in 1989, in the spirit of Nellie Yeoh Whetten, to recognize and encourage excellence by women in graduate studies in the sciences and technologies of interest to the American Vacuum Society. A fund to support the award was established by Timothy J. Whetten, friends and family of Nellie Yeoh Whetten, and the AVS. The award consists of a $1,000 cash prize, a certificate, and support of up to $750 in reimbursed travel expenses to attend the National Symposium.

**1994 NELLIE YEOH WHETTEN WINNER**

Monica Katiyar, University of Illinois

**RUSSELL AND SIGURD VARIAN FELLOWSHIP**

The Russell and Sigurd Varian Fellowship was established in 1982 to commemorate the pioneering work of Russell and Sigurd Varian in the Field of Vacuum Science and Technology. It is presented to recognize and encourage excellence in graduate studies in vacuum science. The Fellowship is supported by Varian Associates and consists of $1,500, a miniature replica of the first Varian pump, a certificate, and support up to $750 in reimbursed travel expenses to attend the National Symposium. The winner is selected from three finalists based on personal interviews with the Scholarships and Awards Committee at the National Symposium and will be announced at the Awards Assembly in October.

**1994 VARIAN FINALISTS**

Britt Turcot, University of Illinois
Robert Turcot, Jr., University of Illinois
Yajun Wang, University of Wisconsin

**AVS GRADUATE RESEARCH AWARDS**

The AVS Graduate Research Award was established in 1984. They are awarded to recognize and encourage excellence in graduate studies in the sciences and technologies of interest to the American Vacuum Society. Each award consists of $1,000, a certificate, and support of up to $750 in reimbursed travel expenses to attend the AVS National Symposium.
1994 GRADUATE RESEARCH AWARD

Thomas R. Bramblett, University of Illinois
Laurie Hackenberger, Pennsylvania State University
Dong Li, Northwestern University
Qian Liu, University of North Carolina
David Patrick, University of Utah
Britt Turkot, University of Illinois
Robert Turkot, Jr., University of Illinois
Yajun Wang, University of Wisconsin
Huasheng Wu, Montana State University
Frank Zimmerman, Cornell University

1994 FELLOWS

Daniel Alpert, University of Illinois
Ernst Bauer, Technical University, Clausthal
Christopher R. Brundle, IBM Research
Rintiant F. Bunshah, University of California
Robert J. Celotta, NIST
John W. Coburn, Fraunhofer Inst fur Angewandte
Festkoerperphysik
Jerome J. Cuomo, IBM Research Center
Paolo della Porta, SAES Milano
Francois M. d'Heurle, IBM Research Center
Charles B. Duke, Xerox Webster Research Center
Charles S. Fadley, Lawrence Berkeley Laboratory
Randall M. Feenstra, IBM Research Center
Maurice H. Francombe, Westinghouse R&D Center
Barbara J. Garrison, Pennsylvania State University
Richard A. Gottscho, AT&T Bell Labs.
John T. Grant, University of Dayton
Marsbed Hablanian, Varian Vacuum Products
Robert J. Hamers, University of Wisconsin
George H. Hass, Radiation Research Lab.
Franz J. Himpsel, IBM Research Center
Paul S. Ho, University of Texas
Leslie Holland
Jack E. Houston, Sandia National Laboratories
Harald Ibach, Forschungszentrum Julich
Hajime Ishimaru, Natl Lab for High-Energy Physics
William J. Kaiser, Jet Propulsion Lab
Harold Kaufman, Front Range Research
Eric Kay, Stanford University
Lawrence Kazmerski, Natl Renewable Energy Lab
Max Lagally, University of Wisconsin
Charles Magee, Evans East
Dennis M. Manos, College of William and Mary
John F. O'Hanlon, University of Arizona
Marjorie Olmstead, University of Washington
Daniel T. Pierce, NIST
Cedric J. Powell, NIST
Stephen M. Rossnagel, IBM Research Center
John E. Rowe, AT&T Bell Labs
Donald Santeler, Process Applications
Martin Seath, National Physical Lab
William Spicer, Stanford University
Michel Van Hove, Lawrence Berkeley Lab
John L. Vossen, John Vossen Associates
Gottfried Wehner
H.H. Wieder, University of California
Jerry M. Woodall, Purdue University
David P. Woodruff, Warwick University
John T. Yates, Jr., University of Pittsburgh
Russell Young, R.D. Young Consultants

AVS FELLOWS

The membership level designated "Fellow of the Society" was established in 1993 to recognize members who have made sustained and outstanding scientific and technical contributions in areas of interest to the AVS. These contributions can be in research, engineering, technical advancement, academic education, or managerial leadership. This is a prestigious membership level to which members are elected. The Fellow will receive a certificate.
DIVISION AWARDS

Morton M. Traum Surface Science Division Student Award

The AVS Surface Science Student Division Award was initiated in 1981. Morton M. Traum, then chair of the Surface Science Division, was the prime motivator in establishing the award. After Mort's untimely death on 1 December 1982, the Executive Committee of the Surface Science Division renamed the award in his memory. The Morton M. Traum Surface Science Division Student Award is presented annually for the best student paper based on work leading to a Ph.D thesis. The papers are judged on technical content and quality of presentation.

The winner of the award will be announced at the beginning of the Postdeadline Discovery Session held on Thursday evening, October 27.

Past winners are:

1981  Eric Stuve  
1982  Steven Gates  
1983  Ann Smith  
1984  Hans Gossman  
1985  Duane Outka  
1986  Greg Sitz  
1987  Michael Henderson  
1988  Jeff Hanson  
1989  Yunong (Neal) Yang  
1990  Benjamin Wieand  
1991  David Peale  
1992  Chaochin Su  
1993  Anna Swan

John Coburn and Harold Winters Student Award in Plasma Science and Technology

The Plasma Science and Technology Division has established the John Coburn and Harold Winters Student Award. John Coburn and Harold Winters have made pioneering contributions to the field of plasma processing and plasma surface interactions. Their work has provided the inspiration for countless students to enter the field. Coburn and Winters have enhanced the graduate experiences of students by both example and by tutorship. In recognition of their work, the Coburn and Winters Student Award will be conferred on the student whose oral presentation of his/her paper at the Annual Symposium is judged most outstanding by a panel selected from the Executive Committee of the PSTD. The student winner will receive a prize of $500.
NEW PRODUCTS POSTER SESSION—SEE THE LATEST IN VACUUM TECHNOLOGY

At this year’s exhibition, poster sessions will present the latest ideas in new technology. Don’t miss the excellent opportunity (3–5 p.m. on Wednesday) to learn about new products being introduced to the market.

SEE THE EXHIBITS—AND DON’T MISS A SINGLE EVENT

This year, you will have more time to tour the exhibit. There will be no plenary lunch-time lectures on exhibit days, and a full three-hour block of time has been opened on Wednesday when there will not be any technical sessions scheduled.

REFRESHMENTS AND SPECIAL SNACKS

There are so many great technical sessions and interesting new technologies to see and so little time! So this year, there will be refreshments and snacks in the exhibition area. You can munch while you browse through the show.

AVS TECHNOLOGY AND EQUIPMENT EXHIBITION

WHY YOU SHOULD ATTEND

This will be an excellent opportunity to see products and services offered by over 150 participating companies. There is no fee to attend the exhibition, and it is open to members and nonmembers of the AVS. Everyone with an interest in keeping abreast of the current innovations in our industry is welcome and encouraged to attend. Many company scientists will be available to answer your questions.

Exhibits will be relevant to engineers, technicians, scientists, and purchasing professionals in the following fields:

- Thin-Film Deposition
- Fusion Technology
- Vacuum Metallurgy
- Nuclear and High-Energy Physics
- Surface Analysis
- Lasers and Optics
- Analytical Instruments
- Vacuum Technology
- Semiconductor Materials and Processing
TECHNOLOGY AND EQUIPMENT EXHIBITORS

NEW EXTENDED SHOW HOURS

A & N Corporation
Academic Press
Advanced Ceramics Corp.
Advanced Energy Industries
AJA International
Alberta Microelectronic Centre
Alcatel Vacuum Products
Alta Group
American Institute of Physics
Ametek/Dycor
Amplifier Research
Angstrom Sciences
APD Cryogenics
Applied Science & Technology
Applied Surface Technologies
Arconium Specialty Alloys - PlasmArc Div.
ATI Instruments N.A.
Austin Scientific Co.
Balzers
Bertan High Voltage
Bi-Braze
Brooks Instruments
Burleigh Instruments
Cambridge Mill Products
Cerac, Inc.
Ceramaseal
CHA Industries
Commonwealth Scientific
Cooke Vacuum Products
CTI-Cryogenics
CVC Products
CVI Inc.
Danielson Associates
Denton Vacuum, Inc.
Digital Instruments
Dunway Stockroom
Dynavac
Edwards High Vacuum Int'l.
Elnik Systems
Evac International
Charles Evans & Associates
Extrel Mass Spectrometry
FEI Co.
Ferrofluidics
Fil-Tech, Inc.
Fisons Instruments
Fuji Seiki Inc.
Geller Microanalytical
GNB Corporation
Granville-Phillips
Helicoflex Co.
Hiden Analytical Ltd.
High Vacuum Apparatus Mfg.
IBM Analytical Services
Inland Vacuum
Innotec Group Inc.
Integrated Vacuum Technology
Ion Tech Inc.
Jen-USA, Inc.
John Crane Belfab
KDF Electronic & Vacuum Svces.
Key High Vacuum Products
Kimball Physics
Kratos Analytical
L.D.S. Vacuum Products
Larson Electronic Glass
Kurt J. Lesker Co.
Leybold Infield Inc.
Leybold Technologies
Leybold Vacuum Products
LK Technologies
Luxtron Corporation
Mass-Vac, Inc.
Mat-Vac Technology
Materials Science Inc.
R.D. Mathis Co.
Maxtek, Inc.
McAllister Technical Services
MDC Vacuum Products
Melvac, Inc.
Metroline Industries
MKS Instruments
NIST/Standard Reference Data
Nor-Cal Products
Nordiko USA, Inc.
Nuvac Innovations
OCI Vacuum Microengineering
Onimicron Associates
Osaka Vacuum Ltd.
Oxford Instruments
Park Scientific Instruments
Perkin-Elmer
Phelps Electronics
Phillips Semiconductors
Plasma-Therm, Inc.
Plasmquest, Inc.
Plasmatrak, Inc.
Polyacool Systems
Precision Flow Devices
Precision Plus Vacuum Parts
Princeton Research Instruments
Princeton Scientific Corp.
Process Systems Int'l.
Pure Tech Inc.
Quantar Technology
Rees Instruments
Research & PVD Materials
RF Power Products
RF Services Inc.

The hours are as follows:

Tuesday 11 a.m.-6 p.m.
Wednesday 10 a.m.-5 p.m.
Thursday 10 a.m.-3 p.m.

RHK Technology
Rocky Mountain Laboratories
Rushmore Technology Inc.
SAES Getters U.S.A.
Sciens Institute AB
Seiko Seiki Co., Ltd.
Senior Flexonic Systems
Service Physics
Soleris Ltd.
Spees GmbH
Spectra Instruments
Spectravac Inc.
Staib Instrumente
Stephens Analytical
Stokes Vacuum Inc.
Super Conductor Materials
Surface/Interface
Swagelok Co.
Sycon Instruments
T-M Vacuum Products Inc.
Target Materials, Inc.
TechnoTrade International
Techware Systems
Teledyne Brown Engineering
Telemark
Televac
Tencor Instruments
Terranova Scientific
TFS Technologies
Thermionics Lab.
TopoMetrix
Tylan General
U-C Components, Inc.
US Thin Film Products
UTI Instruments
Vacuum Engineering & MAT
Vacuum Research Corp.
Vacuum Technology
Varian Associates
VAT, Inc.
Veeco Instruments
VSI Vacuum Science Instruments GmbH
John Wiley & Sons, Inc.
Williams Advanced Materials
Willson Scientific Glass
J.A. Woollam Co.
World Scientific Publ.
Yeagle Technology
The American Vacuum Society will hold its National Symposium and Topical Conferences at the Colorado Convention Center. This Symposium is the main gathering for scientists and technologists whose research interests or processing needs demand reduced pressure environments. Last year's meeting in Orlando attracted over 3000 registrants from academia, industry, and government.

Technical Program

The meeting features a dynamic program comprised of 120 technical sessions including 18 poster sessions and 3 topical conferences — combined to offer a total of over 1000 papers. Distinguished invited speakers will keynote many of the technical sessions. A lunch-time plenary lecture on the world technological balance of power is scheduled to complement the technical program on the first day of the symposium.

Exhibits

A very important part of each AVS National Symposium is the exhibit of vacuum and deposition equipment, vacuum system components, analysis systems, and vacuum-related services. As in previous years, an extensive commercial exhibit of current products will highlight progress in vacuum hardware and services. In Denver, the exhibits will be open Tuesday through Thursday (11 a.m.-6 p.m.; 10 a.m.-5 p.m.; 10 a.m.-3 p.m.). To encourage everyone to visit the exhibits, we have expanded the lunch hour on Wednesday, giving a full three hours for lunch and interaction with exhibitors.

Morning Coffee/Tea and Announcements

We will be offering free coffee and tea in the foyer above the registration area on Monday through Friday before the first technical session (7:30-8:20). In addition, on Tuesday through Thursday free coffee and tea will be offered during the exhibit hours. Posters will be strategically located in the registration area of the Convention Center that will announce last minute changes in the sessions or other activities. Please check them.

Short Courses

The Convention Center will also be the site for the short course program where courses on a variety of topics will be offered. These courses will run concurrently with the AVS Symposium. Full details are available in the preliminary program on pages 117.

DIVISIONAL PROGRAM

The preliminary program is listed below for the 41st National Symposium of the American Vacuum Society. The program is organized by the Society's eight divisions. Various technical sessions will be keynoted by invited speakers of international reputation.

Applied Surface Science Division

Session AS-MoM presents overviews of recent advances in Imaging and Small Area Analysis using ion, photon, and electron beams. Major topics include SIMS imaging techniques emphasizing organic surface characterization and advances in small area XPS and scanning AES instrumentation. Micro-Raman spectroscopy for strain imaging analysis will also be discussed.

The latest developments in data banks, data processing and reference materials for surface analysis will be presented in Session AS-MoA, Data Processing and Reference Methods. This is an area receiving a lot of attention worldwide. Information on a data model and data dictionary will be presented in a Tuesday poster session. Session AS-TuP illustrates the diversity of analytical techniques and applications. Areas of emphasis are: conductive polymers; organic surfaces and self-assembled monolayers; siloxane coatings; nanotopography and nanostructures, and metal oxide surfaces. Spectroscopic and imaging techniques include XPS, SIMS and AFM.

The location and identification of particulate contamination on surfaces are addressed in Session AS-TuM, Surface Chemistry and Contamination. This information has become critical in the area of manufacturing as the sizes of semiconductor devices have been reduced. The session on Electrochemistry and Liquid/Solid Interfaces, AS-TuA, will be joint with the Surface Science Division. The presentations will focus on: characterization of the liquid/solid interface, especially using scanning probe methods; passivation and corrosion of metal surfaces, adsorption from solution, and factors controlling surface reactivity.

Session AS-ThA, Polymer/Organic Surfaces, emphasizes ana-
lytical investigations of polymers such as: surface modification (ion beam, corona treatments), polymer interfaces (co-polymers), vibrational characterization (EELS), tacticity (SIMS), nanomechanical properties (AFM), and additives (silica in silicones, lubricants, plasticizers). XPS and/or TOF-SIMS are applied to various classes of polymers including halogenated species, polyethylenes, polystyrenes, polypropylenes and polymethacrylates.

The session on Adhesion and Adhesive Bonding, AS-FrM, will cover many aspects of adhesion including surface preparation and primers, characterization of polymer/metal interfaces and interfacial bond strengths. Applications range from structural bonding to microelectronics devices.

<table>
<thead>
<tr>
<th>Code</th>
<th>Session</th>
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<tbody>
<tr>
<td>AS-MoM</td>
<td>Imaging and Small Area Analysis</td>
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<tr>
<td></td>
<td>H. N. Migeon, Centre Univ. - Lux.</td>
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<tr>
<td>AS-MoA</td>
<td>Data Processing and Reference Methods</td>
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<tr>
<td></td>
<td>R. W. Linton, Univ. North Carolina</td>
</tr>
<tr>
<td>AS-TuM</td>
<td>Surface Chemistry and Contamination</td>
</tr>
<tr>
<td></td>
<td>T. Francis, Applied Materials</td>
</tr>
<tr>
<td>ASSS-TuA</td>
<td>Electrochemistry and Liquid/Solid Interfaces</td>
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<tr>
<td></td>
<td>D. Baer, Pacific Northwest Lab</td>
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<tr>
<td>AS-WeM</td>
<td>Quantitative Analysis, Emphasizing Angle-Resolved XPS</td>
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<tr>
<td>AS-WeA</td>
<td>Depth Profiling</td>
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<td></td>
<td>E. H. Cirlin, Hughes</td>
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<tr>
<td>AS-ThM</td>
<td>Self-Assembled Monolayers</td>
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<td>A. Ulman, Eastman Kodak</td>
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<tr>
<td>AS-ThA</td>
<td>Polymer/Organic Surfaces</td>
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<tr>
<td>AS-FrM</td>
<td>Adhesion and Adhesive Bonding</td>
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Electronic Materials and Processing Division

The Electronic Materials and Processing Division provides a forum for the discussion of research on electronic, optical, physical and chemical properties of semiconductors, metals and dielectrics and their interfaces, and on processing technologies for these materials. In this year's program, oral and poster sessions provide an exciting overview of the multiple connections between materials growth, structure and processing, and device fabrication and performance.

The EMPD program features sessions in all four of the Symposium's Focus Areas. Three sessions are devoted to Si based optoelectronics. The first is an all-invited session with overview talks describing the leading edge of optoelectronic device technologies. It is followed by two sessions on heterostructures for optoelectronics and wide band gap nitride materials which feature advances in II-VI and III-V blue/green emitters and lasers. In conjunction with other sessions focusing on sensors and diagnostics, EMPD will present a session on in-situ optical diagnostics for materials processing. It highlights a number of real-time techniques for characterization in situ, with a special focus on spectroscopic ellipsometry. A session on processing and characterization of materials for nanostructures is coordinated with a Surface Science session on nanoscale measurements on surfaces and a Nanometer-scale Science and Technology session on the definition of nanoscale structures by lithography. Finally, a session on surface preparation and passivation will examine the current state of the art in surface cleaning and thin gate oxide formation. It is jointly sponsored by EMPD and the Manufacturing Science topical symposium, which will have a complementary session on tooling and manufacturing processes for wafer cleaning.

In conjunction with the topics covered in the focus areas, important progress in the growth, characterization and use of thin films in device structures is presented in two sessions covering materials for device integration, and thin film heterostructures. Areas highlighted include characterization and application of heterostructures, issues in interconnects and metallization, and new dielectric materials. Exciting progress in the science of electronic materials growth, characterization and processing is also presented in this year's program. Three sessions jointly sponsored by EMPD and the Surface Science Division will feature recent advances in fundamental understanding of microscopic mechanisms of surface modifications during materials processing, including important new insights made possible by use of the STM to characterize chemically modified surfaces. They are complemented by a session on characterization, presenting the state of the art in spectroscopic structural probes of heterogeneous interfaces. It will honor Marjorie Olmstead, 1994 Peter Mark Award winner.

In addition to the oral program, two exciting poster sessions will feature papers on core topics in this year's Electronic Materials and Processing Division program, as well as a wide range of other areas of interest to the division.

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<th>Code</th>
<th>Session</th>
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<tr>
<td>EM-MoM</td>
<td>Materials for Device Integration</td>
</tr>
<tr>
<td></td>
<td>B. Gnade, Texas Inst.</td>
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<tr>
<td>EM-MoA</td>
<td>Thin Film Heterostructures</td>
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<tr>
<td></td>
<td>R. Ludeke, IBM; M. Asada, Tokyo Inst. of Tech.</td>
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<tr>
<td>EMMS-TuM</td>
<td>Surface Preparation and Passivation</td>
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<tr>
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<td>T. Ohmi, Tohoku Univ.</td>
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<tr>
<td>EM-TuA</td>
<td>Silicon-based Optoelectronics</td>
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<td>S. Yokoyama, Hiroshima Univ.; C. Henry, AT&amp;T; M. Koyonagi, Hiroshima Univ.;</td>
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<td>J. Michel, MIT; Y. Shiraki, Univ. Tokyo</td>
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<tr>
<td>EM-WeM</td>
<td>Heterostructures for Optoelectronics</td>
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<td>M. Ikeda, Sony; J. DePuydt, 3M; K. Bachmann, N. Carolina State Univ.</td>
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<tr>
<td>EM-WeA</td>
<td>Wide-bandgap Nitrides</td>
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<td>S. Nakamura, Nichia Chemical</td>
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<tr>
<td>EM-ThM</td>
<td>Optical Diagnostics for Materials Processing</td>
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<td>G. Maracas, Arizona State Univ.</td>
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<tr>
<td>EMSS-ThM</td>
<td>Semiconductor Surface Reactions III</td>
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<td>J. Weaver, Univ. Minnesota</td>
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<tr>
<td>EM-ThA</td>
<td>Materials for Nanostructures</td>
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<td>K. Martin, Georgia Inst. Tech.</td>
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<tr>
<td>EM-FrM</td>
<td>Interface Characterization</td>
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<td>R. Headrick, Cornell Univ.; M. Olmstead, Univ. Washington</td>
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The Nanometer-Scale Science and Technology
NANO 3

This year, the Nanometer-scale Science and Technology Division (NSTD) will host the Third International Conference on Nanometer-scale Science and Technology (NANO 3). The NANO 3/NSTD program provides a multi-disciplinary forum focused on the science and technology of nanometer-scale phenomena in fields such as biology, chemistry, electronics, engineering, fabrication, materials science, medicine, metrology, optics, and physics.

NANO 3 follows the first conference held with STM 90 in Baltimore and NANO 2 held last year in Moscow. It was organized by a joint NSTD/international program committee. Members of the NSTD program committee include Rich Colton (Chair), Bob Hamers, Mark Reed, and Clayton Teague. The international committee members include Chair Christie Marrian (USA), Haroon Ahmed (UK), Chunli Bai (China), John Dagata (USA), Masa Hara (Japan), Young Kuk (Korea), Huguette Launois (France), Shakra Prokes (USA), Jurgen Rahe (Germany), Dan Rugar (USA), and Hiroshi Tokuhoto (Japan). In addition to NSTD support, NANO 3 is being sponsored by the Office of Naval Research, the U.S. Army Research Office and the National Science Foundation.

The program begins Monday morning with an exceptional plenary session featuring three world-renowned keynote speakers: Prof. Wolfgang Göpel (Univ. Tübingen) on "Nanostuctural Sensors for Molecular Recognition," Dr. Don Eigler (IBM Almaden Research Center) on "Quantum Corrals," and Dr. Yoshinobu Sugiyama (ETL) on "Recent Progress on Magnetic Sensors and Nanostructures and Applications." Briefly, Prof. Göpel is Director of the Institute of Physical Chemistry at the University of Tübingen. His work deals with the development of novel sensors based on the self-assembly of molecules at interfaces. Dr. Eigler is an IBM Fellow at the IBM Almaden Research Center. Using a cryogenic scanning tunneling microscope, he has shown that it is possible to individually move atoms to form structures that do not form naturally. Dr. Sugiyama is at the Electrochemical Laboratory of the Ministry of International Trade and Industry in Japan. He has worked extensively on semiconductor magnetic sensors including the quantum Hall probe.

The invited and contributed program includes 16 oral and 4 poster sessions featuring 19 invited speakers and over 225 contributed papers. There will be poster sessions devoted to all aspects of nanometer-scale science and technology: Instrumentation and Metrology (Mo), Materials Characterization (Tu), Nanometer-scale Science and Technology (We), Nanoelectronics and Nanofabrication (Th).

The oral session are organized as follows. The Monday afternoon session focuses on advances in metrology at the nanometer-scale featuring an invited talk by Dr. J. Griffith (AT&T Bell Laboratories) on "Dimensional Metrology with Scanning Probe Microscopes." The session also includes talks on methods for fabricating and characterizing scanning probe tips and algorithms for overcoming some of the effects of finite tip geometry. The second session is the first of two sessions on the material properties of nanostructures including chemical, electrochemical, electronic, magnet, optoelectronic, and structural properties. This session focuses on electronic and structural properties of nanostructures exhibiting quantum size effects and is highlighted by an invited talk by Prof. H. Ahmed (Cambridge University).

On Tuesday morning, we begin a series of sessions devoted to the materials science aspects of systems where structure and composition are controlled on the nanometer-scale. The session on nanostructured and nanocrystalline materials is headed by an invited talk by Prof. H.-E. Schaefer (University Stuttgart) on "Nanostructured Solids as Interface-detemined Systems." Nanoelectronics devices based on quantum effects such as resonant tunneling are close to 'real' applications. These recent advances are highlighted in a session on nanoelectronics with invited talks by Dr. J. Randall (Texas Instruments) on "Applications of Nanoelectronic Devices" and Dr. M. Pechenok (NRL) on "Manufacturing Considerations for Quantum-Effects Devices." The session concludes with a special presentation by Dr. V.N. Af财产 who will review recent developments in nanotechnology in Russia. On Tuesday afternoon, the sessions on material properties of nanostructures continue with a focus on chemical and electrochemical systems. The session features an invited talk by Dr. J. Frommer (IBM Almaden Research Center) on "Scanning Probe Microscopy on Organic Materials." We also feature part I of two sessions on nanomechanics and nanotribology with an invited talk by Prof. J.-M. Georges (University Lyon) on "Nanomechanics and Nanotribology of Lubricant Films."

Material manipulation has evolved to the point where structures can be assembled atom by atom and chemical functionality of surfaces and interfaces can be modified with molecular precision. On Wednesday morning, we begin a series of nanofabrication sessions with the topic of atomic manipulation. Invited speakers for this session are Prof. J. Lyding (University Illinois) speaking on "Nanometer-scale Patternning of Si Surfaces with an UHV STM" and J. McClelland (NIST) on "Nanofabrication with Laser Focussed Atomic Deposition." We also explore various industrial applications of proximal probes in a session devoted to this topic. Invited speakers include Prof. H. Fuchs (University Munster) on "Industrial Application of Proximal Probes" and Dr. J. Mamin (IBM Almaden) on "Ultra-High Density Data Storage Based on Proximal Probes." This session also features a series of talks on using SPM-related techniques for 1-D and 2-D dopant profiling of semiconductor device structures. Since the discovery of photoluminescence from porous silicon, the role of surfaces and interfaces in determining the optical properties of silicon nanostructures has been recognized as a key issue. These effects are important in fabricated silicon nanostructures due to their large surface-to-volume ratio. A session on this topic will be held on Wednesday afternoon with an invited talk by Prof. H. Koch (Technical University Munich) on "Light from Silicon." The NANO 3 is also co-sponsoring three joint sessions with the Topical Conference on Biomaterial Interfaces. This afternoon we feature the first of two sessions on Biology at the Nanoscale with an invited talk by Dr. E. Betzig (AT&T Bell Labs) on "Scanning Near-Field Optical Microscopy."

On Thursday morning, the second session on biology at the nanoscale will feature an invited talk by Prof. L. Bottomley (Georgia Tech) on "SPM Visualization of Electrostatically Immobilized Interctalcal Drug-Nucleic Acid Complexes." A second nanofabrication session devoted to proximal prob lithography will feature an invited talk by Prof. C. Van Haesendonck (Katholieke Univ-Leuven) on "Nanolithographic Patternning of Metal Films with the STM." This session will also feature several
talks on quantum conductance through thin wires. The afternoon session features part II of the session on nanomechanics and nanotribology. In addition, the biology joint sessions conclude with a session on micro-instrumentation and sensors which features an invited talk by Prof. G. Kovacs (Stanford University) on “Micromachined Neural Interface Technology.”

On Friday we conclude our conference with two strong sessions devoted to innovations in proximal probe technology and novel nanofabrication techniques. Applications of proximal probes will be highlighted along with innovations such as instrumentation with high spatial and temporal resolution. Dr. G. McClelland (IBM Almaden) will deliver an invited talk on “Continuous Observation of the Motion of Single Adsorbed Atoms and Molecules with Picosecond and Sub-nanometer Resolution.” The nanofabrication session will feature invited talks by J. Calvert (NRL) on “Chemistry and Applications of Self-assembled Films,” and S. Fujima (NEC) on “Carbon Nanotubes.”

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<th>Code</th>
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<tr>
<td>NS1-MoM</td>
<td>NANO 3 Plenary Session</td>
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<td>W. Göpel, Univ. Tübingen; Y. Sugiyama, Electrotech. Lab.; D. Eigler, IBM</td>
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<td>NS1-MoA</td>
<td>Nanostructure Properties: Structural and Electronic</td>
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<td>J. Griffith, AT&amp;T</td>
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<td>J. Randall, Texas Instr.; M. Peckvar, Naval Res. Lab</td>
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<td>J. Georges, Ecole Centrale de Lyon</td>
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<td>NSMS-WeM</td>
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<td>H. Fuchs, Univ. Muenster; J. Mannin, IBM</td>
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<td>G. Kovacs, Stanford Univ.</td>
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<td>NS2-ThA</td>
<td>Nanomechanics and Nanotribology: II</td>
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**NS1-FrM** Novel Materials and Methods for Nanofabrication

*J. Calvert, Naval Res. Lab.; S. Iijima, NEC*

**NS2-FrM** Novel Probes

*G. McClelland, IBM*

### The Plasma Science and Technology Division

The Plasma Science and Technology Division will host eight oral sessions on the plasma processing of materials. The division will also sponsor one oral session on target fabrication for inertial confinement fusion. Two poster sessions will be offered. Eight students will compete for the John Coburn and Harold Winters Student Award in Plasma Science and Technology.

Rick Gottscho, the 1993 Division chairman, will lead off with an invited review of some of the key issues raised last year to help set the stage for this year. This talk will kick off the first two sessions on plasma etching and deposition, and one of the themes will be selective oxide etching. Yoshuhiro Horiike of Hiroshima University will present his work on highly selective oxide etching in an inductively coupled plasma. In the area of plasma deposition, Tom Mantei of the University of Cincinnati will provide an overview of diamond-like carbon film deposition, emphasizing plasma physics and chemistry aspects. The contributed papers in this session range over many of the issues involved with plasma generation, transport and surface interactions. Plasma deposition of metallic films for trench lining, trench filling, and contact via formation is addressed in a series of contributed papers.

Analysis and design of advanced plasma reactors continues to be an important topic in plasma materials processing. The contributed papers in this session examine problems and features in established designs such as ECR and ICP, along with some ideas for new reactor designs. The results of these studies are relevant to both deposition and etching.

Diagnostics are essential for understanding and controlling processing plasmas. Hideo Sugai of Nagoya University will present his work on diagnostic measurements of neutral and ionic radicals in an inductively coupled plasma reactor. The contributed papers include talks on gas phase and surface species detection. This session is one of the two focus areas in the 1994 Symposium in which PSTD is involved. Diagnostic sessions will be offered by seven other divisions.

Plasma-induced charging and contamination effects are among the most serious problems facing plasma technology. The session on these topics features an invited talk by Kouichi Hashimoto of Fujitsu, Ltd. on local differential charging of features, thought to be a new mechanism for gate oxide breakdown. Other papers in this session involve studies of profile distortion due to charging and charge-induced damage. Plasma-induced contamination, especially particle dynamics in plasma, is the focus of other contributed papers in this session. Surface contamination and control is the second of the two focus areas in which PSTD is included. Three other divisions will offer sessions related to this topic.

Charge free processing is an area attracting increasing attention, partly because of the charge damage and processing complications presented in the previous session. Chemical downstream etch and neutral beams are addressed in this session. Herb Sawin of MIT will present an overview of charge free processing applications,
including the current understanding of neutral-surface chemistry in charge free processing. Takachi Yunogami of Hitachi Ltd. will review recent work in the production of neutral beams, and their use for oxide etching. Among the key issues addressed in this talk are generation of high flux beams with large area uniformity.

Plasma-surface interactions are of course at the heart of plasma materials processing technologies. This year's session features an invited talk by Vince Donnelly of Bell Laboratories on measurements of spatially resolved surface adsorbate composition, termed 'chemical topography.' Contributed papers include experimental and theoretical studies of neutral and ionic species interacting with surfaces.

The session on target fabrication for inertial confinement fusion includes two invited talks. Michael Tobin of Lawrence Livermore National Laboratory will address target area design and system performance for the National Ignition Facility. Uichi Kubo of Kinki University will cover the historical development of microencapsulation for ICP capsules.

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<td>Plasma Etching &amp; Deposition</td>
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<td>R. Gottsch, AT&amp;T; Y. Horiike, Hiroshima Univ.</td>
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<td>PS-MoA</td>
<td>Plasma Deposition</td>
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<td>PS-TuM</td>
<td>Plasma Process &amp; Reactor Modeling</td>
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<td>PS-WeA</td>
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<td>K. Hashimoto, Fujitsu</td>
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<td>PS1-ThA</td>
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<td>PS2-ThA</td>
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<td>M. Tobin, Lawrence Livermore Nat. Lab.; U. Kubo, Kinki Univ</td>
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**Surface Science Division**

The Surface Science Division is the largest division within the American Vacuum Society. One quarter of all the abstracts submitted for presentation at the symposium were submitted to the Division. Within the society, the Division has its main focus in the area of fundamental research. In this year's program, there will be presentations on all areas in surface chemistry and physics.

Two of the major awards given by the society this year are to individuals active in the Division. The Welch Award, given to recognize and encourage outstanding research in fields of interest to the society, will be awarded to Professor John T. Yates, Jr. from the University of Pittsburgh. Professor Yates will give an invited talk on his recent research. The Gaede-Langmuir Award, given to recognize and encourage outstanding discoveries in the science and technologies of interest to the AVS, will be awarded jointly to Dr. Daniel Pierce and Dr. Robert Celotta, both from NIST. They are co-inventors of a new high resolution method of imaging magnetic domains, which will be discussed in their invited presentations.

A number of special sessions have been organized to focus attention on areas in which recent research has led to significant new understanding. Among these are sessions on the liquid/solid interface in which the development of atomic and molecular level structural probes now allow comparisons to be made with vacuum studies. This generates a close interaction between formerly disparate fields. Sessions on surface magnetism feature, among other topics, the discovery of a new type of magnetic material. It is a magnetic surface alloy which has potential applications to magnetic storage. Sessions on semiconductor surface reactions highlight recent experiments which give new insights into the role of atomic level steps in the oxidation and the etching of silicon surfaces by halogens.

Competition for the Morton M. Traum Award will focus on ten finalists who will be judged on their poster presentations on Monday and Tuesday afternoon.

In addition to these special sessions, the division has a very strong program in its traditional areas of surface structure, reactivity and characterization by spectroscopic means. These sessions are divided into groupings centered around materials and processes. They include sessions on surface reactions on metals, alloy and compound surface structure, surface electronic structure, semiconductor surface reactions, nucleation and growth of metals and semiconductors, heteroepitaxy, nanoscale measurements, and surface dynamics which include both thermal and non-thermal excitations.

On Thursday evening, a post deadline discovery session concerned with late breaking exciting or controversial recent developments in surface science will feature 15 minute presentations, accompanied by pretzels and beer. Abstracts for the post deadline session should be submitted on AVS forms to arrive by **Friday, September 2, 1994** to:

John C. Hemminger
Department of Chemistry
University of California, Irvine
Irvine, CA 92717

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<td>SS2-MoM</td>
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<td>S. Semancik, NIST</td>
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<td>SS2-TuM</td>
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<td>D. Jacobs, Univ. Notre Dame</td>
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<td>TFVM-MoA</td>
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<td>J. Howard, IBM</td>
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<td>TFVM-TuA</td>
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<td>A. MacLeod, Univ. Arizona; A. Kingon, North Carolina State Univ.</td>
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<td>J. Bilello, Univ. Michigan; R. Bradt, Univ. Nevada, Reno</td>
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<td>TF-ThA</td>
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<td>J. Bates, Oak Ridge Nat. Lab.; R. Goldner, Tufts Univ.</td>
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<td>TF-FrM</td>
<td>In Situ Thin Film Characterization</td>
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<td>M. Hammond, SL Diamond; S. Barnett, Northwestern Univ.</td>
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**Thin Film Division**

The Thin Film Division is sponsoring/co-sponsoring a number of sessions in the areas of innovative film deposition and characterization techniques and novel and practical applications.

Sessions with invited and contributed papers include:
- Thin Films for Energy Conversion and Efficiency/Active Films.
- Optical, Piezoelectric and Ferroelectric Films

In conjunction with the Vacuum Metallurgy Division, the Thin Film Division is sponsoring two sessions:
- Energetic Condensation: Process, Properties, and Products
- Diamond, Cubic Boron Nitride and other Ultra-Hard Films

Thin Film Division is also participating in the Sensors and Nanostructures. Focus Sessions with papers in the areas of:
- Thin Film Sensors
- In-situ Thin Film Characterization
- Deposition and Characterization Techniques of Nanostructures in Thin Films

The invited talks are focused on new and innovative applications for thin films as well as general review talks to help educate newcomers in the field. Finally, there will be an opportunity for extended interaction between presenters and audience in social sessions presented in the poster format and schedule for Wednesday and Thursday afternoons with focus in the areas of Post-deposition Film Characterization Techniques and Thin Film Applications.

**Vacuum Metallurgy Division**

The Vacuum Metallurgy Division presents a session on mechanisms of thin film microstructure evolution covering effects of stress, surface thermodynamics as well as energetic bombardment on nucleation, texture, grain size, grain boundaries, etc. A special session is devoted to the novel area of pulsed laser and pulsed ion techniques for film deposition and surface modification. The session on surface engineering for wear and corrosion applications includes papers on protective coating techniques and treatments, particularly plasma-based process. The high level of interest in practical manufacturing technology for production of coatings and thin films prompted a special session in this area. Emphasized in the talks are processes for the deposition of large-area coatings or films. Also, an invited paper describes environmentally compliant coating processes to replace some electroplating processes that are no longer environmentally acceptable. VMD co-sponsors two sessions on ultra-hard coatings including diamond, cubic boron nitride, carbon nitride, and multilayer nitride films. Also of special interest is the session on energetic condensation which includes the John Thornton Award Lecture by D.W. Hoffman.
VM-ThM  Pulsed Laser and Pulsed Ion Technology for Film Deposition and Surface Modification  
L. Greer, Raytheon; D. McIntyre, Sandia Nat. Labs

VM-ThA  Manufacturing Technology for Coatings  
K. Legg, Northwestern Univ.; D. Glocke, Eastman Kodak; P. LeFebvre, OCLI

VM-FrM  Surface Engineering for Wear and Corrosion Protection  
A. Matthews, Univ. Hull

- Code: VT-MoM  Session: Total Pressure Gauging  
S. Cho, Draper Labs

- Code: VT-MoA  Session: Gas Flow, Partial Pressure Analysis, and Leak Detection  
M. Bockermann, Vacuum Technology, Inc.; A. Fine, EPA

- Code: VT-TuM  Session: Vacuum Systems for Accelerators and Fusion  
P. LaMarche, Princeton Plasma Phys. Lab; R. Warrick, Sandia Nat. Lab.

- Code: VT-TuA  Session: Vacuum System Outgassing and Cleaning  
S. Tison, NIST

Vacuum Technology Division

The 1994 Vacuum Technology Division program covers vacuum science and technology issues that are important to all users of vacuum equipment and processes. These topics include fundamentals of the production, measurement and control of the vacuum environment.

The program begins Monday morning with a session on total pressure measurement. Characterization of any system or process usually requires a reliable measurement of the pressure. This session presents pressure gauge calibration and reliability measurements and several promising new measurement methods. An invited paper and several contributed papers highlight silicon-based micro sensors. Partial pressure, gas flow, and leak detection methods are covered in Monday afternoon's session. Invited papers cover gas sampling techniques and the development of advanced vacuum insulation for use in energy conservation.

New to the Vacuum Technology Division program will be a technical poster session on Monday afternoon covering many aspects of vacuum science and technology. This session will provide excellent opportunity for questions and discussions with the presenters.

The large national and international facilities for particle physics and fusion research require complicated and high performance vacuum systems. Tuesday morning's session describes progress in the specialized materials, vacuum components, and vacuum vessel conditioning techniques developed for these large facilities that often find use in smaller systems for research and manufacturing. Invited talks will summarize the D-T experiments at Princeton which recently produced world's record fusion power with the TFTR tokamak, and the planned design of a new National Ignition Facility for inertial fusion research.

The understanding of the source of gas phase and surface contaminants is important for the design and operation of vacuum systems where impurity control is a critical parameter. The VTD's Focus Session on contamination control leads off with an invited talk by S. Tison from NIST who will summarize a recently held workshop sponsored by the Division on the measurement and control of water in vacuum systems. Related papers will discuss novel surface preparation methods and conditioning techniques which minimize thermal outgassing. The session concludes with a cluster of papers discussing new developments in vacuum materials for use in the next generation of storage rings for minimizing photodesorption gas loads.

Topical Conferences

Manufacturing Science and Technology

Building on last year’s successful Topical Conference on manufacturing, and as a prelude to the recent establishment of a new Manufacturing Science and Technology Group within the AVS, this Second Topical Conference will comprise 3 1/2 days of technical sessions. The technical program of the Topical Conference has been jointly planned with the IEEE, which is a Technical Co-Sponsor.

The Topical Conference, focusing primarily on the microelectronics industry, is aimed at elucidating the scientific and technical issues which underpin effective manufacturing. An Overview session of primarily invited speakers will highlight the role of equipment, future manufacturing concepts, research opportunities, and environmental issues.

Two sessions are devoted to Advanced Manufacturing Equipment for both thermal and plasma processes, underscoring the importance of the equipment industry to microelectronics manufacturing. The equipment industry represents a critical component to microelectronics manufacturing competitiveness, and the AVS hopes to contribute significantly in this area, building on its scientific strengths and on the long-standing alliance between its university, government, and industrial members.

A session on Diagnostics, Sensors, and Control represents a crucial element for manufacturing and also draws on a traditional strength of the AVS; this session is part of a multi-session focus for the entire National Symposium. The session on Micro-Contamination and Defects, part of different multi-session focus in the meeting on surface contamination and control, will address particulate and molecular contamination, defects, and their relation to equipment and process. The Process and Equipment Modeling session will include an invited talk on the role of equipment modeling and process synthesis, as well as talks on both reactor-scale and microfabricate-scale issues in chemical processes.

A joint session on Vacuum Process Control for manufacturing will emphasize aspects of vacuum technology with major manufacturing impact in microelectronics, particularly process and equipment control, ergonomics, and diagnostic sensors. Finally, the Topical Conference is co-sponsoring other sessions in the Symposium on Industrial Applications of Scanning Probe Microscopy and on Surface Preparation and Passivation.
Biomaterials Interfaces

The Second Biomaterials Interfaces Topical Conference has been organized to provide a special forum where biologists and interface scientists can share their unique perspectives to the R&D opportunities in biomaterials. This mixing of disciplines is important because many biological phenomena occur at the solid-liquid interface. Two sessions are devoted to the interactions of proteins and cells with solid-state interfaces. The perspectives of the speakers in these two sessions range from fundamental interactions to developed applications. A third session addresses the issues involved in sensors’ interactions with biological systems, from design to applications. The fourth session, which is new to the Conference this year, described progress in the field of artificial cellular assemblies. The dominant theme in this session is the creation of artificial neuronal networks and their characterization. Most of the top groups in the world involved in this exciting frontier research are represented.

The conference also includes three joint sessions with the NANO 3 Conference. Two sessions deal with Biology at the Nanoscale, a collection of work that features applications of recent breakthroughs in Scanning Tunneling Microscopy/Spectroscopy, Force Microscopy, Near Field Optical Microscopy, and related technology. The investigation of DNA binding interactions is highlighted in one of these sessions. The final joint session lists developments in proximal probes, including medical and neuronal devices. The NANO 3 Conference also features related work in surface modification, nanomechanics, and nanotribology. In all, the combination of sessions give a good overview of the exciting area of biological-solid state interactions, with direct impact on human health issues and biotechnology.

Focus Areas

“Focus Areas” are a new concept we are introducing at this meeting in order to highlight new technical areas that will likely be important topics at future symposia, and to highlight areas of common interest among a number of different divisions. We have scheduled sessions within each focus area to minimize conflicts, so that the attendee can obtain diverse perspectives from a number of AVS Divisions. The Focus Areas and sessions included in them are listed below. These sessions are also highlighted on the “Program at a Glance” in the rear of the program book.

Si-based Optoelectronics

Sponsored primarily by the Electronic Materials and Processing Division, talks include materials, structures and devices for optoelectronics, based on silicon technology, including waveguides, light emitters, interconnects, and system applications.

Sensors, in-situ Diagnostics and Process Control

This broadly defined area includes sessions on sensor materials and devices, use of sensors and other diagnostics in the process environment, and control of processes using in-situ diagnostic information.
EM-ThM  Optical Diagnostics for Materials Processing
AS-ThM  Self-Assembled Monolayers
NSBI-ThA Micro-Instrumentation and Sensors
TF-FrM  In Situ Thin Film Characterization

Nanostructure Fabrication and Atomic-scale Manipulation of Surfaces

This area includes sessions on nanostructure fabrication processes and devices.

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Surface Contamination and Control

Here we focus on cleaning, passivation, particulate control, diagnostics, and contamination-free manufacturing.

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<td>Plasma-Induced Charging and Contamination Effects</td>
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<td>MS-ThM</td>
<td>Micro-Contamination and Defects</td>
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"The Role of Research in the Technological Balance of Power"

No region of the modern developed world is able, or should seek, to dominate the world high-technology economy. What is desirable is that there be a stable technological balance of power among major economic regions. What contribution can research make to the achievement of such a balance? What can be done about the increasing difficulty of ensuring that the society which funds basic research will reap its share of the returns on new knowledge it has paid for?

John Armstrong, IBM Vice President, Science and Technology (retired) was born in Schenectady, NY, in 1934. He received an A.B. in Physics from Harvard College in 1956, and a Ph.D. from Harvard in 1961. He remained there as a Research Fellow working with Prof. N. Bloembergen in nonlinear optics until 1963, when he joined IBM as a Research Staff Member.

Between 1976 and 1980, as Director of Physical Sciences, he was responsible for a major part of the physics, chemistry, and materials science at IBM Research. In 1981 he moved to the IBM East Fishkill development lab as Manager of Materials and Technology Development, working on advanced bipolar semiconductor and packaging technology. In 1986 he was named IBM Director of Research, and in 1987 was elected an IBM Vice President. In 1989, he was elected a member of the Corporate Management Board and named Vice President, Science and Technology. He retired from IBM in 1993.

Armstrong is the author or co-author of more than 50 papers on nuclear resonance, nonlinear optics, the photon statistics of lasers picosecond pulse measurements, the multiphoton spectroscopy of atoms, the management of research in industry, and issues of science and technology policy. He is a Fellow of numerous professional societies, was a member of the National Advisory Committee for Semiconductors, was a member of the National Science Board Special Commission on the Future of the NSF, and is a member of the NRC Board on Science, Technology and Economic Policy. He is Chair of the NRC study on The Future of Space Science.

He is a member of the National Academy of Engineering and of the Royal Swedish Academy of Engineering Sciences, and is a member of the Harvard University Board of Overseers. He received the 1989 George E. Pake Prize of the American Physical Society. In 1992, he was Regents' Lecturer of UCSB, and he was 1993-1994 Karl T. Compton Visiting Lecturer at MIT. He is currently Visiting Professor of Electrical Engineering and Computer Science at the University of Virginia.
Association of Vacuum Equipment Manufacturers
Annual Seminar
held during the AVS 41st National Symposium
at the
Radisson Hotel Denver
Tuesday, October 25, 1994 8:00 a.m. - 11:00 a.m.
"Forecasting the Future Needs and Challenges for the Vacuum Industry"

7:30 a.m.  Registration (refreshments served)
8:00 a.m.  Welcome Address
Joel McFadden, President, Stokes Vacuum, Inc. and President, AVEM

8:05 a.m.  Opening Remarks: Proactive Collaboration between AVS and the Vacuum Equipment Manufacturers
John Noonan, Argonne National Laboratory and President, AVS

8:20 a.m.  A Forecast of the U.S. Economy with an Inside Look at the Vacuum Technology/Semiconductor Industry
Kermit Baker, Director of the Economics Dept. Cahners Publishing Company
Sponsored by R&D Magazine

Cahners Publishing Company publishes more than 80 magazines and directories covering virtually every sector of the U.S. economy, including R&D Magazine. Cahners Economics is the research and forecasting arm of Cahners Publishing and uses a wide range of microeconomic indicators for forecasting business trends. Dr. Baker will describe his forecast for the U.S. economy in general and will then focus on prospects for the field of vacuum technology and the semiconductor industry.

8:55 a.m.  The Changing Face of NIST
David W. Norcross, Acting Director, NIST

The National Institute of Standards and Technology (NIST) began life in 1901 as the National Bureau of Standards. For 87 years, it was a respected measurement laboratory for the U.S. Government. In 1988 its name was changed and it was given an expanded mission - to work more directly with American industry. The Clinton Administration has made NIST the lead agency in helping industry modernize and solve tricky technical problems. In addition to its traditional laboratory programs, NIST now manages the Advanced Technology Program, the Manufacturing Extension Partnership, and the Malcolm Baldrige National Quality Award. NIST's budget is scheduled to grow from a modest $250 million just a few years ago, to $1.4 billion by 1997 - most of that new money will go for direct support to American Industry.

9:30 a.m. - 9:50 a.m.  Coffee break

9:50 a.m.  USDC - A Key Ingredient to FPD Success
M. Robert Pimel, Chief Technical Officer, U.S. Display Consortium

The U.S. flat panel display (FPD) Industry is entering a critical period which may represent the final opportunity for U.S. industry to be a significant player in this market. Among the factors which will determine success or failure for U.S. manufacturing capability in FPDs, will be the availability and supply of state-of-the-art equipment, materials, and process technology. The recognition of this issue led to the formation of the United States Display Consortium (USDC) and to the definition of its mission. Among the topics to be explored are the USDC's span of activities, its organizational structure, the approaches and key procedures established to fulfill its mission, the work projects undertaken, and an analysis of impact and benefits. A case will be made for the consortium approach as an effective solution to some of the significant barriers facing the U.S. FPD Industry.

10:25 a.m.  A Review of Capital Equipment Requirements from Component Products and Suppliers
Alex Glew, Core Technologist, Applied Materials

In order for capital equipment suppliers to develop and introduce new equipment to the semiconductor industry, they must work closely with component suppliers. It is important to set expectations for component suppliers that will allow successful integration during equipment manufacture, a long robust component life during service, and overall product value. In order to achieve these goals in the ever shrinking development time cycles, the component supplier must develop products, plan capacity, distribution and service prior to or concurrently with the equipment developer. The component performance and reliability must be characterized by the component supplier in order for the equipment supplier to perform system level characterizations and cost of ownership models.

11:00 a.m.  Adjourn Seminar
AVS Equipment Show Opens

11:30 a.m.  Luncheon and AVEM Annual Business Meeting
(Non-members of AVEM are welcome. For the convenience of attendees, there are separate registration fees for the AVEM Seminar and the luncheon).

For registration information, contact the Association of Vacuum Equipment Manufacturers
Telephone 505/856-6924; FAX 505/856-6716
SUNDAY, OCTOBER 23, 1994
8:30 a.m.  Tutorial: Nanostructures Fabrication and Characterization ................................................................. Terrace Room (H)
ASTM E-42 Subcommittee and Task Group Meetings
3:00 p.m.-4:00 p.m.  E42.03 AES Task Groups .................................................................................................................. Ballroom Section A (H)
3:15 p.m.-4:00 p.m.  E42.11 Standard Reference Data ..................................................................................................... Capitol Room (H)
4:00 p.m.-4:30 p.m.  E42.09 Standard Reference Materials .................................................................................................. Capitol Room (H)
4:40 p.m.-5:00 p.m.  E42.03 AES ............................................................................................................................................ Ballroom Section A (H)
4:30 p.m.-5:30 p.m.  E42.08 Ion Beam Sputtering ......................................................................................................... Capitol Room (H)
5:00 p.m.-5:30 p.m.  E42.02 Terminology ....................................................................................................................... Capitol Room (H)
5:00 p.m.-6:00 p.m.  E42.06 SIMS ................................................................................................................................. Ballroom Section A (H)
5:30 p.m.-6:00 p.m.  E42.01 Editorial .......................................................................................................................... Capitol Room (H)
7:30 p.m.-8:30 p.m.  E42.04 XPS ........................................................................................................................................... Capitol Room (H)
7:00 p.m.-8:30 p.m.  E42.14 STM/AFM ................................................................................................................. Ballroom Section A (H)
8:30 p.m.-9:15 p.m.  E42.00 Main Committee ............................................................................................................... Capitol Room (H)
9:15 p.m.-10:00 p.m. E42 Executive Committee ....................................................................................................... Capitol Room (H)
1:00 p.m.  Town Hall Meeting ............................................................................................................................... Grand Ballroom (H)
1:00 p.m.  Tutorial: A Guideline for TQM: A Stepping Stone to ISO 9000 ......................................................... Columbine Room (H)
1:00 p.m.  Tutorial: Surface, Biology, and Biomaterials .......................................................................................... Jr. Ballroom (H)

MONDAY, OCTOBER 24, 1994
9:00 a.m.  Science Educators Workshop .................................................................................................................. Denver Room (H)
12:00 Noon Science Educators Lunch ......................................................................................................................... Spruce Room (H)
12:45 p.m.  Plenary Lecture “The Role of Research in the Technological Balance of Power”
           Dr. John Armstrong, Vice President, IBM (retired)
2:00 p.m.  Topical Conference on Biomaterials Interfaces .......................................................................................... Room A106 (CC)
2:00 p.m.  Topical Conference on Manufacturing Science and Technology ......................................................... Room A110 (CC)
8:30 a.m.-5:00 p.m.  Short Course Program ........................................................................................................ Various Rooms (CC)

TUESDAY, OCTOBER 25, 1994
8:20 a.m.  Topical Conference on Biomaterials Interfaces .......................................................................................... Room A106 (CC)
8:20 a.m.  Topical Conference on Manufacturing Science and Technology ......................................................... Room A110 (CC)
9:00 a.m.  Science Educators Workshop .................................................................................................................. Denver Room (H)
11:00 a.m.-6:00 p.m.  Vacuum Equipment Exhibition .............................................................................................. Exhibit Hall A
12:00 Noon Science Educators Luncheon ...................................................................................................................... Spruce Room (H)
2:00 p.m.  Topical Conference on Biomaterials Interfaces .......................................................................................... Room A106 (CC)
2:00 p.m.  Topical Conference on Manufacturing Science and Technology ......................................................... Room A110 (CC)
6:30 p.m.  Placement Center Workshop ..................................................................................................................... Columbine Room (H)
8:00 p.m.  ASTM E.42/ASSD Workshop .................................................................................................................. Jr. Ballroom (H)
8:30 a.m.-5:00 p.m.  Short Course Program ........................................................................................................ Various Rooms (CC)

WEDNESDAY, OCTOBER 26, 1994
8:20 a.m.  Topical Conference on Biomaterials Interfaces .......................................................................................... Room A106 (CC)
8:20 a.m.  Topical Conference on Manufacturing Science and Technology ......................................................... Room A110 (CC)
10:00 a.m.-5:00 p.m.  Vacuum Equipment Exhibition .............................................................................................. Exhibit Hall A
3:00 p.m.-5:00 p.m.  New Vacuum Products Poster Session ....................................................................................... Ballroom 4 (CC)
6:45 p.m.  AVS Awards Assembly and Reception ...................................................................................................... Ballroom 1,2,3 (CC)
8:30 a.m.-5:00 p.m.  Short Course Program ........................................................................................................ Various Rooms (CC)

THURSDAY, OCTOBER 27, 1994
10:00 a.m.-5:00 p.m.  Vacuum Equipment Exhibition .............................................................................................. Exhibit Hall A
8:00 p.m.  Surface Science "Post Deadline Discovery Session" .................................................................................. Grand Ballroom (H)
8:30 a.m.-5:00 p.m.  Short Course Program ........................................................................................................ Various Rooms (CC)

FRIDAY, OCTOBER 28, 1994
8:30 a.m.-5:00 p.m.  Short Course Program ........................................................................................................ Various Rooms (CC)

H=Radisson Hotel
CC=Colorado Convention Center
## BUSINESS AND COMMITTEE MEETINGS

### SATURDAY, OCTOBER 22, 1994

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>4:00 p.m.</td>
<td>Education Committee Meeting and Dinner</td>
<td>Gold Room (H)</td>
</tr>
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### SUNDAY, OCTOBER 23, 1994

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>8:00 a.m.</td>
<td>Short Course Executive Committee Meeting</td>
<td>Birch Room (H)</td>
</tr>
<tr>
<td>8:30 a.m.</td>
<td>Tutorial: Nanostructures Fabrication and Characterization</td>
<td>Gold Room (H)</td>
</tr>
<tr>
<td>11:00 a.m.</td>
<td>History Committee Meeting</td>
<td>Colorado Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>History Committee Luncheon</td>
<td>Aspen Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>Companions Program</td>
<td>Silver Room (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>Tutorial: A Guideline For TQM: A Stepping Stone to ISO 9000</td>
<td>Spruce Room (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>Tutorial: Surface, Biology, and Biomaterials</td>
<td>Century Ballroom (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>Town Hall Meeting</td>
<td>Grand Ballroom (H)</td>
</tr>
</tbody>
</table>

### MONDAY, OCTOBER 24, 1994

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
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<tbody>
<tr>
<td>7:00 a.m.</td>
<td>Companions Program (see insert)</td>
<td>Silver Room (H)</td>
</tr>
<tr>
<td>8:00 a.m.</td>
<td>Chapters &amp; Divisions Newsletter Breakfast</td>
<td>Columbine Room (H)</td>
</tr>
<tr>
<td>8:30 a.m.</td>
<td>Fundamentals of Surface Science Short Course</td>
<td>Capitol Room (H)</td>
</tr>
<tr>
<td>9:00 a.m.</td>
<td>Science Educators Workshop</td>
<td>Denver Room (H)</td>
</tr>
<tr>
<td>9:00 a.m.</td>
<td>PPA Sampling</td>
<td>Gold Room (H)</td>
</tr>
<tr>
<td>9:00 a.m.</td>
<td>Cryo Pumps</td>
<td>Aspen Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>Science Educators Workshop Luncheon</td>
<td>Spruce Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>JVST Luncheon</td>
<td>Century Ballroom (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>ICMC Abstract Selection Meeting</td>
<td>Colorado Room (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>Vacuum Symbols</td>
<td>Birch Room (H)</td>
</tr>
<tr>
<td>1:00 p.m.</td>
<td>Leak Detection</td>
<td>Cedar Room (H)</td>
</tr>
<tr>
<td>5:00 p.m.</td>
<td>1995 Program Committee Meeting</td>
<td>Columbine Room (H)</td>
</tr>
<tr>
<td>6:00 p.m.</td>
<td>Chapters &amp; Divisions Reception</td>
<td>S. Convention Lobby (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Publications Committee Dinner</td>
<td>Century Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Applied Surface Science Executive Committee Meeting &amp; Dinner</td>
<td>Cedar Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Manufacturing Science and Technology Group Meeting and Dinner</td>
<td>Spruce Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Vacuum Technology Executive Committee Meeting &amp; Dinner</td>
<td>Gold Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Student Reception</td>
<td>A Upper Lobby (CC)</td>
</tr>
<tr>
<td>7:00 p.m.</td>
<td>Chapters and Divisions Dinner</td>
<td>Jr. Ballroom (H)</td>
</tr>
<tr>
<td>7:00 p.m.</td>
<td>NANO 3 Reception</td>
<td>Majestic Ballroom (H)</td>
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</tbody>
</table>

### TUESDAY, OCTOBER 25, 1994

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
<th>Location</th>
</tr>
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<tbody>
<tr>
<td>7:00 a.m.</td>
<td>Companion's Program (see insert)</td>
<td>Silver Room (H)</td>
</tr>
<tr>
<td>8:00 a.m.</td>
<td>AVEM Seminar</td>
<td>Ballroom Section D (H)</td>
</tr>
<tr>
<td>9:00 a.m.</td>
<td>Science Educators Workshop</td>
<td>Denver Room (H)</td>
</tr>
<tr>
<td>9:00 a.m.</td>
<td>Ion Gauges</td>
<td>Birch Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>Science Educators Luncheon</td>
<td>Spruce Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>Topical Conferences Luncheon</td>
<td>Colorado Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>Recommended Practices Executive Committee Luncheon</td>
<td>Aspen Room (H)</td>
</tr>
<tr>
<td>12:00 Noon</td>
<td>AVEM Luncheon</td>
<td>Gr. Ballroom Section A (H)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Applied Surface Science Division Business Meeting</td>
<td>Room A101 (CC)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Electronic Materials &amp; Processing Division Business Meeting</td>
<td>Room A108 (CC)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Nanometer-Scale Science &amp; Technology Division Business Meeting</td>
<td>Room A207 (CC)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Surface Science Division Business Meeting</td>
<td>Room A205 (CC)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Thin Film Division Business Meeting</td>
<td>Room A105 (CC)</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Vacuum Technology Division Business Meeting</td>
<td>Room A102 (CC)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Vacuum Metallurgy Division Executive Committee Meeting</td>
<td>Birch Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>EMPD Executive Committee Meeting &amp; Dinner</td>
<td>Century Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Recommended Practices Committee Meeting &amp; Dinner</td>
<td>Gr. Ballroom Section B (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Surface Science Division Executive Committee Meeting &amp; Dinner</td>
<td>Gold Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Thin Film Division Executive Committee Meeting &amp; Dinner</td>
<td>Aspen Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Nanometer Science and Technology Division Executive Committee Meeting &amp; Dinner</td>
<td>Cedar Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Plasma Science &amp; Technology Division Executive Committee Meeting &amp; Dinner</td>
<td>Colorado Room (H)</td>
</tr>
<tr>
<td>6:30 p.m.</td>
<td>Placement Center Workshop</td>
<td>Colorado Room (H)</td>
</tr>
<tr>
<td>8:00 p.m.</td>
<td>ASTM E42/ASSD Workshop</td>
<td>Jr. Ballroom (H)</td>
</tr>
</tbody>
</table>
WEDNESDAY, OCTOBER 26, 1994
7:00 a.m. Companions Program (see insert) ......................................................... Silver Room (H)
8:00 a.m. Newsletter/Publicity Breakfast ............................................................... Colorado Room (H)
9:00 a.m. Low Pressure Gauges Subcommittee ...................................................... Spruce Room (H)
10:00 a.m. Scholarship Meeting ................................................................. Birch Room (H)
12:00 Noon Scholarship Luncheon ................................................................. Aspen Room (H)
5:20 p.m. Plasma Science and Technology Division Business Meeting .............. Room A109 (CC)
5:20 p.m. Vacuum Metallurgy Division Business Meeting ................................. Room A106 (CC)

THURSDAY, OCTOBER 27, 1994
7:00 a.m. Companions Program (see insert) ......................................................... Silver Room (H)
8:30 a.m. Exhibitors Breakfast ................................................................. Room C201 (CC)
12:00 Noon 1995 Program Committee Chairs ............................................... Aspen Room (H)
12:15 p.m. AVS Business Meeting ................................................................. Room A101 (CC)
6:00 p.m. Surface Science Spectra Editorial Board Meeting & Dinner ................ Gold Room (H)
6:00 p.m. 1994-95 LAC/Program Committee Reception .................................... Jr. Ballroom D (H)
7:00 p.m. 1994-95 LAC/Program Committee Dinner ......................................... Jr. Ballroom E (H)
8:00 p.m. Surface Science Postdeadline Discovery Session ............................... Grand Ballroom (H)

FRIDAY, OCTOBER 28, 1994
7:00 a.m. Companions Program (see insert) ......................................................... Silver Room (H)
8:00 a.m. Surface Science Database Committee Meeting ................................. Denver Room (H)
12:00 Noon Foreign Interactions ................................................................. Gold Room (H)

H=Radisson Hotel
CC=Colorado Convention Center

TECHNICAL SESSIONS
KEY TO SESSION/PAPER NUMBERS:

AS—Applied Surface Science Division
EM—Electronic Materials and Processing Division
NS—Nanometer Science and Technology Division and NANO 3
PS—Plasma Science and Technology Division
SS—Surface Science Division
MS—Topical Conference on Manufacturing Science and Technology
BI—Topical Conference on Biomaterials Interfaces
TF—Thin Film Division
VM—Vacuum Metallurgy Division
VT—Vacuum Technology Division

Sessions sponsored by two divisions are labeled with both acronyms (e.g. SSEM)
then: a number to indicate parallel sessions sponsored by the same Division (e.g. SS1, SS2)
then: Monday, Tuesday, Wednesday, Thursday, Friday
then: Morning, Afternoon, Evening, Poster then: a number to indicate the time slot scheduled for each paper
Example: SS1-MOM9 (Surface Science, Monday morning, 11:00 a.m.)
**SURFACE SCIENCE**  
*Room A205 - Session SS1- MoM pg. 114*

**Reactions on Metals: Hydrocarbons**

**Moderator:** D. Dywer, University of Maine.

<table>
<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:00 am</td>
<td>SS1-MoM3 Anharmonicity on the NIAI(111) Bimetallic Alloy Surface.</td>
<td>A.P. RADDORF, Oak Ridge National Laboratory. A.T. HANICKI, University of Pennsylvania.</td>
</tr>
<tr>
<td>10:00 am</td>
<td>SS1-MoM6 Acetylene Di- and Trimerization Reactions at Pt(111) and the (3x3)R30°-Sn/Pt(111).</td>
<td>J. SZANYI, M.T. PAFFETT, Los Alamos National Laboratory.</td>
</tr>
<tr>
<td>10:20 am</td>
<td>SS1-MoM7 The Surface Chemistry of 1,1-dimethylhydrazine on Pt and Ag-Modified Pt.</td>
<td>A.L. SCHWANGER, M. KOVAR, D.J. ALBERAS, J.M. WHITE, University of Texas, Austin.</td>
</tr>
<tr>
<td>11:00 am</td>
<td>SS1-MoM9 The Chemistry of Phthalic Anhydride at a Clean and Oxygen Covered Copper Single Crystal Surface.</td>
<td>S. KAO, R.C. BAINBRIDGE, N.V. RICHARDSON, University of Liverpool, United Kingdom.</td>
</tr>
<tr>
<td>11:20 am</td>
<td>SS1-MoM10 Cu(100) as a Model for the Methanol Catalyst.</td>
<td>I. CHORZERONOFF, J. WAMBACH, M. KAZUTA, P.B. RASMUSSEN, Technical University of Denmark.</td>
</tr>
</tbody>
</table>

**SURFACE SCIENCE**  
*Room A201 - Session SS2-MoM pg. 115*

**Alloy and Compound Surface Structure**

**Moderator:** B.E. KOEL, University of Southern California.

<table>
<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8:20 am</td>
<td>SS2-MoM1 Invited Surface Structure Investigation with Scanning Tunneling Microscopy (STM) and Low Energy Ion Backscattering.</td>
<td>H. MIEBUS, Humboldt-Universitat zu Berlin, Germany.</td>
</tr>
<tr>
<td>9:00 am</td>
<td>SS2-MoM2 Invited talk continued.</td>
<td></td>
</tr>
<tr>
<td>9:20 am</td>
<td>SS2-MoM3 Structure of NIO(111) Films on a Ni(100) Substrate.</td>
<td>G.L. WARREN, P.A. THEIL, Iowa State University.</td>
</tr>
<tr>
<td>9:40 am</td>
<td>SS2-MoM4 Detailed Surface Structures of Ultrathin Films of Ice (H2O) and Rust (Fe3O4) Grown on Pt(111). Determined by Automated Tensor LEED.</td>
<td>M.A. VANO HOVE, N.B. BARBIERI, N. MATERER, U. STARKE, W. WEISS, M. RITTER, G.A. SOMORJAI, Lawrence Berkeley Laboratory.</td>
</tr>
<tr>
<td>10:00 am</td>
<td>SS2-MoM5 Structural Studies of Quasicrystals.</td>
<td>W.-B. CHIN, C.J. JENKS, S.-L. CHANG, C.-M. ZHANG, P.A. THEIL, Iowa State University.</td>
</tr>
<tr>
<td>10:40 am</td>
<td>SS2-MoM7 The Surface Chemistry of 1,1-dimethylhydrazine on Pt and Ag-Modified Pt.</td>
<td>A.L. SCHWANGER, M. KOVAR, D.J. ALBERAS, J.M. WHITE, University of Texas, Austin.</td>
</tr>
<tr>
<td>11:00 am</td>
<td>SS2-MoM9 Structure Determination of the InP(001) Surface.</td>
<td>M.M. SUNG, J.W. RABALAES, University of Houston.</td>
</tr>
<tr>
<td>11:40 am</td>
<td>SS2-MoM11 Structure of the Ga-rich GaAs(001) 4×2 and 4×6 Phases.</td>
<td>Q.K. XUE, T. HASHIYU, T. ZHOU, T. SAKATA, T. SAKURAI, Tohoku University, Japan.</td>
</tr>
</tbody>
</table>

**12:45 pm Plenary Lecture**

"The Role of Research in the Technological Balance of Power"

**Dr. John A. Armstrong**

Ballroom 1

*Medard W. Welch Award Winner*
<table>
<thead>
<tr>
<th>Time</th>
<th>Session Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>8:20 am</td>
<td>Introduction and Welcome: C.R.K. Mariann, Naval Research Laboratory.</td>
</tr>
<tr>
<td>8:40 am</td>
<td>AS-MeM1 INVITED Static and Dynamic SIMS with Sub-Micron Resolution: Sensitivity and Quantification Aspects. Y. Gao, F. Saldi, H.-N. Migeon, CRP-CU, Luxembourg.</td>
</tr>
<tr>
<td>9:00 am</td>
<td>NS1-MoM3 INVITED Nanostructural Sensors for Molecular Recognition. W. Göpel, University of Tübingen, Germany.</td>
</tr>
<tr>
<td>11:00 am</td>
<td>NS1-MoM9 INVITED Quantum Corrals. D.M. Eigel, IBM Almaden Research Center.</td>
</tr>
<tr>
<td></td>
<td>AS-MeM10 SAM and XPS Studies of Roman Bronzes. E. Paparazzo, L. Moretto, ISM-CNR, Italy. P. Northover, University of Oxford, United Kingdom. C. D'Anato, Museo della Civiltà Romana, Italy. E. Severini, CSM SpA, Italy. A. Palmiere, ITABC-CNR, Italy.</td>
</tr>
<tr>
<td></td>
<td>AS-MeM11 Strain Imaging Analysis using Raman Microscope. J.P.H. Sukamoto, K. Ajito, K. Hashimoto, A. Fujishima, University of Tokyo, Japan.</td>
</tr>
<tr>
<td>Time</td>
<td>Session</td>
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<tr>
<td>8:40</td>
<td>PS-MeM3</td>
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<tr>
<td>9:00</td>
<td>PS-MoM4</td>
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<tr>
<td>11:00</td>
<td>PS-MoM9</td>
</tr>
<tr>
<td>11:40</td>
<td>PS-MoM11</td>
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*Albert Nieren Award Winner*
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<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Authors</th>
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<tbody>
<tr>
<td>8:20 am</td>
<td>EM-MoM1</td>
<td>Invited Performance and Integration of Copper VLSI/ULSI Interconnects</td>
<td>D.C. Edelstein, IBM TJ Watson Research Center</td>
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<tr>
<td>8:40 am</td>
<td></td>
<td>Invited talk continued.</td>
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<tr>
<td>9:00 am</td>
<td>EM-MoM3</td>
<td>Optimization of a Contact Metallization Scheme for 0.35 AsIC Technology</td>
<td>S. Bothra, T. Thowbridge, VLSI Technology Inc</td>
</tr>
<tr>
<td>10:00 am</td>
<td>EM-MoM6</td>
<td>Chemical Vapor Deposited TiCN - A New Barrier Metallization for Sub-micron Via and Contact Applications</td>
<td>M. Eizenberg, K. Littau, S. Ghanahein, A. Mak, Y. Naeda, M. Chang, A.K. Sinha, Applied Materials</td>
</tr>
<tr>
<td>10:20 am</td>
<td>EM-MoM7</td>
<td>Experiments and Simulations of High Pressure Chemical Vapor Deposition of TIN from Tetrakis-dimethylaminothiuramic and Ammonia in Vias</td>
<td>H. Liao, Arizona State University, R. Gordon, Harvard University, A. Toprac, SEMATECH, T.S. Gale, Arizona State University</td>
</tr>
<tr>
<td>11:00 am</td>
<td>EM-MoM9</td>
<td>Invited Processing and Device Issues of High Permittivity Materials for DRAM Memories</td>
<td>B.E. Gnade, Texas Instruments</td>
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<tr>
<td>11:20 am</td>
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<td>Invited talk continued.</td>
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## MY SCHEDULE
Monday Morning, October 24, 1994

<table>
<thead>
<tr>
<th>TIME</th>
<th>SESSION</th>
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## OTHER EVENTS MONDAY
- **7:00 a.m.** Companions Program (See insert) (Silver Room (H))
- **8:00 a.m.** Chapters & Divisions Newsletter Breakfast (Columbine Room (H))
- **9:00 a.m.** Science Educators Workshop (Denver Room (H))
- **9:00 a.m.** PPA Sampling (Gold Room (H))
- **9:00 a.m.** Cryo Pumps (Aspen Room (H))
- **12:00 Noon** Science Educators Workshop Luncheon (Spruce Room (H))
- **12:00 Noon** JYST Luncheon (Century Room (H))

H=Radisson Hotel
CC=Colorado Convention Hotel

## SHORT COURSES MONDAY
- **8:30 a.m.** Fundamentals of Surface Science (Vail Room (H))
- **8:30 a.m.** Vacuum Technology (Room C109 (CC))
- **8:30 a.m.** A Comprehensive Course on Surface Analysis AES, XPS, SIMS, Profiling, & ISS/RBS (Room C107 (CC))
- **8:30 a.m.** Basics of Radio Frequency (RF) Technology (Room C106 (CC))
- **8:30 a.m.** Operation and Maintenance of Vacuum Pumping Systems (Room C108 (CC))
- **8:30 a.m.** Sputter Deposition (Room C101 (CC))
- **8:30 a.m.** Auger Electron Spectroscopy (Room C107 (CC))
- **8:30 a.m.** Controlling Contamination in Vacuum System (Room C112 (CC))
- **8:30 a.m.** CVD for Microelectronics (Room C119 (CC))
- **8:30 a.m.** An Introduction to Ion Sources: Principles and Techniques (Room C108 (CC))
- **8:30 a.m.** An Overview of Thin-Film Deposition and Etching Techniques (Room C106 (CC))
- **8:30 a.m.** Technological Aspects of Metal Semiconductor Contacts (Room C104 (CC))
- **8:30 a.m.** Understanding Thin-Film Optics (Room C102 (CC))

## Special or Focus Area Sessions
- **KS1-MoM** — Ballroom 1. NANO 3 Plenary Session
## MY SCHEDULE
Monday Afternoon, October 24, 1994

<table>
<thead>
<tr>
<th>TIME</th>
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<td>5:00 pm</td>
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</table>

## OTHER EVENTS MONDAY
1:00 p.m.  ICMC Abstract Selection Meeting (Colorado Room (H))
1:00 p.m.  Vacuum Symbols (Birch Room (H))
1:00 p.m.  Leak Detection (Cedar Room (H))
5:00 p.m.  1995 Program Committee Meeting (Columbine Room (H))
6:00 p.m.  Chapters & Divisions Reception (S. Convention Lobby (H))
6:30 p.m.  Publications Committee Dinner (Century Room (H))
6:30 p.m.  ASS Executive Committee Meeting & Dinner (Cedar Room (H))
6:30 p.m.  Manufacturing Science and Technology Group Meeting and Dinner (Spruce Room (H))
6:30 p.m.  Vacuum Technology Executive Committee Meeting & Dinner (Gold Room (H))
6:30 p.m.  Student Reception (A Upper Lobby (CC))
7:00 p.m.  Chapters and Divisions Dinner (Jr. Ballroom (H))
7:00 p.m.  NANO 3 Reception (Majestic Ballroom (H))

H=Radisson Hotel
CC=Colorado Convention Hotel

## SHORT COURSES MONDAY
8:30 a.m.  Fundamentals of Surface Science (Vail Room (H))
8:30 a.m.  Vacuum Technology (Room C109 (CC))
8:30 a.m.  A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RBS (Room C107 (CC))
8:30 a.m.  Basics of Radio Frequency (RF) Technology (Room C105 (CC))
8:30 a.m.  Operation and Maintenance of Vacuum Pumping Systems (Room C103 (CC))
8:30 a.m.  Sputter Deposition (Room C101 (CC))
8:30 a.m.  Auger Electron Spectroscopy (Room C107 (CC))
8:30 a.m.  Controlling Contamination in Vacuum System (Room C112 (CC))
8:30 a.m.  CVD for Microelectronics (Room C116 (CC))
8:30 a.m.  An Introduction to Ion Sources: Principles and Techniques (Room C108 (CC))
8:30 a.m.  An Overview of Thin-Film Deposition and Etching Techniques (Room C106 (CC))
8:30 a.m.  Technological Aspects of Metal Semiconductor Contacts (Room C104 (CC))
8:30 a.m.  Understanding Thin-Film Optics (Room C102 (CC))

### Special or Focus Area Sessions

**Sensors, In-situ Diagnostics, Process Control**

**SS1-MoA**  Surface Mechanisms and Materials for Chemical Sensors
<table>
<thead>
<tr>
<th>Time</th>
<th>Session SS1-MoA</th>
<th>Title</th>
<th>Speaker(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2:00 pm</td>
<td>SS1-MoA1</td>
<td>Surface Chemistry of Gold Doped WO3 Hydrogen Sulfide Sensors</td>
<td>B. FRIERBERGER, D.J. DYER, University of Maine.</td>
</tr>
<tr>
<td>2:40 pm</td>
<td>SS1-MoA3</td>
<td>Surface, Interface and Thin Film Effects in Solid State Chemical Sensing</td>
<td>S. SEMANICK, R. CAVICCHI, E.P. POIRIER, J.S. SUEHEL, M. GAITAN, National Institute of Standards &amp; Technology</td>
</tr>
<tr>
<td>3:00 pm</td>
<td>Invite</td>
<td>Invited talk continued.</td>
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<tr>
<td>4:00 pm</td>
<td>SS1-MoA6</td>
<td>Structure and Chemical Sensing Properties of Vanadium Oxide Sol–Gel Films</td>
<td>J.S. LEDFORD, P.A. ASKELAND, Michigan State University.</td>
</tr>
<tr>
<td>5:00 pm</td>
<td>SS1-MoA9</td>
<td>Synthesis of Ordered Ultra-thin Al2O3 Films on Ru(0001) and Re(0001) Surfaces</td>
<td>Y. WU, E. GARFINKEL, T.E. MADEY, Rutgers University.</td>
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<thead>
<tr>
<th>Time</th>
<th>Session SS2-MoA</th>
<th>Title</th>
<th>Speaker(s)</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>SS2-MoA3</td>
<td>Mixed-Morphology MTP Growth of Ag on Si(100)-2x1 Surface. G. JAYARAM, N. DORAIWAMY, L.D. MARKS, Northwestern University.</td>
<td></td>
</tr>
<tr>
<td>2:20 pm</td>
<td>SS2-MoA4</td>
<td>Modification of Overlayer Growth Kinetics by Surface Interlayers: The Si(111)x7x3 – Indium Surface. S. SUREVE, J. KRAFT, F.P. NETZER, University Graz, Austria.</td>
<td></td>
</tr>
<tr>
<td>2:40 pm</td>
<td>SS2-MoA5</td>
<td>Islands on Si(111): Morphology Changes Induced by O Exposure. B. LAMONTAGNE, F. SEMONDO, D. ROY, Université Laval, Canada.</td>
<td></td>
</tr>
<tr>
<td>3:00 pm</td>
<td>SS2-MoA6</td>
<td>Kinetic Control in Epitaxial Growth: Chemisorption to Heteroepitaxy on MoP (111)</td>
<td>M. O. KISHINO, Y. HANADA, Tokyo University, Japan; T. HASHIMOTO, Nagoya University, Japan. T. SAKURAI, Tokyo University, Japan.</td>
</tr>
<tr>
<td>3:40 pm</td>
<td>SS2-MoA8</td>
<td>Structure of MBE-grown As-rich GaAs(001) Phases. T. HASHIMOTO, Y. KISHINO, Y. HANADA, Tokyo University, Japan. T. SAKURAI, Tokyo University, Japan.</td>
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</tr>
<tr>
<td>5:00 pm</td>
<td>SS2-MoA12</td>
<td>Diffusion Barriers on GaAs:Sn (100) Surfaces. S.M. SWEET, A.M. DABIRAN, X. SONG, B.E. ISHAG, P.J. COHEN, University of Minnesota.</td>
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<td>Time</td>
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<td>Speaker/Institution</td>
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<td>3:00 pm</td>
<td>Invited talk continued.</td>
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<tr>
<td>3:20 pm</td>
<td>NS1-MoA5</td>
<td>Voltage Oscillation Observed in Two Dimensional Nanostructures using Liquid Crystal Molecules at Room Temperature. N. NEJOM, M. AONO, M. TSUKADA, JRDC, RIKEN, and University of Tokyo, Japan.</td>
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<tr>
<td>4:00 pm</td>
<td>NS1-MoA7</td>
<td>Strain Relieving of Epitaxially Grown Ge Layers on Si Mesa Structure. Y.-H. KHANG, Y. KUK, Seoul National University, Korea.</td>
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<td>NS2-MoA10</td>
<td>An Optical Fiber Based Shear Force Microscope for Nanometer-Scale Dimensional Metrology on Large Samples. H.M. MARCHMAN, AT&amp;T Bell Laboratories.</td>
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<td>Time</td>
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<td>2:00 pm</td>
<td>AS-MoA1</td>
<td>Auger Electron Spectrometry Data Bank</td>
<td>M.P. SEAH, National Physical Laboratory, United Kingdom.</td>
</tr>
<tr>
<td>2:20 pm</td>
<td>AS-MoA2</td>
<td>Multi-spectral Scanning Auger Microscopy of Rough, Chemically Inhomogeneous Samples</td>
<td>I.R. BARKSHIRE, M. PRUTTON, University of York, United Kingdom.</td>
</tr>
<tr>
<td>2:40 pm</td>
<td>AS-MoA3</td>
<td>Surface Microanalysis and Imaging: Data Visualization and Pattern Recognition Applications</td>
<td>R.W. LINTON, University of North Carolina, Chapel Hill.</td>
</tr>
<tr>
<td>3:00 pm</td>
<td>AS-MoA4</td>
<td>IMERD: An Investigation of SiO₂ PECVD using Attenuated Total Reflection Fourier Transform Infrared Spectroscopy</td>
<td>S.C. DESHMUKH, E.S. AYDIL, University of California, Santa Barbara.</td>
</tr>
<tr>
<td>3:40 pm</td>
<td>AS-MoA6</td>
<td>SIMS vs. the TRIM Code for Depth Profiles of 100 to 350 keV Phosphorus in Silicon</td>
<td>K. MIEHE, W.H. GRIES, Deutsche Bundespost, Aachen, Germany; H. STRUSNÝ, Technische Universität, Prague, Czech Republic.</td>
</tr>
<tr>
<td>5:00 pm</td>
<td>AS-MoA10</td>
<td>The Effects of the Process Parameters on the Low Temperature Si/SlGe Epitaxy by UHV-ECR CVD</td>
<td>S.J. JOO, E. YOON, K.H. Hwang, Y.P. ED, K.W. Whang, Seoul National University, Korea.</td>
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<td>Time</td>
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<td>2:00 pm</td>
<td>VT-MoA1</td>
<td>Transition Gas Flow in Drag Pumps and Capillary Leaks</td>
<td>J.C. HELMER, G. LEVI, Varian S.p.A. Italy.</td>
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<tr>
<td>2:40 pm</td>
<td>VT-MoA3</td>
<td>Invited: High Precision, Gas Analysis System used to Analyze Grab Samples from the Space Shuttle Engine Compartment.</td>
<td>G.M. SOLOMON, M.G. BOECKMANN, Vacuum Technology, Inc.</td>
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<td>3:00 pm</td>
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<td>Invited talk continued</td>
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<td>3:40 pm</td>
<td>VT-MoA6</td>
<td>Fast Fine Leak Testing of Very Large Vessels</td>
<td>L. BERGST, Y.T. SASAKI, Quantum Mechanics Corp.</td>
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<td>4:20 pm</td>
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<td>Invited talk continued</td>
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<td>5:00 pm</td>
<td>VT-MoA10</td>
<td>Measurement of Metal's Vapour Pressure as Function of Temperature.</td>
<td>V.I. RAKHOVSKY, Research Center for Surface and Vacuum Investigation, Russia.</td>
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<td>TFVM-MoA4</td>
<td>Diamond Nucleation by Seeding from the Gas Phase.</td>
<td>Z. AJJI, M. BUCK, Universität Heidelberg, Germany. H. SCHWENK, Wackel-Chemicin, Germany. CH. WÜLL, Universität Heidelberg, Germany.</td>
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<td>Time</td>
<td>Session EM-MoA</td>
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<td>4:40 pm</td>
<td>EM-MoA9</td>
<td>INVITED Metal (CoSi\textsubscript{2})/Insulator (CaF\textsubscript{2}) Resonant Tunneling Diodes. M. ASADA, M. WATANABE, T. SUEMASU, Y. KOHNO, Tokyo Institute of Technology, Japan.</td>
<td>MS-MoA9 Deposition and Flow Planarization of Glasses. H. LIAO, T.S. CALE, Arizona State University.</td>
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<td>5:00 pm</td>
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<td>Invited talk continued.</td>
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<tr>
<td>Time</td>
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<td>2:00 pm</td>
<td>Bi-MoA1</td>
<td>The Bone Cell/Biomaterial Interface</td>
<td>J.E. Davies, University of Toronto, Canada.</td>
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<td>2:20 pm</td>
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<td>Invited talk continued.</td>
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<td>3:00 pm</td>
<td>Bi-MoA4</td>
<td>Nanofabricated Structures for the Measurement of Signals from Single Cells.</td>
<td>J.M. Cooper, A. Griffiths, H. Morgan, University of Glasgow, United Kingdom.</td>
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<tr>
<td>3:20 pm</td>
<td>Bi-MoA5</td>
<td>Invited</td>
<td>D.A. Oronsky, Xyronix, Inc.</td>
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<td>4:00 pm</td>
<td>Bi-MoA7</td>
<td>Nanofabricated Fibers for Studying the Phagocytosis of Inorganic Particulates by Macrophages.</td>
<td>J. Gold, B. Nilsson, B. Kasemo, Chalmers Univ. of Technology and Univ. of Goteborg, Sweden.</td>
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<tr>
<td>4:40 pm</td>
<td>Bi-MoA9</td>
<td>Effect of Surface Mobility on Biointeractions.</td>
<td>C.D. Ratner, C.D. Tidwell, University of Washington.</td>
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<tr>
<td>5:00 pm</td>
<td>Bi-MoA10</td>
<td>Secondary Ion Mass Spectrometry Studies of Covalently Bound Peptides to a Fluoropolymer: Imaging and Quantitation.</td>
<td>E.J. Bekos, P.V. Bright, J.A. Garibella, Jr., State University of New York, Buffalo.</td>
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</table>
SURFACE SCIENCE
Room BR4 - Session SS-MoP pg. 144

Aspects of Surface Science
Moderator: J. C. Hemminger, University of California, Irvine.

Presenters present: 5:00–6:30 pm.


SS-MoP2 Multi-Bounce Direct Scattering of N₂ from Cu(110). J. L. W. SIDERS, S. G. SITZ, University of Texas, Austin.

SS-MoP3 Dynamics of D₂ Recombinative Desorption from Ag(111). R. N. CARTER, F. HEALEY, A. HODGSON, University of Liverpool, United Kingdom.


SS-MoP7 Interaction of Fullerene with the Cu(111) and Ag(111) Surfaces. T. SAKURAI, X.-D. WANG, T. HASHIZUME, J. KISHIMOTO, S. YAMAZAKI, V. YUROV, Tohoku University, Japan. H. SHINOHARA, Nagoya University, Japan. H. W. PICKERING, Pennsylvania State University.


SS-MoP20 Selective Oxidation Reactions on Rh(111)–p(2x1)–O. C. W. BOL, C. M. FRIEND, Harvard University.


SS-MoP26 Scattering of 3He from the NO(001) Surface. B. KÄR, G. G. BISHOP, E. S. GILLMAN, S. A. SIFNO, M. S. SKOFRONJUK, Florida State University.

SS-MoP27 The Chemisorption of F₂ and O₂ on Si(110) 2x1 and Si(111) 7x7. H. C. FLAMM, E. B. BEHRINGER, E. J. LANZENDORF, D. MASSON, D. J. SULLIVAN, A. C. KUMMEL, University of California, San Diego.

SS-MoP28 Isothermal H₂ Desorption Kinetics from Si(100) 2x1: Dependence on Disilane and Atomic Hydrogen Precursors. L. A. OKADA, M. L. WISE, S. M. GEORGE, University of Colorado, Boulder.

SS-MoP29 Molecular Beam Reaction Studies of Trimethylgallium (TMGa) on Ga- rich GaAs(100). B. A. BANSEMAUER, C. E. NELSON, University of Wisconsin, Eau Claire. J. R. CROCKETT, Sandia National Laboratories.


SS-MoP31 Evolution of Surface Morphology of Si(100)–(2x1) during Oxygen Adsorption at Elevated Temperatures. Y. HONG, K. WURM, Y. WEI, I. S. TSONG, Arizona State University. R. KLÜEWE, B. RÖTTSER, H. NEDERMEYER, Ruhr-Universität Bochum, Germany.

SS-MoP32 Analysis of Chemisorption Sites: Cl₂ on Si(111)–7x7. C. YAN, J. A. JENSEN, A. C. KUMMEL, University of California, San Diego.

SS-MoP33 Resonantly Enhanced Multiphoton Ionization of SiO Desorbing from Si(111) in Reaction with O₂. K. S. NAKAMURA, M. KITAJIMA, National Research Institute for Metals, Japan.


SS-MoP35 The Site–Specific Neutralization Behavior of 2.0 keV Li+ Ions Scattered from Alkali/Al(100) Surfaces. C. B. WEARE, K. A. GERMAN, J. A. YARMOFF, University of California, Riverside & Lawrence Berkeley Lab.


*Finalist for the Morton M. Traum Award
NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room BR4 - Session NS-MoP pg. 150

Instrumentation and Metrology
Moderator: E.C. Teague, National Institute of Standards and Technology.

Presenters present: 5:00-6:30 pm.

**NS-MoP1** Quantum Friction Observed with Two-Dimensional Frictional Force Microscope. S. FUJIBARA, E. KISHI, Y. SUGAWARA, S. MURATA, Hiroshima University, Japan.

**NS-MoP2** Microtribological Study of Lubricants using Friction Force Microscopy. B. BRUSHAN, V.N. KONKAR, Ohio State University.

**NS-MoP3** Determining the Local Mechanical Properties of a Material using a Scanning Tunneling Microscope as a Surface Acoustic Wave Detector. B.M. SCHAEPFER, R.J. COLTON, S.M. HÜES, Naval Research Laboratory.

**NS-MoP4** Modelling of the Nanindentation of Thin SiO₂ Layers on Si using Finite Element Analysis. C.F. BRAFAR, Vanderbilt University, D.M. SCHAEPFER, R.J. COLTON, S.M. HÜES, Naval Research Laboratory.


**NS-MoP6** Ultrasound Influence on the Tribological Properties of CVD Diamond. V. ŠNITKA, Research Institute “Vibrotechnika”, Lithuania. V. BARANAUSSKAS, State University of Campinas, Brazil.

**NS-MoP7** Surface Roughness Characterization of Soft X-ray Multilayer Films on the Nanometer Scale. J. YU, Chubu University, Japan and SKLAO, P.R. China. J.L. CAO, SKLAO, P.R. China, Y. NAMBA, Chubu University, Japan. Y.Y. MA, SKLAO, P.R. China.

**NS-MoP8** Step Height Measurement using a Scanning Tunneling Microscope Equipped with a Crystalline Lattice Scale Reference and Interferometer. T. FUJII, M. SUZUKI, Nikon Corporation, Japan. H. KOGAMI, H. KAWAKATSU, T. HIGUCHI, University of Tokyo, Japan.

**NS-MoP9** Comparison of Magnetic Force Microscopy Techniques. P. RICE, J. MORELAND, National Institute of Standards & Technology.


**NS-MoP13** Ar Atomic Emission Microscopy. P.D. VAN PATTEN, J.D. NOLL, M.L. MYRICK, University of South Carolina.


**NS-MoP15** AFM Tip-Sample/Sample-Tip Deconvolution. P.C. MARKIEWICZ, M.C. DOH, University of Toronto, Canada.


**NS-MoP17** Analysis of Resist Artifacts by AFM for use in SEM and In-house Line Width Standards. D.A. CHERNOFF, Advanced Surface Microscopy, Inc.


**NS-MoP20** UHV Setup with Original STM for Nanostructures Creating and Study. K.N. ELTSOV, A.N. KLIYOV, A.M. PROKHOROV, V.M. SHEVLYUGA, V.V. YURCH, Russian Academy of Sciences.


Monday Afternoon Poster Sessions

PLASMA SCIENCE
Room BR4 – Session PS-MoP pg. 153

Plasma Etching and Deposition
Moderator: D.B. Graves, University of California, Berkeley.

Presenters present: 5:00–6:30 pm.


PS-MoP2  Silicon Oxide Deposition in an ECR Plasma with Microwave Spectroscopy as a Diagnostic. K.H. CHEW, J. CHEN, R.C. WOODS, J.P. SHOD, University of Wisconsin, Madison.


PS-MoP5  The Ion Energy Distribution at the Substrate Surface in a RF Induction Plasma Source. J.B. GAUGRAN, Oak Ridge National Laboratory.


PS-MoP8  Synergistic Sputtering Effects during Ion Bombardment with Two Ion Species. S. BERG, I.V. KARDOVE, Uppsala University, Sweden.


PS-MoP22  Spatially Averaged (Global) Model of Time Modulated High Density Electronegative Plasmas. S. ASHIDA, C. LEE, M.A. LIEBERMAN, University of California, Berkeley.

PS-MoP23  2D RF Sheath Structure due to Geometrical Discontinuities Outside the Wafer Edge. B. LANE, Plasma Dynamics.

VACUUM TECHNOLOGY
Room BR4 - Session VT-MoP pg. 157
Vacuum Science and Technology
Moderator: I.D. Hinkle, MKS Instruments Inc.
Presenters present: 5:00-6:30 pm.


VT-MoP2 Measurement of Xe Pressure by Photoelectron Counting with the Imaging Counter. S. SEKINE, S. ICHIMURA, Electrotechnical Laboratory, Japan.


VT-MoP5 Outgassing of Type 304 Stainless Steel by Surface Oxidation in Air. K. ODAKA, S. UEDA, Hitachi Ltd., Japan.


BIOMATERIAL INTERFACES
Room BR4 - Session BI-MoP pg. 160
Biomaterial Interfaces
Moderator: M.J. Tarlov, National Institute of Standards and Technology.
Presenters present: 5:00-6:30 pm.


BI-MoP11 Surface—Angstrom Movements of Biological Specimens Detected by Microwave. P. GUDULICH, G. ASCHERO, Dip. Fisiopatologia Clinica, Italy, F. MANG, Scuola Normale Superiore, Italy.


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<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Authors/Institutions</th>
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<tbody>
<tr>
<td>8:20</td>
<td>SS1-TuM1</td>
<td>Dynamics of the Si(100) Surface</td>
<td>C. Pearson, R. Curtis, X. Shi, E. Ganz, University of Minnesota.</td>
</tr>
<tr>
<td>8:40</td>
<td>SS1-TuM2</td>
<td>STM Studies of Vacancy–Step Interaction Kinetics during Low–Energy Ion Bombardment of the Si(001) Surface</td>
<td>B.S. Swartzentruber, Sandia National Laboratories; C.M. Matzke, University of New Mexico; J.E. Houston, Sandia National Laboratories; D.L. Kendall, University of New Mexico.</td>
</tr>
<tr>
<td>9:00</td>
<td>SS1-TuM3</td>
<td>Stress–Induced Step Bunching on Vicinal Strained Layers</td>
<td>Z. Zhang, University of Wisconsin, Madison; J.D. Tersoff, IBM TJ Watson Research Center; Y. Hong, M.G. Lagally, University of Wisconsin, Madison.</td>
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<tr>
<td>9:40</td>
<td>SS1-TuM5</td>
<td>Kinetic Smoothing of Vicinal Si(111).</td>
<td>H.-C. Kan, R.J. Phaneuf, E.D. Williams, University of Maryland.</td>
</tr>
<tr>
<td>10:00</td>
<td>SS1-TuM6</td>
<td>Fractal Growth on Metal Surfaces</td>
<td>Z.G. Zhang, X. Chen, M.G. Lagally, University of Wisconsin, Madison.</td>
</tr>
<tr>
<td>10:20</td>
<td>SS1-TuM7</td>
<td>Computer Simulations of Diffusion and Island Growth</td>
<td>G. Vidal, Syracuse University; D. Bird, Syracuse University; and Hebrew University; M. Karimi, Indiana University of Pennsylvania; R. Kimmel, Sun Microsystems; H. Zeng, Syracuse University.</td>
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<tr>
<td>11:00</td>
<td>SS1-TuM9</td>
<td>Surface Morphology Changes Upon Laser Heating of Pt(111)</td>
<td>J. Frohn, J. Reynolds, T. Engel, University of Washington.</td>
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<td>11:20</td>
<td>SS1-TuM10</td>
<td>Configurational Stability and Surface Diffusion of Rhodium Clusters on Rh(100).</td>
<td>G.L. Kellogg, Sandia National Laboratories.</td>
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<tr>
<td>8:40</td>
<td>SS2-TuM2</td>
<td>Dynamics of Surface Photochemistry: CO + O2 on Pt(111).</td>
<td>D.S. Busch, W. Ho, Cornell University.</td>
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<tr>
<td>9:00</td>
<td>SS2-TuM3</td>
<td>Invited Reactions of Hyperthermal Energy (5–100 eV), Molecular Ions with Metal and Oxide Surfaces.</td>
<td>D.C. Jacobs, University of Notre Dame.</td>
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<tr>
<td>10:00</td>
<td>SS2-TuM6</td>
<td>Models for Positive Ion Emission by Photon Excitation at the (001) Surface of MgO.</td>
<td>A. Gibson, J.A. Lapenta, Pacifc Northwest Laboratories. J.F. Dickinson, Washington State University.</td>
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<td>11:00</td>
<td>SS2-TuM9</td>
<td>TOF and Internal State Distribution of Photodesorbed Species from N2/O/Pt(111) by 193 nm Light.</td>
<td>D.P. Masson, E.J. Langanke, A.C. Kimmel, University of California, San Diego.</td>
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<td>8:20 am</td>
<td>NS1-TuM1</td>
<td>Nanocluster Surface Science by LDA Theory. D.R. JENDRISCH, S. NISDN, Y.S. Li, Biosym Technologies, Inc.</td>
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<tr>
<td>8:40 am</td>
<td>NS1-TuM2</td>
<td>Kinetics and Thermodynamics of Organic Nanocluster Formation. P.V. SHIBAEV, Y. SHIBAEV, K. SCHAUMBURG, K. BRUNFELDT, C.S.</td>
<td>Moscow State University, Russia; S. SCHMIDT, M.P. SEARS, T. KLITSCH, L. FRENKEL, Sandia National Laboratories.</td>
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<td>9:00 am</td>
<td>NS1-TuM3</td>
<td>Nanostructured Materials as Interface-determined Systems. H.-E. SCHAEPER, Stuttgart University, Germany.</td>
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<td>9:20 am</td>
<td>NS1-TuM4</td>
<td>The Surface Composition of Semiconductor Nanocrystals. J.E. BOWEN KATARI, V.L. COLVIN, A.P. ALIVISATOS, University of California, Berkeley.</td>
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<tr>
<td>11:00 am</td>
<td>NS1-TuM9</td>
<td>Invited Talk continued.</td>
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<tr>
<td>11:40 am</td>
<td>NS1-TuM11</td>
<td>Invited Talk continued.</td>
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### Tuesday Morning, October 25, 1994

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<thead>
<tr>
<th>Time</th>
<th>Session</th>
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<th>Speaker(s)</th>
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<tbody>
<tr>
<td>8:20 am</td>
<td>AS-TuM1</td>
<td>Overview of Particle Detection on Silicon Wafers. T. Francis</td>
<td>Applied Materials.</td>
<td>Kent State University.</td>
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<td>8:40 am</td>
<td>Invited</td>
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<td>9:00 am</td>
<td>AS-TuM3</td>
<td>Limitations of Surface Analytical Techniques for Determining the</td>
<td>A.G. Sault, Sandia National Laboratories.</td>
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<td>Surface Composition of Bimetallic Particles.</td>
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<td>SIMS and Laser – SIMS.</td>
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<td>Membranes.</td>
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<td>NASA Langley Research Center.</td>
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<td>10:00 am</td>
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<td>Oxidation of Gold by UV and Ozone at 25°C. D.E. King</td>
<td>National Renewable Energy Laboratory.</td>
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<td>Vanhommegic, A.W. Denier Van Der Gon, H.H. Bronkersma.</td>
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<td>Glasses.</td>
<td>Fisk University. R.H. Maunder, R.S. White, R.A. Zahr. Oak Ridge National</td>
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<td>11:20 am</td>
<td>AS-TuM10</td>
<td>Investigations of the Surface Chemistry of Pathogenic Silicates.</td>
<td>S. Seal, S. Hardcastle, T.L. Barr, University of Wisconsin, Milwaukee.</td>
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<td>H. He, J. Klinowski, P. Evans, University of Cambridge, United Kingdom.</td>
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<td>Pennsylvania State University.</td>
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**Plasma Science**

- **Room A109 – Session PS-TuM** pg. 171

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<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Speaker(s)</th>
<th>Institution</th>
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<tbody>
<tr>
<td>9:00 am</td>
<td>PS-TuM2</td>
<td>Design of a Surface Reflection Neutral Beam Source for Semiconductor</td>
<td>C.A. Nichols, D.M. Manos, College of William &amp; Mary.</td>
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<td>Processing.</td>
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<td>Density Plasma Sources.</td>
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<td>9:40 am</td>
<td>PS-TuM4</td>
<td>The Effect of Time Varying Sheaths on Radially Dependent Ion</td>
<td>R.J. Hoenstra, M.J. Kushner, University of Illinois, Urbana.</td>
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<td>Energy Distributions in Inductively Coupled Plasmas.</td>
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<td>10:00 am</td>
<td>PS-TuM5</td>
<td>Modelling of Plasma Etching Discharges. T.J. Bartel, Sandia National</td>
<td>D. Economou, University of Houston.</td>
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<td>Reactor Model with Comparison to Experimental Measurements.</td>
<td>Lam Research Corporation.</td>
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<td>10:40 am</td>
<td>PS-TuM7</td>
<td>Role of Etch Products in Si Etching by Cl₂ in High Density Plasma</td>
<td>C. Lee, M.A. Lieberman, D.B. Graves, University of California, Berkeley.</td>
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<td>11:00 am</td>
<td>PS-TuM8</td>
<td>Molecular Dynamics Simulations of Si Etching. N. Feil, Philips</td>
<td>M. Albert, Research Laboratory, The Netherlands.</td>
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<td>Research Laboratories.</td>
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<td>11:20 am</td>
<td>PS-TuM9</td>
<td>Invited talk continued.</td>
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<td>11:40 am</td>
<td>PS-TuM10</td>
<td>Invited talk continued.</td>
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<td>8.20 am</td>
<td>VT-TuM1</td>
<td>Theoretical Submonolayer Adsorption Isotherms for Hydrogen on a Heterogeneous Surface. J.P. HOBSON, National Vacuum Technologies, Inc.</td>
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<td>9.00 am</td>
<td>VT-TuM3</td>
<td>INVITED D-T Experiments in TFTR. P.H. LA MARCHE, Princeton University.</td>
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<td>9:20 am</td>
<td>INVITED</td>
<td>Invited talk continued.</td>
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<td>11:20 am</td>
<td>INVITED</td>
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<td>11:40 am</td>
<td>VT-TuM11</td>
<td>A Non-circular UHV Seal that Functions between ±200°C. R.A. CHILDS, J.E. RICE, Massachusetts Institute of Technology.</td>
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<td>8:20 am</td>
<td>EMMS-TuM1</td>
<td>INVITED Very High Quality Thin Gate Oxide Film Formation Technology. T. OHMI, Tohoku University, Japan.</td>
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<td>8:40 am</td>
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<td>Invited talk continued.</td>
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<tr>
<td>9:00 am</td>
<td>EMMS-TuM3</td>
<td>STM/XPS Investigation on the Chemical Oxidation of Hydrogen Terminated Si(111) Surface. U. NEUWALD, U. MEINMESS, R.J. BECK, Universität Ulm, Germany.</td>
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<tr>
<td>9:40 am</td>
<td>EMMS-TuM5</td>
<td>Controlled Nitrogen-Atom Incorporation at Si-SiO2 Interfaces by a Low-Temperature (500°C) Pre-Deposition Remote Plasma Oxidation Using N2O. B.R. LEE, G. LUCOVSKY, North Carolina State University.</td>
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<tr>
<td>10:20 am</td>
<td>EMMS-TuM7</td>
<td>Ion Beam Characterization of Si(100) Surfaces during Wet Chemical Cleaning. V. ATURI, N. HERBST, P. YE, R.J. CULBERTSON, Arizona State University.</td>
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<tr>
<td>11:00 am</td>
<td>EMMS-TuM9</td>
<td>Low Damage Surface Cleaning of CdTe by Hydrogen ECR Plasma. Y. LUO, P. LASKY, M. CHANG SHIH, R.M. OSGOOD, JR., Columbia University.</td>
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<tr>
<td>11:20 am</td>
<td>EMMS-TuM10</td>
<td>The Thermal Stability and Effect of Atomic Deuterium Exposure on S-Passivated InP(100)-(1x1). B.W. ANDERSON, M.C. HANF, P.R. NORTON, University of Western Ontario, Canada. Z.H. LU, M.J. GRAHAM, National Research Council, Canada.</td>
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<td>11:40 am</td>
<td>EMMS-TuM11</td>
<td>Thermal Effects on GaAs(C01) Surface Prepared by Deoxygenated and Deionized Water Treatment. Y. NIKOTA, T. GINNO, NTT Basic Research Labs, Japan. Y. WATANABE, M. OSHIMA, NTT Interdisciplinary Research Labs, Japan.</td>
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<td>12:40 am</td>
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<td>Invited talk continued.</td>
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<tbody>
<tr>
<td>9:00 am</td>
<td>MS-TuM3</td>
<td>INVITED Integrating Regulatory Policy &amp; Science — Can It Be Done? B.C. JORDAN, U.S. Environmental Protection Agency.</td>
<td></td>
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<td>9:20 am</td>
<td>MS-TuM5</td>
<td>INVITED Environment, Safety, and Health Issues in Manufacturing. H.R. KEBBY, SEMATECH.</td>
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<td>10:00 am</td>
<td>MS-TuM7</td>
<td>INVITED Factory of the Future: The &quot;Whole Factory&quot; View. S. HARRELL, SEMATECH.</td>
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<tr>
<td>11:00 am</td>
<td>MS-TuM9</td>
<td>Photocatalytic Oxidation for Point-of-Use VOC Abatement in Microelectronics Manufacturing. M. AMEEN, R. VARGHESE, J. NICOLI, B.B. RAUPP, Arizona State University.</td>
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<td>8:20 am</td>
<td>BI-TuM1</td>
<td>INVITED Molecular Recognition at Protein–Biological Composite Interfaces, P.S. STAYTON, R. CLARK, C.L. LONG, L. KLUMBE, A. CHILKOTI, A.A. CAMPBELL, G. DROBNY, University of Washington and Pacific Northwest Laboratories.</td>
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<td>8:40 am</td>
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<td>9:00 am</td>
<td>BI-TuM2</td>
<td>INVITED Surface Plasmon Imaging of Biotin–Streptavidin Binding on UV–Photopatterned Alkanethiol Monolayers Self–Assembled on Gold. D. PISEVIC, Max–Planck Institute for Polymer Research, Germany. M. TANLOV, National Institute of Standards &amp; Technology. W. KNOLL, Max–Planck Institute for Polymer Research, Germany.</td>
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<td>10:40 am</td>
<td>BI-TuM6</td>
<td>INVITED Bioreactivity of Titanium Implant Alloys. S.J. KERBER, Material Interface, Inc.</td>
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<td>11:00 am</td>
<td>BI-TuM7</td>
<td>Electrochemically and Glow Discharge Modified Titanium Surfaces used for Biological Evaluation. B.-O. ARONSSON, J. LAUSMAA, M. RODAHL, B. KASEMO, Chalmers University of Technology, Sweden.</td>
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## MY SCHEDULE
**Tuesday Morning, October 25, 1994**

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### OTHER EVENTS TUESDAY
- **7:00 a.m.** Compassion's Program (see insert) (Silver Room (H))
- **8:00 a.m.** AVEM Seminar (Ballroom Section B (H))
- **9:00 a.m.** Science Educators Workshop (Denver Room (H))
- **9:00 a.m.** Ion Gauges (Birch Room (H))
- **12:00 Noon** Science Educators Luncheon (Spruce Room (H))
- **12:00 Noon** Topical Conferences Luncheon (Colorado Room (H))
- **12:00 Noon** Recommended Practices Executive Committee Luncheon (Aspen Room (H))
- **12:00 Noon** AVEM Luncheon (Gr. Ballroom Section A (H))

H=Radisson Hotel  
CC=Colorado Convention Center

### SHORT COURSES TUESDAY
- **8:30 a.m.** Vacuum Technology (Room C106 (CC))
- **8:30 a.m.** A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RBS (Room C107 (CC))
- **8:30 a.m.** Basics of Radio Frequency (RF) Technology (Room C105 (CC))
- **8:30 a.m.** Operation and Maintenance of Vacuum Pumping Systems (Room C103 (CC))
- **8:30 a.m.** Sputter Deposition (Room C101 (CC))
- **8:30 a.m.** Fundamentals and Process Characterization of Ion Implantation (Room C112 (CC))
- **8:30 a.m.** Fundamentals of Semiconductor Characterization: Electrical and Optical Techniques (Room C110 (CC))
- **8:30 a.m.** Materials Microcharacterization (Room C108 (CC))
- **8:30 a.m.** Safety Concerns in the Use of Vacuum Equipment (Room C104 (CC))
- **8:30 a.m.** X-Ray Photoelectron Spectroscopy (XPS/ESCA) (Room C107 (CC))

### Special or Focus Area Sessions
- **Sensors, In-situ Diagnostics, Process Control**
- **TF-TuM** Thin Films for Sensors
- **Surface Contamination and Control**
- **EMMS-TuM** Surface Preparation and Passivation
### MY SCHEDULE
**Tuesday Afternoon, October 25, 1994**

<table>
<thead>
<tr>
<th>TIME</th>
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<tr>
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### OTHER EVENTS TUESDAY
- **5:20 p.m.** ASS Division Business Meeting *(Room A101 (CC))*
- **5:20 p.m.** EMP Division Business Meeting *(Room A108 (CC))*
- **5:20 p.m.** NST Division Business Meeting *(Room A207 (CC))*
- **5:20 p.m.** SS Division Business Meeting *(Room A285 (CC))*
- **5:20 p.m.** TF Division Business Meeting *(Room A105 (CC))*
- **5:20 p.m.** VT Division Business Meeting *(Room A102 (CC))*
- **6:30 p.m.** VM Division Executive Committee Meeting *(Birch Room (H))*
- **6:30 p.m.** EMPD Executive Committee Meeting & Dinner *(Century Room (H))*
- **6:30 p.m.** Recommended Practices Committee Meeting & Dinner *(Gr. Ballroom Section B (H))*
- **6:30 p.m.** SS Division Executive Committee Meeting & Dinner *(Gold Room (H))*
- **6:30 p.m.** TF Division Executive Committee Meeting & Dinner *(Aspen Room (H))*
- **6:30 p.m.** NST Division Meeting & Dinner *(Cedar Room (H))*
- **6:30 p.m.** PST Division Executive Committee Meeting & Dinner *(Colorado Room (H))*
- **6:30 p.m.** Placement Center Workshop *(Columbine Room (H))*
- **8:00 p.m.** ASTM E.421ASSO Workshop *(Jr. Ballroom (H))*

### SHORT COURSES TUESDAY
- **8:30 a.m.** Vacuum Technology *(Room C199 (CC))*
- **8:30 a.m.** A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RBS *(Room C107 (CC))*
- **8:30 a.m.** Basics of Radio Frequency (RF) Technology *(Room C185 (CC))*
- **8:30 a.m.** Operation and Maintenance of Vacuum Pumping Systems *(Room C183 (CC))*
- **8:30 a.m.** Sputter Deposition *(Room C101 (CC))*
- **8:30 a.m.** Fundamentals and Process Characterization of Ion Implantation *(Room C112 (CC))*
- **8:30 a.m.** Fundamentals of Semiconductor Characterization: Electrical and Optical Techniques *(Room C119 (CC))*
- **8:30 a.m.** Materials Microcharacterization *(Room C108 (CC))*
- **8:30 a.m.** Safety Concerns in the use of Vacuum Equipment *(Room C104 (CC))*
- **8:30 a.m.** Vacuum System Design *(Room C104 (CC))*
- **8:30 a.m.** X-Ray Photoelectron Spectroscopy (XPS/ESCA) *(Room C107 (CC))*

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**Special or Focus Area Sessions**

- **Silicon-based Optoelectronics**
- **EM-TuA** Silicon-based Optoelectronics
- **Sensors, in-situ Diagnostics, Process Control**
- **Bi-TuA** The Biosensor-Biology Interface
- **Surface Contamination and Control**
- **VT-TuA** Vacuum System Outgassing and Cleaning

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H=Radisson Hotel  
CC=Colorado Convention Center
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<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Speaker(s)</th>
<th>Institution(s)</th>
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</thead>
<tbody>
<tr>
<td>2:20 pm</td>
<td>SS1-TuA2</td>
<td>O₂ Adsorption on Silver Surfaces.</td>
<td>F. BUATIER DE MONGEOT, M. ROCCA, U. VALBUSÁ, CFSB-CNRS and INFM, Italy.</td>
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<tr>
<td>3:00 pm</td>
<td>SS1-TuA4</td>
<td>Investigations of the Adsorption and Desorption Dynamics of O₂ at Si Surfaces.</td>
<td>K.W. KOLASINSKI, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Germany.</td>
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<tr>
<td>3:20 pm</td>
<td>SS1-TuA5</td>
<td>Probing Reactive Deposition and Surface Dynamics using In Situ, Real-Time Emission Microscopy.</td>
<td>M.E. KORDESCH, Ohio University.</td>
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<tr>
<td>3:40 pm</td>
<td>SS1-TuA6</td>
<td>Invited talk continued.</td>
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<tr>
<td>4:00 pm</td>
<td>SS1-TuA7</td>
<td>Simulation of Complex Reaction/Desorption Kinetics using a Simple Stochastic Method.</td>
<td>F.A. ROULE, W.D. HINSBERG, IBM Almaden Research Center.</td>
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<tr>
<td>5:00 pm</td>
<td>SS1-TuA10</td>
<td>A New Technique to Measure Surface Diffusion with the STM.</td>
<td>M.L. LOZANO, M.C. TRINGOLES, Iowa State University.</td>
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<tr>
<td>2:40 pm</td>
<td>SS2-TuA2</td>
<td>Apparent Giant Annihilation of Small 2D Ag Islands on Ag(100).</td>
<td>P.J. BEDROSSIAN, Lawrence Livermore National Laboratory.</td>
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<tr>
<td>3:00 pm</td>
<td>SS2-TuA3</td>
<td>A RHEED Specular Diffraction Spot Intensity Study of Au(111) Homoeptaxy.</td>
<td>K. ROOS, Bradley University, K. STANDLEY, Iowa State University.</td>
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<tr>
<td>3:20 pm</td>
<td>SS2-TuA4</td>
<td>Enhanced 2-Dimensional Growth of Cu on Cu(100) by Seeding.</td>
<td>A.K. SWAN, J.F. WENDELMAN, Oak Ridge National Laboratory.</td>
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<td>4:00 pm</td>
<td>SS2-TuA5</td>
<td>Kinetic Roughening in a Multilayer Nucleation and Growth Model for M/M(100) Homoeptaxy.</td>
<td>M.C. BARTELJ, J.W. EVANS, Iowa State University.</td>
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<tr>
<td>4:20 pm</td>
<td>SS2-TuA6</td>
<td>First-Principles Calculations of Transition-Metal Surface Stress.</td>
<td>P.J. FEIBELMAN, Sandia National Laboratories.</td>
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<tr>
<td>4:40 pm</td>
<td>SS2-TuA7</td>
<td>Strain Induced Alloying of Immiscible Metals in Thin Film Systems.</td>
<td>J.L. STEVENS, R.O. HWANG, Sandia National Laboratories.</td>
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<tr>
<td>5:00 pm</td>
<td>SS2-TuA8</td>
<td>Surface Alloving and Dealloving of Au on Ni(110) and Ni(111) Studied by STM and RBS.</td>
<td>F. Besenbacher, L. Pleth Nielsen, E. Stensgaard, E. Laegsgaard, Aarhus University, Denmark.</td>
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<td>6:00 pm</td>
<td>SS2-TuA10</td>
<td>Long Jumps in the Surface Diffusion of Atoms: Ni, Pd on W(211).</td>
<td>D. Cowell, G. Ehrlich, University of Illinois, Urbana-Champaign.</td>
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<td>2:20 pm</td>
<td>NS1-TuA2</td>
<td>The Nature of Mixed Adlayers on Platinum Single Crystal Electrodes Probed by In Situ STM and Infrared Spectroscopy, T. VILLEGAS, M.J. WEAVER, Purdue University.</td>
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<td>2:40 pm</td>
<td>NS1-TuA3</td>
<td>Scanning Probe Microscopy of Organic Materials, J.E. FROMMER, IBM Almaden Research Center.</td>
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<td>Invited talk continued.</td>
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<td>3:40 pm</td>
<td>NS1-TuA6</td>
<td>Atomic Ordering within AlGaAs Alloys Studied using Cross-Sectional Scanning Tunneling Microscopy, A.R. SMITH, C.K. SHIH, Y.C. SHIH, B.S. STREETMAN, University of Texas, Austin.</td>
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<td>4:00 pm</td>
<td>NS1-TuA7</td>
<td>Characterization of the Gallium Vacancy on GaAs(110), D. LENDEL, M. WILKINS, M. WEIMER, J. DRYKO, R.E. ALLEN, Texas A&amp;M University.</td>
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<td>4:20 pm</td>
<td>NS1-TuA8</td>
<td>Structure and Electronic States on Reduced SrTiO(3)(110) Surface Observed by Scanning Tunneling Microscopy and Spectroscopy, R. BANDO, Y. AIURA, Electrotechnical Laboratory, Japan. Y. HARUYAMA, University of Tokushima, Japan. Y. NISHIHARA, Electrotechnical Laboratory, Japan.</td>
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<td>5:00 pm</td>
<td>NS1-TuA10</td>
<td>Direct Observations of Electric Field Gradients Near Field Emission Cathode Arrays, Y. LIM, University of Pennsylvania. W. GOODHUE, Lincoln Laboratory. D. BONNELL, University of Pennsylvania.</td>
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<tr>
<td>2:20 pm</td>
<td>NS2-TuA2</td>
<td>Molecular Dynamics Simulations of Metal–Oxide Surfaces and Interfaces, F.H. STEINZ, J.W. MINTMIRE, Naval Research Laboratory.</td>
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<tr>
<td>2:40 pm</td>
<td>NS2-TuA3</td>
<td>Electron–Ion Dynamics: A New Technique for Simulating both Electronic Transitions and Atomic Motion in Molecules and Materials, R.E. ALLEN, Texas A&amp;M University.</td>
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<td>3:40 pm</td>
<td>NS2-TuA5</td>
<td>Tribology with the Atomic Force Microscope, D.F. ODGETREE, J. HU, X.-D. XIANG, C. MORANT, M. SALMERON, Lawrence Berkeley Laboratory.</td>
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<td>4:00 pm</td>
<td>NS2-TuA7</td>
<td>Effect of Contact Area on Nanoindentation Curves Obtained using the Atomic Force Microscope, C.F. DRAFER, Vanderbilt University. D.M. SCHAEFER, R.J. COULTON, S.M. HUES, Naval Research Laboratory.</td>
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<td>2:00 pm</td>
<td>ASSS-TuA</td>
<td>Evidence of Specific and Non-Specific Adsorption of ClO₂ on Ag(110). A. KRASNOPOLE, E.M. STUVE, University of Washington.</td>
<td>PS-TuA1 Helicon Plasma Source Excited by Flat Spiral Coll. J.E. STEVENS, Princeton Plasma Physics Laboratory. M.J. SOWA, Princeton University. J.L. CECHI, University of New Mexico.</td>
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<td>3:20 pm</td>
<td>ASSS-TuA5</td>
<td>In Situ Measurement of Thickness Changes with Electrolytic SP. R. HUFFERT, P. HÄRING, R. KOTZ, Swiss Scherrer Institute, Switzerland.</td>
<td>PS-TuA5 Construction and Characterization of a Multidipole-Confining RF Inductively-Coupled Plasma Etching Source. C. LADE, B. BRUNMEIER, R.C. WOODS, University of Wisconsin, Madison.</td>
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<td>3:40 pm</td>
<td>ASSS-TuA6</td>
<td>Electrochemical Etching of Si(111) Surfaces in Fluoride Solutions Studied by STM and AES. R. HUBERZT, Universität des Saarlandes, Germany. U. MEMMERT, Universität Ulm, Germany.</td>
<td>PS-TuA6 Large Area Transformer Coupled Plasma for Microelectronics Processing. Z. YU, G.M. SHAW, G.J. COLLINS, Colorado State University.</td>
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<td>5:00 pm</td>
<td>ASSS-TuA10</td>
<td>In Situ Observation of Hg Amalgamation Formation and Stripping on Au using Atomic Force Microscopy. I.A. NAGAHARA, University of Tokyo, Japan. X. YANG, University of Tokyo, Japan.</td>
<td>PS-TuA10 Performance of a Permanent Magnet ECR Reactor. A. SAPPO, D. DANE, T.D. MANTEI, University of Cincinnati.</td>
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*62* John Coburn and Harold Winters Award Nominee
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<td>2:40 pm</td>
<td>VT-TuA3</td>
<td>TiN Thin Film on Stainless Steel for Extremely High Vacuum Material. K. SASA, S. INAYOSHI, Y. IKEDA, Y. YANG, S. TSUKAHARA, ULVAC Japan, Ltd.</td>
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<tr>
<td>3:00 pm</td>
<td>VT-TuA4</td>
<td>SIMS Analysis for Aluminum Surfaces Treated by Glow Discharge Cleaning. J.R. CHEN, SRRC and National Tsing-Hua University, China. G.Y. HSUONG, Synchrotron Radiation Research Center, China. Y.C. LIU, SRRC and National Tsing-Hua University, China. W.H. LEE, C.C. NEE, National Tsing-Hua University, China.</td>
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<tr>
<td>4:00 pm</td>
<td>VT-TuA7</td>
<td>Thermal Outgassing Studies on Machinable Tungsten and TZM Molybdenum Alloy. D. LIU, J. R. NIELSEN, Y. LI, D. RYDING, T.M. KUZAY, Argonne National Laboratory.</td>
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<td>4:20 pm</td>
<td>VT-TuA8</td>
<td>Photon Stimulated Desorption (PSD) Measurements of Extruded Copper and of Welded Copper Beam Chambers for the PEP II Asymmetric B-Factory. C.L. FORSTER, C. LANNI, Brookhaven National Laboratory. C. PERKINS, Stanford Linear Accelerator Center. M. CALDERON, Lawrence Livermore National Laboratory.</td>
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<td>4:40 pm</td>
<td>VT-TuA9</td>
<td>Photodesorption from Copper Chamber with Cutting Inner Surface by Broaching. T. KOBARI, M. MATUMOTO, N. HIRANO, MELI, Hitachi Ltd., Japan. M. KATAIE, M. MATSUZAKI, Hitachi Works, Japan. Y. HIGRI, M. KOBAYASHI, PF KEK, Japan. M. NAGAI, Hitachi Cable, Japan.</td>
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**Thin Film/Vacuum Metallurgy**

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<tr>
<td>2:00 pm</td>
<td>TFVM-TuA1</td>
<td>INVITED Deposition and Characterization of Cubic Boron Nitride Thin Films. R.F. DAVIS, D.J. KESTER, K.S. AILEY, North Carolina State University.</td>
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<tr>
<td>3:00 pm</td>
<td>TFVM-TuA3</td>
<td>An AES/XPS Study of Ti-B-N Thin Films. M.A. BAKER, A. STEINER, J. HAUPT, W. GIESLER, Institute for Advanced Materials, Italy.</td>
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<tr>
<td>3:40 pm</td>
<td>TFVM-TuA4</td>
<td>Electron Cyclotron Resonance Plasma Assisted Growth of Thin Carbon Nitride Films on Si(100): Film Composition and Structure. A. BOUSETTA, A. BENSAGOLA, M. LU, University of Houston.</td>
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<tr>
<td>4:40 pm</td>
<td>TFVM-TuA6</td>
<td>Thermal Behavior of Carbon Nitride and TiN/NbN Superlattices. S. LOPEZ, M.S. WONG, W.D. SPROUL, Northwestern University.</td>
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<tr>
<td>5:00 pm</td>
<td>TFVM-TuA7</td>
<td>Deposition and Properties of Polycrystalline VN/NbN Superlattices. X. CHU, M.S. WONG, W.D. SPROUL, S.A. BARNETT, Northwestern University.</td>
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<tr>
<td>6:00 pm</td>
<td>TFVM-TuA9</td>
<td>Thickness-Distribution Control of Large-Area DLC Films Formed by CH4/H2 Supermagnetron Plasma. M. KINOSHITA, S. NOMURA, M. HONDA, Shizuoka University, Japan.</td>
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<td>Authors/Institutions</td>
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<td>2:00 pm</td>
<td>EM-TuA1</td>
<td>INVITED Optical Waveguides on Silicon Chips.</td>
<td>S. YOKOKUMA, Y. KURUDA, T. MIYAMOTO, T. DOI, T. NAMBA, T. NAGATA, K. MIYAKE, S. MIYAZAKI, M. KOYANAGI, M. HIROSE, Hiroshima University, Japan.</td>
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<td>2:40 pm</td>
<td>EM-TuA3</td>
<td>INVITED Silicon Optical Bench Waveguide Technology.</td>
<td>C.H. HENRY, AT&amp;T Bell Laboratories.</td>
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<td>3:20 pm</td>
<td>EM-TuA5</td>
<td>INVITED Novel LSI Memories with Optical Interconnections.</td>
<td>M. KOYANAGI, Tohoku University, Japan.</td>
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<td>Invited talk continued.</td>
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<tr>
<td>4:00 pm</td>
<td>EM-TuA7</td>
<td>INVITED Rare Earth Doped Silicon Emitters.</td>
<td>J. MICHEL, L.C. KIMERLING, Massachusetts Institute of Technology.</td>
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<td>4:20 pm</td>
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<td>Invited talk continued.</td>
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<td>4:40 pm</td>
<td>EM-TuA9</td>
<td>INVITED SiGe/Si Quantum Well Light Emitters.</td>
<td>Y. SHIRAKI, S. FUKATSU, University of Tokyo, Japan.</td>
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<td>5:00 pm</td>
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<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td>MS-TuA5</td>
<td>MESC Cluster Tools for Advanced Metallization.</td>
<td>P.H. BALLENTINE, T. OMSTEAD, M. MOSLEHI, CVC Products, Inc.</td>
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<td>4:20 pm</td>
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<td>Invited talk continued.</td>
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<tr>
<td>4:40 pm</td>
<td>MS-TuA6</td>
<td>Application of Motorola IRONMAN Methodology to Equipment Reliability Improvement.</td>
<td>R. DUFFIN, K. MCCORMACK, Motorola SPS.</td>
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<td>5:00 pm</td>
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<td>Invited talk continued.</td>
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<tr>
<td>4:00 pm</td>
<td>MS-TuA7</td>
<td>INVITED The Development of the Fast Thermal Processor (FTP).</td>
<td>K. OKUMURA, Toshiba.</td>
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<td>4:20 pm</td>
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<td>Invited talk continued.</td>
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<td>5:00 pm</td>
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<td>Invited talk continued.</td>
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<td>Time</td>
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<td>Authors/Institutions</td>
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<td>Invited talk continued.</td>
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<tr>
<td>3:00 pm</td>
<td>BI-TuA4</td>
<td>Micrometre Resolution Molecular Patterning at Transducer Surfaces</td>
<td>J.M. COOPER, H. MORGAN, D.J. PRITCHARD, University of Glasgow, United Kingdom.</td>
</tr>
<tr>
<td>4:00 pm</td>
<td>BI-TuA7</td>
<td>Invited: Molecular Assemblies of Functionalized Polydiacetylenes</td>
<td>D.H. CHARCH, A. REICHERT, Lawrence Berkeley Laboratory. R. STEVENS, G. KUZEMKO, University of California, Berkeley. J. NAGY, Lawrence Berkeley Laboratory.</td>
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<td>4:20 pm</td>
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<td>Invited talk continued.</td>
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<tr>
<td>4:40 pm</td>
<td>BI-TuA9</td>
<td>Preparation of an Immunosensing Surface by a Mixed Self-Assembled Monolayer</td>
<td>P. HEIDUSKHA, J. RICKERT, W. BECK, G. JUNG, W. GROPEL, University of Tübingen, Germany.</td>
</tr>
<tr>
<td>5:00 pm</td>
<td>BI-TuA10</td>
<td>Hydration and Dehydration of Adsorbed Protein Monolayers on Different Surfaces</td>
<td>M. RODAHL, F. HÖK, B. KASEMÖ, Chalmers Univ. of Technology and Univ. of Göteborg, Sweden.</td>
</tr>
</tbody>
</table>
Tuesday Afternoon Poster Sessions

SURFACE SCIENCE
Room BR4 - Session SS-TuP pg. 198

Aspects of Surface Science
Moderator: J.C. Hemminger, University of California, Irvine.
Presenters present: 5:00-6:30 pm.

SS-TuP1 Simulation Studies of Electron Transfer at a Metal–Aqueous Electrolyte Interface. B.B. SMITH, J.W. HALLE, University of Minnesota.


SS-TuP4 The Interaction of HCl with Ultrathin Ice Films: Observation of Adsorbed and Absorbed States. J.D. GRAHAM, J.T. ROBERTS, University of Minnesota.

SS-TuP5 Differences in the Surface Chemistry of Amorphous and Crystalline Ice Thin Films. J.E. SCHAFF, J.T. ROBERTS, University of Minnesota.

SS-TuP6 SPA–RHEED – Spot Profile Analysis for In Situ Characterization. B. MÜLLER, M. HENZLER, Universität Hannover, Germany.


SS-TuP9 Atomically Resolved STM Study of the Interaction of Boron with Si(001). Y. WANG, R.J. HAMERS, University of Wisconsin, Madison.


SS-TuP13 STM Observations of Hydrogen–Induced Ag Cluster Formation on the Si(111)x3x3(R30°)–Ag Surface. K. KATAYAMA, Y. OHBA, Osaka Institute of Technology, Japan. H. OKISHI, Y. YAMAMOTO, K. OYUKI, Osaka University, Japan.

SS-TuP14 The Evolution of Growth Front in a Heteroepitaxial System: Pb on Cu(100). H. ZENG, G. VIDAL, Syracuse University.


SS-TuP18 Identification of Metastable bcc Ni Film Growth on Si(001) Surfaces Using High-Energy Ion Scattering, X-ray Photoelectron Diffraction, and X-ray Photoelectron Spectroscopy. V. SHUTHANANDAN, A.A. SALEM, R.J. SMITH, Montana State University.

SS-TuP19 The Initial Growth of Ultrathin Pd Films on Cu(001). J. YAO, Y.G. SHEN, D.J. O’CONNOR, University of Newcastle, Australia.


SS-TuP21 The Thermochromism of CBr4 on GaAs(100). M.T. MCELLESTRECK, D. SLOAN, J.M. WITHE, University of Texas, Austin.

SS-TuP22 Surface Chemistry of Dimethyl Hydrazine on GaAs(100). Y.-M. SUN, D. SLOAN, A. SCHWACHER, J.M. WHITE, University of Texas, Austin.

SS-TuP23 The Adsorption of Si on GaAs(001)–c(4x4): STM Evidence for Si Induced Disruption of As Layers. T.S. JONES, A.R. ALVERZ, D.M. HOLMES, M.R. FANT, B.A. JOYCE, Imperial College, United Kingdom.

SS-TuP24 Adsorption and Decomposition of Ammonia and Hydrazine on the GaAs(100)–c(8x2) Surface. E. APEN, J.L. GLAND, University of Michigan.

SS-TuP25 Chemical Reactions of Triethylammonium on GaAs(100). J.M. HEITZINGER, J.G. EKEROT, University of Texas, Austin.

SS-TuP26 The Interaction of Hydrogen Atoms with Ethyl Groups on Si(100). P.A. STEINER, J.M. HEITZINGER, M. MCELLESTRECK, D.J. BONNER, J.M. WHITE, University of Texas, Austin.


SS-TuP28 Surface Chemistry of Potential CVD Precursors. Dithylsilane and Dithiethylgermane on Ge(100). A. MAHJAN, B.K. KELLERMAN, S. RUSSELL, S. DANDRE J.E. EKEROT, A. TASCHEL, J.M. WHITE, University of Texas, Austin.

SS-TuP29 Surface Reactions of Ethylgermanes on Si(100). L. CHEN, L.A. KELLING, J.R. GU, C.M. GREENLEF, University of Missouri, Columbia.

SS-TuP30 An STM Study of Hydrogen Adsorption on Si(112). A.A. BASKI, L.J. WHITTMAN, Naval Research Laboratory.

*Finalist for the Morton M. Traum Award
NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room BR4 - Session NS-TuP pg. 203

Materials Characterization

Moderator: R.J. Hamers, University of Wisconsin, Madison.

Presenters present: 5:00-6:30 pm.


NS-TuP2 Chemistry in Nanoporous Materials: Semiconductor Particles Formed within a Solid Template. C.M. ZELENSKI, P.K. DORNOUT, Colorado State University.


NS-TuP5 The Origin of Uniaxiality in the Top Layer Relaxation of Zincblende (110) and Wurtzite (1010) Surfaces. G. BROWN, M. WELLEN, Texas A&M University.


NS-TuP8 Luminescence of Nanostructures Induced by Carriers Injected from a Scanning Tunneling Microscope Emitter. L. MONTEJUD, J. LINDEK, M.E. PISTUL, L. SAMUELSON, Lund University, Sweden.


NS-TuP18 Nanomaterials: Secondary boil corrosion of surfaces of conductive materials. R.Z. BAKHTIZIN, S.S. GHTS, Bashkir State University, Russia.

NS-TuP19 Many-Particle Effects in Tunneling of Electrons from Metal and Semiconductor Surfaces. M.A. KOZULSHINER, G.K. IVANOV, Russian Academy of Sciences.

NS-TuP20 STM/STS Investigation of Ion-Exchanged Polymer Multilayer LB Films: 20 Conductivity, Resonance Tunneling, Charge Superstructure. N. MASLOVA, Y.N. MOISEEV, V. NIKANDROV, V. PAVLOV, S. SAVINOV, R. YUSUPOV, Moscow State University, Russia.

NS-TuP21 Determination of Large Heterogeneous Rate Constants using Nanoelectrode Ensembles. V.P. MENON, C.R. MARTIN, Colorado State University.
Tuesday Afternoon Poster Sessions

APPLIED SURFACE SCIENCE
Room BR4 - Session AS-TuP pg. 206

Aspects of Applied Surface Science
Moderator: D.R. Baer, Battelle Pacific Northwest Laboratories.
Presenters present: 5:00-6:30 pm.

AS-TuP1 A Study of Vapor-Deposited Polythiophene Thin Films.

AS-TuP2 Highly Ordered Thin Films of Phthalocyanines Created by Self-Assembly and Langmuir Blodgett Deposition.

AS-TuP3 Metal Overlayers on Self-Assembled Monolayers: 5. ISS and XPS of the Ag/COOH Interface.
B.C. HEDIG, A.V. CZANDER-NA, National Renewable Energy Laboratory.

AS-TuP4 Quantitative Analysis by Static SIMS of Mixed Self-Assembled Monolayer Films on Metal Substrates.
Y.-Z. DU, R.W. JOHNSON, J.R. GARDELLA, State University of New York, Buffalo.

AS-TuP5 Temperature Controlled TOF-SIMS Investigations of Polymer Materials.
M. DEIMEL, B. HAGENHOFF, A. BENNINGHOVEN, Universität Münster, Germany.

AS-TuP6 Radiation Damage on Some Organic Materials during S-SIMS and XPS Analysis.
M. KUDD, T. NISHIHARA, Seikei University, Japan. T. HOSHI, ULVAC-PHI, Inc., Japan.

AS-TuP7 XPS Characterization of Nylons.
S.N. RAMAN, Monsanto.

AS-TuP8 Surface Characterization of Silylated Substrates by TOF-SIMS.
B. HAGENHOFF, M. DEIMEL, D. RADING, A. BENNINGHOVEN, J. GROBE, Universität Münster, Germany.

AS-TuP9 Characterization of an Abrasion Resistant Coating on Polycarbonate.
R.M. FRIEDMAN, Monsanto Corporate Research. R.W. LINTON, M.P. MAVIN, University of North Carolina, Chapel Hill.

AS-TuP10 Fundamental Characterization of Siloxane Adhesion Promoters on Metal and Metal Oxide Surfaces.
S. MISHRAM, J.J. WEIMER, University of Alabama, Huntsville.

AS-TuP11 Film Growth and Surface Structure of Methylene Blue on Mica.
G. HÄHNERT, W.R. CASEY, N.D. SPENCER, ETH Zürich, Switzerland.

AS-TuP12 Nanometer Resolution Imaging of the Growth of Water Monolayers on Mica from its Vapor.
J. HU, S. XIAO, D.F. OGLETREE, M. SALMERÓN, Lawrence Berkeley Laboratory.

AS-TuP13 Surface Finish Characterization of Potassium Dihydrogen Phosphate (KDP) for Nonlinear Optical Applications.
E.C. HONEA, R.L. MOEACHEN, Lawrence Livermore National Laboratory.

AS-TuP14 Counterion and Dopant Induced Effects on the Structure of Electropolymerized Polyaniline Thin Films.
T.H. PORTER, A.G. SYKES, Northern Arizona University.

AS-TuP15 Contribution of Surface Roughness in the Nanometer Regime to the Depth Resolution in Sputter Profiling.

AS-TuP16 Influence of Ion Species, Ion Energy and Substrate on Sputtering-Induced Ripple Topography.

AS-TuP17 Use of Resonance Ionization Microprobe Analysis in Material Sciences.
H.F. ARLINGHAUS, C.F. JOYNER, T.J. WHITAKER, Atom Sciences, Inc.

AS-TuP18 Depth-Resolved Vibrational Characterization of Buried Interfaces.
N.H. FONTAINE, I.E. FURTAK, Colorado School of Mines.

AS-TuP19 Thickness Determination of Uniform Overlayers on Rough Substrates by Angle Dependent X-ray Photoelectron Spectroscopy.

AS-TuP20 Angle-resolved XPS Data Acquisition.
B.J. TIELSCH, J.E. FULGHEY, Kent State University.

AS-TuP21 XPS Studies of Compositional Changes Induced by Ion Bombardment on LaPO4 Surface.
D.P. IVANOVA, A.V. NAUMKIN, I.A. VASILEYEV, V.I. RAKHOVSKY, Research Center for Surface and Vacuum Investigation, Russia.

S.J. SCHMIDT, D.N. BELLON, D.R. MONROE, General Motors Research & Development Center.

M. WATAMORI, K. ONDA, Osaka University, Japan. T. NAKAMURA, Sumitomo Electric Industries, Ltd., Japan.

AS-TuP24 Negative Charge Production from Bombardment of a Cu Surface by Low Energy Oxygen Atoms and Ions.
T.M. STEPHEN, R.C. AMME, B. VAN ZYL, University of Denver.

AS-TuP25 The Effect of Copper Overlayers on the Thermal Stability of Chromium Carbide on HOPG.

AS-TuP26 Electric States of Segregated Metal Atom on Metal Surfaces and Potential Use for Field Emitter.
M. YOSHITAKE, K. YOSHITAKE, National Research Institute for Metals, Japan.

AS-TuP27 The Effect of Sample Bias on the Nitrogen Etching Products of Aluminum.
M.A. ROEKE, P.M.A. SHERWOOD, Kansas State University.

AS-TuP28 Local Density of State Analysis by AES.
H.J. ZHOU, Y.C. WANG, W. HSING, Tsinghua University, China.

AS-TuP29 The Development of an Electron Spectroscopy Data Dictionary for the AVS Surface Science Spectral Database.
E.W. GARRENSTROM, General Motors R&D Center.

AS-TuP30 A Generic Analytical Data Model: Ensuring that Surface Science Data Meet Analytical Chemistry Standards.
R.N. LEE, Naval Surface Warfare Center.

#Candidate for the Applied Surface Science Division Award
EM-TuP1  Thermal Oxidation of Heteroepitaxial Si$_{1-x}$Ge$_x$C$_y$ Alloy Thin Films. J. Xiang, N. Herbots, P. Ye, S. Hearne, Arizona State University.

EM-TuP2  The Role of Temperature during Synthesis of Heteroepitaxial Si$_{1-x}$Ge$_x$C$_y$/Si(001) Synthesized by Combined Ion and Molecular Beam Deposition (CIMD). N. Herbots, P. Ye, J. Xiang, S. Hearne, Arizona State University.

EM-TuP3  Growth of Si$_{1-x}$Ge$_x$ Multiple Quantum Wells using Molecular Beam Epitaxy for Infrared Absorption. T. Krefeld, Air Force Institute of Technology, P.E. Thompson, Naval Research Laboratory.


EM-TuP9  Structural and Chemical Changes during Annealing of Gas-Phase Polysulfide Passivated InP. R.W.M. Kwok, G. Jin, The Chinese University of Hong Kong, L. Huang, W.M. Lau, The University of Western Ontario, Canada, C.C. Hsu, Institute of Semiconductors, PR China.


EM-TuP16  Comparison of Trisdimethylaminoantimony and Sb as Sources for Growth of GaSb, AlSb and InSb by Metalorganic Molecular Beam Epitaxy. C.R. Aberti, University of Florida, Gainesville.


EM-TuP22  Low Pressure Metalorganic Chemical Vapor Deposition of GaN on c-plane (0001) Sapphire Substrates. C.Y. Huang, W.I. Mayo, Y. Lu, Rutgers University, H. Liu, Emcore Corporation.

EM-TuP23  Growth of Crystalline 3C–SiC Films on 6H–SiC at 900°C by Chemical Vapor Deposition. C. Yuan, A.J. Steckly, University of Cincinnati, J. Chaudhuri, R. Ghokal, Wichita State University, M.J. Loboda, Dow Corning Corporation.


MANUFACTURING SCIENCE AND TECHNOLOGY
Room BR4 - Session MS-TuP  pg. 215

Manufacturing Science and Technology
Moderator: G.W. Rubloff, North Carolina State University.

Presenters present: 5:00-6:30 pm.


MS-TuP3  Magnetron RIE without Charge-up Damage Using a Dipole-ring Magnet. T. SAKAI, M. SAKAE, M. SEKINE, S. KOBAYASHI, N. KANAZAWA, TOYOTA, Japan.


THE AMERICAN VACUUM SOCIETY

cordially invites you
to attend
the Awards Assembly
on
Wednesday, October 26, 1994
in
Ballroom 1
Colorado Convention Center
at 6:45 p.m.
followed by the
Awards Reception
<table>
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<tr>
<th>Time</th>
<th>Session</th>
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<th>Authors/Institutions</th>
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<tr>
<td>8:00 am</td>
<td>SS1-WeM3</td>
<td>Spin-Polarized Electron Measurements: Foundations of Surface Magnetism Research.</td>
<td>R.J. Celotta*, B.T. Pierce*, National Institute of Standards &amp; Technology</td>
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<td>Invited talk continued.</td>
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<td>S. Chiang, H. Poppa, IBM Almaden Research Center.</td>
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<tr>
<td>9:00 am</td>
<td>SS1-WeM3</td>
<td>Scanning Ion Microscopy with Polarization Analysis (SIMPA) to Study Domain Structures of</td>
<td>N.L. Nunes, Rice University.</td>
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<td>Metal Overlayers on Fe and Pd Single Crystal Surfaces.</td>
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<td>H.P. Oepen, 10V-XFA Julich, Germany.</td>
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<td>G. Steierl, C. Rau, Rice University.</td>
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<td>9:20 am</td>
<td>SS1-WeM4</td>
<td>Spin-Resolved X-ray Photoelectron Spectroscopy Study of the 2p_{3/2} Level of Ferromagnetic</td>
<td>A.K. Lee, L.E. Klemeloff, Lehigh University.</td>
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<td>10:00 am</td>
<td>SS1-WeM6</td>
<td>Ferromagnetic Phases at the Fe/Si(111) Interface.</td>
<td>J.P. Woods, Y.L. He, O. Wu, University of Nebraska.</td>
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<tr>
<td>10:20 am</td>
<td>SS1-WeM7</td>
<td>Circular Dichroism in Core-Level Photoemission.</td>
<td>W. Xiao, Univ. of California. Davis &amp; Lawrence Berkeley Laboratory.</td>
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<td>A.P. Kadowela, M.A. Van Hove, Lawrence Berkeley Laboratory.</td>
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<td>C.S. Fadley, Univ. of California. Davis &amp; Lawrence Berkeley Laboratory.</td>
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<td>10:40 am</td>
<td>SS1-WeM8</td>
<td>Spin-Specific Photoelectron Diffraction Using Magnetic X-ray Circular Dichroism.</td>
<td>J.B. Tobi, G.D. Waddill, Lawrence Livermore National Laboratory.</td>
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<td>X. Liu, S.Y. Tong, University of Wisconsin, Milwaukee.</td>
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<td>8:00 am</td>
<td>NSMS-WeM0</td>
<td>Industrial Applications of Friction Force Microscopy</td>
<td>J. Berger, A. Lejali, M. Bruggeli, R. Christof, CSEM, Switzerland</td>
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<tr>
<td>8:20 am</td>
<td>NSMS-WeM1</td>
<td>Invited: Industrial Applications of Proximal Probes</td>
<td>H. Fuchs, Universität Münster, Germany</td>
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<td>8:40 am</td>
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<td>Invited talk continued</td>
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<td>9:00 am</td>
<td>NSMS-WeM3</td>
<td>Lateral and Vertical Dopant Profiling in Semiconductors by</td>
<td>P. de Wolf, T. Clarisse, W. Vandervorst, IMEC, Belgium, L. Hellemans, J. Enauwer Vending, University of Leuven, Belgium</td>
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<td>9:20 am</td>
<td>NSMS-WeM4</td>
<td>Scanning Tunneling Microscopy/Spectroscopy Doping Determination of Si and GaAs Bulk Materials and Device Structures</td>
<td>R.M. Silver, J.A. Dagata, W. Tseeng, National Institute of Standards &amp; Technology</td>
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<tr>
<td>9:40 am</td>
<td>NSMS-WeM5</td>
<td>Invited: Ultra-High Density Data Storage Based on Proximal Probes</td>
<td>H.J. Marin, B. Terris, S. Hoem, L.S. Fan, D. Rugan, IBM Almaden Research Center</td>
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<td>10:00 am</td>
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<td>Invited talk continued</td>
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<td>10:20 am</td>
<td>NSMS-WeM7</td>
<td>Quantitative Dopant Profile Measurements on Si by Scanning Capacitance Microscopy</td>
<td>Y. Huang, C.C. Williams, University of Utah, J. Sun Man, IBM Microelectronics</td>
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<td>10:40 am</td>
<td>NSMS-WeM8</td>
<td>Two-Dimensional Delineation of Semiconductor Doping by Scanning Resistance Microscopy</td>
<td>J.N. Xu Malo, D. Shimizu, C. Shafai, D.J. Thomson, University of Manitoba, Canada</td>
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<td>8:00 am</td>
<td>AS-WeM0</td>
<td>Depth-Profiling by Angle-Resolved XPS and AES; Elastic Scattering Corrections, and Intrinsic Limits on Depth-Resolution</td>
<td>P.J. Cumpson, National Physical Laboratory, United Kingdom.</td>
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<tr>
<td>8:20 am</td>
<td>AS-WeM1</td>
<td>Assessment of Overlayer Thickness Determination Model by Controlled Monolayers</td>
<td>B.C. Beard, Akzo Nobel, R.A. Brizolara, Naval Surface Warfare Center.</td>
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<tr>
<td>8:40 am</td>
<td>AS-WeM2</td>
<td>Inelastic Mean Free Pathlengths of X-ray Photoelectrons for Quantitative Investigation of Technological Ultrathin Layers</td>
<td>W.H. Gries, Deutsche Bundespost Telekom, Germany.</td>
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<tr>
<td>9:00 am</td>
<td>AS-WeM3</td>
<td>Corrections to the Angle-Resolved X-ray Photoelectron Spectroscopy with Consideration of the Solid Acceptance Angle</td>
<td>K.W. Wong, R.W.M. Kwok, The Chinese University of Hong Kong.</td>
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<tr>
<td>9:20 am</td>
<td>AS-WeM4</td>
<td>Variation of Effective Sampling Depths with Direction in Core Photoelectron Emission from Single Crystals</td>
<td>R.X. Ynunza, Univ. of California, Davis &amp; Lawrence Berkeley Laboratory.</td>
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<tr>
<td>9:40 am</td>
<td>AS-WeM5</td>
<td>A Novel Algorithm for Quantitative Elemental Depth Profiling by Angle-Resolved XPS</td>
<td>J.M. Williams, T.P. Beebe, Jr., University of Utah.</td>
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<tr>
<td>10:00 am</td>
<td>AS-WeM6</td>
<td>A Comparison of XPS Quantitation using Electrostatic and Magnetic Lens-Based Analyses</td>
<td>D.J. Surman, A.R. Walker, Kratos Analytical Inc.</td>
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<tr>
<td>10:20 am</td>
<td>AS-WeM7</td>
<td>Quantification in Molecular SIMS</td>
<td>B. Hagenhoff, K. Meyer, M. Beinmel, D. Rading, W. Sichtermann, A. Benninshoven, Universitat Munster, Germany.</td>
</tr>
<tr>
<td>11:00 am</td>
<td>PS-WeM9</td>
<td>Laser-Induced Thermal Desorption as an In Situ Surface Probe during Plasma Processing</td>
<td>I.F. Herman, AT&amp;T Bell Laboratories and Columbia University.</td>
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<tr>
<td>11:20 am</td>
<td>PS-WeM11</td>
<td>Transport of Ions through High Aspect Ratio Apertures: Simultaneous, High Resolution Measurement of Angular and Energy Distribution Functions</td>
<td>B. Quinio, Lawrence Livermore National Laboratory.</td>
</tr>
<tr>
<td>11:40 am</td>
<td>PS-WeM2</td>
<td>Measurement of the Electron Energy Distribution Function with a Differentially Pumped Spherical Sector Energy Analyzer</td>
<td>K.D. Schatz, D.N. Rusig, University of Illinois, Urbana.</td>
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<tr>
<td>11:40 am</td>
<td>PS-WeM4</td>
<td>Spatial Distribution of Argon Metastables in RF Plasmas Measured by 2-D LIF Imaging</td>
<td>B.K. McMillin, M.R. Zachariah, National Institute of Standards &amp; Technology.</td>
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<tr>
<td>11:40 am</td>
<td>PS-WeM5</td>
<td>Invited</td>
<td>Diagnostics and Control of Radicals in an Inductively-Coupled Etching Reactor</td>
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<td>11:40 am</td>
<td>PS-WeM7</td>
<td>The Third Peak in 'Bimodal' Ion Energy Distributions</td>
<td>D. Vender, R.J.M.M. Snijders, G.W.M. Kroesen, F.J. De Hoo, Eindhoven University of Technology, The Netherlands.</td>
</tr>
<tr>
<td>11:40 am</td>
<td>PS-WeM8</td>
<td>Monitoring InP and GaAs Etched in Cl2/Ar Plasma Generated by Electron Cyclotron Resonance Source using Optical Emission Spectroscopy</td>
<td>S. Thomas III, K.K. Ko, S.W. Pang, University of Michigan.</td>
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* John Coburn and Harold Waters Award Nominee
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<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Presenter(s)</th>
<th>Location</th>
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<tbody>
<tr>
<td>8:00 am</td>
<td>SSEM-WeMo</td>
<td>Structure Sensitivity of Trimethylgallium Chemisorption on GaAs Surfaces</td>
<td>J.R. CREIGHTON, Sandia National Laboratories</td>
<td>Room A102 - Session SSEM-WeMo</td>
</tr>
<tr>
<td>8:20 am</td>
<td>SSEM-WeM1</td>
<td>Molecular Beam Scattering as a Probe of Thin Film Deposition Processes</td>
<td>J.R. ENGSTROM, Cornell University</td>
<td>Room A105 - Session TF-WeM</td>
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<tr>
<td>8:40 am</td>
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<td>Invited talk continued.</td>
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<tr>
<td>9:00 am</td>
<td>SSEM-WeM3</td>
<td>GaF₃ Thin Films Grown on GaAs via Exposure to XeF₂</td>
<td>W.C. SIMPSON, P.R. VAREKAMP, J.A. YARMOFF, University of California,</td>
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<td>Riverside &amp; Lawrence Berkeley Lab</td>
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<td>9:20 am</td>
<td>SSEM-WeM4</td>
<td>Initial Stages of Phosphorous Desorption from InP(110) Surfaces</td>
<td>PH. EBERT, University of Wisconsin, Madison, M. HEUROCH, M. SIMON, K. URBAN,</td>
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<td>Institut für Festkörperforschung, Germany, M.G. LAGALLY, University of</td>
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<td>Wisconsin, Madison</td>
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<td>9:40 am</td>
<td>SSEM-WeM5</td>
<td>Interaction of 50 eV Electrons with D₂O on GaAs(100): Electron and Thermal Induced Oxidation</td>
<td>D.W. SLOAN, Y.-M. SUN, J.M. WHITE, University of Texas, Austin</td>
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<tr>
<td>10:00 am</td>
<td>SSEM-WeM6</td>
<td>Characterization of Silicon Surfaces and Interfaces by Vibrational Spectroscopy</td>
<td>Y.J. CHABAL, M.A. HINES, D. FEUDD, AT&amp;T Bell Laboratories</td>
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<td>10:20 am</td>
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<td>Invited talk continued.</td>
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<td>10:40 am</td>
<td>SSEM-WeM8</td>
<td>Surface IR Investigation of Arsine Adsorption on GaAs(001)</td>
<td>H. DI, P.E. GEE, R.F. HICKS, University of California, Los Angeles.</td>
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<tr>
<td>8:00 am</td>
<td>EM-WeM5</td>
<td>MBE Growth of II–VI Blue/Green Laser Diodes</td>
<td>D.C. Girollo, M.D. Ringle, J. Han, R.L. Gunshor, G.C. Hua, Purdue University, A. Salokave, H. Jeon, A. V. Nurmikko, Brown University.</td>
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<tr>
<td>8:20 am</td>
<td>EM-WeM1</td>
<td>Invited: MBE Growth of ZnMgSSe and its Application to Blue and Green Laser Diodes.</td>
<td>M. Ikeda, A. Ishibashi, Sony Corporation Research Center.</td>
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<td>8:40 am</td>
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<td>Invited talk continued.</td>
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<td>9:00 am</td>
<td>EM-WeM3</td>
<td>Structural and Compositional Characterization of HgSe Contacts to p-ZnSe.</td>
<td>J.J. Furl, P.H. Holloway, J. Trexler, J. Kim, K. Jones, University of Florida, Gainesville.</td>
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<tr>
<td>9:20 am</td>
<td>EM-WeM4</td>
<td>Deep Level Interface States Near ZnSe/GaAs(100) Heterointerfaces.</td>
<td>A.D. Rasanen, L.J. Brillson, Xerox Webster Research Center, G. Bratina, L. Sorba, A. Franciosi, University of Minnesota.</td>
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<td>10:00 am</td>
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**MANUFACTURING SCIENCE AND TECHNOLOGY**

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<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Authors</th>
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<tbody>
<tr>
<td>8:00 am</td>
<td>MS-WeM0</td>
<td>Long-throw Low-pressure Sputtering Technology for ULSI Devices.</td>
<td>Y. Kadokura, Y. Kashimoto, T. Kiyota, N. Motebi, I. Nakayama, ULVAC Japan, Ltd.</td>
</tr>
<tr>
<td>8:40 am</td>
<td>MS-WeM2</td>
<td>Interlayer Dielectrics with Low Dielectric Constant for Multilevel Interconnection.</td>
<td>R. Aoki, N. Hayasaka, Y. Nishiya, A. Miyajima, Y. Nakasak, H. Okano, Toshiba Corporation, Japan.</td>
</tr>
<tr>
<td>9:00 am</td>
<td>MS-WeM3</td>
<td>Manufacturing Issues of Electrostatic Chucks.</td>
<td>D.R. Wright, L. Chen, P. Federlin, K. Forbes, SEMATECH.</td>
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<tr>
<td>10:00 am</td>
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<td>Invited talk continued.</td>
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<tr>
<td>10:20 am</td>
<td>MS-WeM8</td>
<td>The Complex Impedance of a Dusty Processing Plasma.</td>
<td>W.C. Roth, R.H. Carlile, University of Arizona.</td>
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<td>Time</td>
<td>Speaker(s) and Title</td>
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<tr>
<td>8:00 am</td>
<td><strong>BINS-WeM0</strong> Electrical Characterization of Artificial Neuronal Networks. J.J. HICKMAN, K.E. FOSTER, R.C. OPRISON, Science Applications International Corporation, D.A. STENGER, Naval Research Laboratory. A.E. SHAFFNER, J.L. BARKER, National Institutes of Health.</td>
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<tr>
<td>8:20 am</td>
<td><strong>BINS-WeM1</strong> Neuronal Cells Cultured on Modified Microelectronic Device Surfaces. A. OFFENHAUSSE, J. RUHE, Frontier Research Program, RIKEN, Japan. W. KNOLL, RIKEN, Japan &amp; Max-Planck-Institute of Polymer Res., Germany.</td>
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<td>8:40 am</td>
<td><strong>BINS-WeM2</strong> Using both Topographic Control and Micropatterned Protein Substrates in Controlling Neuron Extension and Connection in Culture. A.S.G. CURTIS, S. BRITLAND, C.D.W. WILKINSON, University of Glasgow, United Kingdom.</td>
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<td>9:00 am</td>
<td><strong>INVITED</strong> Photocoehemically-driven Surface Modifications and Cellular and Biomolecular Assemblies with Micron-order Precision. T. MATSUOA, National Cardiovascular Center Research Institute, Japan.</td>
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<td>9:20 am</td>
<td>Invited talk continued.</td>
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<td>9:40 am</td>
<td><strong>BINS-WeM5</strong> Cell Patterns and Whole-Cell Biosensors using Photonic Tweezers Technology. J. RENKEN, S. SEEGER, Universität Heidelberg, Germany.</td>
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<td>10:00 am</td>
<td><strong>BINS-WeM6</strong> Modification and Refunctionalization of Fluoropolymers for Controlling and Directing Neurons at Interfaces. T.G. VARGO, et al., State University of New York, Buffalo.</td>
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<tr>
<td>10:40 am</td>
<td><strong>BINS-WeM8</strong> Patterning of Microtubules Observed with Atomic Force Microscopy. D.C. TURNER, C. CHANG, S.L. BRANDOW, Naval Research Laboratory. O.B. MURPHY, Johns Hopkins University Medical School.</td>
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MY SCHEDULE
Wednesday Morning, October 26, 1994

**TIME** | **SESSION** | **ROOM**
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8:20 am | | |
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**OTHER EVENTS WEDNESDAY**
7:00 a.m. Companions Program (see insert) (Silver Room (H))
8:00 a.m. Newsletter/Publicity Breakfast (Colorado Room (H))
9:00 a.m. Low Pressure Gauges Subcommittee (Spruce Room (H))
10:00 a.m. Scholarship Luncheon (Birch Room (H))
12:00 Noon Scholarship Luncheon (Aspen Room (H))

H-Radisson Hotel
CC—Colorado Convention Center

**SHORT COURSES WEDNESDAY**
8:30 a.m. Vacuum Technology (Room C109 (CC))
8:30 a.m. A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RBS (Room C107 (CC))
8:30 a.m. Physics, Chemistry, and Mechanics of Adhesion (New) (Room C106 (CC))
8:30 a.m. Cryopump Technology (Room C103 (CC))
8:30 a.m. Partial Pressure Analyzers: Analysis and Applications (Room C101 (CC))
8:30 a.m. Vacuum Sealing and Joining Techniques (Room C108 (CC))
8:30 a.m. An Introduction to Ellipsometry (Room C106 (CC))
8:30 a.m. Plasma Etching and RIE (Room C104 (CC))
8:30 a.m. Secondary Ion and Neutral Mass Spectrometry (SIMS, SIMS, SALI) (Room C107 (CC))
8:30 a.m. Surface Preparation for Thin-Film Deposition (Room C110 (CC))

**Special or Focus Area Sessions**
Silicon-based Optoelectronics
EM-WeM Heterostructures for Optoelectronics
Sensors, in-situ Diagnostics, Process Control
PS-WeM Plasma Diagnostics
Nanostructure Fabrication and Atomic-scale Manipulation
NS2-WeM Atomic Manipulation
### MY SCHEDULE
**Wednesday Afternoon, October 26, 1994**

<table>
<thead>
<tr>
<th>TIME</th>
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### OTHER EVENTS WEDNESDAY
- 5:20 p.m. PST Division Business Meeting *(Room A109 (CC))*
- 5:20 p.m. Vacuum Metallurgy Division Business Meeting *(Room A106 (CC))*

H=Radisson Hotel  
CC=Colorado Convention Center

### SHORT COURSES WEDNESDAY
- 8:30 a.m. Vacuum Technology *(Room C109 (CC))*
- 8:30 a.m. A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RBS *(Room C107 (CC))*
- 8:30 a.m. Physics, Chemistry, and Mechanics of Adhesion *(New)* *(Room C105 (CC))*
- 8:30 a.m. Cryoupd Technology *(Room C103 (CC))*
- 8:30 a.m. Partial Pressure Analyzers: Analysis and Applications *(Room C101 (CC))*
- 8:30 a.m. Vacuum Sealing and Joining Techniques *(Room C108 (CC))*
- 8:30 a.m. An Introduction to Ellipsometry *(Room C106 (CC))*
- 8:30 a.m. Plasma Etching and RIE *(Room C104 (CC))*
- 8:30 a.m. Secondary Ion and Neutral Mass Spectrometries (SIMS, SIMS, SALI) *(Room C107 (CC))*
- 8:30 a.m. Surface Preparation for Thin-Film Deposition *(Room C110 (CC))*

### Special or Focus Area Sessions
- **Silicon-based Optoelectronics**
- **EM-WeA** Wide-bandgap Nitrides
- **NS2-WeA** Optical Properties of Silicon Nanostructures
- **Sensors, In-situ Diagnostics, Process Control**
- **MS-WeA** Diagnostics, Sensors, and Control
- **Surface Contamination and Control**
- **PS-WeA** Plasma-Induced Charging and Contamination Effects
- **Nanostructure Fabrication and Atomic-Scale Manipulation**
- **TF-WeA** Deposition and Characterization Techniques of Nanostructures in Thin Films
**Wednesday Afternoon, October 26, 1994**

**SURFACE SCIENCE**  
Room A205 - Session SS1-WeA  pg. 233

**Surface Magnetism II**  
**Moderator:** B.P. Tonner, University of Wisconsin, Milwaukee.

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<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker(s)</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>Experimental Studies of Surface and Interface Magnetism. H. HÖPSTEIN</td>
<td>University of California, Irvine.</td>
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<td>2:20 pm</td>
<td>Invited talk continued</td>
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<td>2:40 pm</td>
<td>Strain Accommodation and Atomic Order in the Non-pseudomorphic Growth</td>
<td>S. E. FOWLER, J. V. BARTH, IBM Research Division</td>
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<td>of Ultrathin Fe Films on Cu(001).</td>
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<tr>
<td>3:00 pm</td>
<td>Magnetic Structure of Ultra-thin Mn Films on Fe(100)/Pd(100).</td>
<td>H. G. STEIER, C. R. AU, Rice University.</td>
</tr>
<tr>
<td>3:20 pm</td>
<td>Mössbauer Spectroscopy Study of Tetragonal-Distorted fcc Fe(100).</td>
<td>J. W. FREELAND, D. F. STORM, I. L. GRIBOROV,</td>
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<td>Invited</td>
<td>D. J. KEAVNEY, J. C. WALKER, Johns Hopkins University.</td>
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<tr>
<td>3:40 pm</td>
<td>Magnetic Properties of Fe and Mn Overlayers on Ir(111) by Soft X-ray</td>
<td>W. L. O'BRIEN, B. P. TONNER, University of Wisconsin, Milwaukee.</td>
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<td></td>
<td>Circular Dichroism.</td>
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<td>4:00 pm</td>
<td>Impact of Magnetism on the Stability of Ultrathin Films: The</td>
<td>S. BLÜGEL, IFF, Forschungszentrum Jülich, Germany.</td>
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<td>Magnetically Driven Two Dimensional Surface Alloy.</td>
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<td>4:20 pm</td>
<td>Invited talk continued</td>
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<td>4:40 pm</td>
<td>Enhanced Exchange Splitting of Gd at the Monolayer Limit.</td>
<td>D. LI, Argonne National Laboratory. J. ZHANG,</td>
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<td>Invited</td>
<td>P. A. DOWBEN, University of Nebraska, Lincoln.</td>
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<td>M. SNELLION, University of Wisconsin, Madison.</td>
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<tr>
<td>5:00 pm</td>
<td>The Connection between Morphology and Magnetic Characteristics for</td>
<td>S. E. TÖBER, R. K. YAMAZAKI, Lawrence Berkeley Laboratory. C. S. FAOLEY, University of California, Davi</td>
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<td>Gd Thin Films Grown on W(110).</td>
<td>Lawrence Berkeley Laboratory.</td>
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<td>Invited</td>
<td>University of California, Davis &amp; Lawrence Berkeley Laboratory.</td>
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**SURFACE SCIENCE**  
Room A201 - Session SS2-WeA  pg. 234

**Solid Liquid Interfaces**  
**Moderator:** E. M. Stuve, University of Washington.

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<th>Time</th>
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<th>Speaker(s)</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>Surface Reconstruction at the Metal-Electrolyte Interface. D. M.</td>
<td>KOLB, University of Ulm, Germany.</td>
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<td>INvited</td>
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<td>Electrode in Sulfate Media.</td>
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<td>3:00 pm</td>
<td>Characterization of Carbon and Sulfur Covered Pt(111) and their</td>
<td>D. E. SAUER, R. L. BORUP, E. M. STUVE, University of Washington.</td>
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<td>Influence on CO Adsorption and Electrooxidation.</td>
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<tr>
<td>3:40 pm</td>
<td>Magnetic Properties of Fe and Mn Overlayers on Ir(111) by Soft X-ray</td>
<td>W. L. O'BRIEN, B. P. TONNER, University of Wisconsin, Milwaukee.</td>
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<td>Circular Dichroism.</td>
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<td>4:00 pm</td>
<td>In Situ EXAFS of Cu on Pt under Electrochemical Control. L. WANG,</td>
<td>T. B. LÉVY, L. P. PAINT, R. M. HAYES, Rensselaer Polytechnic Institute.</td>
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<td>4:20 pm</td>
<td>Model Electrochemical Interfaces in Ultrahigh Vacuum: Ionic and</td>
<td>N. KIZHVERAVIAM, I. VILLEGAS, Purdue University.</td>
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<td>Surface Solvation Probed by Infrared Spectroscopy. M. J.</td>
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### NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY/BIOMATERIAL INTERFACES  
Room A209 - Session NSBI-WeA  pg. 236

**Biology at the Nanoscale: I**  
Moderator: L. Bottomley, Georgia Institute of Technology.

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<th>Time</th>
<th>Session</th>
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<th>Author/Institution</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>NSBI-WeA1</td>
<td>Non-contact Scanning Force Microscopy of F-actin. D. BRAUNSTEIN, J.A. SPUDICH, Stanford University.</td>
<td></td>
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<tr>
<td>2:40 pm</td>
<td>NSBI-WeA3</td>
<td>Imaging Biological Systems with Near-Field Scanning Optical Microscopy. J. HWANG, The Johns Hopkins University; E. BETZIS, Bell Laboratories; M. EDIDIN, The Johns Hopkins University.</td>
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<td>3:00 pm</td>
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<td>Invited talk continued.</td>
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<td>3:20 pm</td>
<td>NSBI-WeA5</td>
<td>Imaging Individual Protein Molecules by Scanning Probe Microscopy. S.L. TANG, A.J. MCQHIE, DuPont Central Research and Development.</td>
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<tr>
<td>3:40 pm</td>
<td>NSBI-WeA6</td>
<td>Nanometer-Scale Modification of Biological Membranes by Field Emission Scanning Tunneling Microscopy. J. TAMAYO, R. GARCEA, Centro Nacional de Microelectrónica, Spain.</td>
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<td>4:00 pm</td>
<td>NSBI-WeA7</td>
<td>SPM Studies of Supramolecular Architecture at Biological Interfaces. M. HARA, W. KNOLL, RIKEN, Japan.</td>
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<td>4:20 pm</td>
<td>NSBI-WeA8</td>
<td>Control of the Adsorption of the Photoreactive Biological Purple Membrane to Surfaces using Self-Assembled Monolayers. R.A. BRIZZOLARA, Naval Surface Warfare Center.</td>
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<tr>
<td>4:40 pm</td>
<td>NSBI-WeA9</td>
<td>Force Modulation Imaging of Protein Membranes. H. YAMADA, Joint Research Center for Atom Technology, Japan; Y. HIRATA, J. MIYAKE, National Institute for Bioscience &amp; Human-technology, Japan.</td>
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<tr>
<td>5:00 pm</td>
<td>NSBI-WeA10</td>
<td>LB Films of Disintegrated Purple Membranes: Photo-electrical Properties and STM Investigation. E.A. FEDOROV, V.V. KISLOV, V.P. PANOV, A.A. KONONENKO, E.P. LUKASHEV, D.S. CHERNAV-SKI, Russian Academy of Science.</td>
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### NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY  
Room A207 - Session NS2-WeA  pg. 237

**Optical Properties of Silicon Nanostructures**  
Moderator: D.J. Glembocki, Naval Research Laboratory.

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<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Author/Institution</th>
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<tr>
<td>2:00 pm</td>
<td>NS2-WeA1</td>
<td>Invited talk continued.</td>
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<tr>
<td>2:20 pm</td>
<td>NS2-WeA3</td>
<td>Visible Luminescence from Plasma Deposited Nanocrystalline Silicon Thin Films. R.E. HOLLINGSWORTH, Materials Research Group, Inc; M. ESTES, University of Colorado; Boulder; C. DENT, P.K. BHAT, Materials Research Group, Inc.</td>
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<tr>
<td>2:40 pm</td>
<td>NS2-WeA4</td>
<td>The Mechanism of Room Temperature Red Light Emission in Porous Silicon and Fabricated Silicon Nanostructures. S.M. PROKES, W.E. CARLOS, O.J. GLEMBOCKI, Naval Research Laboratory.</td>
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<tr>
<td>3:00 pm</td>
<td>NS2-WeA5</td>
<td>Properties of Ultrathin Films of Porous Silicon. J. VON BEHREN, Univ. of Rochester &amp; Technical University of Munich, Germany; Y. KOSTOLAS, L. TSYBEKOV, University of Rochester; J.V. VANDYSHEV, University of Rochester and Moscow State University, Russia. P.M. FAUCHET, University of Rochester.</td>
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<tr>
<td>3:40 pm</td>
<td>NS2-WeA7</td>
<td>Visible Electroluminescence from Ultrathin Stain-Etched Porous Films. J. XU, A.J. STECKL, University of Cincinnati.</td>
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<tr>
<td>4:00 pm</td>
<td>NS2-WeA8</td>
<td>Properties of Partially Oxidized Porous Silicon. L. TSYBEKOV, P.M. FAUCHET, University of Rochester.</td>
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<tr>
<td>4:20 pm</td>
<td>NS2-WeA9</td>
<td>Enhancement and Suppression of the Formation of Porous Silicon. S.P. DUTTADUPTA, C. TENIS, P.M. FAUCHET, University of Rochester; S.K. KURNEC, Rochester Institute of Technology; T. BLANTON, Eastman Kodak Company.</td>
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<td>Time</td>
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<td>2:00 pm</td>
<td>AS-WeA1</td>
<td>Invited: Fundamental Limits to Sputter Depth Profiling: Atomic Force</td>
<td>E.-H. Girlin, Hughes Research Laboratories</td>
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<td>Microscopy of Ion Beam-Induced Topography.</td>
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<td>2:20 pm</td>
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<td>Invited talk continued.</td>
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<tr>
<td>2:40 pm</td>
<td>AS-WeA5</td>
<td>Determination of the Electron Attenuation Length from High Resolution</td>
<td>S. Hofmann, Max-Planck-Institut für Metallforschung, Germany</td>
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<td>AES Depth Profils.</td>
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<tr>
<td>2:40 pm</td>
<td>AS-WeA4</td>
<td>Resolution in Sputter Depth Profiling Assessed by AlAs/GaAs</td>
<td>K. Kaigawa, Sony Corporation, Japan; R. Shimizu, Osaka University, Japan</td>
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<td>Superlattices</td>
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<td>3:00 pm</td>
<td>AS-WeA5</td>
<td>The Effects of Misorientation of the GaAs(100) Surface on the</td>
<td>K. J. Lee, S.H. Kim, W.J. Choi, C.Y. Kim, S.T. Kim, Goldstar Central Research</td>
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<td>Secondary Ion Yield during O_2^+ or Cs^+ Sputtering.</td>
<td>Laboratory, Korea</td>
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<td>3:40 pm</td>
<td>AS-WeA7</td>
<td>The Effect of Tilt Angle on As Implants in Si</td>
<td>J.M. Anthony, J.A. Keenan, Texas Instruments</td>
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<tr>
<td>4:00 pm</td>
<td>AS-WeA8</td>
<td>Nondestructive and Quantitative Depth Profiling Analysis of Ion</td>
<td>J.C. Lee, H.J. Kang, H.K. Kim, D.W. Moon, Korea Research Institute of Standards and</td>
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<td>Bombarded Surfaces by Medium Energy Ion Scattering Spectroscopy.</td>
<td>Science</td>
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<tr>
<td>4:20 pm</td>
<td>AS-WeA9</td>
<td>Nondestructive EPMA Depth Profiling of Buried Oxide Layers in</td>
<td>A.P. Alexeyev, Research Centre for Surface and Vacuum Investigation, Russia</td>
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<td>Silicon</td>
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<tr>
<td>4:40 pm</td>
<td>AS-WeA10</td>
<td>Real Time Monitoring of Depth Profiling.</td>
<td>L.A. Vasileyev, V.V. Rakushsky, Research Centre for Surface and Vacuum Investigation,</td>
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<td>Russia</td>
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<tr>
<td>5:00 pm</td>
<td>AS-WeA10</td>
<td>Post-Plasma Particle Dynamics in a Gaseous Electronics Conference</td>
<td>S.M. Collins, J.F. O'Mallon, R.N. Carlile, University of Arizona; D.A. Brown, Sandia</td>
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<td>Standard Cell</td>
<td>National Laboratories</td>
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*John Coburn and Harold Winters Award Nominees*
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<tr>
<th>Time</th>
<th>Session 1: Surface Science/Electronic Materials</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td><strong>SSEM-WeA1</strong> Thermal Stability of Hydroxyl Species on the SiO₂ Surface. D. SNEH, S.M. GEORGE, University of Colorado, Boulder.</td>
</tr>
<tr>
<td>2:20 pm</td>
<td><strong>SSEM-WeA2</strong> Correlation of Surface Core Levels and Structural Building Blocks through High Resolution Core Level Spectroscopy: Si(111)-(7x7). J.J. FAEGER, W. THEIS, K. HORN, Fritz-Haber-Institut der MPG, Germany; C. JUNG, C. HELLWIG, M. PETERSEN, BESSY GmbH, Germany.</td>
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<tr>
<td>2:40 pm</td>
<td><strong>SSEM-WeA3</strong> Abstraction of H from Group IV Surfaces by Atomic Hydrogen. D.D. KOLESKE, S.M. GATES, IBM TJ Watson Research Center; B. JACKSON, University of Massachusetts.</td>
</tr>
<tr>
<td>3:00 pm</td>
<td><strong>SSEM-WeA4</strong> Local Adsorbate Structures of F₂ Chemisorbed onto the Si(111)-(7x7) Surface: A Molecular Beam/STM Study. J.A. JENSEN, C. YAM, A.C. KUMMEL, University of California, San Diego.</td>
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<tr>
<td>3:20 pm</td>
<td><strong>SSEM-WeA5</strong> First-Principles Study of Structures and Reactions on Si/SiO₂ Surfaces. K. RAGHAVACHARI, AT&amp;T Bell Laboratories.</td>
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<tr>
<td>3:40 pm</td>
<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td><strong>SSEM-WeA7</strong> Translational-Energy-Induced Etching of n⁺Si(100) by Hyperthermal Cl₂ Molecular Beams. Y. TERAOKA, I. NISHIYAMA, NEC Corporation, Japan.</td>
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<tr>
<td>4:20 pm</td>
<td><strong>SSEM-WeA8</strong> Vacancy-Mediated and Ion-Enhanced Surface Diffusion on Semiconductors. C.E. ALLEN, E.G. SEEBAUER, University of Illinois, Urbana; I.I. SUNJ, Clarkson University.</td>
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<tr>
<td>4:40 pm</td>
<td><strong>SSEM-WeA9</strong> Thermal and Photochemical Reactions of HN₃ on Ge(100): A Lower Temperature Route to Germanium Nitride. C. THIBAULT, J.C. HEMMININGER, University of California, Irvine.</td>
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<tr>
<td>5:00 pm</td>
<td><strong>SSEM-WeA10</strong> Surface Phase Transformations in the Ni/Si(111) System: Real-Time Observations using LEEM and STM. P.A. BENNETT, M.Y. LEE, S.A. PARKH; Arizona State University; K. WORM, University of Clausthal, Germany; R.J. PHANEUF, University of Maryland.</td>
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<th>Time</th>
<th>Session 2: Thin Film</th>
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<tr>
<td>2:00 pm</td>
<td><strong>TF-WeA1</strong> Deposition and Characterization Techniques for Nanostructures in Thin Films. J.C. BILELLO, S.M. YALISOVE, University of Michigan.</td>
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<tr>
<td>2:20 pm</td>
<td><strong>TF-WeA2</strong> Invited talk continued.</td>
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<td>3:00 pm</td>
<td><strong>TF-WeA3</strong> Crystallographic Relations in Zirconia-Alumina Multilayer Nanolaminates. M. GAJARDZISKAJA-JOJNOVSKA, C.R. AITA, University of Wisconsin, Milwaukee.</td>
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<tr>
<td>3:40 pm</td>
<td><strong>TF-WeA5</strong> Fabrication of Thin Films with Highly Porous Microstructures. K. ROBBIE, S.K. DEW, University of Alberta, Canada; T. SMY, Carleton University, Canada; M.J. BRETT, University of Alberta, Canada.</td>
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<tr>
<td>4:20 pm</td>
<td><strong>TF-WeA7</strong> Compositional and Microstructural Characterization of Fe-N Thin Films for Recording Sensor Applications. Y.K. KIM, P.B. NARAYAN, Rocky Mountain Magnetics, Inc.</td>
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<tr>
<td>4:40 pm</td>
<td><strong>TF-WeA8</strong> Positron Annihilation Studies of Diamond-like Nanocomposite (DLN) Films. P. ASOBA-KUMAR, Brookhaven National Laboratory; B.F. DORFMAN, M.E. ABRAZOV, Polytechnic University; D. YAN, F.H. POLLAK, Brooklyn College of CUNY.</td>
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<td>5:00 pm</td>
<td><strong>TF-WeA9</strong> Invited talk continued.</td>
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**THIN FILM Session:**

- TF-WeA2: Invited talk continued.
- TF-WeA5: Fabrication of Thin Films with Highly Porous Microstructures. K. Robbie, S.K. Dew, University of Alberta, Canada; T. Smy, Carleton University, Canada; M.J. Brett, University of Alberta, Canada.
- TF-WeA8: Positron Annihilation Studies of Diamond-like Nanocomposite (DLN) Films. P. Asoka-Kumar, Brookhaven National Laboratory; B.F. Dorfman, M.E. Abrazov, Polytechnic University; D. Yan, F.H. Pollak, Brooklyn College of CUNY.
- TF-WeA9: Invited talk continued.
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<th>Time</th>
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<tr>
<td>2:00 pm</td>
<td>EM-WeA1</td>
<td>Low Carrier Concentration GaN Grown by MOCVD at Low Temperature (720ø)</td>
<td>J.C. CHENG, B. YANG, University of Maryland, Baltimore, Maryland.</td>
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<td>P.D. Holloway, University of Florida, Gainesville.</td>
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<td>2:20 pm</td>
<td>EM-WeA2</td>
<td>Ohmic Contacts to Intrinsic n-Type GaN.</td>
<td>S.J. MILLER, P.H. HOLLOWAY, University of Florida, Gainesville.</td>
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<td>J. PANKOVE, University of Colorado, Boulder.</td>
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<td>2:40 pm</td>
<td>EM-WeA3</td>
<td>INVITED: InGaN/AlGaN Blue-Light-Emitting Diodes.</td>
<td>S. NAKAMURA, Nichia Chemical Industries Ltd., Japan.</td>
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<td>3:00 pm</td>
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<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td>EM-WeA6</td>
<td>5(111) Surfaces.</td>
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<td>4:40 pm</td>
<td>EM-WeA8</td>
<td>Outdiffusion of Deuterium from GaN, AlN and InN.</td>
<td>R.G. WILSON, Hughes Research Laboratories, University of Florida, Gainesville.</td>
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<tr>
<td>5:00 pm</td>
<td>EM-WeA9</td>
<td>Dry Etching of GaN and AlGaN.</td>
<td>I. ADESAKO, A.T. RING, University of Illinois, Urbana-Champaign, M. ASAT KHAN, D.T. OLSON, J.N. KUZIA, APA Optics, Inc.</td>
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<td>MS-WeA2</td>
<td>A Production Demonstration of Wafer-to-Wafer Plasma Gate Etch Control</td>
<td>E.A. RIETMAN, AT&amp;T Bell Laboratories, S. H. PATEL, AT&amp;T Microelectronics.</td>
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<td>by Adaptive Real-Time Computation of the Over-Etch Time from In Situ Process Signals.</td>
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<td>Etching.</td>
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<td>MS-WeA4</td>
<td>The Application of Real-Time Optical Monitors to Semiconductor</td>
<td>J. O'NEILL, IBM Microelectronics.</td>
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<td>Manufacturing.</td>
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<td>MS-WeA6</td>
<td>Mass Spectrometric In Situ Process Monitoring Applied to Silicon Dioxide Electron Cyclotron Resonance Chemical Vapor Deposition.</td>
<td>L.M. WILLIAMS, Lam Research Corp, L.C. FREES, T. VO, Leybold Intron Inc.</td>
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<td>MS-WeA7</td>
<td>INVITED: Microsensors for Process Control and Monitoring.</td>
<td>R.C. HUGHES, J.J. WICZER, Sandia National Laboratories.</td>
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<td>2:20 pm</td>
<td>VM-WeA2</td>
<td>The Effect of 20–95 eV Ar Ion Bombardment on Molecular Beam Epitaxy of GaAs(100). J. Mirecki Millunchick, S.A. Barnett, Northwestern University; L. Hultman, Linköping University, Sweden.</td>
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<td>2:40 pm</td>
<td>VM-WeA3</td>
<td>INVITED Theoretical Considerations on Stress Effects on Thin Film Microstructure. D.J. Sholovitz, University of Michigan.</td>
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<td>3:00 pm</td>
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<td>Invited talk continued.</td>
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<td>3:20 pm</td>
<td>VM-WeA5</td>
<td>INVITED Surface Thermodynamics Effects on Thin Film Microstructure. R.C. Cammarata, Johns Hopkins University.</td>
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<td>3:40 pm</td>
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<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td>VM-WeA7</td>
<td>A Molecular Dynamics Study of Transient Processes during Deposition on (001) FCC Metal Surfaces. C.M. Gilmore, The George Washington University &amp; Naval Research Laboratory; J.A. Sprague, Naval Research Laboratory.</td>
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<tr>
<td>4:20 pm</td>
<td>VM-WeA8</td>
<td>Synthesis of Epitaxial SnGe&lt;sub&gt;1-x&lt;/sub&gt; Alloy Films by Ion Assisted Molecular Beam Epitaxy and Pulsed Laser Deposition. G. He, M.E. Taylor, H.A. Atwater, California Institute of Technology.</td>
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<tr>
<td>5:00 pm</td>
<td>VM-WeA10</td>
<td>Structure–Property Relationships in Compositionally Modulated Titanium–Aluminum Thin Films. R. Ahuja, H.L. Fraser, The Ohio State University.</td>
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Wednesday Afternoon Poster Sessions

SURFACE SCIENCE
Room BR4 - Session SS-WeP pp. 250

Aspects of Surface Science
Moderator: J.C. Hemminger, University of California, Irvine.
Presenters present: 5:00-6:30 pm.

SS-WeP1 Elevated Temperature Structures of the Ag on Si(111) System at Submonolayer Coverages. G. NAVROTSKI, J.M. BLAKEY, Cornell University.

SS-WeP2 Surface Morphology Induced by Ga and Sn Overlayers on Si(100) and Si(311) Surfaces. L. LI, Y. WEI, I.T.S. TSONG, Arizona State University.

SS-WeP3 STM Studies of Benzene Adsorption on both Clean and O-preadsorbed Ni(110). L. RUAN, I. STENSSGAARD, F. BESENBACHER, E. LÆGSGAARD, Aarhus University, Denmark.

SS-WeP4 Determination of the Reconstruction of Pt(100) Surface on Atomic Scale by Scanning Tunneling Microscope. C.S. CHANG, W.B. SU, T.T. TSONG, Academia Sinica, ROC.


SS-WeP6 Cu Deposition on Ru(0001) by Low Energy Li+ Ion Scattering. Y.H. SHEN, D.J. O'CONNOR, J. YAO, R.J. MACDONALD, University of Newcastle, Australia; H. VAN ZEE, Eindhoven University of Technology, The Netherlands; R.H. ROBERTS, University of Newcastle, Australia; M. WANDEN, Universität Bonn, Germany.


SS-WeP8 Highly Angular Resolved Patterns of Photo- and Auger-Electron Diffraction from MgO(001). Y. NISHI, H. ISHII, M. OWARI, Y. ICHIKAWA, University of Tokyo, Japan.

SS-WeP9 Structural Surface Phase Transitions during Segregation Competition. M. MÜLLER, The University of British Columbia, Canada; S. HOFMANN, Max-Planck-Institut für Metallforschung, Germany.


SS-WeP11 Magneto-Optical Kerr Effect of Exchange Coupling in Co/Cr/Co Films on Pd(111) by Pd(100) Substrates. D.J. MCKINLEY, C. RAU, Rice University.


SS-WeP13 Experimental Fermi Surface of Magnetic Multilayers. C. CASADO, J. AVILA, A. VIJATON, Université Paris-Sud, France; F. SOUFI, Instituto de Ciencia de Materiales de Madrid, Spain; M.C. ASSENSIO, Université Paris-Sud, France.

SS-WeP14 Combined XPD, STM, and LEED Study of Iron Oxide Films on Pt(111). Y.J. KIM, Lawrence Berkeley Laboratory & University of Hawaii; C. WESTPHAL, Lawrence Berkeley Laboratory and Univ.-GHS-Essen, Germany; R.X. YUNZUNGA, H. XIAO, Z. WANG, Lawrence Berkeley Laboratory & Univ. of California, Davis; H.C. GALLUNAY, Lawrence Berkeley Laboratory & Univ. of California, Berkeley; M. SALMERON, M.A. VAN HOVE, Lawrence Berkeley Laboratory; C.S. FADLEY, Lawrence Berkeley Laboratory & Univ. of California, Davis.

SS-WeP15 Surface-Specific Magnetic Order Transitions for Ising Ferrimagnets and Antiferromagnets: Implications for Spin-Polarized Photoelectron Diffraction. F. ZHANG, Univ. of California, Davis & Lawrence Berkeley Laboratory. S. THEVUTHASAN, Pacific Northwest Laboratory. R. SCALETAR, R. SINGH, University of California, Davis; C.S. FADLEY, Univ. of California, Davis & Lawrence Berkeley Laboratory.

SS-WeP16 Imaging Short-Range Magnetic Order by Spin-Polarized Photoelectron Holography. Z. WANG, Univ. of California, Davis & Lawrence Berkeley Laboratory. A.P. KADYUNA, Lawrence Berkeley Laboratory. S. THEVUTHASAN, Pacific Northwest Laboratory. M.A. VAN HOVE, Lawrence Berkeley Laboratory; C.S. FADLEY, Univ. of California, Davis & Lawrence Berkeley Laboratory.


SS-WeP19 Reaction of HN3 with Al(111): Growth of an AlN Film. J.N. RUSSELL, Jr., Naval Research Laboratory.

SS-WeP20 Tailoring Sensing Film Selectivity and Sensitivity using Principles from the Hard/Soft Lewis Acid–Base (HSAB) Paradigm. V. THOMAS, J.W. SCHWANK, J.L. GLAND, University of Michigan.


SS-WeP23 Thermal and Electron–Induced Chemistry of CF3I on Ni(100). M.B. JENSEN, P.A. THIEL, Iowa State University.

SS-WeP24 Thermally Stimulated Desorption of Neutral CF3 from CF3 on Ag(111). K.H. JUNKER, Z.-J. SUN, T.B. SCOBING, J.M. WHITE, University of Texas, Austin.

SS-WeP25 Cyclopentyl on Cu(111): Area Selective Preparation and Vibrational Analysis Using a Dispersion–Compensation HREELS Spectrometer. B. MARTEL, A.P. ROCHEFORT, P.H. MCBREEN, Université Laval, Canada.


SS-WeP27 Kinetics of Propene Desorption from Pd(111). N.A. THORNBURG, T.M. ABDELREHIM, C.M. GERTH, K.A. POCIOTTO, D.P. LAND, University of California, Davis.

SS-WeP28 Adsorption and Film Growth of BTA on the Clean and Oxygen Adsorbed Cu(110) Surfaces. K. CHO, J. KISHIMOTO, T. HASHIZUME, Tohoku University, Japan; H.W. PICKERING, Pennsylvania State University, T. SAKURAI, Tohoku University, Japan.


SS-WeP30 First IR Spectroscopic Evidence of Strained Cyclic Reaction Sites on the Borosilicate Glass (BSG) Surface. I.L. TENDER, J.E. CROWELL, University of California, San Diego. K.J. URAM, Lam Research Corporation.
NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room BR4 - Session NS-WeP pg. 254

Nanometer-Scale Science and Technology
Moderator: R.J. Colton, Naval Research Laboratory.

Presenters present: 5:00-6:30 pm.

NS-WeP1  The Nanometer-scale Structure of Isocyanide Functionalized Polyaniline Thin Films. T.L. PORTER, A.G. SYKES, Y. SHI, G. CAPEL, Northern Arizona University.


NS-WeP3  Nucleation and Growth Kinetics of Monolayer Organic Films Studied with Graphite Etch Pits and STM. V.J. CEE, D.L. PATRICK, T.P. BEEBE, J.R. University of Utah.

NS-WeP4  Scanning Force Microscopy of Quasi-single Crystalline Polyethylene and Nylon 6 Produced by Channel Dye Compression. C. WANG, R.-E. RIEHANN, M. DRECHSLER, H.-J. CANTOW, Albert-Ludwigs University, Germany.


NS-WeP6  Amorphous and Ordered Layers of Oligothiophenes: A Combined AFM-, UPS-, HREELS-, and Conductivity Study. O. BOHME, D. OETER, C. ZIEGLER, W. GOPEL, University of Tϋbingen, Germany.

NS-WeP7  Substrate Effects on Two-Dimensional Ordering of Self-Assembled Layers on TMDs by STM. D.L. SAMPSON, B.A. PARKINSON, Colorado State University.

NS-WeP8  Scanning Force Microscopy of Stearic Acid LB Film Deposited on MgO. J. LI, W. YAN, S. XI, E. WANG, Chinese Academy of Sciences.


NS-WeP11  Structure and Electrical Property of C60Ag Ultraline Particle–Polymer Thin Films. H.J. GAO, Z.D. XUE, Q.D. WU, Peking University, P.R. China. S. PANG, Academia Sinica, P.R. China.

NS-WeP12  The AFM as a Tool for Metal Surface Modification. H. GÖBEL, P. VON BLAHLKENHAGEN, W. SCHOMMERS, Kernforschungszentrum Karlsruhe, Germany.

NS-WeP13  Nanometer Scale Modifications of Gold Surfaces by STM. A. OHI, JRACAT-ATP, Japan. W. MIZUTANI, H. TOKUMOTO, JRACAT-NAIR, Japan.


NS-WeP15  Nanofabrication of Gold Surface with Scanning Tunneling Microscope by Combination of Mechanical and Electrical Method. Y. ISHIKAWA, N. UMEDA, A. TAKAYANAGI, Tokyo University of Agriculture and Technology, Japan.


NS-WeP17  Nanostucture of Cleaved Surface of Bi Crystal. A.M. TROFANOVSKII, V.S. EDELMAN, Academy of Science of Russia.

NS-WeP18  Scanning Tunneling Microscopy of Transition-Metal Clusters. P.E. QUEENBERY, T.A. HANN, P.N. FIRST, Georgia Institute of Technology.


NS-WeP23  Structural Analysis of Domain Boundaries on Si(111)/7x7 Surfaces by STM. D.J. GU, Z.L. MA, N. LIU, X. GE, W.B. ZHAO, X. CHU, Z.D. XUE, S. PAN, Academia Sinica, P.R. China.


Wednesday Afternoon Poster Sessions

PLASMA SCIENCE
Room BR4 - Session PS-WeP pg. 258

Plasma Etching and Inertial Confinement Fusion Targets
Moderator: J.L. Cecchi, University of New Mexico.

Presenters present: 5:00–5:30 pm.

PS-WeP1 Extraction of Oxygen for CO₂ using Glow-Discharge and Permeation Techniques. D. WU, R.A. OUTFLOW, R.L. ASH, Old Dominion University.


PS-WeP3 Reactive Ion Etching-Induced Damages in GaAs/AlGaAs Quantum Well Structures and Recovery by Rapid Thermal Annealing and Hydrogen Passivation. B.-S. YOO, S.-J. PARK, K.-H. PARK, Electronics and Telecommunications Research Institute, Korea.

PS-WeP4 Characterization of ECR Plasmas with a Quadrupole Mass Spectrometer and Cylindrical Mirror Energy Analyzer. S. BERENBAUM, B. BELLO, H. IBRAHIM, W.M. LAU, University of Western Ontario, Canada.


PS-WeP6 Power versus Time in Pulsed, Parallel Plate rf Discharges. L.J. OVERZET, University of Texas, Dallas.


PS-WeP12 Simulations of Damage Profiles due to Reactive Ion Etching and Ion-assisted Etching. R.J. DAVIS, P. JHA, Columbia University.


THIN FILM
Room BR4 – Session TF-WeP pg. 262
Aspects of Thin Films
Moderator: F. Sequeda, Conner Peripherals.
Presenters present: 5:00–6:30 pm.

TF-WeP1 A Novel Approach to the Calculation of ITO Films' Figures of Merit. S.A. KNICKERBOCKER, A.K. KULKARNI, Michigan Technological University.

TF-WeP2 Protecting Silver Polymer Mirrors with Oxide Coatings for Solar Applications. C.E. KENNEDY, National Renewable Energy Laboratory.


TF-WeP6 The Structure and Magneto–Optical Properties of Ta/Fe and Ta/Co Bilayers. E.E. SHALIGINA, L.V. KOZLOVSKY, University of Daugavpils, Latvia.


TF-WeP9 Low Temperature Formation of Textured ZnO Transparent Electrodes by Magnetron Sputtering. T. MINAMI, H. SUGIHARA, S. TAKATA, I. FUKUDA, Kanazawa Institute of Technology, Japan.

TF-WeP10 Vacuum Deposition Parameters for Thin Film Shape Memory Alloys. A.P. JARDINE, R. DANNENBERG, State University of New York, Stony Brook.


TF-WeP12 A Compact Negative Metal Ion Beam Source for Low Energy Thin Film Deposition. S.I. KIM, Y.C. AHN, SKION Corporation.


TF-WeP14 Modelling and Experimental Studies of Multi–Phase Formation in Reactive Sputtering. C. NENDER, T. NYBERG, S. BERG, Uppsala University, Sweden. K.D. LEGG, M. OTHMAN, P.J. RUUKKI, M.S. WONG, W.D. SPROUL, Northwestern University.


TF-WeP17 Development of Vacuum Arc Metal Ion Sources and Techniques TAMEK for Material Surface Modification. A.M. TOLIPOA, Ukraine Academy of Science and Pan TAMEK, Russia.

TF-WeP18 Synthesis and Properties of Polycrystalline CrN/TiN Superlattices. F. YASHAR, X. CHU, M.S. WONG, W.D. SPROUL, S.A. BARNETT, Northwestern University.

TF-WeP19 Neutralized Beam Assisted Evaporation of Cubic Boron Nitride on Si(100). M. LIU, A. BOURJET, A. BENDOULA, University of Houston. K. WATERS, A. SCHULTZ, Ionwerks.


TF-WeP22 The Effect of the Physical Vapor Deposition Techniques of Sputtering and Evaporation of Ti on the Formation and Transformation of C49 to C54 Ti3Si2. C. CARRAL, R.A. RYDE, L.A. CLEVERGER, K.N. SAMS, IBM T.J. Watson Research Center.


VACUUM TECHNOLOGY
Room BR4 – Session NP-WeP pg. 266
New Products
Moderator: W. Weed, Sandia National Laboratories.
Presenters present: 5:00–6:30 pm.


NP-WeP3 Reliable In–Situ Gas Analysis for Aggressive Gas Processes. W. EISINGER, Leybold Italia Inc.


NP-WeP5 New Compact Total Pressure Gauges. A. SCHMIDT, R. Stocker, Balzers Limited, Liechtenstein.

NP-WeP6 Combination Valve Enables In–Situ Zeroing and Calibration of Capacitance Manometers. J. SKUBA, HPS Division of MKS Instruments. J. GUNN, MKS Instruments.
<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Title</th>
<th>Authors/Institutions</th>
</tr>
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<tbody>
<tr>
<td>8:20 am</td>
<td>SS1-ThM1</td>
<td>Invited Talk</td>
<td>Nanoscale Measurements by Diffusion Controlled Growth. K. Kern, École Polytechnique de Lausanne</td>
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<tr>
<td>8:40 am</td>
<td>SS2-ThM1</td>
<td>Invited Talk Continued</td>
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<tr>
<td>9:00 am</td>
<td>SS1-ThM2</td>
<td>Observation of Quantum Size Effects at Room Temperature on Metal Surfaces with STM. Ph. Avouris, I.-W. Lyo, Y. Hasegawa, IBM TJ Watson Research Center</td>
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<tr>
<td>9:20 am</td>
<td>SS1-ThM3</td>
<td>STM of Thin Film MgO Grown on Mo(001). M.C. Gallagher, M. Pyfield, J.P. Cowin, S.A. Joyce, Pacific Northwest Laboratories</td>
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<td>9:40 am</td>
<td>SS1-ThM4</td>
<td>Nanosphere Lithography. J.C. Hulteen, R.P. Van Duyne, Northwestern University</td>
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<td>10:00 am</td>
<td>SS1-ThM5</td>
<td>Nanofabrication on Electron Beam Resist Using Scanning Tunneling Microscopy. M.H. Nayfeh, A. Archer, J.M. Hetrick, I. Adesida, University of Illinois, Urbana-Champaign</td>
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<tr>
<td>10:20 am</td>
<td>SS1-ThM6</td>
<td>Nanometer-Scale STM Electrochemical Syntheses of Electronic Materials. W. Li, J. Zoval, J.A. Virtanen, R.M. Penner, University of California, Irvine</td>
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<tr>
<td>10:40 am</td>
<td>SS2-ThM7</td>
<td>Invited Talk Continued</td>
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<tr>
<td>11:00 am</td>
<td>SS1-ThM8</td>
<td>Self-Assembled Monolayers of Pendant Monomers: A Basis for Fabrication with the Scanning Tunneling Microscope. R.J. Willcut, R.L. McGarvey, Louisiana State University</td>
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<tr>
<td>11:40 am</td>
<td>SS2-ThM11</td>
<td>The Synthesis of Ammonia on Ru(0001). P.A. Taylor, E. Tönnesvist, Haldor Topsoe Research Laboratories, Denmark</td>
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*Finalist for the Morton M. Taup Award
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<tr>
<td>8:20 am</td>
<td>NSB1-ThM1</td>
<td>Molecular Recognition between DNA Base Pairs by AFM</td>
<td>B.D. RATNER, T. BOLAND</td>
<td>University of Washington</td>
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<tr>
<td>8:40 am</td>
<td>NSB1-ThM2</td>
<td>Direct Measurement of the Interaction Forces between Complementary Strands of DNA with the Atomic Force Microscope</td>
<td>G.U. LEE, L.A. CHRISEY, R.J. COLTON</td>
<td>Naval Research Laboratory</td>
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<tr>
<td>9:00 am</td>
<td>NSB1-ThM3</td>
<td>Scanning Probe Microscopic Visualization of Electrostatically Immobilized Interacting Drug–Nucleic Acid Complexes</td>
<td>L.A. BOT-TOMLEY, J.E. COURT, G. GARDNER, E.A. HANDLEY, L.D. WILLIAMS</td>
<td>Georgia Institute of Technology</td>
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<td>9:20 am</td>
<td>Invited</td>
<td>Invited talk continued.</td>
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<td>9:40 am</td>
<td>NSB1-ThM4</td>
<td>Sequence Effects in the Images and Bonding of Single Stranded DNA on Au(111) Observed by Electrochemical STM</td>
<td>Y. LYUBCHENKO, D. REKES, T.W. JING, S.M. LINDSAY</td>
<td>Arizona State University</td>
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<tr>
<td>10:00 am</td>
<td>NSB1-ThM5</td>
<td>Imaging Polyelectrolyte Chromosomes with the Atomic Force Microscope</td>
<td>D.M. JONDE, L. AMBROSIO, J. VESENKA, E. HENDERSON</td>
<td>Iowa State University</td>
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<tr>
<td>10:20 am</td>
<td>NSB1-ThM6</td>
<td>The Topology of Supercoiled DNA by SFM Imaging</td>
<td>R. SAMORI, C. NIGRO, I. MUZZALupo, G. ZUCCHERI, C. QUAGLIARIELLO</td>
<td>University of Catania, Italy</td>
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<tr>
<td>10:40 am</td>
<td>NSB1-ThM7</td>
<td>Scanning Probe Microscopy Studies of Macromolecular Interactions</td>
<td>S.J.B. TENDLER, C.J. ROBERTS, P.M. WILLIAMS, M.C. DAVIES, D.E. JACKSON</td>
<td>University of Nottingham, United Kingdom</td>
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<td>11:00 am</td>
<td>NSB1-ThM8</td>
<td>DNA Surface Attachment Schemes for Scanning Probe Microscopy</td>
<td>L.A. WENZLER, T.P. BEEBE-JR.</td>
<td>University of Utah</td>
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<td>11:20 am</td>
<td>NSB1-ThM9</td>
<td>Studying the Dynamics of Polymer Surface Degradation by Scanning Force Microscopy</td>
<td>K.M. SHAKESHEFF, M.C. DAVIES</td>
<td>University of Nottingham, United Kingdom</td>
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<td>11:40 am</td>
<td>NSB1-ThM10</td>
<td>In Situ Observation of the Protonation of Cytosine on Au(111) by Electrochemical STM</td>
<td>D. LAMPNER, T.W. JING, J. PAN, S.M. LINDSAY</td>
<td>Arizona State University</td>
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<td>11:20 am</td>
<td>NSB1-ThM11</td>
<td>Electrical and Mechanical Properties of Metallic Nanowires: Conductance Quantization and Localization</td>
<td>J.I. PASCUAL, J. MENDEZ, J. GÓMEZ-HERRERO, A.M. BARÓ, N. GARCÍA</td>
<td>Universidad Autónoma de Madrid, Spain</td>
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<td>11:40 am</td>
<td>NSB1-ThM12</td>
<td>Quantized Conductance in an Atom-Sized Point Contact</td>
<td>F. BESENGHIERI, L. OLESEN, E. LAEGSGAARD, I. STENSGAARD</td>
<td>Aarhus University, Denmark</td>
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<td>12:00 am</td>
<td>NSB1-ThM13</td>
<td>Nanoscale Mechanical and Chemical Processing of Surfaces: Molecular Dynamics Simulations</td>
<td>S.B. HAYKIN, R.I. COOK</td>
<td>C.T. WHITE, D.W. BRENNER</td>
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<td>12:20 pm</td>
<td>NSB1-ThM14</td>
<td>Electrical Measurements on STM Patterned Silicon MOSFETs</td>
<td>T. PAYFIELD, T.K. HIGMAN</td>
<td>University of Minnesota</td>
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<td>12:40 pm</td>
<td>NSB1-ThM15</td>
<td>Nanolithographic Patterning of Metal Films with the STM</td>
<td>C. VAN HAENSEN</td>
<td>L. STOCKMAN, G. NEUTIENS, C. STRUNK, P. BREUNSIGAARD</td>
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<td>1:00 pm</td>
<td>NSB1-ThM16</td>
<td>Spectroscopic Investigation of PEDA, an Ultrathin Resist for STM Lithography</td>
<td>F.K. PERKINS, E.A. DOBISZ, M.-S. CHEN, J.M. CALVERT, C.R.K. MARRIAN</td>
<td>Naval Research Laboratory</td>
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<td>1:40 pm</td>
<td>NSB1-ThM18</td>
<td>Cyanide Etching of n-Alkanethiol–Modified Au (111) Surfaces Studied by Electrochemical Scanning Tunneling Microscopy</td>
<td>Y.Q. LI, D. CHAILLAFAK, R.M. CROOKS</td>
<td>Texas A&amp;M University</td>
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<td>2:00 pm</td>
<td>NSB1-ThM19</td>
<td>Nanometer Structure Fabrication on Insulator and Magnetic Material using Scanning Probe Microscope</td>
<td>S. NOZAKA, H. HAJIDE, A. KIKUHARA, M. MIYAMOTO, T. SHINTANI, K. NAKAMURA, J. BRUGGER, M. AMURA</td>
<td>Hitachi Ltd., Japan</td>
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### Applied Surface Science

**Room A101 - Session AS-ThM**  
*Page 274*

**Self-Assembled Monolayers**  
**Moderator:** A.W. Czanderia, National Renewable Energy Laboratory.

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<thead>
<tr>
<th>Time</th>
<th>AS-ThM1</th>
<th>PS-ThM1</th>
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</table>
| 8:20 am | **AS-ThM1**  
*Invited*  
Surface Engineering using Mixed Alkanethiolate Monolayers on Gold.  
A. ULMAN, Polytechnic University. | **PS-ThM1**  
*Invited*  
H.N. SAWIN, Massachusetts Institute of Technology. |

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<th>Time</th>
<th>AS-ThM3</th>
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| 9:00 am | **AS-ThM3**  
Probing the Phase Transition in Alkyl Thiol Assemblies on Gold Surfaces.  
F. BENSEBA, T. ELLIS, Univ. de Montreal, Canada.  
A. BADIA, B. LENNOX, Univ. de McGill, Canada. |

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<th>Time</th>
<th>AS-ThM4</th>
<th>PS-ThM4</th>
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| 9:20 am | **AS-ThM4**  
A NEXAFS-Investigation of the Adsorption of Pyromellitic Dianhydride (PMDA) on Pt(111).  
C. THUMMELER, T. STRUNKUS, A. SCHERTEL, C.W. HUTCHINGS, H. WEGNER, CH. WÜLL, M. GRUNZIE, Universität Heidelberg, Germany. | **PS-ThM4**  
M.G. BLAIN, T.L. MEISENHEIMER, Sandia National Laboratories.  
A.O. NEILL, IBM Microelectronics.  
J.A. BONIER, P.K. AIUM, T. TA, SEMATECH. |

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| 9:40 am | **AS-ThM5**  
Structure and Dynamics of Alkanethiol Self-Assembled Monolayers on Au(111) Characterized by Scanning Tunneling Microscopy.  
G.E. POUHIER, N.J. TARLOV, H.E. RUTHEIMER, National Institute of Standards & Technology. | **PS-ThM5**  
Effects of Reactor Wall on Downstream Stripping.  
M. NAKAMURA, K. NISHIKAWA, K. SHINAGAWA, Fujitsu Ltd., Japan. |

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<th>Time</th>
<th>AS-ThM6</th>
<th>PS-ThM7</th>
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| 10:00 am | **AS-ThM6**  
Metal Overlayers on Self-Assembled Monolayers: 6.  
XPS of Cr/COOH on Mercapthexadecanoic Acid.  
D.R. JUNG, A.W. CZANDERIA, National Renewable Energy Laboratory. | **PS-ThM7**  
*Invited*  
Neutral-Beam-Assisted Etching.  
Y. YUNODAMI, K. YOKOGAWA, T. MIZUTANI, Hitachi Ltd., Japan. |

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<th>Time</th>
<th>AS-ThM7</th>
<th>PS-ThM8</th>
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| 10:20 am | **AS-ThM7**  
G. LIU, University of California, Berkeley.  
C.D. CHIDSEY, Stanford University.  
D.F. O'BRIEN, M.B. SALMORINA, Lawrence Berkeley Laboratory. | **PS-ThM8**  
Hyperthermal Neutral Beam Etching.  
K.P. GIAPIS, T.A. MOORE, T.K. MINTON, California Institute of Technology. |

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<th>Time</th>
<th>AS-ThM8</th>
<th>PS-ThM9</th>
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| 10:40 am | **AS-ThM8**  
The Surface Chemistry of Self-Assembled and Vapor Deposited Perfluorinated n-Alkanic Acid Monolayers on Native Aluminum–oxide.  
R.M. WALLACE, S.A. HENCK, P. CHEN, B.A. WEBB, Texas Instruments Inc. | **PS-ThM9**  
Quantitative Analysis of Monolayer Molecular Films using "Molecule Corrals" and STM.  
D.L. PATRICK, V.J. CEE, T.P. BEEBE, J.R., University of Utah. |

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<th>Time</th>
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| 11:00 am | **AS-ThM9**  
In Situ AFM/STM Study of Nucleation, Growth and Electron Transfer Reactions of Purines at the Graphite-Water Interface.  

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<th>Time</th>
<th>AS-ThM10</th>
<th>PS-ThM10</th>
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</table>
| 11:20 am | **AS-ThM10**  
Hyperthermal Neutral Beam Anisotropic Etching for Submicron Patterning of Electronic Materials.  
J.B. CROSS, M.A. HOFFBAUER, Los Alamos National Laboratory. | **PS-ThM10**  
A Thermalized Neutral Radical Source for Charge-Free Processing.  
L. CHEN, D. YANG, D.R. WRIGHT, K.L. MAXWELL, SEMATECH. |

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<tr>
<th>Time</th>
<th>AS-ThM11</th>
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| 11:40 am | **AS-ThM11**  
Multi-Step Assembly of Polymeric Monolayers.  
D.W. GRANGIER, G. MAO, F. SUN, Colorado State University.  
D.G. CASTNER, University of Washington. | **PS-ThM11**  
*Invited*  
A Thermalized Neutral Radical Source for Charge-Free Processing.  
L. CHEN, D. YANG, D.R. WRIGHT, K.L. MAXWELL, SEMATECH. |
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<tr>
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<th>Speaker(s)</th>
<th>Location/Institution</th>
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<tr>
<td>8:20 am</td>
<td>EMSS-ThM1</td>
<td>Sputtering of GaAs(110) Studied with Scanning Tunneling Microscopy. R.J. Pechman, X.-S. Wang, J.H. Weaver, University of Minnesota.</td>
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<tr>
<td>8:40 am</td>
<td>EMSS-ThM2</td>
<td>Atomically-resolved Decomposition of Phosphine and Formation of P-terminated Layers on Si(001). R.J. Haners, Y. Wang, University of Wisconsin, Madison.</td>
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<td>9:00 am</td>
<td>EMSS-ThM3</td>
<td>INVITED SEMiconductor Etching with Halogens: Scanning Tunneling Microscopy Investigations. J.H. Weaver, University of Minnesota.</td>
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<td>9:20 am</td>
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<td>Invited talk continued.</td>
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<tr>
<td>9:40 am</td>
<td>EMSS-ThM5</td>
<td>Analysis of Nanometer-Scale Surface Roughness with Reflection Inelastic Electron Scattering from Silicon Surfaces. H.N. Frase, H.A. Atwater, S.S. Wong, C.C. Ahr, California Institute of Technology.</td>
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<tr>
<td>10:00 am</td>
<td>EMSS-ThM6</td>
<td>Sub-surface Diffusion of Ni on Si(100) and (111). M.Y. Lee, P.A. Bennett, Arizona State University.</td>
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<td>10:20 am</td>
<td>EMSS-ThM7</td>
<td>Chemical Vapor Deposition of TiSi$_2$ using SiH$_4$ and TiCl$_4$, M.A. Menticcio, R.P. Southwell, E.G. Seebauer, University of Illinois, Urbana.</td>
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<tr>
<td>11:00 am</td>
<td>EMSS-ThM9</td>
<td>Investigation of the Chemical Mechanisms of Ta/Ta-oxide Halogen Etch Selectivity. L.A. DeLouis, Xerox Webster Research Center.</td>
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<td>8:20 am</td>
<td>EM-ThM1</td>
<td>Invited talk continued</td>
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<tr>
<td>10:00 am</td>
<td>EM-ThM6</td>
<td>Real Time Monitoring of the Electron Cyclotron Resonance Etching of Semiconductors by In Situ Spectroscopic Ellipsometry.</td>
<td>N.J. IANNO, P.G. SNYDER, S. AHMER, University of Nebraska, Lincoln. S. PITEL, B. JOHNS, J.A. WOLLMAN, J.A. Wollam Co.</td>
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<tr>
<td>10:40 am</td>
<td>EM-ThM8</td>
<td>Monitoring of Deposition and Dry Etching of Si/SiGe Multiple Stacks.</td>
<td>B. TILLACK, G. RITTER, H.H. RICHTER, A. WOLFF, G. MÖGENSTERN, Institute for Halbleiterphysik, Germany.</td>
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<tr>
<td>11:00 am</td>
<td>EM-ThM9</td>
<td>Observation of Etching Reaction using Second Harmonic Generation.</td>
<td>S. HARAICH, F. SASAKI, S. KOBAYASHI, M. KOMURO, T. TANI, Electrotechnical Laboratory, Japan.</td>
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<tr>
<td>11:20 am</td>
<td>EM-ThM10</td>
<td>Hydrogen Annealing Effect on SHG from SiO₂/Si(111) Interfaces.</td>
<td>H. HIRAYAMA, F. ITO, K. WATANABE, NEC Corporation, Japan.</td>
<td></td>
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<tr>
<td>11:40 am</td>
<td>MS-ThM1</td>
<td>The Effect of Contamination on the Reliability of Magnetic Materials and Storage Disks.</td>
<td>B. HERMSEIER, IBM Advanced Magnetic Recording Lab.</td>
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</tr>
<tr>
<td>12:00 am</td>
<td>MS-ThM3</td>
<td>An In Situ XPS Study of Metal Surface Recontamination and Hollow Cathode Plasma Cleaning.</td>
<td>H. LI, A. BELKANDI, Z. ORBAN, BOC Group Technical Center, F. JANSEN, Arco Coating Technology.</td>
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<tr>
<td>12:40 am</td>
<td>MS-ThM5</td>
<td>Rapid Yield Learning.</td>
<td>D.M.H. WALKER, Texas AM University.</td>
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<tr>
<td>1:00 am</td>
<td>MS-ThM7</td>
<td>New Ashing Technology with Multi-Plasma-Mode Reactor.</td>
<td>R.L. BERSIN, M. KIKUCHI, I. NAKAYAMA, ULYAC Technologies, Inc.</td>
<td></td>
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<tr>
<td>1:20 am</td>
<td>MS-ThM8</td>
<td>Vapor Phase SiO₂ Etching and Pre-gate Oxide Cleaning in an Integrated Cluster System.</td>
<td>Y. MA, M.S. GREEN, AT&amp;T Bell Laboratories, M. MARCH, Submicron System, Inc. K. HANSON, J. SAVETTA, D. BRASEN, AT&amp;T Bell Laboratories.</td>
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<tr>
<td>Time</td>
<td>Session</td>
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<td>Authors</td>
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<tr>
<td>8:40 am</td>
<td>VM-ThM2</td>
<td>Nanostructured Films and Particles Produced by Femtosecond Pulsed-Laser Ablation.</td>
<td>S.L. Rohde, A. Latef, B. Robertson, T. Voiles, D. Derr, D.R. Alexander, University of Nebraska.</td>
<td></td>
</tr>
<tr>
<td>9:00 am</td>
<td>VM-ThM3</td>
<td>Large-Area Pulsed Laser Deposition: Techniques and Applications.</td>
<td>J.A. Green, M.D. Tabat, Ratheon Company - Research Division.</td>
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<tr>
<td>9:20 am</td>
<td>Invited</td>
<td>Invited talk continued.</td>
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<tr>
<td>10:00 am</td>
<td>VM-ThM6</td>
<td>GeO&lt;sub&gt;2&lt;/sub&gt; Films Prepared by Pulsed Laser Deposition.</td>
<td>B.M. Patterson, P.J. Wolf, M. Scott, USAF Academy, T. Christensen, University of Colorado, Colorado Springs.</td>
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<tr>
<td>10:40 am</td>
<td>Invited</td>
<td>Invited talk continued.</td>
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<tr>
<td>11:00 am</td>
<td>VM-ThM9</td>
<td>Carbon and Nitrogen Implantation in a Large-Scale PSI&lt;sub&gt;II&lt;/sub&gt; Experiment.</td>
<td>B.P. Wood, J.T. Scheuer, D.J. Rej, I. Henins, W.A. Reass, R.J. Faehl, K.C. Walter, M.A. Nastasi, Los Alamos National Laboratory.</td>
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### MY SCHEDULE
**Thursday Morning, October 27, 1994**

<table>
<thead>
<tr>
<th>TIME</th>
<th>SESSION</th>
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<tr>
<td>8:20 am</td>
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**Lunch**
When: 
Where: 

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### OTHER EVENTS THURSDAY

- **7:00 a.m.** Companions Program (see insert) (Silver Room (H))
- **8:30 a.m.** Exhibitors Breakfast (Room C207 (CC))
- **12:00 Noon** 1995 Program Committee Chairs (Aspen Room (H))

**H:** Radisson Hotel  
**CC:** Colorado Convention Center

---

### SHORT COURSES THURSDAY

- **8:30 a.m.** Vacuum Technology (Room C106 (CC))
- **8:30 a.m.** A Comprehensive Course on Surface Analysis: AES,XPS,SIMS, Depth Profiling, AES RBS (Room C107 (CC))
- **8:30 a.m.** Physics, Chemistry, and Mechanics of Adhesion (New) (Room C105 (CC))
- **8:33 a.m.** Hard Coatings by PVD Methods (Room C103 (CC))
- **8:33 a.m.** Depth Profiling (Room C107 (CC))
- **8:33 a.m.** Mass Flow Controllers: Fundamentals, Techniques, and Applications (Room C106 (CC))
- **8:33 a.m.** Plasma-Enhanced CVD: Fundamentals, Techniques, and Applications (Room C106 (CC))
- **8:33 a.m.** Total Pressure Gauging Techniques (Room C106 (CC))
- **8:33 a.m.** Transparent Conducting Oxides: Their Science, Fabrication, Properties, and Applications (Room C106 (CC))
- **8:33 a.m.** Vacuum Evaporation/Deposition Technology (Room C109 (CC))
- **8:33 a.m.** Vacuum Leak Detection (Room C112 (CC))

---

### Special or Focus Area Sessions

- **Sensors, in-situ Diagnostics, Process Control**
- **EM-ThM** Optical Diagnostics for Materials Processing
- **AS-ThM** Self-Assembled Monolayers
- **Surface Contamination and Control**
- **MS-ThM** Micro-Contamination and Defects
- **Nanostructure Fabrication and Atomic-Scale Manipulation**
- **NS2-ThM** Proximal Probe Based Fabrication
- **SS1-ThM** Nanoscale Measurements
## MY SCHEDULE
### Thursday Afternoon, October 27, 1994

<table>
<thead>
<tr>
<th>TIME</th>
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<td>2:00 pm</td>
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</table>

### OTHER EVENTS THURSDAY

- **12:15 p.m.** AVS Business Meeting *(Room A101 (CG))*
- **6:00 p.m.** Surface Science Spectra Editorial Board Meeting & Dinner *(Gold Room (HI))*
- **6:00 p.m.** 1994-95 LAC/Program Committee Reception *(Jr. Ballroom D (HI))*
- **7:00 p.m.** 1994-95 LAC/Program Committee Dinner *(Jr. Ballroom E (HI))*
- **8:00 p.m.** Surface Science Postdeadline Discovery Session *(Grand Ballroom (HI))*

**H=Radisson Hotel  
CC=Colorado Convention Center**

### SHORT COURSES THURSDAY

- **8:30 a.m.** Vacuum Technology *(Room C109 (CC))*
- **8:30 a.m.** A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling; & ISS/RBS *(Room C107 (CC))*
- **8:30 a.m.** Physics, Chemistry, and Mechanics of Adhesion (New) *(Room C105 (CC))*
- **8:30 a.m.** Hard Coatings by PVD Methods *(Room C103 (CC))*
- **8:30 a.m.** Depth Profiling *(Room C107 (CC))*
- **8:30 a.m.** Mass Flow Controllers: Fundamentals, Techniques, and Applications *(Room C101 (CC))*
- **8:30 a.m.** Plasma-Enhanced CVD: Fundamentals, Techniques, and Applications *(Room C108 (CC))*
- **8:30 a.m.** Total Pressure Gauging Techniques *(Room C106 (CC))*
- **8:30 a.m.** Transparent Conducting Oxides: Their Science, Fabrication, Properties, and Applications *(Room C104 (CC))*
- **8:30 a.m.** Vacuum Evaporation/Deposition Technology *(Room C110 (CC))*
- **8:30 a.m.** Vacuum Leak Detection *(Room C112 (CC))*

### Special or Focus Area Sessions

- **Sensors, In-situ Diagnostics, Process Control**
- **NSBI-TTA** Micro-Instrumentation and Sensors
- **Nanostructure Fabrication and Atomic-Scale Manipulation**
- **EM-TTA** Materials for Nanostructures
<table>
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<th>Time</th>
<th>Title</th>
<th>Authors</th>
<th>Institution</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>Molecular Beam Homoeptaxial Growth and Surface Characterization of MgO(001)</td>
<td>T.T. Tran, T.A. Hileman, S.A. Chambers</td>
<td>Pacific Northwest Laboratory</td>
</tr>
<tr>
<td>2:20 pm</td>
<td>Step Fluctuation Kinetics and the Formation of (n)(x)=0/Ag(110)</td>
<td>W.W. Pail, M.R. Peng, N.C. Bartelt, J.E. Reutt-Robey</td>
<td>University of Maryland</td>
</tr>
<tr>
<td>2:40 pm</td>
<td>Oxidation Behavior of the (100) and (110) FeAl Surfaces</td>
<td>H. Graupner, L. Hammer, K. Muller, D.M. Zehner</td>
<td>University of Erlangen, Germany</td>
</tr>
<tr>
<td>3:00 pm</td>
<td>Electron Stimulated Oxidation of Metals at Low Temperature: N(111) at 120K</td>
<td>W. Li, M.J. Stimson, S.J. Sibener</td>
<td>University of Chicago</td>
</tr>
<tr>
<td>3:20 pm</td>
<td>Cesium/Oxide Interactions for Ultrathin Films on an α-Al$_2$O$_3$(0001)</td>
<td>K.R. Zavadil, Sandia National Laboratories, J.L. Ing, XIA Technology</td>
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<tr>
<td>4:00 pm</td>
<td>Infrared Spectroscopy of Oxygen and Formate on Cu(100): Broadband Reflectance and Low-Frequency Vibrations</td>
<td>K.C. Lin, R.G. Togin, Michigan State University, P. Dumas, Lure, France</td>
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<tr>
<td>4:20 pm</td>
<td>The Structure and Stability of Methyl Thiocarbonate on Ni(111)</td>
<td>D.R. Huntley, D.R. Mullins, S.H. Overbury</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>4:40 pm</td>
<td>A Comparative Study of P(CH$_3$)$_3$ and PHF$_3$ Decomposition on Ru(0001)</td>
<td>H.S. Tao, T.E. Madey, D. Diebold, Tulane University, N.D. Shinn, Sandia National Laboratories</td>
<td></td>
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<tr>
<td>5:00 pm</td>
<td>Long Range Periodicity and Local Order in Complex Molecule Chemisorption</td>
<td>B.G. Fredericks, F.M. Lembre, S. Dhesi, M.B. Lee, K. Kitching, N.V. Richardson</td>
<td>University of Liverpool, United Kingdom</td>
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<td>Time</td>
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<td>Speaker(s)</td>
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<td>2:00 pm</td>
<td>NSBI-ThA1</td>
<td>Micromachined Neural Interface Technology. G.T.A. KOVACS, Stanford University.</td>
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<td>Invited talk continued.</td>
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<tr>
<td>3:40 pm</td>
<td>NSBI-ThA7</td>
<td>Initial Tests of a Micromachined SEM. D.A. CREWE, A.D. FEINERMAN, University of Illinois, Chicago.</td>
<td></td>
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<tr>
<td>4:00 pm</td>
<td>NSBI-ThA8</td>
<td>Localized Photodiode for Near-Field Photodetection Optical Microscopy. R.C. DAVIS, C.C. WILLIAMS, University of Utah.</td>
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<td>NSBI-ThA9</td>
<td>Influence of Water Vapor on Nanotribology Studied by Friction Force Microscopy. M. BINGELI, C. MUTSCH,Streamline for Tribology, Switzerland. C.M. MATE, IBM Almaden Research Center.</td>
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<td>NSBI-ThA4</td>
<td>Nanotribology in Electrochemically Controlled Environment. M. BINGELI, J. BURGER, R. CHRISTOF, Dr. Suisse de Electronique et de Microtechnologie, Switzerland.</td>
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<td>NSBI-ThA7</td>
<td>Orientational Ordering of Polymers with A.F.M. in Contact Mode: An Application to Conjugated Polymers. Z. ELMAAKOUR, J.P. AYNE, T. BOUCHACINA, C. ODIN, Universite Bordeaux I, France. T. MASUDA, Kyto University, Japan.</td>
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<td>Time</td>
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<td>2:00 pm</td>
<td>AS-ThA1</td>
<td>Polymer Surface Damage by X-ray Photoelectron Spectroscopy.</td>
<td>W.F. STICKLE, P.E. SOBOLO, H. IWAI, Physical Electronics Laboratories.</td>
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<tr>
<td>3:00 pm</td>
<td>AS-ThA4</td>
<td>High Resolution XPS and ToFSIMS Studies of Polyamide Molecules.</td>
<td>A.G. SHARP, M.C. DAVIES, J.B. TENDLER, University of Nottingham, United Kingdom.</td>
</tr>
<tr>
<td>3:40 pm</td>
<td>AS-ThA6</td>
<td>Addition of a Single Chemical Functional Group to a Polymer Surface with a Mass-Separated Low Energy Ion Beam.</td>
<td>P. NOWAK, N.S. MCINTYRE, Surface Science Western and Univ. of Western Ontario, Canada.</td>
</tr>
<tr>
<td>4:00 pm</td>
<td>AS-ThA7</td>
<td>Polyfluoroether Lubricant Analysis by TOF-SIMS.</td>
<td>P. KASA, A.M. SPOOL, IBM Storage Systems Division.</td>
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**Thursday Afternoon, October 27, 1994**

**APPLIED SURFACE SCIENCE** Room A101 - Session AS-ThA pg. 293
Moderator: A.B. Ulman, Eastman Kodak Company.

**PLASMA SCIENCE** Room A109 - Session PS1-ThA pg. 294
Moderator: M. Nakamura, Fujitsu Ltd., Japan.

**PS1-ThA1** INVITED
Chemical Topography of Masked Poly-Si Films Etched in Cl2 and HBr-Containing, High Density Plasmas. V.M. DONNELLY, C.C. CHENG, D.V. GUNN, AT&T Bell Laboratories. E.P. HERMAN, AT&T Bell Laboratories & Columbia University.

*Invited talk continued.*


**PS1-ThA4** Molecular Dynamics Simulation of Atomic Layer Etching of Si(100). S.D. ATHAVELE, D.J. ECONOMOU, University of Houston.


**PS1-ThA6** Aspect Ratio Dependent Etching of Polysilicon. T.J. DALTON, H.H. SAWIN, Massachusetts Institute of Technology.


**PS1-ThA9** Characterization of Ion Directionality in Plasma Enhanced Chemical Vapor Deposition of Silicon Dioxide. J. LI, C. CHANG, J.P. MCVITIE, K.C. SARASWAT, Stanford University.

**PS1-ThA10** An Auger and XPS Study of Ar+ Sputtering Yield and Angular Distribution of Al. P.C. SMITH, R.B. TURKOT, J.P. KELLY, D.N. RIZIC, University of Illinois, Urbana.
<table>
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<th>Speaker(s)</th>
<th>Institution</th>
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<tbody>
<tr>
<td>2:00 pm</td>
<td>PS2-ThA1</td>
<td>Target Area Design Basis and System Performance for the National Ignition Facility</td>
<td>M.T. Tobin, V. Karpenko, K. Hagan, A. Anderson</td>
<td>Lawrence Livermore National Laboratory, R. Wavrik, R. Garcia, Sandia National Laboratories</td>
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<td>2:20 pm</td>
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<td>Invited talk continued.</td>
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<td>2:40 pm</td>
<td>PS2-ThA3</td>
<td>Precision Shell Characterization using Radial Averaging of X-ray Images</td>
<td>R.B. Stephens</td>
<td>General Atomics</td>
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<tr>
<td>3:00 pm</td>
<td>PS2-ThA4</td>
<td>High Yield Fabrication of Uniform, Large Diameter Foam Shells for Laser Fusion Targets using Polymerization by Photo Initiation with UV Light</td>
<td>M. Takagi, Y. Kobayashi, T. Norimatsu, Y. Iwata, S. Nakai</td>
<td>Osaka University, Japan</td>
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<tr>
<td>3:20 pm</td>
<td>PS2-ThA5</td>
<td>The Historical Development of the Microencapsulation Technique used to Fabricate ICF Capsules</td>
<td>U. Kubo, H. Nakano, H. Tsukubihara, Kinki University</td>
<td>Japan</td>
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<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td>PS2-ThA7</td>
<td>Vapor Deposited Solid Hydrogen Crystals: Size, Structure, and Roughening</td>
<td>G.W. Collins, E. Mapoles, W. Units, T. Bernat,</td>
<td>Lawrence Livermore National Laboratory</td>
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<tr>
<td>4:20 pm</td>
<td>PS2-ThA8</td>
<td>The Design, Performance, and Application of an Atomic-Force Microscope-based Profilityometer</td>
<td>R.L. Mceachern, C.E. Moore, R.J. Wallace</td>
<td>Lawrence Livermore National Laboratory</td>
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<td>4:40 pm</td>
<td>PS2-ThA9</td>
<td>Cryogenically Reusable Vacuum Seals</td>
<td>N.B. Alexander</td>
<td>General Atomics</td>
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<td>5:00 pm</td>
<td>PS2-ThA10</td>
<td>Estimation for Heating Uniformity on a Solid Fuel Layer by a Glow Discharge Plasma in Plasma Layering Technique to Make a Uniform Cryogenic Fuel Layer Inside an ICF Target</td>
<td>T. Norimatsu, M. Ishihara, M. Takagi, Y. Iwata, S. Nakai</td>
<td>Osaka University, Japan</td>
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<td>TF-ThA2</td>
<td>Sputter Deposition of Cermet Fuel Electrodes for Solid Oxide Fuel Cells</td>
<td>T. Tsai, S.A. Barnett</td>
<td>Northwestern University</td>
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<td>3:00 pm</td>
<td>TF-ThA4</td>
<td>Morphology of Precursors and CuIn_{x}Ga_{1-x}Se_{2} Thin Films Prepared by Two-Stage Seleniumization Process</td>
<td>N.O. Drene, S. Kutath, Florida Solar Energy Center</td>
<td>H.R. Moutinho, National Renewable Energy Laboratory</td>
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<tr>
<td>3:40 pm</td>
<td>TF-ThA5</td>
<td>Structure, Morphology, and Properties of CuIn_{x}Ga_{1-x}Se_{2} Epitaxial Layers on GaAs</td>
<td>S.A. Berry, D. Schroeder, L.-C. Yang, H.Z. Xiao, A. Rockett</td>
<td>University of Illinois, Urbana</td>
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<tr>
<td>4:00 pm</td>
<td>TF-ThA6</td>
<td>Growth of Chalcoprite Cu(In,ln)Se_{2}/CuIn_{x}Se_{2} Absorbers by r.f. Sputtering</td>
<td>J.L. Hernandez-Rojas, I. Martinez, G. Gonzalez-Diaz, J. Santamaria, F. Sanchez-Quesada</td>
<td>Universidad Complutense, Spain</td>
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<tr>
<td>4:40 pm</td>
<td>TF-ThA10</td>
<td>Low Temperature, High Rate Deposition of Electrochromic Materials/Devices with MetaMode</td>
<td>N.A. Q'Brien, J.G.H. Mathew, B.P. Hichwa, Optical Coating Laboratory, Inc</td>
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<tr>
<td>5:00 pm</td>
<td>TF-ThA10</td>
<td>Properties of Transparent Conducting ZnO-Stannite Films Prepared by RF Magnetron Sputtering</td>
<td>T. Minami, S. Takata, H. Satoh, H. Sonoda</td>
<td>Kanazawa Institute of Technology</td>
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<td>2:00 pm</td>
<td>EM-ThA1</td>
<td>Invited Magnetotransport in Semiconductor Nanostructures</td>
<td>K.P. Martin, Georgia Institute of Technology</td>
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<td>Invited talk continued</td>
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<td>2:40 pm</td>
<td>EM-ThA3</td>
<td>Nanoscale Structures in III-V Semiconductors Using Sidewall Masking</td>
<td>F. Ren, AT&amp;T Bell Laboratories; S.J. Pearton, C.R. Abernathy, University of Florida; Gainesville; J.R. Latham, AT&amp;T Bell Laboratories</td>
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<td>and High Ion Density Dry Etching</td>
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<td>Fabricated by Neutral Atom Etching at Cryogenic Temperatures</td>
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<td>3:20 pm</td>
<td>EM-ThA5</td>
<td>Room Temperature Photoluminescence and Electroluminescence from Ge</td>
<td>J.R. Myone, N. Chaudhry, R. Tellevan, University of Michigan</td>
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<td>Nanocrystals in SiOx formed by Ion Implantation and Precipitation</td>
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<td>with Scanning Tunneling Microscope</td>
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<td>4:00 pm</td>
<td>EM-ThA7</td>
<td>Temporal Evolution of Nanoscale Interfacial Phases between GaAs and</td>
<td>T.-J. Kim, P.H. Holloway, University of Florida, Gainesville</td>
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<td>Metal Films</td>
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<td>4:20 pm</td>
<td>EM-ThA8</td>
<td>Selective Chemical Vapor Deposition of Copper on Pd-Activated Self-Assembled Films</td>
<td>S.J. Potoczniak, D.S.Y. Hsu, J.M. Calvert, P.E. Persson, Naval Research Laboratory</td>
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<td>An Improved Method of Non-Intrusive Deposition Rate Monitoring by Atomic Absorption Spectroscopy</td>
<td>C. Liu, Y. Guan, Intelligent Sensor Technology, Inc.</td>
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<td>4:40 pm</td>
<td>EM-ThA9</td>
<td>Surface Chemistry of Al2O3 Deposition using Al(CH3)3 and H2O in a Binary Reaction Sequence</td>
<td>A.W. Ott, A.C. Dillion, S.M. George, University of Colorado, Boulder; J.D. Way, Colorado School of Mines</td>
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<td>A Model Based Technique for Estimation of Fluorine in a CF4/Ar Plasma</td>
<td>P.D. Hanish, J.W. Grizzle, M.O. Giles, University of Michigan</td>
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<td>Role of Inert Carrier Gases in Modeling, Design and Operation of a Single Waler APCVD Reactor for Manufacturing</td>
<td>P.N. Garbi, Queen's University, Canada</td>
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<td>2:00 pm</td>
<td><strong>VM-ThA1</strong> INVITED</td>
<td>Environmentally Compatible Coating Technology. K.O. LEGG, A. ADAMSKI, C. WEST, P. RUDNICK, Northwestern University, F. RASTADAR, Cummins Piston Ring Division, J. SCHELL, GE Aircraft Engines, A. GONZALES, Corpus Christi Army Depot, B. SARTWELL, Naval Research Laboratory.</td>
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<td>2:20 pm</td>
<td>Invited talk continued.</td>
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<td>2:40 pm</td>
<td><strong>VM-ThA3</strong> INVITED</td>
<td>Development of a Manufacturing Process to Sputter AlN Barrier Layers on MO Recording Disks. D.A. GLOCKER, Eastman Kodak Company.</td>
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<td>3:00 pm</td>
<td>Invited talk continued.</td>
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<td>4:00 pm</td>
<td><strong>VM-ThA7</strong> INVITED</td>
<td>Recent Technology Advancements in Optical Thin Film Manufacturing. P.M. LEFEBVRE, B.P. HICKWA, R.W. ADAIR, Optical Coating Laboratory Inc.</td>
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<td>4:20 pm</td>
<td>Invited talk continued.</td>
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<td>4:40 pm</td>
<td><strong>VM-ThA9</strong></td>
<td>Sputter Deposition of Indium Tin Oxide (ITO) from a Cylindrical Ceramic Target. K.P. GIBBONS, T. VAN SKIKE, C.K. CARNIGLIA, Airco Coating Technology.</td>
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NANO 3/NANOMETER–SCALE SCIENCE AND TECHNOLOGY
Room BR4 - Session NS–ThP, pg. 304

Nanoelectronics and Nanofabrication
Moderator: J.D. Dagata, National Institute of Standards and Technology.

Presenters present: 5:00–6:30 pm.

NS-ThP1 Voltage Controlled Nanometer Scale Oxidation of Si(100) Surface by STM. E. Pérez-Murano, G. Abadal, N. Barniol, X. Aymerich, Universitat Autónoma Barcelona, Spain.

NS-ThP2 Local Electrical Switching Effects in CuTCNO Films Studied by Surface Modification with STM. L. Wang, C.W. Yuan, J.W. Liang, H. Chen, Y. Wei, Southeast University, PR China.


NS-ThP4 Measurement of the Piezoelectricity of Thin Films with STM. R. Winters, M. Feinerman, C. Ems, G. Weiss, Universität Heidelberg, Germany.


NS-ThP6 Nanometer–Scale Definition of Conductors on Insulators using the AFM. M. Falvo, D. Glick, R. Superfine, University of North Carolina, Chapel Hill.


NS-ThP8 Local Fusion of Metallic Surfaces and Dots Formation using STM. L. Libidoule, J.-M. Gilles, Faculté Notre-Dame de la Paix, Belgium.


NS-ThP13 Submicron Pattern Transfers in HgCdTe and GaAs by Reactive–Ion Etching through Nanochannel Glass Lithographic Masks. C.R. Eddy, Jr., R.J. Tonucci, Naval Research Laboratory.

NS-ThP14 The Technological Aspects of Cluster Film Application in Nanoelectronics with Use of STM. P.N. Lukinovich, E.E. Butman, I.A. Ryzhikov, “Beta” R&D Institute, Russia.


NS-ThP16 Ambient Dependence of Nanometer Scale Writing on Silicon (100) Surfaces by an Atomic Force Microscope. L. Tsau, D. Wang, K.L. Wang, University of California, Los Angeles.

NS-ThP17 The Formation of Nanometer–Scale Molecular Memory by STM. K. Matsushige, Kyushu University, Japan. S. Tak, Kyushu University, Japan.

NS-ThP18 Using Scanning Tunneling Microscopy for Control and Reversal of Strong Rifle Surfaces of Plane Carbon Field Emitters Modified by Ion Beams. A.L. Gudorov, V.V. Protsenko, V.D. Stolyarova, N.P. Shishkin, Institute of Theoretical & Experimental Physics, Russia.


Næs, Danish Institute for Fundamental Metrology, Denmark. N.B. Larsen, University of Copenhagen, Denmark. T. Bjørnholm, Danish Institute for Fundamental Metrology, Denmark.


NS-ThP24 Nanoscale Lithography and Pattern Transfer on Si(100) with a UHV Scanning Tunneling Microscope. C. Wang, J.R. Tucker, J.W. Lyding, University of Illinois, Urbana–Champaign.
Aspects of Electronic Materials and Processing II

Moderator: A. Rockett, University of Illinois, Urbana.

Presenters present: 5:00–6:30 pm.

EM-ThP1 Study of Damage Caused by W Etchback and Effect on Subsequent AlGaAs Deposition. I.R. ALLEN, J.M. GRANT, Sharp Microelectronics Technology, Inc.


EM-ThP3 Excimer Laser Induced Deposition of Tungsten on GaAs from WF₆ and SiH₄. M. TABBAR, M. MEUNIER, R. IZQUIEDO, A. YELON, Ecole Polytechnique de Montreal, Canada.

EM-ThP4 The Effect of Chemical Etchants on Back Contact Formation to CdTe-Based Solar Cells. X. LI, D. NILES, F. HASOON, P. SHELDON, National Renewable Energy Laboratory.

EM-ThP5 STM Observations of Sub-Surface Donor and Acceptors in GaAs. J.-F. ZENG, D. OSWAL, E. WEBER, M. SALMERON, Lawrence Berkeley Lab & University of California, Berkeley.


EM-ThP9 Interface Exciton Luminescence: Indication of Interface Inhomogeneities in Single GaAs/GaAlAs Heterostructures. V.N. BESPOLOV, V.V. EVLISTROPOV, M.V. LEBEDEV, V.V. ROSSIN, A.F. Ioffe Physico-Technical Institute, Russia.

EM-ThP10 The Nature of Ambient (100) GaAs Surfaces. O.J. GLEMBOCKI, J.A. TUUCH, Naval Research Laboratory. K.K. KO, S.W. PANG, University of Michigan. J.A. DAGATA, Naval Research Laboratory. R. KAPLAN, Naval Research Laboratory. C.E. STULTZ, Wright Patterson Laboratories.

EM-ThP11 Surface Studies of MBE Grown GaAs(111)A by HREELS, XPS, UPS and LEED. J. WU, G.J. LAPEYRE, Montana State University.

EM-ThP12 Geometric and Electronic Structure of Ni Silicide on Si(110). Y. KHANG, Seoul National University, Korea. D. JEON, Myongji University, Korea. Y. KUK, Seoul National University, Korea.

EM-ThP13 Electronic Band Structure of Epitaxial $\gamma$-FeSi₂(111)2x2. J. ALVAREZ, C. LIMONES, E.G. MICHEL, R. MIRANDA, M.E. DAVILA, J. MARTIN-GASIO, Universidad Autonoma de Madrid, Spain.


EM-ThP18 Adsorption and Co-adsorption of Boron and Oxygen on Ordered α-SiC Surfaces. V.M. BERMUDEZ, Naval Research Laboratory.


EM-ThP20 Oxidation-Induced Roughening and Oxide Nucleation on Si(011)–2x1 Surfaces: An STM Study. J.P. PELT, J.V. SEIPLE, Ohio State University.


EM-ThP22 Aged-20 Years, Resolved STM Study of the Interaction of Trimethyl Gallium with Si(001). M.J. BRONIKOWSKI, R.J. HAMERS, University of Wisconsin, Madison.


EM-ThP24 Synchrotron Radiation Induced Decomposition of closo-1,2-dicabado-decaborane. D. BYUN, S.-D. HWANG, University of Nebraska, Lincoln and Syracuse University. J. ZHANG, University of Nebraska, Lincoln. H. ZENG, Syracuse University. F.K. PERKINS, Naval Research Laboratory. G. VIDALI, Syracuse University. P.A. DOWDEN, University of Nebraska, Lincoln.


EM-ThP26 Chemisorption and Thermally Activated Etching of Iodine on Si(110)–2x1. D. RIUQ, F. STEPMAN, R.J. PEDDIE, J.H. WEAVER, University of Minnesota.
Thursday Afternoon Poster Sessions

TF-TP1 Thin Film Characterization and Sensors Applications
Moderator: A.C. Wall, IBM Corporation.

Presenters present: 5:00-6:30 pm.

TF-TP1 Auger Electron Spectroscopy as a Real-Time Probe of Film Composition during MBE Growth. S.A. CHAMBERS, T.T. TRAN, Pacific Northwest Laboratory.


TF-TP3 Mass Spectroscopy of Recoiled Ions (MSRI) as a Tool for Surface Analysis of Boron Nitride Thin Films during Deposition and EX SITU. L. WOLVERTON, J.A. SCHULTZ, K. EPERIS-SMITH, K. WATERS, IONWORKS.

TF-TP4 Deposition of Polysilicon by ECR CVD and RTO CVD. P. MÜLLER, E. CONRAD, Hahn-Meitner-Institut, Germany. T.R. OMSTAD, CVI Products, Inc.

TF-TP5 Oxygen Content of Indium Tin Oxide Films Fabricated by Reactive Sputtering. S. HONDA, A. TSUJIMOTO, M. WATAMORI, K. OURA, Osaka University, Japan. F. SHOJ, Kyushu Kyeritsu University, Japan.

TF-TP6 Thin-Film and Buried Interface Characterization using High Brightness Synchrotron Radiation. J.J. JIA, T.A. CALLCOTT, University of Tennessee; F.J. HUMPER, H. AKATSU, IBM TJ Watson Research Center; M.O. GEMAN, J. STOUT, IBM Almaden Research Center; O.L. EDERER, Tulane University; J.A. CARLISLE, E.A. HUDSON, L.L. TERMINELLO, Lawrence Livermore National Laboratory; R.C.C. PERERA, D.K. SHUJ, Lawrence Berkeley Laboratory.


TF-TP8 Ion Beam-Induced SiC Crystallization of Carbon Implanted Layers in Si(100) Studied by Electron Spectroscopy. H.J. STEFFEN, V. HEERA, R. KOEGLER, W. SKORUPA, Research Center Rossendorf Inc., Germany.


TF-TP10 Study of Adsorption and Desorption of 1,5-Cyclooctadiene on Cu(111) Surface. O. CHENG, K. GRIFFITHS, S. SERSHINSKI, Z. YAN, P.R. NORTON, R.J. PUDDEPHATT, University of Western Ontario, Canada.


TF-TP12 Synchrotron Radiation Photoelectron Emission Microscopy of Natural and Chemical Vapor Deposited Diamond Surfaces. J.D. SHOVIN, M.E. KORDESCH, Ohio University; D. DUNHAM, B.P. TONER, University of Wisconsin, Milwaukee; W. ENGEL, Fritz Haber Institute, Germany.


TF-TP14 Phase Stability and Al Solubility in Epitaxial Nd1.5Al0.5N Films Grown on MgO(001) by Reactive Sputtering. T.I. SELNUNGER, D.J. MILLER, K.E. GRAY, Argonne National Laboratory; L. HULTMAN, M.R. SARELA, Jr., Linköping University, Sweden.

TF-TP15 Growth and Structure of Silicon Oxide Thin Films on Polymers by AFM and XPS. H. LI, S. KRAMMERHOEK, E. EZZEL, ROC Group Technical Center, F. JANSSEN, Airco Cooling Technology.

TF-TP16 Characterization of Aluminum based Oxide Layers formed by Microwave Plasma. Z. KATZ, Ben-Gurion-University, Israel; A. RAVEN, NRC-Regen, Israel.


TF-TP18 ReSiOx Thin Film Infrared Detectors. J.P. BECKER, J.E. MAHAN, Colorado State University; R.S. LONG, Hewlett-Packard Co.

TF-TP19 Detection of Chlorinated Hydrocarbon Vapors using 1,3, bis-(1-pyrene)propane in a Poly (vinyl alcohol) Film. S.L. ROSE-PEHRSSON, J. KRECH, Naval Research Laboratory.


TF-TP23 AlN Thin Film Structure Development and Quality during Magnetron Sputter Deposition of Si(100) and Si(111), Studied by XRD, AES, and FE SEM. B. LADZ, M. CHASON, S. VOIGT, Motorola.

TF-TP24 Microstructural Characterization of Pd/InP and Ag/InGaAs Films Grown at Room Temperature and 77K. M.E. HAWLEY, D.X. JIA, Los Alamos National Laboratory; J. PALMER, H.J. LEE, W. ANDERSON, State University of New York, Buffalo; D. HOELZER, Alfred University.

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### Surface Science
Room A205 – Session SS1-FrM pg. 317

**Surface Electronic Structure**

Moderator: R. Hwang, Sandia National Laboratories.

- **8:20 am** SS1-FrM1 Theory of Clean and H-covered Be(0001). R. STUMPF, P.J. FEIBELMAN, Sandia National Laboratories.
- **8:40 am** SS1-FrM2 Core-Level Spectroscopy of Metal Monolayers and Interfaces. N.D. SHINN, Sandia National Laboratories. B. KIM, Pohang University of Science & Technology, Korea. J.K. KIM, Kon-Kuk University, Korea. T.-H. KANG, Pohang University of Science & Technology, Korea. J.L. ERSKINE, University of Texas, Austin.
- **9:00 am** SS1-FrM3 Core Level Shifts in Trimetallic Systems. D.R. RAINER, J.S. CORNEILLE, D.W. GOODMAN, Texas A&M University.
- **9:40 am** SS1-FrM5 Nature of the 4s-derived States of Adsorbed K Studied by X-ray Absorption Spectroscopy and the Core Hole Clock Method. A. SANDELL, A. NILSSON, P.A. BRÜHLWILER, N. MARTENSSON, Uppsala University, Sweden.
- **10:00 am** SS1-FrM6 Photomission Study of Alkali Metal Adsorption on the Passivated Si(111)1x1–As Surface. M.C. HÄNNSSON, M. JOHANSSON, L.S.O. JOHANSSON, University of Lund, Sweden.
- **11:00 am** SS1-FrM9 Inverse Photoemission Study of CO Chemisorption on Metallic Quantum Wells. F.G. CURTI, R.A. BARTYNSKI, Rutgers University.

### Surface Science
Room A201 – Session SS2-FrM pg. 319

**Group IV Semiconductor Surface Structure**

Moderator: R. Hamers, University of Wisconsin.

- **8:40 am** SS2-FrM2 High Resolution TEM Determination of the (5x2) Au/Si(111) Structure. R. PLASS, L.D. MARKS, Northwestern University.
- **9:20 am** SS2-FrM4 Boron Induced Structures on the Si(111) Surface. C. WANG, T.C. SHEN, J.W. LYDING, J.R. TUCKER, University of Illinois, Urbana-Champaign.
- **9:40 am** SS2-FrM5 Surface-Induced Optical Anisotropies of Single-Domain (2x1)-Reconstructed (001) Si and Ge Surfaces. L. MANTESE, T. YASUDA, D.E. ASPHINX, North Carolina State University.
- **10:00 am** SS2-FrM6 High-Temperature Structural Phases of the Si(111) Surface Studied with High-Resolution, Energy-Resolved Helium Atom Scattering. C.A. MEUL, Naval Research Laboratory. G. LANGE, J.P. TOE- NIES, Max-Planck-Institut für Strömungsforschung, Germany.
- **10:40 am** SS2-FrM8 Strain-induced Surface Morphology on Si(111)-(7x7). Y. WEI, L. LI, I.S.T. TSONG, Arizona State University.  
- **11:00 am** SS2-FrM9 A Novel Facet Reconstruction of Si(112) Revealed by STM. A.A. BASKI, L.J. WHITMAN, Naval Research Laboratory.

*Finalist for the Morton M. Traum Award*
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<tr>
<td>8:20 am</td>
<td>NS1-FrM1</td>
<td>INVITED Chemistry and Applications of Self-Assembled Films. J.M. CALVERT</td>
<td>Naval Research Laboratory.</td>
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<td>8:40 am</td>
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<td>9:40 am</td>
<td>NS1-FrM5</td>
<td>Fabrication of 10nm Holes on a 20nm Hexagonal Lattice in Si(100). T.A. WINNINGHAM, S.D. WILLIAMS, K. DOUGLAS, University of Colorado, Boulder. D. CHUOTOV, J.D. PIPER, K.P. MARTIN, H.P. GILLIS, Georgia Institute of Technology.</td>
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<td>10:00 am</td>
<td>NS1-FrM6</td>
<td>New Compound Quantum Dot Materials Produced by Electron-Beam Induced Deposition. M. WEBER, Technische Hochschule Darmstadt, Germany. H.W.P. KOOPS, Deutsche Bundespost Telekom, Germany. M. RUDDOLPH, J. KRETZ, Technische Hochschule Darmstadt, Germany. G. SCHMIDT, Universitat Hamburg, Germany.</td>
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<td>10:20 am</td>
<td>NS1-FrM7</td>
<td>Synthesis of Polymers at Highly Ordered Pyrolytic Graphite Templates via Flow Injection Scanning Tunneling Microscopy. M.L. MYRICK, P.G. VAN PATTEN, J.D. NOLL, University of South Carolina.</td>
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<td>10:40 am</td>
<td>NS1-FrM8</td>
<td>Nanaarchitectures of Poly(di-n-alkylsillylene)s. S. SHEIKO, H. FREY, M. MOLLER, Universitat Ulm, Germany.</td>
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<td>11:00 am</td>
<td>NS1-FrM9</td>
<td>INVITED Carbon Nanotubes. S. IJIMA, NEC R&amp;D Group, Japan.</td>
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<td>11:20 am</td>
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<td>invited talk continued</td>
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<td>11:40 am</td>
<td>NS1-FrM10</td>
<td>C$_x$ on Silicon: Self-Organization, Film Growth, and SiC Formation. D. SARIO, D. CHEN, University of Arizona, Tucson.</td>
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<td>NS2-FrM4</td>
<td>Room Temperature Quantum Mechanical Capacitance Measured with the Ultrastack Scanning Probe Microscope. D. BOTKIN, S. WEISS, D.F. OGLE, M. SALMERON, D.S. CHEMLA, University of California, Berkeley.</td>
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<td>NS2-FrM5</td>
<td>INVITED Continuous Observation of the Motion of Single Adsorbed Atoms and Molecules with Picosecond and Sub-nanometer Resolution. G.M. MCELLELAND, F. WATANABE, H. HEINZELMANN, IBM Almaden Research Center.</td>
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<td>10:00</td>
<td>AS-FrM6</td>
<td>Strained Siloxane Rings in the Surface of Silica: Their Reaction with Organosiloxanes. A. Rabe, T.A. Michalske, W.L. Smith, Sandia National Laboratories.</td>
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<td>10:40</td>
<td>AS-FrM8</td>
<td>Improved Adhesion at the Copper/Polyimide Interface using an Organometallic Additive. D. Coulman, The DuPont Company.</td>
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<td>TF-FrM1</td>
<td>In Situ Ellipsometric Monitoring and Post-Deposition Characterization of Dielectric Optical Multilayers. P. He, S. Pittal, B. Johnson, Y.H. Woolam, J.A. Woolam Co., Inc. J.H. Kim, CVI Laser Corp.</td>
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<td>TF-FrM2</td>
<td>Real Time Monitoring of the Deposition and Growth of Thin Organic Films by In Situ Ellipsometry. J.F. Wall, E. Clauberg, R.W. Murray, J.A. Irene, University of North Carolina, Chapel Hill.</td>
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<td>TF-FrM4</td>
<td>Deposition and Characterization of Transition-Metal Nitride Superlattices. S.A. Barnett, Northwestern University.</td>
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<td>TF-FrM5</td>
<td>Spectroscopic Ellipsometry of Thin Films on Transparent Substrates: A Formalism for Data Interpretation. Y.H. Yang, J.R. Aebelen, University of Illinois, Urbana.</td>
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<td>TF-FrM6</td>
<td>Optical Emission Spectroscopy of Plasmas for Diamond Growth. V. Manuonda, R. Dillon, University of Nebraska.</td>
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<td>TF-FrM7</td>
<td>Optical Second Harmonic Generation (SHG) as an In Situ Technique to Monitor the Growth and to Analyse the Structure of Thin Films. M. Buck, Ch. Dressler, M. Grunze, Universitat Heidelberg, Germany. F. Trager, Universitat Kassel, Germany.</td>
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### ELECTRONIC MATERIALS
Room A108 – Session EM-FrM pg. 327

**Interface Characterization**

**Moderator:** S. A. Chambers, Pacific Northwest Laboratory.

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<td>8:20 am</td>
<td><strong>EM-FrM1 INVITED</strong> High Resolution X-ray Reflectivity Characterization of Interface Roughness and Correlation.</td>
<td>R.L. HEADRICK, Cornell University.</td>
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<td>Invited talk continued.</td>
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<tr>
<td>9:00 am</td>
<td><strong>EM-FrM3</strong> Buffer Layer– Superlattice Interactions in the AIAs/GaAs Superlattice System.</td>
<td>J.G. PELLEGRINO, National Institute of Standards &amp; Technology, S.B. QADRI, Naval Research Laboratory, B. ROUGHANI, GM Engineering and Management Institute, C.A. RICHTER, SRC Research Associate.</td>
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<td>9:20 am</td>
<td><strong>EM-FrM4</strong> Interfacial Properties of Metal–Insulator Semiconductor Capacitors on GaAs(110).</td>
<td>L.J. HUANG, R. KRISINAMURTHY, W.M. LAU, University of Western Ontario, Canada. S. INGREGY, Bell Northern Research, Canada. D. LAUMHEER, J.-P. NOEL, National Research Council, Canada.</td>
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<td>10:20 am</td>
<td><strong>EM-FrM7</strong> Modified Surface Charge Spectroscopy for the Characterization of Insulator–Semiconductor Structures.</td>
<td>W.M. LAU, I.J. HUANG, University of Western Ontario, Canada. R.W.M. KWOK, G. JIN, Chinese University of Hong Kong.</td>
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<td>10:40 am</td>
<td><strong>EM-FrM8</strong> Buried Contaminant Structure Determination with Component-Resolved X-ray Photoelectron Diffraction in CaF₂/O/Si(111) Heterostructures.</td>
<td>M. LESKOVAR, U. HESSINGER, M.A. OLMSHEAD, University of Washington.</td>
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<td>11:00 am</td>
<td><strong>EM-FrM9</strong> A Study of Thermal Oxidation of Rough Silicon Surfaces.</td>
<td>D. LIU, L. SPANOS, E.A. IRENE, University of North Carolina, Chapel Hill.</td>
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*Peter Mark Award Winner

### VACUUM METALLURGY
Room A106 – Session VM-FrM pg. 329

**Surface Engineering for Wear and Corrosion Protection**

**Moderator:** C.R. Parent, The Gillette Company.

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<th>Time</th>
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<td>8:20 am</td>
<td><strong>VM-FrM1</strong> Friction and Wear of Gas Lubricated SiC/Mo Couples in Sliding Contact.</td>
<td>L.L. SINGER, Naval Research Laboratory. TH. LE MOIGNE, CH. DONNET, J.M. MARTIN, Ecole Centrale de Lyon, France.</td>
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<td><strong>VM-FrM2</strong> Microstructural Effects on Tribological Properties of Electron Enhanced Magnetron Sputtered Coatings.</td>
<td>J.M. SCHNEIDER, A.A. VOEVODIN, C. REBHOLZ, A. MATTHEWS, University of Hull, United Kingdom. D.B. LEWIS, Sheffield Hallam University, United Kingdom.</td>
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<td>9:20 am</td>
<td><strong>VM-FrM3</strong> Coatings Defects on Corrosion Behavior of Hard Nitride Coatings on Steel.</td>
<td>Y. B. WANG, M.S. WONG, R. KRUEGER, W.D. SPROUL, T.J. BARLO, Northwestern University.</td>
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<td>11:00 am</td>
<td><strong>VM-FrM7</strong> Vacuum Technology for Coating TiCN-Based Cermet.</td>
<td>I.Y. KONYASHIN, Hardmetal Technologies, Russia.</td>
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<td>11:20 am</td>
<td><strong>VM-FrM8</strong> Fluoroplastics Coating of Cold Drying – &quot;TETRON&quot;.</td>
<td>V.I. DEMIN, V.I. RAKHOVSKY, Research Center for Surface and Vacuum Investigation, Russia.</td>
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*Peter Mark Award Winner*
MY SCHEDULE
Friday Morning, October 28, 1994

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OTHER EVENTS FRIDAY
7:00 a.m. Companions Program (see insert) (Silver Room (H))
8:00 a.m. Surface Science Database Committee Meeting (Denver Room (H))
12:00 Noon Foreign Interactions (Gold Room (H))

SHORT COURSES FRIDAY
8:30 a.m. Vacuum Technology (Room C109 (CC))
8:30 a.m. A Comprehensive Course on Surface Analysis: AES, XPS, SIMS, Depth Profiling, & ISS/RES (Room C107 (CC))
8:30 a.m. Hard Coatings by PVD Methods (Room C103 (CC))
8:30 a.m. Gas Flow in Vacuum Systems (Room C112 (CC))
8:30 a.m. Ion Scattering and Rutherford Backscattering Spectroscopies (Room C107 (CC))
8:30 a.m. Nucleation, Growth, and Microstructure Evolution (Room C110 (CC))
8:30 a.m. Optical Diagnostic Techniques for Plasma Processing (Room C104 (CC))
8:30 a.m. Pumping Hazardous Gases (Room C108 (CC))
8:30 a.m. Rapid Thermal Processing: Equipment, Technology, and Process (Room C108 (CC))
8:30 a.m. Scanning Tunneling and Atomic Force Microscopy (Room C108 (CC))
8:30 a.m. Throughput Pump Technology: Mechanical, Turbomolecular, and Diffusion Pumps (New) (Room C102 (CC))
8:30 a.m. UHV Design and Practices (Room C101 (CC))

Special or Focus Area Sessions
Sensors, in-situ Diagnostics, Process Control
TF-FrM In-situ Thin Film Characterization
H additional structure Fabrication and Atomic-Scale Manipulation
NS1-FrM Novel Materials and Methods for Nanofabrication
AVS SHORT COURSE GENERAL INFORMATION

Location
Colorado Convention Center
700 14th Street
Denver, CO 80202
303/640-8000
The short courses, symposium technical sessions, and equipment exhibit will take place at the Colorado Convention Center.

Schedule
All courses will begin at 8:30 a.m. and finish at 5:00 p.m. with a hour break for lunch. Lunch is not included.

Certificates
Certificates of completion will be given to all students attending the full course.

Textbook and Course Notes
The fee for most courses includes a set of class notes. With many of the short courses offered, the fee for the course includes the cost of a published, hardcover textbook. Plain notebooks are not provided.

Registration Locations and Hours
All short courses will take place at the Colorado Convention Center. All pre-registered students must check in at the Short Course Registration desk located in ??? to collect their course materials and badges.
Registration hours are as follows:
Sun, October 23  4:00 p.m. to 7:00 p.m.
Mon, October 24  7:00 a.m. to 5:00 p.m.
Tues, October 25–Thurs, October 27  7:30 a.m. to 5:00 p.m.
Fri, October 28  7:30 a.m. to 12:00 noon

Further Information
For further information contact:
Margaret Stringer, AVS, 120 Wall Street, 32nd Floor, New York, NY 10005; 212/248-0326.
H. S. Taylor in 1925. Modern surface spectroscopic and structural methods have addressed this problem in more recent times. In this talk, a model chemical reaction, the formation of CO from CO and oxygen on stepped Pt single crystal surfaces, will be described, identifying the most active site on two related Pt single crystal surfaces. In addition, step defect sites have been used for trapping mobile molecules to observe and characterize a new surface phenomenon, electron stimulated migration, ESM.

INVITED

*Work supported by the Office of Basic Energy Sciences (DOE), and The Air Force Office of Scientific Research (AFOSR).


Laser-induced thermal desorption with post-ionization and FT mass spectrometry (LITD/FTMS) is utilized to study the cyclotrimination of acetylene to benzene on Pd(111). A Nd:YAG laser (1064 nm, 5 ns, ~10¹⁰ W/cm²) is used to desorb neutrals without substrate ablating. A 70 eV electron beam post-ionizes the neutrals and FTMS is used for detection of the ionized species. LITD/FTMS yields the desorption of intact products and intermediates.

Heating acetylene on Pd(111) causes cyclotrimination to benzene even at 10⁻¹⁰ pressures. In addition, 6L exposure of acetylene with 30-monolayer coverages of sulfur (0.6 ML) produce low temperature (120 K) formation of thiophene, as well as benzene. These desorbs in the gas phase, however, very little is observed in TDS. Kinetic studies of benzene formation from acetylene (2.5 L) on clean Pd(111) were initiated resulting from our preliminary TDS and LITD/FTMS surveys. Isothermal rate investigations were conducted at temperatures ranging from 170 K to 205 K. The results indicate that benzene formation has an activation energy of 10 ± 1 kcal/mol and a preexponential factor of 9 × 10⁻⁹ s⁻¹. The effects of sulfur on the rates and selectivity of benzene and thiophene formation will also be reported.


10:00 am S11-MoM6 Acetylene Di- and Trimerization Reactions on Pd(111) and the (3 × 3)R30°-Sn/Pd(111) Surface Alloy, Janos Szanyi and M. T. Paffett, CST-1, Los Alamos National Laboratory, Los Alamos, NM 87545.

The di- and trimerization reactions of acetylene were studied over Pd(111) and (3 × 3)R30°-Sn/Pd(111) model catalysts at moderate pressures (20–100 Torr). The overall reaction activity of (3 × 3)R30°-Sn/Pd(111) surface alloy was ~4–5 times higher than that of Pd(111). Both surfaces produced C₂ and chain and cyclic Cₙ hydrocarbons as di- and trimerization products with C₂ production rates being about an order of magnitude higher than that for Cₙ hydrocarbons. For both the C₂ and Cₙ product groups the degree of unsaturation of the hydrocarbon molecules depended upon the experimental conditions applied (Pₚ/Cₚ/Cₚ). The formation of unsaturated hydrocarbon molecules often seen under all experimental conditions did not occur in this study. The investigation of the behavior of these hydrocarbons and their precursors for surface nitridation, and more stable than hydrocanadene. In addition, Cₙ surface bonds, which are used in catalysis to improve selectivity in hydrogenation reactions, can be formed. To further our understanding of alkylhydrazine surface chemistry, the thermal decomposition of 1,1-dimethylhydrazine (DMH), (CH₃)₂N-NH₂, at 100 K on Pt and Ag-modified Pt surfaces

10:20 am S11-MoM7 The Surface Chemistry of 1,1-Dimethylhydrazine on Pt and Ag-Modified Pt, A. L. Schwaner, M. Kozar, Diann J. Albera, and J. M. White, Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712.

The role of defect sites on metal surfaces has been of interest since the historical postulate of the active site by
has been studied by temperature programmed desorption, temperature programmed static secondary ion mass spectrometry, high resolution electron energy loss spectroscopy, X-ray and ultraviolet photoelectron spectroscopy. Our interests centered on the thermal desorption pathway and dehydrogenation of DMH, and how the dehydrogenation properties of Pt are influenced by increasing Ag coverage (0.5–14 ML). On clean Pt, DMH adsorbs molecularly at coverages >1 ML, and dissociatively at <1 ML. Thermal N-N bond scission occurs at 300 K creating two surface fragments, -NH₂ and (CH₃)₂N-. Each fragment has its own independent desorption pathway. Interestingly, hydrogen (320 K), ammonia (340 K) and nitrogen (855 K) desorb via thermal desorption of -NH₂ while desorption of (CH₃)₂N yields hydrogen (430 K), cyanide (610 K), cyanoegen (780 K) desorption products and surface carbon (>1000 K). On thickly Ag-covered Pt surfaces, the parent desorption was observed. At 8 °C coverages <1 ML, similar products to those observed desorbing from Pt were detected. However, the desorption temperatures were higher on Ag/ Pt than clean Pt; thus, we believe that Ag lowers the reactivity of Pt. The chemistry associated with the desorption of DMH on Pt and Ag-modified Pt will be described, and our results compared with thermal desorption of other alkylated amines and hydrazines. Supported by the U.S. Department of Energy, Office of Basic Sciences.

10:40 am SSI-MoM8 Adsorption and Aggregation of HCN on Pt(111), D. Jentsz, H. Celio, and M. Treuney, Department of Chemistry, M/C (111), University of Illinois at Chicago, 845 W. Taylor St., Room 4500, Chicago, IL 60607-7061.

We have investigated the adsorption of HCN on Pt(111) at 85 K, 200 K and 300 K from low to high exposures by Fourier transform infrared reflection absorption spectroscopy (FT-IRAS) and thermal desorption spectroscopy (TDS). Based on the IR spectra we conclude that at 85 K HCN weakly adsorbs onto the surface at low exposures since the spectra resemble that reported for HCN in an Argon matrix isolation study. Higher exposures (>1 L) aggregation occurs which is determined by the formation of hydrogen bonded species on the surface which leads to a shifting of vCN to lower frequencies and a broadening of the band. These multimers continue to form until at 20 L the IR spectrum resembles that for solid HCN which is composed of long chains of hydrogen bonded linear HCN. Upon annealing to 300 K, HCN irreversibly rehydridizes and forms a bent di-s bonded species with the C-H bond parallel to the surface. This is determined by the appearance of an intense band at ~1570 cm⁻¹ characteristic of a C-N double bond. High exposures at 300 K, hydrogen isocyanide monomer and HCN multimers form and exist on the surface simultaneously with the HCN monomer. At all temperatures, strong adsorbate-adsorbate interactions occur which lead to aggregation; however, above 200 K the surface mediates these interactions by strongly interacting with the adsorbate leading to irreversible rehydridization of the HCN monomer.

structure investigations. The determination of structure parameters of metal, metal alloy and semiconductor surfaces can be made element specific and well resolved for the first few surface layers with NICISS. The obtained lateral averaged surface data have been used for a proposal of a structure model, which in turn can be verified and complemented by atomic-resolution resolved microscopy for measuring unit cell parameters, and additionally with no need of atomic resolution to show morphological features as growth mechanisms, step or other defect distributions. The usefulness of the combination of methods will be demonstrated at metal and metal alloy surfaces, i.e. at Cu-Au. After prolonged annealing of Cu-Au(111) at a LEED (4 × 1) superstructure appears at room temperature. The (4 × 1) also shows up in the STM images. From the NICISS investigation the gold rich termination has been found. Upon oxygen exposure at 330K and subsequent annealing at 800K a segregation of Cu atoms to the surface occurs. In case of nitrogen at Cu-Au(110), the STM images indicate the same chain like structure elements as at Cu-Au(110)(2 × 3)-N surfaces. Structure models based on NICISS and STM measurements will be discussed.

9:00 am SS2-MoM3 Anharmonicity on the NiAl(110) Bimetallic Alloy Surface, Arthur P. Baddorf, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6024, and Aubrey T. Hambicki, Univ. of Pennsylvania, Philadelphia, PA.

Anharmonicity of an alloy surface has been directly measured for the first time by examining the temperature dependence of the energy and lifetime of a surface phonon on NiAl(110). This surface consists of an ordered array of Ni and Al atoms, is ruffled with the Ni contracted and the Al expanded, and exhibits an optical phonon at the center zone whose energy lies in the bulk band gap. High-resolution electron energy loss spectroscopy was employed to determine the energy and linewidth (lifetimes) of the optical phonon between 40 and 960 K. Over this range, the phonon energy decreases from 27.6 to 25.1 meV, following a functional form which is a signature for anharmonicity: little change at low T, and linear dependence at high T. The rate of decrease observed is 2.3/2 that found at the zone center on Cu(110). Since the two materials have a similar bulk thermal expansion, the enhancement of anharmonicity by the surface appears to be smaller on NiAl(110). Based on this comparison, the NiAl(110) surface appears to be about 50% more anharmonic than bulk NiAl.


Recent experimental and theoretical studies of the (3 × 3)R30° structures formed by adsorption of Na on Ag and Rb on Al(111) at room temperature have surprisingly revealed that for each system the alkali atoms adsorb in substitutional sites formed by disrupting the outermost substrate layer. No such consistent picture has yet emerged for the even more complicated structures which form at coverages other than 1/3 monolayer. We report here on a LEED determination of the geometry of the (2 × 2) structure formed by adsorption of 1/2 monolayer Na. The LEED results are shown to be in very good agreement with the results of ab initio calculations, with SEXAFS results, and with previous core level photoemission measurements. The structure, which can be best described as a surface alloy, contains two (2 × 2) Na layers interspersed by a (2 × 2) Al layer. Na atoms in the lower Na layer are adsorbed in substitutional sites on the first, reconstructed layer of the substrate.


9:40 am SS2-MoM5 Structure of NiO(111) Films on a Ni(100) Substrate, O. L. Warren and P. A. Thiel, Department of Chemistry and Ames Laboratory, Iowa State University, Ames, IA 50011 USA.

NiO can be grown on the (111) epitaxy on metallic Ni substrates. We have performed a full dynamical LEED structure analysis of such a film on Ni(100). The film is sufficiently thick and contiguous that metallic Ni is not probed by the electron beam. The oxide is oxygen-terminated with oxygen in fcc sites, and with a 15% contraction in the first interlayer spacing (relative to the bulk spacing). Dependent layers retain cubic symmetry while compressing to align uniaxially with the metallic substrate. Comparison with other work suggests that oxygen termination, strong contraction in the first interlayer spacing, and retention of cubic symmetry in deeper layers may be general features of metal oxide, rocksalt films with unreconstructed, (111) surface orientations.

10:00 am SS2-MoM6 Detailed Surface Structures of Ultrathin Films of Ice (H2O) and Xe (Fex) Grown on Pt(111), Determined by Automated Tensor LEED, M. A. Van Hove, A. B. Barbier, N. Mather, U. Starke, W. Weiss, M. Ritter and G.A. Somorjai, Lawrence Berkeley Laboratory, University of California, Berkeley, CA 94720, USA.

Ice was grown at 90 K by deposition of water vapor on Pt(111), until the Pt(111) LEED pattern could no longer be seen. Announcing to 140 K produced a moderately sharp LEED pattern, corresponding to a (1 × 1) termination of the oriented hexagonal (h) or cubic (c) bulk phases of ice. Fitting of LEED IV curves with automated tensor LEED favors the (0001) face of the hexagonal form (most common in nature) with minimal relaxations from the bulk structure and undetectable hydrogen atoms. The data are most satisfactorily explained by a full-bilayer termination in which the outermost half-bilayer of H2O molecules vibrates so strongly at 90 K as to become invisible to LEED.

Iron oxide was grown on Pt(111) by successive cycles of monolayer iron deposition and oxidation, until the Pt(111) LEED pattern could no longer be seen. The resulting moderately sharp LEED pattern corresponds to an unreconstructed (111) termination of bulk Fe3O4. With automated tensor LEED, 6 possible bulk terminations and many other models were tested. The favored structure having bulk termination with an exposed “1/4” monolayer of Fe atoms on a dense monolayer of O atoms. Strong relaxations of atomic positions perpendicular to the surface are found, up to about 0.5 Å from bulk positions: these are likely related to strong local elastic distortions.

10:20 am SS2-MoM7 Structural Studies of Quasicrystals, W.-B. Chin, C. J. Jenks, S.-L. Chang, C.-M. Zang, and P. A. Thiel, Department of Chemistry and Ames Laboratory, Iowa State University, Ames, IA 50011.

Quasicrystals are materials with long range aperiodic order and crystallographically forbidden rotational symmetries. Quasicrystals show great promise for use as protective coatings because of their hardness, low coefficient of friction, resistance to oxidation, and more environmentally friendly manner in which they can coat metals compared to standard chromium coatings. Although many studies have been conducted on the bulk properties of quasicrystals, little has been done to determine the surface properties of quasicrystals. We have investigated the surface properties of the five-fold surface of a “single crystal” of Al₆Pd₄Cu₆ in the presence of adsorbing low energy electron diffraction and thermal desorption spectroscopy. Our results show that oxygen affects the quasicrystal's surface structure while hydrogen does not. Implications for the resistance of quasicrystals to oxidation will be discussed.


A new type of UHV Atomic Force Microscope (AFM) which operates at variable temperatures (100K-500K) has been developed. The instrument has been used in the study of KMF₅ surfaces cleaned under UHV condition. It has been found that after cleavage, two different types of terminations, KF and MnF₂, coexist on the surface. One type of termination forms large domains (≥1000 Å), while the other appears as small islands, about 200 Å across. AFM images with atomic resolution taken on both of these two terminations show no lattice constant nor lattice orientation changes. It is hence proposed that on KF surface, because the ionic radius of K⁺ and F⁻ are very close, they are both imaged by the AFM tip, while on MnF₂ surface, since Mn²⁺ is much smaller than F⁻, only F⁻ is imaged. Friction images taken on both of these terminations indicate that friction force is different for the two surfaces with different chemical compositions.

11:00 am SS2-MoM9 Structure Determination of the InP(100) Surface, M. M. Sung, H. Bu, and J. W. Rabalais, Department of Chemistry, University of Houston, Houston, TX 77204-5641.
NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room BR1 - Session NS1-MoM

NANO 3 Plenary Session
Moderator: C. R. K. Marrian, Naval Research Laboratory

9:00 am NS1-MoM3 Molecular Recognition of Valence Bands (VOCs) with Self-Assembled Monolayers of Supramolecular Compounds, Wolfgang Göpel, Klaus-Dieter Schierbaum, Tito Weiss, and David N. Reinhoudt, Institute of Physical and Theoretical Chemistry, University of Tübingen, Auf der Morgenstelle 8, D-72076 Tübingen (FRG), Department of Organic Chemistry, MESA Research Institute, PO Box 217, 5000 AE Enschede (Netherlands).

Monolayers of resinoc[4]arenes on clean Au surfaces have been prepared by self-assembly through thiol bonds in ethanol/chloroform solutions. Angle-resolved photoelectron spectra give evidence that the monolayers are well-ordered and that resinoc[4]arene macrocycles determine the outermost surface. Their interaction with organic molecules like C6Cl4 was studied with quartz microbalance oscillators. It leads to a fast and reversible mass increase. The latter results from an incorporation of C6Cl4 within the molecular “cavities” formed by the resinoc[4]arenes. Comparative thermal desorption spectra indicate binding sites with high interaction energies of C6Cl4 molecules. These monolayers serve as a model supramolecular compound for SX/M studies which provide well-defined surface recognition structures for organic molecules.

11:20 am SS2-MoM10 Surface Relaxation of PbTe(100), C. B. Duke, A. Lazurides, A. Paton, Xerox Webster Research Center, Webster, NY 14580; A. Kahn, Princeton University, Princeton, NJ.

An analysis of surface core level shifts [G. Allen, Phys. Rev. B 43 (1990), 9594] from rock salt structure PbTe(100) suggests large contractions of the top layer of this material, in contradiction to expectations from other binary cubic semiconductors like MgO. To test this hypothesis we have performed a structure analysis on PbTe(100) using eight beams of diffracted low energy electrons (50 eV < E < 250 eV) from PbTe(100) at 100 K. An intensity analysis, based on relativistic potentials shown to be accurate for other fifth row elements in addition to Pb, leads to the conclusion that the Pb sublattice in the top layer is contracted by 0.24 Å (7%), whereas the Te species in the top layer is unrelaxed. A search for second layer relaxations did not improve the description of the measured intensities. An error analysis, performed using a new procedure yielding statistically significant errors, suggests a 95% confidence in this structure to within +/-0.08 Å for both layers. Thus, our analysis gives results analogous to those found for the (100) surfaces of other cubic materials rather than the large uniform top-layer contraction suggested by the analysis of the core-level shifts.


Among a wide variety of reconstructions being realized using molecular-beam epitaxy (MBE), the Ga-rich GaAs(001) surfaces has not been systematically studied, while the As-rich 2 × 4 reconstruction is extensively studied. By taking advantage of the migration enhanced epitaxy (MEE) technique, we have investigated the 4 × 2 and 4 × 6 phases using a high-performance MBE-STM instrument, which is a combination of a MBE and the FI-STM (field-ion scanning tunneling microscope).

We have found that the 4 × 2 structure is so-called “a bilayer model” with the unit structure of two Ga dimers and another Ga dimer in the third layer. In the high resolution STM images of the filled states, the Ga dimers in the first layer are observed as the bifurcated dim protrusions, while the dangling bond-derived filled states of the second layer As appears bright, much brighter than the first layer Ga dimers. By changing the As/Ga flux ratio in MEE growth or post annealing temperature, we have shown that the 4 × 6 phase is more Ga-rich than the 4 × 2 phase, which has been reported otherwise in some of the previous publications. In the case of the 4 × 6 filled states images, we observe the Ga dimers brighter than the As dangling bond-derived feature, which is explained by a charge transfer from the extra Ga atoms to the empty band localized at the dangling bond of the Ga dimers.

*Permanent address: Faculty of General Science, Osaka Prefecture University, Sakai 591, Japan.

10:00 am NS1-MoM6 Recent Progress on Magnetic Sensors with Monolayers and Applications, Yoshinobu Sugiyama, Electrotechnical Laboratory, 1-1-4, Umezono, Tsukuba 305, Japan.

A billion-per-year of semiconductor Hall devices and magnetoresistors are used for a displacement sensor of brushless motor in a video tape recorder and a disk driver, a banknote detector of vending machine and a contactless switch. The conventional magnetic sensor has a few hundred microns of active area and a micron thickness. Recently, a two-dimensional Hall device made of InAs/GaAs pseudo-morphic heterostructure semiconductors with high electron mobility of 16,000 cm²/Vs at 300 K and 160,000 cm²/Vs at 10 K has been developed. The new device has a very thin active layer of two-dimensional electron gas (2DEG), and the high device performance with a large dynamic sensitivity from nano tesla to a few tesla and a wide temperature range from cryogenic temperature to some hundred centigrade. A nanoscale of 2DEG Hall sensor with a very thin active layer of about 10 nm can be designed. The 1/f current noise decreases with decreasing the device dimension, that is the decrease of Hooge's parameter. The high signal-to-noise ratio given by the ratio of the large magnetic sensitivity to the small current noise is promising on the low dimensional microsensor. A magnetic image sensor made of one- or two-dimensional array integration of three-terminal Hall elements are very useful for an inspection of magnetic microdomain. The operating principle is based on the differential detection with one reference element integrated monolithically. The linear magnetic property is shown in this device, but a half sensitivity.

Another new type of magnetic sensor is based on galvanomagnetic effects in vacuum. The vacuum magnetic sensor (VMS) is made of three metal electrodes on the quartz substrate, that is a comb-shaped microstructure emitter, a high-field gate and a pair of splitted collectors. An electron emitted in vacuum by high electric field is deflected by an external magnetic field. The device has higher magnetic sensitivity of 1,000 %/T compared with semiconductor sensors.

INVITED

11:00 am NS1-MoM9 Quantum Corrals, D. M. Eigler, IBM Research Division, Almaden Research Center, 650 Harry Rd., San Jose, CA 95120.

Electrons occupying surface states on the closepacked faces of noble metals form a two-dimensional (2-d) nearly-free electron gas. Because this system is accessible to the scanning tunneling microscope (STM), it provides a unique opportunity to study the local properties of electrons in reduced dimensions. We have observed standing wave patterns in the surface local density of states due to the interference of 2-d surface state electrons scattering off of step edges and adsorbate atoms. Analysis of the energy dependence of the periodicity of the standing waves gives an independent measure of the surface state dispersion. We have found that Fe adatoms strongly scatter the surface state
Applied Surface Science
Room A101 – Session AS–MoM

Imaging and Small Area Analysis
Moderator: R. W. Linton, University of North Carolina, Chapel Hill.

8:20 am AS-MoM1 Static and Dynamic SIMS with Sub-Micron Resolution: Sensitivity and Quantification Aspects, Y. Gao, F. Salich, H.-N. Migeon, Laboratoire d’Analyse des Matériaux, CRP-CU 162a, avenue de la Falenciere, L-1511 Luxembourg.

The latest developments in SIMS instrumentation have made possible the analysis with lateral resolutions in the 100 to 200 nm range. Together with this resolution obtained by means of finely focused ion beams (Gallium, Cesium, Oxygen) the use of secondary optical systems allowing high transmission and high mass resolution makes useful SIMS analysis with good lateral resolution.

In this paper, we intend to review the capabilities for both static and dynamic SIMS applications in terms of ionisation efficiencies under different recording conditions, ionisation efficiencies of different elements and their influence on detection limits in microvolumes, image acquisition time. The figures of merit of double focusing magnetic sector and Time of Flight spectrometers will be outlined for static, dynamic and intermediate regimes.

If those performances can be predicted when dealing with SIMS measurements either in the static or in the dynamic regimes, the major limitation for the use of SIMS on laterally heterogeneous samples is related to the matrix effect. Some examples of such artefacts will be pointed out under different bombarding conditions. In order to minimise the matrix effect in dynamic SIMS we will concentrate on two alternatives, cationisation (MCs+) and SNMS, as well as laser positionisation for the static SIMS measurements.

9:00 am AS-MoM3 Surface Trimer Crystallization on Poly(Ethylene Terephthalate) Studied by Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS), S. Reichmuth1, S. R. Bryan2, and D. Briggs2, 1) Perkin-Elmer Physical Electronics Division, Eden Prairie, MN 55344, 2) ICI plc., Wilton Research Centre, Middlesbrough, Cleveland TS90 8JE.

Film poly (ethylene terephthalate) (PET) is used as substrate material in a great variety of applications, because of its excellent physical properties. Film strength is an important requirement for magnetic recording tapes as used in every VCR. Surface properties like adhesion and friction are also extremely crucial in this application. Under certain thermal treatments or exposure to some solvents PET is known to crystallize oligomeric material on the surface. This oligomeric material is dominated by the cyclic trimer. We investigated the generation and spatial distribution of the cyclic trimer on PET film samples. The film samples were heat treated in air, in vacuum at 117°C for different periods of time. Exact distributions of trimer crystals were observed for both materials even after only 1h of treatment. Domains with different characteristic were observed under an optical microscope and crystal size varied from 1µm to 50µm. The surfaces were then chemically characterized using TOF-SIMS. High mass resolution allows unambiguous assignments of the observed surface species and was used to verify previous observations using a quadrupole SIMS instrument1. The high sensitivity and transmission of the TOF analyzer also allowed chemical imaging of these surfaces. Retrospective image analysis was used to determine mass spectral differences between the PET substrate surface and the crystallize surface. Relative peak intensities from these areas were used to investigate the presence of non crystallized trimer in the areas between the crystals.


Surface properties like adhesion, wetting behaviour, friction and corrosion play a key role in many fields of technology. As these properties are determined by the molecular composition and structure in the uppermost monolayer an analytical technique is required which allows to localize, identify and quantify the molecules present in this layer.

We applied imaging TOF-SIMS to the characterization of structured SA monolayers (domain structure, monitoring of patterning processes), coated fibers (surface coverage, molecular distribution in two component systems) and defects in car paint (failure analysis). Although TOF-SIMS instruments equipped with liquid nitrogen-cooled ion guns meanwhile allow lateral resolutions of ≤ 100 nm we found that the useful lateral resolution for organic imaging in most cases is limited to 1 µm. This is mainly due to the static conditions required for organic SIMS (only the uppermost monolayer can be consumed if intact molecules have to be detected) and the generally low transformation probabilities of molecular species (P(M – X) ≤ 10–7 – 10–8). The results presented will, however, show that imaging TOF-SIMS nevertheless can offer most valuable information which is not accessible with other analytical techniques.


Self-assembly of organic monolayers (SAMs) via silane or sulfur anchor groups provides a versatile method of preparing bound thin films with well-defined surface chemistry. Variation of monolayer structure in the vertical direction can be obtained by assembly of multifunctional polymers onto surfaces. This study examines methods of preparing well-defined lateral patterns of surface chemistry. A series of 20 µm long lines varying in width from 0.2 to 2 µm were etched into SAMs of octadecane thiol and perfluoro siloxane polymers on Au with a 20 keV Ga+ focused ion beam. The octadecane thiol SAM was then exposed to perfluorobutane to etch the regions. The etched perfluoro siloxane was imaged without further treatment. The ToF SIMS images were obtained in the static mode (ion dose ≤ 1015) using a 25 keV Ga+ source. All lines were readily resolved on both samples. The perfluoro siloxane polymer was also self-assembled onto a SiO2 substrate with 2 µm wide Au lines. ToF SIMS images showed the SAM was only present on the Au lines. Full mass spectral analysis in the different regions of the patterned SAMs confirmed the surface structure of each region. This selected area analysis also provided information about contamination and oxidation processes occurring on these samples. These SIMS results show patterned SAMs can be prepared with submicron resolution. Additional characterization of the SAMs has been done with XPS, ellipsometry, contact angle, and FTIR.

10:00 am AS-MoM6 Scanning Photoelectron Microscope with Submicron Lateral Resolution Using Wolter-type X-ray Focusing Mirror, Ken Ninomiya, and Masaki Hasegawa, Central Research Laboratory, Hitachi, Ltd., Kokubunji, Tokyo 185, Japan.

Photoelectron microscopy is a powerful tool providing detailed chemical and electronic state information at high spatial resolution. We have recently improved lateral resolution of a scanning photoelectron microscope (SPM) in the soft X-ray beamline of the 2.4 GeV electron storage ring at the Photon Factory. The microscope is characterized by the use of a Wolter-type grazing incidence X-ray mirror for generating a focused soft X-ray microbeam.1 The mirror, fabricated by epoxy resin method2 in combination with vacuum application, has a demagnification of 1/29.8 and an X-ray reflectivity of 77% for 150 eV soft X-rays. When the microbeam (sagittal beam size: 1.8 µm) one-dimensionally scans aluminum stripe patterns formed on a SiO2 wafer, the 4 to 0.6 µm stripes are clearly detected using the total yield of photoelectrons with modulations between 0.33 and 0.08. In addition, the 16%-84% width of the photoelectron count rate change at the pattern edge gives a still higher resolution of 0.5 µm.
These results indicate that our SPEM has a lateral resolution better than 0.6 µm. The present study demonstrates photoelectron imaging at the highest lateral resolution that has ever been achieved using grazing incidence mirrors in the soft X-ray region. A submicron focused beam is also presented that will soon be used for higher-resolution photoelectron imaging. This work has been performed under the approval of the PAC of the Photon Factory (Proposal No. 93-Y003).


10:20 am AS-MoM9 First Results from the Spectro-Microscopy Undulator Photoemission Beamline, J. Denlinger, E. Rotenberg, A. Warwick, S. Kesan, and B. P. Toner, Advanced Light Source, Berkeley, CA 94720, Univ. of Oregon, Eugene, OR, Univ. of Wisconsin-Milwaukee, Milwaukee, WI.

The term "ultraESCA" is used to refer to a high performance photoemission spectroscopy system, which optimizes small area analysis, spectral resolution, atomic species detection sensitivity, and count rate. The implementation at the Advanced Light Source is based on a 5 cm period undulator and spherical grating monochromator with adaptive focusing optics, coupled with a high efficiency electron energy analyzer. The first results show simultaneous achievement of analysis areas below 50 micron, energy resolution below 0.2 eV, and count rates above 1 MHz. The tunable photon energy permits optimization of surface or near-surface sensitivity, and cross-section enhancement of dilute species by orders of magnitude in comparison to typical fixed-wavelength laboratory instruments. A spectrum showing surface core level shifts from Si(111)-7 x 7 is shown here, with a total collection time of 20 seconds. This corresponds to an enhancement of a factor of 800 over Al kα sources. Many applications will be discussed.


Traditionally the major uses of Auger electron spectroscopy (AES) have been in the analysis of conducting samples requiring high spatial resolution. The electron beam primary source with its relatively large dissipation of energy into the sample can lead to serious charging problems on insulating samples. The analysis of insulating samples requires the ability to use either very high (25 keV) or very low (3 keV) primary beam energies and the geometry of the instrument must allow a range of sample tilt angles to satisfy each individual sample requirement. The MICROLAB 310-F is the Fisons Instruments dedicated Auger microprobe with a Schottky field emission electron source which is capable of giving high spatial resolution even at low beam energies. It also has a co-planar geometry which allows a wide range of sample tilt angles to be used in orienting the sample to combat charging effects without compromising the sensitivity of the instrument.

Examples will be shown of the spectroscopy and mapping capabilities of this instrument on ceramics and insulating composite materials showing the analysis of sub-micron sized grains and describing the contribution this capability can make to solving surface related problems on insulating samples with small features.


A series of Roman bronzes of the 3rd century B.C. is studied with scanning Auger microscopy (SAM) and x-ray photoemission spectroscopy (XPS). The object is to characterize the chemical species present at the surface and to highlight their lateral distribution. Cu oxides, SnO2, PbO, PbCl2, and carbonates are the main corrosion products, and the surface composition is related to bulk quantitative analysis obtained with the electron microprobe and atomic emission spectrometry. A huge lateral homogeneity is found when Sn sputter-profiles obtained with XPS in large area (1 cm²) analysis are compared with those given by Auger point spectra recorded over an area 1 µm² large. The microchemistry of the materials is further explored using Auger imaging, which highlights Sn, Cu and O segregations with a spatial resolution of 0.5 µm. We show that SAM and XPS techniques feature unique advantages in the study of the surface microchemistry of archaeological objects, markedly as regards the direct identification of chemical species and the quantitation of light elements.


Micro-Raman spectroscopy has been used to study the local mechanical stress in micro fabricated structures on a silicon substrate. Large stress can give rise to defects in the silicon substrate and even to structural defects such as breaking of an oxide pad, which makes further processing of these structures impossible. Furthermore, mechanical stress has a strong influence on the electrical characteristics and the degradation behavior of metal-oxide-semiconductor (MOS) integrated circuits. In order to investigate the local stress, we used a Raman mapping system which combined a commercial high light throughput spectrometer with a cold CCD detector and a XYZ stepper stage.

We present the ability to image stress fields (2-D) using Raman microprobe. Images of the stress field of a Si wafer will be presented. The local stress field was obtained from the shift of 520 cm⁻¹ Si peak as calculated using a curve fitting program. A shift in the Raman peak, corresponding to a compressive strain, was found in regions where SOI was thermally grown on the Si wafer. Discussion of how resolution in relation to the standard deviation of the peak position as calculated by a Monte Carlo method will be presented. The resolution of our system is about 0.1 cm⁻¹. The total exposure time to generate a strain in 500 by 500 µm image consisting of 2500 data points (50 by 50) was 625 seconds.

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10:40 am AS-MoM8 Identification and Quantification of Hidden Features using Imaging XPS, J. E. Fulghum, L. Paleoudis and T. A. Zupp, Chemistry Department, Kent State University, Kent, OH 44242 and D. J. Surman, Kratos Analytical, 535 East Crescent Ave., Ramsey, NJ 07446.

XPS images are predominantly used to display changes in surface composition or to locate features for small area analysis. However, a more three-dimensional picture of the sample can be developed in some cases. Vertical and horizontal changes in composition contained within the XPS sampling depth can be quantitatively evaluated using XPS images from different sample components. Step heights, layer thicknesses, and feature sizes can be determined if changes in composition occur within the first ~10 nm in depth or within the image field of view (up to 10 mm²). Information which can be obtained will be demonstrated using both organic and inorganic samples containing buried layers and patterns. Quantitative information will be compared to line scans and small area analyses.

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PLASMA SCIENCE
Room A109 – Session PS–MoM

Plasma Etching & Deposition
Moderator: K. L. Seaward, Hewlett Packard Laboratories

8:20 am  PS-MoM1 Recent Developments in Plasma Processing,
Richard A. Gottscho, AT&T Bell Laboratories, Murray Hill, NJ 07974.

The purpose of this talk is to highlight advances presented at last year’s national symposium of the American Vacuum Society and those that will be presented during this week’s symposium. I will also venture slightly beyond this mandate to outline future directions for the plasma science and technology community to consider.

Last year’s meeting featured some healthy controversy and discussion on the relationships between ion and neutral transport, etching mechanisms, and that troublesome problem referred to as microloading, RIE lag, aspect ratio dependent etching etc; in my mind, seemingly contradictory results were reconciled by considering the competition between etching and deposition and their different dependencies on the ion to neutral flux ratio. I will review last year’s talks along these lines and shed new light on the subject.

In going forth, we are witnessing the advent of large area, high performance plasma processing. While Si wafer sizes are increasing to 300 and 400 mm diameters, flat panel displays are already made on 360 x 485 mm glass plates. These sizes place unprecedented demands on etching uniformity and throughput while linewidth control and selectivity demands also grow more stringent. As a result, the plasma processing and reactor design communities are being challenged as never before. I will highlight some of the approaches being taken in dealing with etching and deposition processes and where significant challenges remain.

9:00 am  PS-MoM3 Self-Aligned-Contact (SAC) Dry Etch Process for 0.5µ SRAM Technology, J. E. Nulty, P. S. Trammel, Cypress Semiconductor, San Jose, CA 95134.

Self-Aligned-Contact (SAC) technology is a preferred method to reduce cell size in CMOS SRAM technology while maintaining low manufacturing costs. Key to this technology is the SAC etch, which is a blanket etch-stop layer to protect underlying topography from oxide etch plasma during contact hole formation. We report a SAC etch technology utilizing BPTEOS as an oxide dielectric and CVD Nitride as a blanket etch stop. CVD Nitride is used extensively in existing technologies, demonstrates good step coverage in high aspect ratios, and is a good insulator for semiconductor devices. In the course of our development, we addressed the following key issues: ARDE effects, flat substrate nitride loss, and alignment-sensitive topography-corner nitride loss. Solutions to each issue will be discussed.

We will present both SEM and electrical test structure verification results for our work as applied to 0.5µ SRAM.


This talk will report the connection between the chemical predictions based on thermodynamics and the experimentally observed selective oxide etching in a high wall commercial HDP system. The relation between the protective carbon-based deposit on non-oxide surfaces (Si, TiN etc.) and the theoretically predicted selective deposition of carbon will be explored. While carbon is deposited on both Si and SiO2, the oxygen in the SiO2 leads to the selective removal of the carbon so that plasma etching of the oxide can continue. It will be shown that CO and the chamber/wafer temperature play a major role in this carbon deposition. The chemical role of H in increasing this deposition will also be examined. The model predicts that the HCO responsible for the carbon deposition can have many collisions with the SiO2 sidewalls but will only deposit on the non-oxide at the bottom of an opened contact. This is consistent with the experimental observation that increasing chamber temperature increases the protective deposition, but does not change the sidewall angle.


The kinetics of high selectivity of SiO2 etching achieved by inductively coupled plasma (ICP) employing CF4 + H2 mixture will be discussed. Selectivity depended strongly on the gap distance, d between a quartz plate on which a single turn antenna coupled with 15.56 MHz power was set, and a wafer stage biased by 1000 KHz power. This resulted from a shadow-like structure of ICP. Higher electron density in narrower gap of d < 50 mm dissociated HF products to produce F atoms which were responsible for the Si etching, while HF products were exhausted readily at the downstream diffusive region in wider gap of d > 75 mm, thus increasing the selectivity. The effect was confirmed by the result that selectivity increased with increasing flow rate. Scavenging of F atoms also resulted in deposition of carbon-rich polymer films on the Si surface which protected the Si surface from ion bombardment. Selective deposition of the films in contact holes with feature size less than 0.8 µm which led to infinite selectivity was observed. The SEM cross sectional pictures demonstrated that films covered uniformly whole wall surfaces of holes for 30% H2 addition to CF4, while films deposition was limited to opening areas of holes for CF4 alone. Since film thickness on hole bottom increased with increasing self bias voltage, resputtered side wall films was considered to accumulate on the bottom of holes. Appearance voltage mass analysis of radicals in the CF4/H2 plasma showed that CF4 and CF2 radicals were dominant in spite of with and without H2 addition to CF4. Above drastic change of step coverage of films on wall surfaces in the presence of 30% H2 may be ascribable to a reaction between introduction of CF radicals into hole and H atoms adsorbed on inner wall surfaces.


The dependance of selectivity on rf power

Tungsten (W) is widely used as wire materials instead of aluminum alloys or poly-Si due to its low resistivity, good step coverage and electromigration resistance. However, in W etching, it was difficult
to realize high selectivity to photoresist and anisotropic etching simultaneously. The electron cyclotron resonance (ECR) plasma is one of the suitable methods to realize highly selective etching. In this paper, we study W etching by the ECR plasma using SF$_6$/C$_F_4$ gas mixture. Samples used in this experiment were chemical vapor deposition (CVD) W films on TaIn films masked with photoresist pattern. Stage temperature and gas pressure was set at 30°C and at 0.26Pa, respectively. The selectivity to photoresist increases with decreasing of rf power. The highest value of selectivity is 7.9, which is obtained with applying no rf-bias. Furthermore, the selectivity increases with increasing magnetic field where the sample was located. The reason for this result is supposed that sheath potential decreases as magnetic field increases. Concerning etch profiles, in the case of etching using pure SF$_6$ gas, the side etching is observed in sidewall of W. This side etching is reduced by C$_F_4$ gas addition, and an anisotropic etching is achieved with addition of 36% C$_F_4$. Moreover, it is clear that the addition of C$_F_4$ reduce reactive ion etching (RIE) lag in W etching. The reason for the decrease of RIE lag is that the flux of deposition precursors, which is generated by C$_F_4$ decomposition, decreases due to low conductivity in high aspect ratio patterns.


A permanent magnet electron cyclotron resonance (ECR) plasma source has been redesigned to accommodate powers up to 5 kW and used with a solid metal sputter target for Cu deposition over 200-mm diameters. Sputter target current densities of ~40 mA/cm$^2$ have been measured at 1400 W, and the results of operation at higher powers, along with thin-film deposition rates, uniformities, and resistivities, will be reported. Measurements of the ion energy required for no net deposition have been used to deduce the ratio of Ar ions to all Cu species. At pressures ~4 mTorr and microwave powers ~1000 W, the ratio is ~1.5-3. The ratio of the Cu ion to neutral flux has been estimated using observations on the filling characteristics of substrates, 1:1 to 3:1 aspect ratio features combined with modeling.

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A new electron cyclotron resonance (ECR) plasma source for conductive film deposition has been developed. In this source, 2.45 GHz microwaves are divided into two directions and transported into the composer through quartz windows. The windows are arranged in the blind space from the ECR plasma and in a region where the magnetic field is higher than that of the ECR condition (875 Gauss). Two coils with different inner diameters are set for the generation of highly uniform plasma. High ion current densities above 10 mA/cm$^2$ and good uniformity of 4.5% over a 6-inch diameter area have been obtained. TiN films were deposited by ECR plasma deposition using a sputtering material supply. The ratio of Ti to N was about 0.9 at a N$_2$ partial pressure of 0.01 Pa, and a resistivity of only 25 $\mu$cm was obtained with Ar/N$_2$ plasma at 300°C. Advantageous features, good step coverage and filling to the bottom, were obtained by using a rotating inclined substrate holder. The target current was very stable for deposition over 100 hours. In applying this source to CVD, SiC films were deposited with high reliability using C$_2$H$_2$/SiH$_4$ plasma.

11:20 am PS-MoM10 Micro-Profile Simulations of Metal Ion and Neutral Deposition, S. Hamaguchi and S. M. Rossnagl, IBM, Thomas J. Watson Research Center, P.O. Box 218, Yorktown Heights, N.Y. 10598.

Numerical simulations in parallel with experimental depositions have been performed to model the deposition of metal neutrals and ions into trench structures. This type of topographical feature is commonly used for interconnect metallization in high performance integrated circuits. The work is intended to describe the deposition and etching dynamics occurring deposition from metal plasmas, such as in the case of metal ECR plasmas or Ionized Magnetron Sputter Deposition systems. The numerical model is based on the shock-tracking algorithms for a moving boundary and the etch/deposition rate is calculated from the fluxes of metal ions, metal neutrals and inert gas ions incident on the sample surface. The model is versatile enough to simulate semi-isotropic deposition of neutral metal atoms (conventional sputter deposition), deposition of angle-limited neutral metal atoms (collimated sputtering) and the deposition and etching dynamics of depositing metal ions (ionized magnetron sputter deposition or metal ECR plasma deposition), which may also cause local sputtering and redistribution at the sample surface. Combinations of these effects are also describable. Comparison with experimental depositions in each of these three areas shows good agreement. The model is used to optimize trench/via filling as a function of feature size and aspect ratio for the case of neutral and metal ion deposition at various flux ratios and energies.

11:40 am PS-MoM11 High Density Metal Plasma Formation During DC Magnetron Sputtering, W. M. Posadowski and Z. J. Radziszewski, Institute of Electron Technology, Warsaw University of Technology, Warsaw, Poland; Analytical Instrumentation Facility, North Carolina State University, Raleigh, NC 27609.

During a conventional DC magnetron sputtering with an inert gas, a target material is sputtered in form of neutrals and ions. Majority of sputtered species are neutrals which travelling through the plasma region above the magnetron source may be ionized. If high ionization is guaranteed then one may expect that a high ratio of target ions to neutrals can be achieved. With an electric potential across the dark space field in the order of few hundreds eV, the target ions will have enough energy to sputter target material. If a self-sputtering yield is high enough the process can transition without an inert gas into the self-sustained mode. A high ionization efficiency of target secondary species which has been achieved in the ETERNA$^\text{TM}$ 2.5, 4 and 8" diameter sources enables one to perform sputtering in self-sustained mode for a variety of materials at the chamber background pressure of 8 x 10$^{-6}$ Torr. During such sputtering, the target species are the primary component of plasma. It will be shown that, the plasma impedance, calculated as a ratio of target voltage to target current can be a good indicator of these threshold conditions for self-sustained sputtering, i.e. the formation of high density metal plasma. An interesting tendency can be observed when the plasma impedance is plotted vs. working pressure with target current as the parameter. The plasma impedance is almost independent of working pressure in a wide range from 10$^{-2}$ to 10$^{-6}$ Torr for materials with high sputtering yield. The results will be presented for various materials (e.g. stainless steel, Cu, Ag, Ta, W) using the ETERNA$^\text{TM}$ sources.
VACUUM TECHNOLOGY
Room A102 – Session VT-MoM

Total Pressure Gauging
Moderator: N. T. Peacock, HPS Division of MKS Instruments Inc.


The axial emission gauge (AEG) is an ionization gauge which is used to measure pressure in the ultra high vacuum (UHV) region. The gauge is similar in construction to a Bayard Alpert gauge (BAG), except that the filament and ion collector are located at the two ends, on the symmetry axis of the accelerating grid. As with a BAG, the sensitivity of an AEG is also proportional to the path length within the gauge, which is determined by the electron field present within the gauge. To be able to improve the gauge sensitivity, we have developed a computer model of the AEG to study the trajectories and calculate the total path length of the electrons. Using axial symmetry finite element topography, the potential field used to accelerate the electrons has been calculated by solving the Laplace equation using appropriate boundary conditions. The same topography is then used to compute the electron trajectories and path lengths for both a predetermined direction of electrons and random electron emissions whose speeds have been determined using Monte Carlo techniques assuming a Maxwell velocity distribution. The mathematical model indicates that it is possible to find maximum path length by varying gauge parameters.


We have been studying a Lafferty gauge for extreme high vacuum pressure measurement. The Lafferty gauge has high sensitivity due to electron trapping by applied magnetic field. We found experimentally an electron trapping phenomenon. Ion current of the Lafferty gauge came to zero several minutes after filament power was OFF. The several minutes “life time” of ion current means that electron trapping phenomenon was occurred by magnetic field within the Lafferty gauge. The life time of ion current depended on a magnitude of magnetic field and a field-to-anode voltage. We observed that the longest life time obtained was five minutes on the condition that the field-to-anode voltage: 800 volts, and magnetic field: 400 gauss, and emission: 1.0 mA. Pressure, when the life time measurement was performed, was on the order of 10^{-10} Pa. The life time of ion current has close relationship with the electron storage mechanism within the anode and the electron scattering mechanism to the anode. Therefore experimental results of the life time measurement and theoretical sensitivity analysis by calculating electron’s motion within the gauge including electron-electron scattering, electron-molecule scattering will enable us to realize the sensitivity of the Lafferty gauge.


9:00 am VT-MoM3 The Influence of Filament-Heating Waveform on the Sensitivity of Glass Envelope Bayard-Alpert Gages, P. J. Abbott and J. P. Looney, Thermophysics Division, NIST, Gaithersburg, MD 20899.

Non-linearities of about ten to fifteen percent in the sensitivity of glass enveloped Bayard-Alpert (BA) gages have been observed in the pressure range 10^{-12} to 10^{-5} Pa which are due to a time-dependent potential on the inner glass surface of the gage tube which arises as a result of the use of charged AC filament heating waveforms. These non-linearities were studied using modified BA gage tubes with platinum coatings on their inner glass surfaces and measuring the electron-impact ionization potential of the platinum coating as a function of pressure and waveform characteristics. The sensitivities of the gage systems (gage tube plus controller) were found to depend on the inner surface potential, and this potential was found to depend on the pressure and on the details of the filament heating waveform. It was found that the non-linearities could be minimized by holding the inner surface potential to a fixed DC potential, by modifying the AC filament-heating waveform, or by using a controller that provides a noise free DC filament-heating current.


Over the past 10 years, the Vacuum Standards Laboratory at NIST has performed more than 165 Bayard-Alpert ionization gauge calibrations for a wide variety of industrial and government laboratories. For 16 gauge “tube”/controller combinations the calibration has been carried out more than once, with time intervals typically 1 or 2 years between the repeat calibrations. ThO2/Ir-filament and tungsten-filament gauges of both the nude and glass-tubulated type are included in this group. These calibration results have been analyzed for a given gauge “tube”/controller combination differ by 5% or less from the previous results. In a few cases, this difference is as large as 10%.


In many low pressure industrial applications, there is a need for low cost pressure sensors. Micromechanical pressure sensors offer several advantages over conventional sensing devices, including high sensitivity, wide dynamic range, auto calibration, and self-test. Differential capacitive pressure sensors fabricated from thin silicon membranes can resolve pressures on the order of 1 mTorr, while covering a dynamic range of 6 orders of magnitude. These devices are inherently rugged and can withstand overpressures greater than 1 ATM. Furthermore, unique readout schemes have been developed for this transducer to implement advanced features such as auto-calibration and self-test. Electrostatic signals that simulate pressure impulses are generated in the electronics; these calibrated signals are used as self-test or auto-calibration signals. These devices are fabricated using a novel silicon-on-glass technology. For vacuum applications in the sub-mm and sub-mm Torr operating regions, silicon resonant devices are being used as absolute pressure sensors. The operation of these devices is dependent on the damping of the ambient and are very sensitive to small pressure changes. They are currently being employed as leak detectors for measuring rates much less than standard instrumentation (10^{-14} SCCM).

10:20 am VT-MoM7 Vacuum Gauging with IC Technology Compatible Microsensors, O. Paul, O. Brand, R. Lenggenhager, H. Baltes, Physical Electronics Laboratory, ETH, CH-8093 Zurich, Switzerland.

Micromachined silicon devices used for the measurement of vacuum pressures have a number of advantages such as small size and low cost, and the possibility of batch fabrication within the same fabrication cycle and integration with other signal-conditioning circuits. Such a perspective however requires the compatibility of the devices with commercial IC processes. We have realized thermal vacuum sensors fabricated with the commercial 1.2 µm CMOS process of AMS (Austria Mikro Systeme, Unterpremstaetten, A) and post-processed at our laboratory in two ways. A first category of structures was obtained by silicon bulk micromachining with EDP solution, leaving silicon dioxide cantilevers of physical dimensions 200 × 200 µm². The power dissipated by an integrated polysilicon heater leads to a pressure dependent temperature increase of the device which in turn is measured by integrated thermopiles located on the heater beam itself or on a second beam opposite to it. The range of maximum sensitivity is between 10 and 100 Pa. A second category of structures was obtained by local and selective removal of the lower metalization layer of the CMOS process, creating a 0.6 µm thin air gap between a heated membrane and the silicon substrate. Due to the small size of the gap, the sensitivity of these structures is maximum between 10⁴ and 10⁵ Pa. A third type of devices uses the pressure dependence of the resonant behavior of thermally driven microstructures. The resonators were realized by the anisotropic etching of silicon. Their resonance frequency decreases linearly between 10⁵ and 10⁶ Pa, whereas their quality factor shows a strong decrease between 1 and 10⁶ Hz. By combining the above principles, seven decades in pressure can in principle be covered by a single silicon chip of a few mm².
A new miniaturized silicon based, Pirani type thermal vacuum sensor that detects pressure from 760 Torr to 1 x 10^-7 Torr has been developed. The compact instrument is attitude insensitive, rugged and has rapid response to pressure variations. The small passageways prevent the flow from entering into the viscous flow regime thus resulting in predominately conductive heat transfer over the entire pressure range. This removes the complexities introduced into the pressure versus output curve by convective heat transfer. The output is linear from 1 x 10^-4 up to 0.1 Torr and monotonically increases to 760 Torr.
A unique feature of this instrument is the use of a microprocessor to compute power from the analog voltage and current. In the molecular flow regime the absolute pressure is directly proportional to the power dissipated by the surrounding gas. By measuring the power the sensor rejects most of the errors introduced by ambient temperature variations. The analog to digital converters for both the current and the voltage signals use the S-D conversion method to reject electrical noise by averaging technique. This results in stable signal detection of pressure down to 1 x 10^-7 Torr.
The instrument is thermally stable over an ambient temperature range of 0-50°C when the pressure is between 10^-4 and 760 Torr.

11:00 am VT-MoM9 Long-Term Calibration Stability of Molecular Drag Gage Rotors, R. W. Hyland, and J. P. Looney, Thermophysics Division, NIST, Gaithersburg, MD 20899.
The NIST has been keeping historical records of calibrations of molecular drag gage (MDG) rotors for about 10 years. Over 250 individual calibrations involving over 105 customer rotors, as well as numerous calibrations of NIST-owned rotors, have been performed. These records will be reviewed to determine patterns of long-term stability and ranges of parameters which can be expected with use and handling of the rotors.

Pressure measurements from 10^-7 to 10^-3 Pa were performed by an optical method. In this method, a radiofrequency electrodeless discharge is initiated in a small glass chamber and discharge radiation is sensed by a photosensitive element. The radiation intensity was found to increase initially with an increasing pressure but decrease above a certain pressure threshold. Thus two pressure values are often associated with each radiation intensity reading. However, it was also found that the dc current passing through the radiofrequency oscillator, which is related to the loss in the electric discharge, could be used to identify if the radiation data are taken below or above the "deflection point" pressure in the calibration curve of radiation vs. pressure.
Hence, simultaneous measurements of the radiation intensity and dc oscillator current always give a unique pressure. This optical contactless method is particularly useful for the determination of pressure in chemically aggressive environments, and for automatic pressure stabilization in a radiofrequency plasma system.

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11:40 am VT-MoM11 Validity of the Total Pressure of 5 x 10^-11 Pa Estimated by Laser Ionization Method, S. Ichimura, S. Sekine, and K. Kokubun, Electrotechnical Laboratory, 1-1-4, Umezono, Tsukuba 305, Japan.
Laser ionization method is one of the promising technique for total pressure measurement in extreme high vacuum (XHV) ranges. Using second harmonics of a pico-second YAG laser, we estimated an ultimate pressure in an XHV chamber. Above 7 x 10^-10 Pa (H2 equivalent), which was a detection limit of an ionization (extractor) gage used in this experiment, the number of detected ions changed linearly with pressure. Only ion detection rate decreased further to a minimum level of one count during 500 laser shots. Pressure corresponds to the count rate was estimated by extrapolation to be 5 x 10^-11 Pa.
To verify the validity of the estimated pressure, the size of the ionization region was measured under same experimental condition of incident laser energy (30 mJ/pulse) and focussing with a spherical lens (focal length; 250 mm). An ion imaging detector was used for the measurement. The size of the ionization region having a spindle shape was about 60 μm (maximum diameter) × 2.5 mm (length) at a pressure of 2 x 10^-7 Pa. It was shown that the volume of the ionization region was constant when the product of pressure in the XHV chamber and the number of video images used for the estimation was kept constant. The volume is roughly 2 x 10^-3 mm^3. This small volume supports the fact that we could count only one ion during 500 laser shots, since molecular density at 5 x 10^-11 Pa is about 5 molecules/mm^3.


ELECTRONIC MATERIALS
Room A108 – Session EM-MoM

Materials for Device Integration
Moderator: L. L. Tedder, North Carolina State University.

8:20 am EM-MoM1 Performance and Integration of Copper VLSI/ULSI Interconnects, D. C. Edelstein, IBM T. J. Watson Research Center, Yorktown Heights, NY 10598.
It has long been proposed that on-chip interconnection systems should eventually migrate to copper/low dielectric constant insulators for improved performance. However, advances in CMOS circuit integration density, microprocessor architecture, and custom layout currently afford geometrical performance improvements as compared to more incremental cycle time enhancements predicted from the change in materials. On the other hand, evidence will be presented that migration to copper dual-dama-scene (combined line/stud metal fill and chem-mech polish) interconnects may offer incremental improvements not only in performance but also cost, yield, chip power consumption, and reliability-related impacts. Furthermore, the currently-practiced copper interconnect process may be more extendable to deep-submicron ULSI groundrules and high aspect ratios than aluminum-based technologies. This talk will present data to support the above statements from detailed electrical modeling, high-speed and other electrical measurements, and process integration and materials issues associated with the first successful demonstration of fully-integrated multilevel copper submicron interconnects. INVITED

Cross-section of 4-level copper polyimide VLSI interconnects.

9:00 am EM-MoM3 Optimization of a Contact Metallization Scheme for 0.35 μm ASIC Technology, Sabhas Bothra and Teresa Trowbridge, VLSI Technology Inc., San Jose, CA 95131.
The performance of a contact metallization scheme developed for a 0.35 μm ASIC technology will be presented. The main features of the contact structure are a reactive sputtered TaN glue layer and CVD W plug for contact fill. A DOE approach was used to optimise the contacting layer and process for a low contact resistance with an adequate thermal stability. Since TaN does not form a low contact resistance to p+ and n+ silicon, two silicide contact layers, namely,
PSi and TiSi were investigated. The PtSi contacting layer results in a low contact resistance to both n- and p-type silicon, while TiSi shows a higher contact resistance to p-type silicon. The thicknesses to p-Si on contact diameter were shown below. The barrier properties of the PtSi/TiN and TiSi/TiN were compared by annealing the wafers after subsequent deposition of AlCu alloy metal and patterning. The TiSi/TiN barrier layer was found to be superior and necessary to achieve an adequate barrier over the worst case contact aspect ratio. Further, use of a W plug improved the barrier stability, along with a small lowering in contact resistance. DOE results on the effects of contact sputter etch RTF TiSi formation, TiN barrier thickness, tungsten deposition conditions and stack combinations will be presented.


Titanium disilicide as a refractory metal silicide is ideal for use as a metallization material in VLSI technology, primarily due to its low resistivity, high temperature stability and its compatibility with standard processing techniques. However, the presence of oxygen in the starting films can adversely effect the formation of a uniform and homogeneous silicide. In this paper we describe the results of an extensive experimental study where Ti (300–500 Å) and Si (700–1200 Å) films have been sputter deposited on Si and SiO2 substrates by physical vapor deposition (PVD). Titanium disilicide was formed by use of furnace anneal (FA), rapid thermal anneal (RTA) and in situ high vacuum (UHV) anneal at various temperatures ranging from 400°C to 700°C. The formation of silicide, surface and interface reaction were monitored by use of AES, XPS and RBS. Oxygen and other contaminants in the sputter deposited Si/Ti films were measured by SIMS. Sheet resistance measurements were made by the four-point probe method. The results from FA and RTA thermal treatments indicate that for the amorphous-Si (a-Si) films with O levels above 10^{14} atoms/cm² the Si/Ti reaction did not go to completion due to formation of an SiO interface layer between unreacted a-Si and newly formed TiSi films. These films gave high sheet resistance and had a blue discoloration. Same films when annealed in UHV successfully formed TiSi, with evolution of 0 and SiO into UHV ambient. The paper will further discuss the formation of surface and interface reaction products as TiSi is formed on Si and SiO2 substrates.


Single crystal bcc W layers, 140 nm thick, were grown on Mg(001) substrates by ultra-high-vacuum (UHV) magnetron sputtering at T_S = 600°C. 190-nm-thick Al films, with width (001) preferred orientation and an average grain size of 350 nm, were then deposited at T_A = 100°C without breaking vacuum. Changes in bilayer resistivity during UHV annealing were monitored continuously as a function of temperature T during temperature-ramping and as a function of time t during isothermal annealing. In addition, Rutherford backscattering spectroscopy, x-ray diffraction, transmission line microscopy (TEM), and scanning TEM in which cross-sectional specimens were analyzed by energy-dispersive x-ray analysis with a 1 nm resolution were used to follow area-averaged and local interfacial reaction paths as well as microstructural changes as a function of annealing conditions. The initial reaction products were discontinuous regions of monoclinic structure, W14Al, with the W14Al (240) planes frequently oriented perpendicular to the W interface with a very close lattice match, within 0.5%, to the underlying W (110) planes, bcc W14Al forms at a later stage and grows conformally to cover both W and W14Al. The W14Al and the W14Al phases continue to grow until the Al layer is completely consumed. Information from microchemistry and microstructural analyses was used to model the (0, t) results, based upon a novel multi-element equivalent circuit approach which accounts for the observed non-planar nature of the reaction front, and determine reaction kinetics and activation energies. The results show that the growth of W14Al is diffusion limited with an activation energy E_A of 3.1 eV while the formation of W14Al phases is reaction limited with E_A = 3.3 eV.


High quality chemical vapor deposited (CVD) TiCN films were produced in a single wafer reactor using a metallorganic (TDMAT) precursor. The films have excellent step coverage (>75%) over high aspect-ratio contacts as well as very low particle content. These properties are obtained because the films were deposited under surface reaction controlled conditions; the measured activation energy is 0.9 eV. The stress levels of the films are relatively low, below 5 x 10^5 dyne/cm². The films show also excellent barrier properties against Al and W attack, which are attributed to their amorphous composition, to the high C content of the films, and to the high step coverage. The electrical properties of the CVD TiCN films were evaluated at the via level, and the resistance was shown to be comparable to that of sputtered TiN. These properties make this material a superb barrier material for contact and via applications in ULSI devices.

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1. EVOLVE is a low pressure deposition process simulator developed by T. S. Cale at ASU and Motorola, Inc. with funding from the SRC and NSF.


By combining the low (~2.0) dielectric constant poly(tetrafluoroethylene) (PTFE or Teflon®) with conventional integrated-circuit processing techniques, we have developed processes for the fabrication of fine circuit features directly on PTFE substrates.
Metal features are defined using a lift-off technology, electroless plating, or a combination of electroless and electroplating. The most versatile technique has achieved 4 μm-thick metal features with high lateral resolution (<20 μm, a factor of five smaller than existing printed wiring board-based technology). These processes result in a less expensive process to pattern adherent, conducting metal lines on an etched PTFE substrate. Adhesion of the metal features depends upon successful substrate preparation by etching the PTFE in a sodium naphthalenide solution. Using the processes discussed above, coupled-line quadrature (Lange) couplers that demonstrate equal power splitting between 5 and 14 GHz have been fabricated directly on a PTFE substrate. Lange couplers have extensive applications as passive hybrid device components in microwave and radio-frequency (RF) systems. Details of the developed process sequences and preliminary test results for our Lange couplers on PTFE will be presented. This work demonstrates the utility of PTFE in challenging high frequency device applications where low dielectric constant substrates are essential. This work was performed at Sandia National Laboratories and supported by the U.S. Dept. of Energy under contract DE-AC04-94AL85000.

11:00 am  EM-MoM9  Processing and Device Issues of High Permittivity Materials for Dram Memories, B. E. Gnae, Materials Science Laboratory, Texas Instruments, Dallas, TX 75265.

The development of high permittivity materials for capacitor dielectrics will significantly reduce the cell topography and complexity anticipated for ≥ 256 Mbit DRAMs using current dielectric technology. While cell architectures could be simplified with the introduction of high permittivity materials, there are many materials, processing and device issues which need to be resolved before high permittivity materials will be realized in a ULSI DRAM. This presentation will discuss manufacturing and device requirements which need to be met in order for high permittivity materials to replace SiO₂ and Ta₂O₅ in high density DRAMs.

11:40 am  EM-MoM10  PECVD TiO₂ In Microwave-RF Hybrid Plasma Reactor, Young H. Lee, Michael J. Brady and Kevin K. Chan, IBM T. J. Watson Research Center, Yorktown Heights, NY 10598.

Plasma-enhanced chemical vapor deposition (PECVD) tool and process have been developed for TiO₂ thin films of a high dielectric constant for DRAM storage capacitors. Microwave (2.45 GHz) generates a highly dense plasma (∼ 10¹³ electrons/cm³) through electron cyclotron resonance (ECR) and a RF hollow cathode made of a high-purity titanium sheet for electron confinement in the ECR chamber. Additional RF (13.56 MHz) was applied to the substrate electrode to extract low energy ions (<100 eV) toward a Si substrate during film deposition. Our deposition process includes (1) removal of the Si native oxide in He plasma, (2) regrowth of an ultra thin (<1 nm) SiO₂ buffer layer in O₂/He plasma, and (3) deposition of a 30 nm thick rutile TiO₂ in TiCl₄/0₂/He plasmas. All three process steps were performed near 1 mTorr and monitored by the emission spectroscopy (OES) residual gas mass/energy analyzer (RGA) and 'in situ' ellipsometer. The SiO₂ buffer layer is essential to prevent diffusion of Ti into Si and consequently improves the leakage current below 10⁻¹⁰ A/cm² at the bias of ±2 V with a 'good' thermal stability at 1000°C. X-ray diffraction shows the anatase phase at a low deposition temperature below 200°C but dominantly the rutile phase at higher temperatures. The TiO₂ films deposited at 400°C show the dielectric constant of 60(±5) at 1 MHz, independent of a film thickness (20~200 nm). Low energy ion bombardment has improved the quality and thickness uniformity of TiO₂ films over 125 mm Si wafers.
Monday Afternoon, October 24, 1994

SURFACE SCIENCE
Room A205 – Session SS1-MoA

Surface Mechanisms and Materials for Chemical Sensors
Moderator: R. M. Penner, University of California, Irvine.

2:00 pm SS1-MoA1 Surface Chemistry of Gold Doped WO3 Hydrogen Sulfide Sensors, Bernd Fruhberger and Daniel J. Dwyer, Laboratory for Surface Science and Technology, University of Maine, Orono, ME 04469-5764.

Gas sensors that detect H2S in the ppm range have been developed based on the fact that thin WO3 films doped with gold exhibit resistivity changes upon exposure to H2S. In an attempt to understand the chemical mechanism by which these sensors operate, we have explored the interaction of H2S with a variety of relevant surfaces. These surfaces include: clean Au single crystal surfaces, Au polycrystals, WO3 thin films and Au doped WO3 thin films. These studies used a variety of ultra-high vacuum surface science techniques including X-ray Photoelectron Spectroscopy (XPS), Low Energy Electron Diffraction (LEED), Temperature Programmed Desorption (TPD) and High Resolution Electron Energy Loss Spectroscopy (HREELS). Data obtained from these surface chemistry studies has led us to suggest a chemical mechanism for the sensor. This mechanism involves dissociative adsorption of H2S on the surface of the Au dopant followed by the chemical reduction of the WO3 substrate by a spillover mechanism. Oxygen vacancies produced during the chemical reduction of the WO3 films induce n-type conductivity increases in the films. It is this conductivity increase that triggers the sensor response. The conductivity increase in the sensors is a dynamic process in which the vacancies produced by the reaction with WO3 are re-oxidized by atmospheric oxygen. It is the balance between creation and re-oxidation of the vacancies which defines the dynamic range of the sensors.


Titanium-substituted titanium oxides, in the single phase region at low titanium content (compositions around $Cr_2Ti_{0.9}O_3-x$), act as gas sensitive resistors at elevated temperature with high sensitivity towards H2S. Upon first exposure to ppm levels of H2S in air the materials exhibit an increased baseline resistance. This pretreatment appeared permanent below about 720 K. The surface modification of the material following exposure to H2S was studied by x-ray photoelectron spectroscopy (XPS) and temperature programmed desorption (TPD). The S 2p region indicated a change in the spike of surface sulfur during pre-treatment from, initially, sulfide and elemental sulfur, to sulfate at the completion of the pretreatment, as judged by the effects on the baseline resistance. Vacuum TPD studies of $Cr_2Ti_{0.9}O_3-x$ following H2S exposure at room temperature showed H2S and SO2 desorption peaks at 720 K. This corresponded to the temperature at which the effect of pretreatment was lost. Subsequent exposures to H2S did not reproduce the 720 K SO2 desorption peak unless O2 was co-adsorbed. It was concluded H2S reacted with chemisorbed oxygen during pre-treatment to form sulfur species which resulted in a reduction of electron acceptor states and a decrease in conductivity for this p-type material. The mechanism of response of $Cr_2Ti_{0.9}O_3-x$ was explained in terms of specific surface sites identified by XPS and desorption studies.


The increasing need for sensors that can perform reliable compositional analyses for environmental monitoring, industrial process control, personal safety and a variety of other applications has elevated the level of interest in chemical sensing considerably in recent years. Improvements in the performance characteristics of future generations of chemical sensors can be expected to relate closely to research efforts aimed at understanding transduction mechanisms, exploring new detection schemes and developing and incorporating improved planar materials. After providing a general overview on sensing concepts, we illustrate the importance of surface chemical, structural and electronic characterization in the development of advanced, conductometric microsensor arrays for analyses of gas mixtures. Among the specific topics to be discussed are electron transport properties in crystalline and polycrystalline semiconducting oxides (including ultrathin films), metal/oxide interface formation, approaches used to impart selectivity and microstructure/performance issues. We describe both the challenges encountered and major opportunities realized when active sensing films are combined with micromachined Si array configurations with on-chip circuitry, and also indicate the roles of controlled thermal manipulation of chemisorption phenomena (static and dynamic—i.e., 10⁻¹⁰ sec) and neural networks in attaining optimized performance. Finally, the reciprocal nature of research benefits that can occur between surface science and the evolving field of sensor science are discussed.

3:20 pm SS1-MoA5 Operation of Thin Film Gas Detectors in a Temperature Programmed Mode, R. M. Merchant, W. C. Schwank, J. L. Gundl, K. R. Wise, Chemical Engineering Department, Electrical Engineering and Computer Science Department, University of Michigan, Ann Arbor, MI 48109-2136.

The response of a thin film conductivity based gas detector to aromatic hydrocarbons is monitored by using the detector in both a temperature-programmed desorption and a temperature-programmed reaction mode. The detector structure consists of a Pt/TiO2 sensing film supported by a 1 mm² micromachined dielectric window. A boron-diffused silicon heater beneath the window allows the window temperature to be varied between ambient and 1,000°C. The thin film resistance is measured using Pt/Ti electrodes arranged in a four point probe configuration.

Experimental data of resistance changes induced by adsorption and temperature programmed desorption of aromatic hydrocarbons such as benzene and toluene in UHV will be presented. Use of the detector in a temperature programmed reaction mode will also be discussed. In this mode, the adsorbed species will show a characteristic reaction temperature with a typical “cleaning gas” such as oxygen, giving additional information about the nature of adsorbed species. Results from characterization of the active sensing film using prototypical 1 cm² samples of the active film supported on a silica substrate will also be discussed. The prototypical sensing films are used for ex situ XPS and correlation of temperature programmed desorption to the gas phase with changes in thin film resistance during desorption.


Catalytic-metal-gated field effect devices, such as Pd-MOS capacitors and transistors, can detect small partial pressures of hydrogen in UHV. Their sensitivity and time response are strongly affected by surface impurities and substrate temperature. To help understand how these parameters affect the kinetics of sensor response, we have studied the behavior of Pd-MOS hydrogen sensors in UHV to short pulses (1–200 sec) of H2 gas at pressures in the range of 10–300 µPa as a function of surface condition and temperature. Sputter cleaning of sensor surfaces improves response and recovery times, evidently by removing impurities that impede H2 dissociation and recombination at 300 K. Hydrogen accumulation at the sensing point giving a saturable response initially related to the total gas fluence. At 450 K the hydrogen concentration at the internal interface is more representative of the external partial pressure of hydrogen. These results are interpreted using a model in which hydrogen equilibrates among the internal interface, the metal film, and the internal sensing junction.

This work was supported by the US Department of Energy under contract DE-AC04-94AL85000.

4:00 pm SS1-MoA7 Structure and Chemical Sensing Properties of Vanadium Oxide-Sol-Gel Films, J. S. Ledford, P. A. Askeland, Department of Chemistry and the Center for Fundamental Materials Research, Michigan State University, East Lansing, MI 48823.

The structure of vanadium oxide films prepared by hydrolysis of vanadium isopropoxide oxide has been examined using Fourier trans-
form infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS). Vanadium centers in the bulk of the film are partially reduced and remain in the form of vanadyl ligands. The FTIR analysis shows that the surface of the polymer film is hydroxylated, but contains no alkoxide ligands. Mass spectrometer results for a series of normal alcohols indicate that the vanadium oxide film has molecular sieving properties which lead to the exclusion of alcohols larger than n-propanol from the pores of the film. Film/analyte interactions monitored by FTIR-attenuated total reflection spectroscopy (ATR-FTIR) revealed the formation of water at the vanadium oxide film unpaired electrons of alcohols (C2 and higher) in air and nitrogen has been explained in terms of changes in the proton conductivity of the film that arise from water generated by alcohol dehydrogenation reactions. For methanol, the conductometric response is attributed to reduction of the vanadium oxide lattice. The extent of reduction of the vanadium oxide film in air and nitrogen atmospheres leads to differences in the magnitude of methanol response.


Surface electronic structure of tin oxides (Sn and SnO2) is an important subject of study from point of view of better understanding of their mechanisms in gas sensing and catalytic actions. In order to obtain information on local electronic and geometric structure, around the core-ionized atoms, high resolution core and valence band photoemission as well as photoinduced Auger measurements of Sn metal, SnO and SnO2 samples were performed. SnO surfaces were obtained without the use of ion sputtering by using a special preparation procedure based on in situ surface cleaning. Comparing the valence band spectra to the theoretical spectra calculated by a cluster MO DVX model, unambiguous information was gained on the local geometries. From the Sn core photoemission and Auger measurements it was found, in agreement with the estimation using a simple model, that the initial state effects are the same for SnO and SnO2. This was supported by the chemical state plot as well, indicating the magnitude of the initial state parameters. The oxygen K-Auger parameter shifts show that SnO2 belongs to the class of oxides, hydroxides and metallic anions and indicate larger polarisability, similar to that of NiO. In summary, the use of the Auger parameter approach for obtaining information on initial state effects, polarisability and final state hole repulsion energies has been demonstrated for the case of tin oxides.

4:40 pm SS1-MoA9 Growth and Reactivity of Thin TiO Films on W(110), G. S. Herman and C. H. F. Peden, Pacific Northwest Laboratory, Richland, WA 99352.

The growth and reactivity of thin TiO films, on W(110) have been studied using x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), ion scattering spectroscopy (ISS), low energy electron diffraction (LEED), and temperature programmed desorption (TPD). Growth at different oxygen partial pressures in the 1 x 10^-9 to 1 x 10^-6 range and at different substrate temperatures in the 300 K to 1200 K range resulted in films with varying stoichiometry, and order. Final film stoichiometry was determined by AES and cross-calibrated against Ti 2p/0 Is photoemission intensity ratios. These two techniques also provide Ti oxidation state information. Initial oxidation of the Ti films at 700 K and oxygen partial pressure of 2.5 x 10^-7 Torr resulted in stable TiO Films grown at room temperature, followed by an annealing cycle at 900-1500 K possess long range crystallographic order, as observed by LEED. However, at annealing temperatures above 1500 K tungsten AES and ISS signals can be detected, indicative of the formation of islands. In addition, TPD data indicate that the TiO, films desorb at approximately 1800 K. Finally, the reactivity of these films were investigated using several different probe molecules including D2O and CH3OH.

5:00 pm SS1-MoA10 Synthesis of Ordered Ultra-thin Al2O3 Films on Ru(0001) and Re(0001) Surfaces. Tiantong Wu, Eric Garfinkel and Theodore E. Madey, Departments of Chemistry and Physics, and Laboratory for Surface Modification, Rutgers, the State University of New Jersey, P.O. Box 849, Piscataway, NJ 08855-0849.

Ultra-thin Al2O3 films (about 15-20 Å thick) are synthesized by two different methods on Ru(0001) and Re(0001) surfaces and monitored by XPS, LEIS and LEED. This work is part of an effort to synthesize ultra-thin crystalline films of insulating oxides on conducting substrates. On Ru(0001) post-oxidation of deposited Al films generates Al2O3 films that do not have ordered LEED patterns. When Al is deposited in 1 x 10^-3 Torr oxygen at elevated substrate temperatures (~900°C) the resultant films have hexagonal LEED patterns characteristic of α- or γ-Al2O3. On the Re(001) substrate hexagonal LEED patterns are observed from the films synthesized by both methods. Generally, the films synthesized by evaporation in oxygen show better LEED patterns than those oxidized following deposition, and Re(0001) surfaces yield better LEED structure than Ru(0001). XPS results show that the films are nearly stoichiometric, and LEIS results show that the substrates are well covered. The differences between Ru and Re substrates are discussed in terms of lattice constants and alloying properties between Al and the substrates.

SURFACE SCIENCE Room A201 ~ Session SS2-MoA

Nucleation and Growth: Semiconductors


2:00 pm SS2-MoA1 Mixed-Morphology MTP Growth of Ag on Si(100)-2 x 1 Surface. G. Jayaram, N. Doraiswamy and E. L. Marks, Northwestern University, Dept. of Mat. Sci. and Eng., Evanston, IL 60208.

Although the Ag/Si(100) system has received relatively less attention than Ag/Si(111), more than thirty papers have investigated the growth mode both at the initial stages and for thicker films. STM, LEED and AES studies suggest that the nucleation and growth mode is layer plus island, with a mixture of (100), (110) and (111) epitaxy normal to the surface. All studies have reported, more correctly assumed, a simple structure for these islands. However, little is really known about their structure. We present the first such study of Ag islands grown on Si(100)-2 x 1 surfaces using ultra-high vacuum high resolution transmission electron microscopy (UHV-HREM) imaging and diffraction (TED) techniques.

Several monolayers of Ag were deposited onto clean Si(100)-2 x 1 surfaces at room temperature inside the UHV-HREM facility. TED revealed a complicated pattern which could be interpreted in terms of a mixed epitaxy. HREM images showed that this was due to a mixture of multiply twinned particles (MTP's) and single crystalline islands of Ag which grow with different epitaxies on the Si(100)-2 x 1 surface.

This data demonstrates that one of the most fundamental questions about the growth mode of silver on clean Si(100), i.e. the particle structure, has escaped more than a decade of study. Rather than being a simple Stranski-Krastanov growth system, this is a classic mixed morphology MTP growth system [1].


2:20 pm SS2-MoA2 Modification of Overlayer Growth Kinetics by Surface Interlayers: The Si(111)-7/7 x 7/7-Indium Surface. S. Sanner, J. Kraft and F. P. Netzer, Institut für Experimentalphysik, Universität Graz, A-8010 Graz, Austria.

Indium-on-Silicon is a prototypical metal-semiconductor system which exhibits unusual growth properties in the transition region from 2-D layer to 3-D island growth. Of particular interest in this context is the (1/7 x 1/7) structure of In on Si(111), which is the only known 3-D structure at monolayer coverage for around one monolayer coverage (θ0 = 6/5). The atomic structure of the (1/7 x 1/7) surface has been investigated by STM and LEED: it consists of a densely packed In overlayer with a local arrangement similar to a (001) face of face-centered tetragonal bulk In. AES and thermal desorption measurements indicate that the further In uptake on the (1/7 x 1/7) surface is reduced as compared to In on Si(111) without this ordered interlayer. The mobility of In

*Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RL01830.
on the (\(\sqrt{7} \times \sqrt{3}\)) surface is very high as evidenced by STM, where only few but very large In islands are observed, leaving a major proportion of the surface uncovered even for nominal depositions as high as 500 Å. Epitaxially grown In islands are detected a elevated substrate temperature which displays a hexagonal array of In atoms on their surfaces.

2:40 pm  **SS2-MoA3**  K Islands on Si(111): Morphology Changes Induced by O$_2$ Exposure, Boris Lamontagne, Fabrice Semond, Denis Roy, Laboratoire de Physique Atomique et Moléculaire, Département de Physique, Université Laval, Québec, Canada GIK 7P4.

The morphology of the K overlayer is a controversial issue associated with the research field of Si oxidation promoted by alkali metals. Using SAES getter sources, K overlayers have been deposited in-situ on (\(7 \times 7\)) Si(111) kept at 150 K. Various techniques (XPS, AES, SEM and SIMS) attached to the same UHV system allowed us to characterize precisely the K overlayers. For high K coverages, islands with average dimension of 1-2 μm have been found to coexist and form long parallel islands (\(-10 \mu m\) in length) on the top of a continuous K layer of \(-3\) ML coverage. During a temperature annealing up to 300 K the islands desorb and leave a uniform K monolayer. Surprising and interesting results have been obtained upon the exposure of K islands to O$_2$. K islands are partially levelled under the low pressure O$_2$ exposure (\(-10^{-8}\) mbar) as they freeze under high pressure (\(-10^{-6}\) mbar). The O$_2$ exposure induces a modification of the surface energy via a K interaction. After the high pressure O$_2$ exposure of the K islands, a subsequent annealing to desorb K yields SiO$_2$ islands characterized by SEM, AES and AFM. K islands levelling favors the consequent K desorption as observed with SIMS. While several studies have suggested the formation of K islands, this work is the first of its kind to report the direct observation of K islands as well as the pronounced effect of a subsequent O$_2$ exposure on their morphology.

3:00 pm  **SS2-MoA4**  Kinetic Control in Epitaxial Growth: Chemical Saturation to Heteroepitaxy to Homoeopitaxy in CaF$_2$/Si(111)$^{1}$, Uwe Hessinger, M. Leskovar and Marjorie A. Olmstead, Department of Physics, FM-15, Univ. of Washington, Seattle, WA 98195.

Crystal growth by molecular beam epitaxy is inherently a kinetic process, and the growth mode and morphology depend on the chosen kinetic parameters—flux and substrate temperature—in relation to the material parameters—diffusion barriers, adsorption energies, and island or terrace sizes. For heteroepitaxial growth of dissimilar materials, these parameters will be different for each of the first few layers. We have applied kinetic models to explain the complex system of growth modes we observe with transmission electron microscopy and X-ray photoelectron diffraction for such a heteroepitaxial system, CaF$_2$ on Si(111).

The extent of dissociation of CaF$_2$ to CaF in the first layer can be controlled with temperature. The high temperature (\(\geq 600°C\)) CaF interface exhibits lower adsorption energies and diffusion barriers for second-layer CaF$_2$ molecules than either the adsorbed CaF$_2$ interface (T < 600°C) or adsorbed CaF$_2$ layers. This leads to bilayer growth of the second and third layers at high temperatures, nucleating at surface steps and/or on terraces, depending on the kinetics. The event of nucleating the fourth and subsequent layers with respect to the event of bilayer coalescence determines the extent of a layer-by-layer versus island-grown mode. We extend the model to propose a kinetic range for a layer-by-layer growth mode without bilayer formation.

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$^1$Work supported by the U.S. Dept. of Energy.

3:20 pm  **SS2-MoA5**  The Influence of Surface Topography on Epitaxial Growth, O. P. Karpenko, D. P. Adams and S. M. Yalatsoue, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109-2136.

Growth onto non-planar Si substrates was used to study the relationship between starting surface topography and thin film microstructure for heteroepitaxial growth of CoSi$_2$. In this study, substrates were prepared by lithographically etching trenches (\(\sim 4 \mu m\), depth \(\sim 3000 Å\)) into Si(001) wafers. Following high temperature (T \(\sim 625°C\)) Si buffer layer growth and annealing (T \(\sim 950°C\)) a number of well defined, high index surfaces including Si(117), (115) and (113) were observed at the sidewalls. Subsequently, these substrates were equilibrated to lower temperatures in order to study the growth of CoSi$_2$ using the "Temperature-Dependent" techniques for each of these surfaces. Heteroepitaxy of CoSi$_2$ onto each Si(11x) surface resulted in the nucleation of epitaxial films with different epitaxial orientations. A structure of these films as it relates to the morphology of the starting surface (different Si(hkl) surfaces and the distribution of facets) will be presented. Based on these results, possible mechanisms for the nucleation of misoriented CoSi$_2$(110) grains during heteroepitaxial growth of CoSi$_2$(001) onto planar Si(001) substrates will be suggested. These results will be discussed in the context of other materials systems where the initial surface topography has shown to influence the structure of the resulting film (i.e. Si homoepitaxy).

3:40 pm  **SS2-MoA6**  Structure of MBE-grown As-rich GaAs(001) Phases, T. Hashizume, Q. K. Xue, J. Zhou, A. Ichimiya$^2$ and T. Sueo, Institute for Materials Research (IMR), Tohoku University, Sendai 980-77, Japan, $^1$Department of Applied Physics, Nagoya University, Nagoya 464, Japan.

Understanding the structure of the GaAs(001) surfaces grown by molecular-beam epitaxy (MBE) is one of the most important issues of the recent semiconductor industry. In order to thoroughly study this system, we have applied a MBE-STM instrument, and the FL-STM (field-ion scanning tunnelling microscope) equipped with AES, RHEED.

Three phases of the As-rich (2 x 4); α, β and γ phases, classified by Farrell and Palmstrom, and c(4 x 4) phase are analyzed in detail based on the high resolution STM images, simultaneous RHEED observations and dynamical RHEED calculations. We have found that the unit cell of the (2 x 4)-α, β and γ phases all has the same structure of two As dimers and two dimer vacancies in the topmost layer, in contrast to the general belief that a unit of the three As-dimers and one dimer vacancy is energetically favored. Our systematic study show that the α and β phases have different second and third layer structures which are exposed by the As dimer vacancy rows and that the γ phase is the locally ordered β phase with large open areas exposing the As double layer structures which is the locally ordered c(4 x 4) phase. The two As-dimer structure we propose agrees with the recent theoretical calculation by Northrup and Froyen.$^2$

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Chemical vapor deposition (CVD) represents a complex interplay between chemistry and surface structure, making detailed understanding difficult to achieve. As a step toward this goal we report the first simultaneous measurement of surface chemistry and atomic order using reflectance-difference spectroscopy (RDS) and grazing-incidence X-ray scattering (GIXS), respectively, specifically on (001) GaAs. We use GIXS to determine both short- and long-range order, i.e., surface reconstructions and island sizes, and RDS to determine surface-induced optical anisotropy, which is sensitive to local surface reconstructions and chemical effects. During layer-by-layer growth both GIXS and RDS signals exhibit oscillations whose periods are identical, thereby unambiguously connecting the RDS oscillations to layer-to-layer growth. Using these complementary probes, we have performed a systematic investigation of CVD as a function of temperature, growth rate, V/I ratio, and precursor species. Here, we focus on dynamic measurements during growth, and show that the crossover between layer-by-layer and step-flow growth modes depends on the V/III ratio, and that the amplitudes of GIXS and RDS oscillations depend differently on process parameters, in particular on the partial pressure of tertiarybutylarsine. Additional results that take advantage of this unique combination of data to develop a more complete picture of CVD will also be discussed.

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$^*$Supported by ONR Contract N-00014-93-1-0255.

4:20 pm  **SS2-MoA8**  Heterostructures with Large Lattice Mismatch: Interaction of GaSe with GaAs(111), E. L. Rummaner and F. S. Ouchi, Department of Materials Science and Engineering, University of Washington, FB-10, Seattle, WA 98195.

Van der Waals Epitaxy (VDEW) allows for the growth of heterostructures, where the films are chosen based on the property of interest rather than lattice matching constraints. Films of GaSe have been grown on the surface of GaAs(111) by VDEW, overcoming a lattice
mismatch of 6%. In this study, GaSe was sublimated from a single solid GaSe source. Detailed RHEED and XPS studies of the nucleation of GaSe on the GaAs(111) surface indicates Se termination of the GaAs surface dangling bond occurs prior to GaSe formation. Randomly oriented GaSe islands nucleate at submonolayer coverages and are observed in RHEED by relatively broad, randomly oriented streaks corresponding to the GaSe lattice spacing. While the interaction of the substrate with the film is not strong enough to form an atomic structure, it is strong enough to dictate a favorable orientation relationship. As the nucleated grains grow larger, the broad streaks in the RHEED pattern become narrower and a six-fold rotational symmetry is observed. This corresponds to one favorable orientation of GaSe on the GaAs(111) surface, with the GaSe symmetrically oriented parallel to the underlying GaAs.


We use a scanning tunneling microscope operating in UHV to compare the growth morphology of GaAs(001) prepared using solid-source (As4) and gas-source (AsH3) MBE. We evaluate the effect of temperature and the presence of hydrogen on surface morphology. Films were deposited on the (001) surface miscut 0.25° towards [110] at both 585°C and 650°C with a deposition rate of 1 μm/hr. Samples were cooled from the growth temperature to 400°C in approximately 300 seconds. They were then transferred in UHV from the growth chamber to the STM analysis chamber. Surfaces grown at 585°C using solid-source As4 and an As4 cracker displayed an ordered multilayer system of elongated terraces 5000 × 700 Å, where the large dimension is along the [110] direction. The surface morphology of GaAs(001) prepared using AsH3 is isotropic with irregular shaped steps. Domains of the surface reconstruction on the gas-source grown buffer layer are approximately 600 Å in diameter. At 650°C, island sizes for both the solid-source and gas-source grown surfaces become considerably smaller than the terrace width.

Supported by the US Department of Energy grant no. DEFG02-91-ER45439 through the University of Illinois Materials Research Laboratory.

5:00 pm SS2-MoA10 Diffusion Barriers on GaAs/Sn (100) Surfaces*, S. M. Seutter, A. M. Dubiran, X. Song, B. E. Ishaug, and P. I. Cohen, University of Minnesota, Minneapolis, MN 55455.

Ultra-high vacuum, scanning tunneling microscopy (UHV-STM) and atomic force microscopy (AFM) in air have been used in conjunction with reflection high-energy electron diffraction (RHEED) to study the molecular beam epitaxial growth of GaAs on a delta-doped layer of Sn. Fractional monolayers (ML) of Sn were adsorbed onto singular GaAs(100) surfaces in an As4 flux and at a substrate temperature of 600°C. For growth at these temperatures, Sn is known to segregate to the surface; and dramatic changes in the growth kinetics have been observed by RHEED. The latter includes an enhancement of the layer by layer growth, an increase in Ga and Al surface diffusion, and strong beats in the RHEED intensity oscillations. Sn covered surfaces, with and without GaAs overgrowth, were characterized by AFM and STM. For surfaces without GaAs overgrowth, the surface morphology was relatively unchanged from that without a Sn layer. With a coverage of 20 nm of GaAs growth, the step density increased and monolayer deep holes formed. UHV-STM images showed the surface to be covered with small islands distributed in skewed rows parallel to the step edges. On the upper side of step edges a denuded zone formed while the small islands bunched together on the lower side of step edges and inside the holes. There was no directional anisotropy to the denuded zone. The results are interpreted in terms of diffusion barriers at step edges.

*Partially supported by the National Science Foundation (DMR09-07532).

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A209 – Session NS1-MoA


2:00 pm NS1-MoA1 Scanned-Probe Measurements of CdSe Quantum Dot Structures, B. Alpseron, S. Cohen, G. Hodes, and I. Rubinstein, Weizmann Institute of Science, Rehovot, ISRAEL 76100.

Size-controlled quantum dots provide a means for tailoring band gaps in semiconductor material. We have electrochemically deposited the semiconductor CdSe on gold with densities ranging from isolated clusters with dimensions of several nm, up to coherent coatings of nominal thickness exceeding 30 nm. The electrical characteristics and morphology of these films have been examined using a combined SFM/STM. A standard STM may be problematic in these measurements due to the enormous conductivity differential between the gold substrate and semiconductor, leading to difficulty in selecting bias conditions. STM images also cannot yield accurate height information under these conditions. We developed a fixed-contrast model tip and surface for feedback control, surface topography could be obtained, together with conductivity and I/V behavior. Measurements were performed under ambient conditions. Tunneling resistances measured on the gold indicated that point contact between metallized (Pt) tip and the surface was frequently not observed, but mediated by a contaminating layer. By varying operating conditions and comparing the results from different sized particles, it was possible to deduce information regarding the size-dependent electrical characteristics.


We are using a cryogenic scanning tunneling microscope to study the electrical-transport and structural properties of very thin granular Au/Al2O3 films, having metal volume fractions close to and below the percolation threshold. The granular films typically are ~15 nm thick and sputter-deposited on top of gold films. We have measured simultaneously topographic images and local tunneling current-voltage (I-V) characteristics perpendicular and parallel to film growth anisotropies. The I-V curves were found to vary qualitatively from one tip position to another over distances of the order of a few nanometers, indicating rapid spatial variations of the local transport properties. Some I-V traces show metallic behavior, whereas others exhibit pronounced structure due to single-electron charging effects of the small metal grains. Among these latter traces we observed Coulomb blockade and the Coulomb-staircase, similar to those observed for double-barrier tunnel junction systems. Many of our I-V characteristics exhibit, however, novel Coulomb-staircase structures having unusual variations in step heights and widths due to complex electron tunneling paths. These characteristics cannot be explained assuming a double-barrier tunnel junction geometry. We have developed a triple-barrier tunnel junction model, based on the “orthodox” theory for single electron tunneling, where electrons tunnel through two small metallic grains along their path. This model accounts very well for the experimental results.

2:40 pm NS1-MoA3 Properties of Nanostructures—Electronic, Haroon Ahmed, Cavendish Laboratory, University of Cambridge, UK.

Nanometre scale structures in semiconductors offer possibilities of making electronic devices in the future that overcome some of the limitations that arise when conventional devices are scaled down to smaller and smaller sizes. The transport of electrons in lateral and vertical nanostructures in systems such as 2DEGS can be reduced to 1D or 0D conditions. Novel quantum effects can be observed in such structures such as electron interference and ballistic transport. Another example of such devices exploit the well known Coulomb blockade effect. This effect is observed when current flow is interrupted by the presence of a single electron on an isolated nanoscale island. The requirement is that $e^2/2C$ where $C$ is the island capacitance is much greater than $kT$. The effect is observed in many systems at low temperature but for room temperature observation the islands must be reduced to nanometre scales such that $C < 10^{-15} F$. Another interesting
example of nanotechnology in electronics arises from the need to make artificial silicon structures that mimic the properties of porous silicon. In this case the pores are ~2 nm or less in diameter and therefore nanometers of this size must be created in arrays as artefacts in silicon. There are several fabrication techniques that are contenders for making sub 10 nm structures in semiconductors, electron beam lithography, STM and single atom lithography. With these methods quantum effect devices and single electron devices may be realised.

3:20 pm **NSI-MoA5** Voltage Oscillation Observed in Two Dimensional Nanostructures Using Liquid Crystal Molecules at Room Temperature, H. Nefiih, M. Aono and M. Tsukada, Aono Atomacraft Projects, JRD, 5-9-9 Tohkohali, Tsukuba 300-206 Japan, RIKEN and University of Tokyo.

When liquid crystal (LC) molecules are used as the central electrodes of an array of two-dimensional tunnel junction (TDT) which consists of LC molecules and metal islands, the capacitance between molecules and islands is restricted by the size of the LC molecule (0.5 mm × 1.5 mm). Conventionally, tunnel junction arrays have been fabricated using lithography so that the characteristic size of order of 100 nm. Single electron charging has been observed in such systems only at cryogenic temperatures. Since the size of the TDT using LC molecules is of the order of mm, the corresponding Coulomb energy becomes larger than room temperature. We have observed voltage oscillation across the current biased TDT where the STIM tip was held above an LC molecule and acted as a gate by ramping the tip voltage at room temperature. This structure has the disadvantage that the TDT is not properly isolated from the external circuit. We propose to overcome this problem by using a metal-coated AFM cantilever, where this cantilever will act as a gate and a double tunnel junction will be fabricated by scratching a lead wire with the AFM cantilever and placing LC molecules in the groove.

3:40 pm **NSI-MoA6** Charge Dissipation and its Phase Transition of Densely Contact-Electrified Electrons on a Thin Silicon Oxide, Y. Sugawara, Y. Fukano, T. Uekiibashi, T. Okusako, T. Tsuyuguchi, S. Morita, Y. Yamanishi and T. Oasa*, Department of Physics, Faculty of Science, Hiroshima University, Higashi-Hiroshima, Hiroshima 724, Japan, *Advanced Technology Research Laboratories, Suntomoto Metal Industries, Ltd., Amagasaki, Hyogo 660, Japan.

Recently, Saurenbach and Terris [1] and we [2], independently succeeded in reproducing reproducible and controllable contact electrification of a thin insulator using an atomic force microscope (AFM) with a conductive cantilever.

In the present experiment, we investigated the charge dissipations of the densely contact electrified electrons on a thin silicon oxide surface quantitatively, using the newly developed contact-electrification method. As a result, by increasing the density of the deposited electrons, we observed an appearance of a stable state of the densely deposited electrons and its disappearance due to the charge dissipation, i.e., a kind of stable-unsable phase transition. We also observed a saturation of the density of the deposited electrons with the spatial spread of deposited electrons. We further observed the electrostatic force related to positively charged trap site in a silicon oxide layer in addition to that induced by the deposited electrons on the silicon oxide surface. By comparing the charge dissipation in vacuum with that in air, finally, we discussed the dissipation processes of the contact electrified electrons.


The surface roughness of Si,Ge/Si superlattices grown by molecular-beam epitaxy on vicinal Si(001) has been characterized by ex situ atomic-force microscopy. At high Ge concentration in the alloy layers the surface exhibits a one-dimensional waviness with an average ripple spacing of about 450 nm to 1 μm and an amplitude of ≤ 1 nm. The ripple orientation is always parallel to the substrate steps. The characteristics of this waviness, including cross section of the ripples, domain size of areas with undisturbed waviness, and lateral correlation length of the underlying isotropic roughness are determined as a function of the substrate miscut, the individual-layer thickness, and the number of bilayers. On the basis of these morphological data, together with corresponding x-ray diffraction results on the buried interfaces [1], a model, based on stress accommodation, for the origin of this roughness anisotropy and its spatial evolution is developed.

*Research supported by NSF Grant No. DMR 92-01856. One of us (C. T.) acknowledges support by the German Academic Exchange Service.

4:40 pm **NSI-MoA9** Submicron Lithographically Patterned Magnetic Islands for Magnetic Recording, R. M. H. New, R. F. W. Pease, R. L. White, Stanford University, Stanford, CA 94305.

In conventional hard-disk magnetic recording systems, the signal to noise ratio is limited by “transition” noise which occurs due to the irregular zig-zag domain walls between adjacent recorded bits. In order to address this problem, we have been studying recording media composed of submicron lithographically defined islands of magnetic material. We have developed a procedure using direct write electron-beam lithography for high resolution patterning of magnetic thin films, and we have used this procedure to define large arrays of small magnetic particles. We are studying the characteristics of these particles with both bulk measurement techniques and with high resolution magnetic imaging methods to determine if they will be suitable for high density recording systems. Theory suggests that sufficiently small magnetic islands will behave like single-domain particles with only two stable magnetisation states. Such particles would be ideal for storing a single bit of information.

The scanning electron micrograph to the right shows a patterned thin film of cobalt. The film is 200 Å thick, and the smallest feature size is 0.2 μm. The picture is taken from an angle of 15°, measured from the surface of the film. This work was performed under NSF contract number ECS 8920652.
5:00 pm NS1-M0a10 Ferroelectric Domain Dynamics Investigated By Atomic Force Microscopy, O. Kolosov1, A. Grauerman, J. Hatanaka1, K. Takahashi and H. Tokumo1,2, Mechanical Engineering Laboratory, Tsukuba, Ibaraki 305, Japan, 2Joint Research Center for Atom Technology, ATP and NAIR, Tsukuba, Ibaraki 305, Japan, 3National Institute for Research in Inorganic Materials, Tsukuba, Ibaraki 305, Japan, 4Science University of Tokyo, Faculty of Industrial Science and Technology, Noda, Chiba 278, Japan.
The domain structure in ferroelectric crystal guanidinium aluminum sulfate hexahydrate (GASH) and its evolution under the action of electric field were studied by atomic force microscopy (AFM) with nanoscale resolution. The ferroelectric domains were directly observed on the cleavage surface of crystal using AFM operating in both contact and noncontact modes. To distinguish the contrast associated with topography from that related to electrostatic interaction between tip and polarization, two types of cantilevers were used, namely, standard nonconductive Si3N4 tip and gold coated conductive one. To investigate the domain dynamics a constant potential was applied to the bottom electrode, while conductive tip was held at zero potential.
The domain structure in the form of rows of cylinder domains along with configuration consisting of domains of one sign embedded into the larger domain of opposite sign was observed in topography image. The origin of domain contrast was discussed. The AFM images were compared with domain patterns revealed by powder deposition technique.
The domain dynamics in applied electric field was studied for the first time with nanoscale resolution. It was directly observed that sidewise growth of domains proceeds through nucleation on the domain wall. The directions of nuclei as well as of electric field required for domain growth, showed a good agreement with experimental results. The ability of effective control of the domain structure with AFM was directly demonstrated.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A207 - Session NS2-M0A

Nanometrology

2:00 pm NS2-M0a1 Dimensional Metrology with Scanning Probe Microscopes, J. E. Griffith, ATK Bell Laboratories, Murray Hill, NJ 07974-0636.
By the end of the decade, semiconductor manufacturers will need measurements of surface topography with lateral uncertainties of a few nanometers and vertical uncertainties of less than a nanometer. Scanning probe microscopes have the potential to meet these needs, but only if the measurement "spears" behavior is tightly controlled. The dominant source of measurement error arises from the probe tip, which interacts with the measured surface in an intrinsically nonlinear manner. A fundamental requirement is that the probe shape be stable and well characterized. In our instrument we use conical Ir probes formed with focused ion beams and cylindrical glass probes chemically etched from optical fibers. Our force microscope is able to employ a wide range of probe types because of the force sensor, which does not depend on micromachined topographical patterned cantilevers. We use instead a force-balance scheme that drives a rather large, and rugged, balance beam. The pivots for the beam are small ball bearings constrained by a magnet, which allows easy tip exchange. Balancing of the beam is effected through two capacitors, which also provide force sensing. A set of three capacitors are also used to monitor the position of the scan head. Though capacitors can be extremely precise position sensors, significant position measurement uncertainties can arise from Abbe offsets between the probe and the monitor. The extent to which this affects measurement will be discussed. Our probe microscopy has been used to nondestructively measure high-aspect-ratio features on lithographically patterned photomasks and wafers.

2:40 pm NS2-M0a3 Proximal Probe Characterization, Leigh Ann Files, John N. Randall, Texas Instruments, Inc., and David Keller, University of New Mexico.
One of the primary problems with the transition of Scanning Probe Microscopy (SPMs) from the "picture" to the metrology stage is the difficulty in extracting the true profile of a sample from the apparent profile obtained as a result of convolution of the tip shape with the sample. High aspect ratio tips have been fabricated to help alleviate this problem, but it is difficult to fabricate such tips reproducibly, and they are rather fragile. This can present difficulties when using an SPM to monitor feature sizes in a wafer fabrication area. If the tip breaks while scanning a small feature, much larger dimensions may be measured, and it may erroneously be presumed that the lithography of the sample being scanned is the problem. Thus a method to characterize the tip is needed. It has been proposed that one could scan a standard feature of known dimension and combinations of the scan line to obtain an inverted image or replica of the probe tip. Such a standard could be used to qualify or "grade" tips as they are fabricated to determine the maximum sidewall and minimum dimensions they are suitable for measuring. It could also be used to monitor the tip quality at appropriate intervals during scanning. We have used electron beam lithography to make a standard pattern of features up to 0.5 μm in height and ranging from 0.1 to 5 μm in diameter with a 20 nm thick ledge are formed by anisotropic dry etching followed by isotropic selective etching of a multilayer stack. Envelope image analysis has been used in conjunction with this standard to extract probe shapes from profiles to determine the structure of a number of commercially available probes.


3:00 pm NS2-M0a4 Maximizing the Information Content of Scanning Probe Microscope Data, P. M. Williams, M. C. Davies, T. O. Glasby, D. E. Jackson, C. J. Roberts, K. M. Shakesheff and S. J. B. Tendler, Laboratory of Biophysics and Surface Analysis, Department of Pharmaceutical Sciences, The University of Nottingham, Nottingham, NG7 2RD, UK.
Scanning probe microscopy has revolutionized the field of surface science. Recently, however, much emphasis has been placed on the mechanisms of image formation and the degradations imposed on image data. The effect of probe geometry has been studied by several workers and mechanisms have been proposed and adopted for image refinement. Here we detail a method for the derivation of probe geometry from any probe microscopy image. Two methods eliminate all assumptions concerning probe profile and permits the reconstruction of image surfaces without any prior knowledge of the tip.
An algorithm will be described that extracts a possible probe geometry from an image and is demonstrated on scanning force microscopy studies of semiconductor and polymer samples. The derived probe profiles are then validated by their comparison with electron microscopy images. Furthermore, details will be given of an image reconstruction technique which permits the assignment of true sample features allowing the quantification of image degradation. Such assessments are critical to the rigorous validation of image data by comparison with those from complementary imaging techniques. Finally, recent progress in developing a novel technique for extracting surface selection and compliance data, as an extension to the algorithms described, will be presented.

3:20 pm NS2-M0a5 Estimation of Proximal Probe Microscope Tip Geometry without Calibrated Reference Artifacts, J. S. Villarrubia, National Institute of Standards and Technology, Gaithersburg, MD 20899.
Proximal probe techniques such as scanning tunneling microscopy (STM) and atomic force microscopy (AFM) routinely achieve atomic resolutions of 1 nm or better, making them good candidates for dimensional measurement tools in the otherwise difficult sub-μm regime. However, the tip shape is "convolled" (strictly, dilated) with the surface, resulting in imaging errors which can be significant at the size scales of interest. Parts of the actual surface which were touched by the tip may be reconstructed if the geometry of the tip is known. For this reason, much attention has recently focused on reconstructing tip shapes by imaging known reference artifacts. Mathematically this procedure is well-defined, but calibrating and maintaining stable reference artifacts to nanometer accuracies is not trivial. Fortunately, for many purposes it may not be necessary. Using a mathematical model of the imaging process, I will show that for each point on an image there is a corresponding surface which is an upper bound on the tip. The actual tip must be smaller than or equal to the largest tip which satisfies all of the bounds. Images of surfaces with high relief and narrow, steep features provide tight bounds on the tip geometry. Model calculations indicate good reconstruction of tips for easily manufacturable reference artifacts, the geometry of which need not be known in detail.

Engineering of ultra-sharp tips has made significant progress recently. Procedures encompass oxygen treatment, thermal vacuum, and thermocouple assistance which ease the process of creating point tips with enough sharpness to perform scanning probe microscopy on hard substrates. In this report, we will present the results of our explorations of the effects of different parameters on the tip geometry and its performance. A detailed description of the fabrication process and the new characterization techniques developed to measure the tip geometry and electrical properties will be given. Special emphasis will be given to the applications of these tips to the study of microstructures on hard substrates. The importance of these results for future developments in the field of scanning probe microscopy will be discussed.

Research partially supported by ARPA Contract F49620-93-C-0068.

4:00 pm  NS2-MoA7  Scanning Tunneling Microscope with Three-Dimensional Interferometer for Surface Roughness Measurement, Toru Fuji, Masataka Yamaguchi and Masatoshi Suzuki, Nikon Corporation, 1-10-1, Asanomizai, Sagamihara, Kanagawa 228, JAPAN.

A scanning tunneling microscope (STM) has been well known for its high lateral resolution and non-destructive surface profiling capability but its potential as a profiler for roughness and step height measurement has been rarely exploited because of its unattainable vertical accuracy. We have for the first time developed an STM which is capable of 0.3 µm line scanning secured by a parallel spring mechanism in X direction with interferometric monitoring. STM tip feed-back controlled motion in Z direction along with X and Y raster scanning motion. Step height and pitch measurement on a surface topography standard agrees well with the certified value of the standard. In this study we were successful in combining the superior lateral resolution of the STM with the better vertical accuracy of interferometric measurement. The result of high accuracy roughness measurement with our STM supports the common observation that STM measurement gives larger roughness than interferometric measurement.

Part of this work was conducted in the program: 'Advanced Material Processing and Machining System,' consigned to AMMTRA from MDO, which is carried out under the Industrial Science and Technology Frontier Program enforced by the Agency of Industrial Science and Technology.


In the first part of the paper, automated calibration of the sample image using a dual-tunneling-unit scanning tunneling microscope and a scale reference graphite crystal is discussed. Measurements in the hundred nanometer range showed accuracy of around 99.99%. In the latter part of the paper, the use of scanning force microscopy and a crystal to line laser scale measuring device is introduced. In this technique, the periodic modulating motion of the end point of the tip observed in scanning force microscopy was used. The technique proved to be more robust compared to the method using scanning tunneling microcopy since atomic periodicity is less likely to be lost due to outer disturbances.


In the semiconductor industry the Atomic Force Microscope (AFM) has become an invaluable tool for the measurement of surface micro-roughness and topography with sub-Angstrom resolution. Tunnel and gate oxide interfaces are a strong function of micro-roughness which depends on the physical and chemical processing of the starting Si wafers and oxides. The semiconductor industry is demanding improvements in precision, accuracy, repeatability and reproducibility, so that AFMs can be used as a robust metrology tool for the inline process control. In this paper the results of an AMS sponsored AFM round robin from 9 laboratories are presented and discussed. Starting Si wafers were etched in a solution of 40:1 NH4F:HF to create various grades of micro-roughness from 0.5 Å to 5 Å RMS. In the repeatability study 30 continuous 2µm x 2µm area scans were made using the same probe tip without re-positioning. In the reproducibility study each wafer was sequentially scanned twice using a single tip and re-scanned with a new tip. The results indicate that the participants were able to resolve each grade of roughness. The minimum reported micro-roughness for polished starting wafers was 0.2 Å and maximum was 1.5 Å RMS. For the roughest wafer the reported RMS differed by only 2 x. This paper will discuss microroughness variation from tip to tip and pass to pass, seek correlation between contact and non-contact mode of AFM operation and derive a relationship between RMS and FV.

5:00 pm  NS2-MoA10  An Optical Fiber Based Shear Force Microscope For Nanometer-Scale Dimensional Metrology On Large Samples, H. M. Marchman, AT&T Bell Laboratories, Murray Hill, NJ 07974 USA.

The ability to measure topography of micro-fabricated structures in all three dimensions with nanometer-scale accuracy and precision is crucial for the development of sub micrometer and below device fabrication techniques as well as a product qualification yardstick for existing ones. A shear force microscope has been constructed for performing dimensional measurements of nanometer-scale features on large samples, such as five inch wafers or lithographic masks. Tip-to-sample regulation is accomplished by optically monitoring the change in vibration amplitude of a light-emitting optical fiber probe, oscillating near its mechanical resonance, as it encounters forces from the sample surface. Attractive mode sensing in the lateral scan direction (for sidewalls) is also possible with this system. The frequency and quality factor Q of the mechanical resonance of the light-emitting tip can also be tuned by tailoring the probe shape during fabrication. The cylindrical and undercut etched fiber probes used in this system are also ideal for making accurate dimensional measurements for routine aspect ratio structures. In addition, the Abbe offset error that normally occurs during tip position monitoring is significantly reduced by detecting the position of the light that is emitted near the apex of the tip, or proximal point.

APPLIED SURFACE SCIENCE Room A101 - Session AS-MoA

Data Processing and Reference Methods

2:00 pm  AS-MoA1  Auger Electron Spectrometry Data Bank, M. P. Seab, Division of Materials Metrology, National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK.

An Auger electron spectral data bank has been acquired for most elements in the direct spectral mode at high resolution (0.25 eV with both 5 and 10 keV electron beams). The energy dependence of the spectrometer transmission and its absolute value are calibrated. By removal of (i) the reflected primary electrons, (ii) the secondary electron cascade and (iii) the inelastically scattered Auger electrons, the true Auger electron peak areas may be determined. These true peak area intensities are compared with the theoretical predictions using the best available general sources for the cross sections, backscattering, inelastic mean free paths etc at the absolute level with no adjustable parameters. Agreement is excellent. Divergencies do occur, however, in well-defined regions of kinetic energy and atomic number. The reasons for these divergencies will be discussed with recommendations for more convergent approaches. This analysis gives great confidence in the general use of AES for quantitative analysis since all parameters now have traceability to a basic system of accepted theory or the SI measurement system. Recommendations are given for the best way to use the data bank for quantitative analysis.
Auger microscopy is widely used for surface characterisation. However, the presence of surface topography or sub-surface composition variations can often result in the technique being unable to provide even qualitative information. MULSAM has been developed to allow quantitative microranalysis of rough, chemically inhomogeneous systems. MULSAM is a strategy for the acquisition and analysis of multi-spectral image sets, and involves the simultaneous acquisition of secondary electron, sample absorption current, characteristic X-rays, Auger and backscattered electron signals. The exploitation of correlations between the various signals combined with models enables the removal of artefacts due to both topography and sub-surface composition variations. These techniques will be demonstrated with two distinct types of sample. The first example demonstrates the characterisation of the chemical and topographical variations across wear scars generated using a Plint foil test with model oil formations. The image manipulations prove sufficiently robust to allow meaningful analysis of samples with gross topography. The second example investigates the depth distribution of Al/Cu precipitates formed in 1 µm wide Al/Cu interconnects which have undergone electromigration stressing. Ion beam bevelling is employed to expose laterally the depth distribution of elements along the length of the track. The combination of characteristic X-ray, backscattered electron and Auger images allow the determination of the location of surface and sub-surface precipitates.

AS-MoA3 Surface Microanalysis and Imaging: Data Visualization and Pattern Recognition Applications, R. W. Limton, Department of Chemistry, University of North Carolina, Chapel Hill, NC 27599-3290. A critical issue in the evolution of surface imaging applications is the effective correlation of multi-dimensional data, both for classification and visualization. One illustration involves the use of imaging secondary ion mass spectrometry, for the 3-D dynamic depth profiling of solid surfaces or the 2-D static image in molecular patterns. Even if artifact contrast contributions such as chemical, topographic, and chromatic effects can be evaluated, a problem remains to view 3-D multielemental concentration images with high dynamic range. Possibilities include stereo pairs, holographic display, surface rendering, and true volume rendering using ray tracing. The latter approach is used in combination with shadowing, thresholding and control of voxel opacity to reveal internal structures within a 3-D volume. Pseudocolor is employed to indicate depth scale, multielement correlations, or intensity (concentration) gradients for a given element. Another critical step in the process of reducing SIMS images to chemical or compositional information is segmentation: dividing the images into regions that correspond to structural units. The Lin, intensive nature of SIMS imaging, such as the possibility for acquisition of full mass spectra at each pixel, is a driving force toward the application of multivariate techniques for image classification. This is especially true considering the high dynamic range available in SIMS: the presence or absence of a mass peak often is less informative than the pattern of intensities for various masses. The application of pattern recognition to data classification involves the use of statistical techniques (e.g. principal component analysis, cluster analysis) or artificial neural networks (e.g. Kohonen self-organizing feature maps) to associate each image pixel (or voxel) with a major group or class. Both approaches are of value in correlating mass selected images to localize molecular species on heterogeneous surfaces. INVITED

3:30 pm AS-MoA5 Numerical Analysis of TOF-SIMS Data, 1 Susan G. Mackay, 2 Patrick J. McKewen, 3M Corporation, 3M Center, 201-25-16, St. Paul, MN 55144, 2Perkin-Elmer, Physical Electronics Division, 6509 Flying Cloud Drive, Eden Prairie, MN 55344.

Interpretation of TOF-SIMS data often involves the recognition of a characteristic fragmentation pattern for a particular species. In the cases of heterogeneous samples or complex mixtures, identification of unknown species is complicated by increasing fragment interferences. Currently there is a growing interest in applying numerical analysis techniques to help resolve multicomponent spectra, and to aid in searching of spectral databases. In order for statistical techniques to be successfully applied to TOF-SIMS data analysis, some fundamental data processing issues need to be addressed. Preliminary work has been performed which explores instrumental effects on spectral reproducibility. Data was acquired from a model system (acrylate copolymers) using two different geometry TOF-SIMS instruments. A data matrix was then compiled which combined data acquired on both instruments from identical samples, and from samples from the same copolymer mixtures. The data was then processed and analyzed using partial least squares (PLS) analysis. Various data processing and normalization procedures will be discussed pertaining to both qualitative and quantitative applications.
matters. Plots of differences in BE values from mean values versus BE indicated systematic trends in the BE measurements from each source.


4. B. V. Crist (private communication).


It has become common practice to employ as a binding energy reference for XPS studies of nonconductive materials the C(1s) spectra of the ubiquitous (adventitious) carbon which seems to exhibit an instantaneous presence on all air-exposed materials. Despite this commonality, surface scientists, including many practitioners, have substantial concerns about the validity of this approach. A detailed discussion is presented of the method including a consideration of the types of materials and the electronic energy states involved, e.g., Fermi edges, vacuum levels, etc., and the couplings that must exist between these states. Also described is the physical status that the material being examined and the adventitious carbon must be in for the referencing method to be correctly applied. A number of other surface environments are presented for which the carbon referencing method may be fallacious. This leads to a consideration of the ESCA results for the different types of adventitious species and how the presence of some of these may confuse the use of the method. In this regard, we will also discuss the use of other methods to establish binding energy scales, such as Fermi edge coupling and select doping (e.g., the Au/SiO2 approach). Finally, we will consider the use of the carbon referencing method and the status of adventitious carbon during studies of polymers and other carbonaceous materials.

PLASMA SCIENCE

Room A109 - Session PS-MoA

Plasma Deposition

Moderator: W. Holber, ASTeX, Inc.

2:00 pm PS-MoA1 Plasma Assisted Deposition of Diamond Films, T. D. Mantel, Department of Electrical and Computer Engineering, University of Cincinnati, Cincinnati, Ohio 45221.

A new regime of diamond film applications can be achieved through deposition on carbon-containing gases on a variety of substrates at temperatures below 1000°C and at subatmospheric pressures, with gas activation furnished by a heated filament, laser irradiation, or electron impact in a radio frequency or microwave plasma. In this paper, this will be on plasma assisted CVD of diamond from methane or other gaseous hydrocarbon precursors in a hydrogen plasma environment. Although diamond is metastable with respect to graphite and the kinetics of graphite growth are faster than for diamond, the barrier to diamond growth can be overcome by providing an adequate supply of atomic hydrogen; a principal role of the plasma deposition reactor is to generate sufficient atomic hydrogen while controlling the accompanying ion flux. Current diamond deposition reactors will be compared, including microwave applicators and ECR sources. The effects of additives, including oxygen and halogens, will be considered, and recent results on low temperature and low pressure diamond growth in pulsed magetoactive microwave discharges will be discussed.

2:40 pm PS-MoA3 An Investigation of SiO2 PECVD Using Attenuated Total Reflection Fourier Transform Infrared Spectroscopy, S. C. Deshmukh and E. S. Aydil, Department of Chemical and Nuclear Engineering, University of California Santa Barbara, CA 93106.
We have deposited nearly stoichiometric films, at temperatures below 400°C, with growth rates of 120, 300, and 1800 Å/min for Si-, Ge- and Sn-nitride respectively. The film growth rate was increased by lowering pressure and/or increasing plasma power. The film composition (measured by RBS, Auger and ERS) depended mainly on the ammonia to precursor flow ratio and the substrate temperature. Further characterizations of the films included IR, UV-VIS spectrophotometry and ellipsometry. At optimum deposition conditions, the films were carbon and oxygen free.

4:40 pm PS-MoA9 Fast Deposition of a-Si:H for Solar Cell Application, R. J. Severens, M. C. M. van de Sanden and G. J. Meursen and D. C. Schram, Department of Physics, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands.

Amorphous hydrogenated silicon is used as the intrinsic layer in amorphous silicon solar cells. As the growth rate of a-Si:H using conventional techniques as PECVD is low (typically 0.1 nm/s), a successful commercial introduction of solar energy is hampered. Considerable higher growth rates are therefore desirable. In this paper a newly developed deposition technique, based on an expanding thermal arc in an argon hydrogen mixture seeded with SiH₄, will be discussed. As the opto-electronic properties are essential, the aim was to increase the deposition rate while maintaining the quality of the deposited layers. Properties such as bandgap, carrier lifetime, carrier diffusion length, (SH/SiH₄ ratio) and light to dark conductivity, oxygen and nitrogen impurity content were monitored as function of the deposition parameters. Layers showing good opto-electronic properties (light to dark conductivity >10⁸, n > 3.8, Eₒ = 1.6 – 1.8 eV) were produced at high growth rates (typically > 10 nm/s) as measured with in situ HeNe ellipsometry. The good layers also show low oxygen and nitrogen content. As the ion energy is low (typically < 2 eV) it is demonstrated that the beneficial effect of DC bias in RF deposition is not essential. Also the refractive index seems to be independent of the substrate temperature. The particular role of ions, radicals and the possible presence of clusters is addressed.


The low temperature Si/SiGe epitaxy is investigated using Ultrahigh Vacuum Electron Cyclotron Resonance Chemical Vapor Deposition (UHV-ECR-CVD). The effects of process parameters on the hydrogen plasma cleaning and Si/SiGe epilayer by RHEED, TEM, and dilute Shimmel etching. It is observed that the defect formation on the Si substrates during in situ ECR hydrogen plasma cleaning is strongly dependent on the process parameters such as the microwave power, the remoteness of ECR layer from the substrate, and the substrate DC bias, and the plasma exposure time. The hydrogen ion dose plays a significant role in the low temperature in situ plasma cleaning, and consequently the precise control of both process parameters and the plasma exposure time is very important in preparing the damageless clean Si surfaces prior to epitaxial growth. The defect production in the Si/SiGe epilayers during plasma deposition is mainly caused by the energetic ions impinging on the substrate and can be effectively suppressed by the proper control of the ion energy flux. It has been found that the control of the DC substrate bias can be used for this purpose and a high quality, dislocation-free Si/SiGe epilayers are obtained in the UHV-ECRCVD system under optimum conditions. The effect of process parameters on the Si/SiGe epitaxy will be also reported.

VACUUM TECHNOLOGY
Room A102 – Session VT-MoA

Gas Flow, Partial Pressure Analysis, and Leak Detection
Moderator: J. L. Provo, Martin Marietta Speciality Components Inc.

Modern turbopumps include a drag state that operates in the pressure range of 0.10-1 torr or higher. Flow conditions range from molecular flow at the drag inlet, to viscous flow at the outlet. This is called "transition" flow. Transition flow models of pumps have not been developed. Models of transition conductance in tubes have been considered by Knudsen and others, for capillary leaks to atmosphere. These models add the two conductances of the tube at low and high pressure, multiplied by weighting functions of the average pressure.

If the tube surface moves with a velocity in the axial direction, it becomes a Gaede pump [1]. We have developed a model of the Gaede pump that proceeds from transition flow in a different domain, integrated over the length of the pump. The predictions of this model are modified by a new, "pumping leak" theory of the gas striper, which separates inlet from outlet. The result is compared with experimental measurements.

If the surface velocity is zero, the model predictions agree with Knudsen's data for capillary leaks, while giving a better account of the controversial, "conductance minimum".


2:20 pm VT-MoA2 Calculations of Orifice Flow in the Transition Regime, J. P. Looney, and R. D. Mountain, Thermophysics Division, NIST, Gaithersburg, MD 20899.

The flow of gas through an ideal orifice has been investigated theoretically and experimentally for large Knudsen numbers (Kn = mean free path to orifice diameter ratio) by several investigators since the pioneering work of Knudsen. To date, calculations of the gas flow or orifice conductance for nearly free molecular flow conditions at large Kn either do not exhibit good agreement with experimental data or do not predict the flow in the gas law region. In this talk I will discuss a model that we have developed which is based upon mean free path kinetic theory and the numerical solution of this model through the use of Monte Carlo methods. The physical interpretation of this model is straightforward, and the results of these calculations are in excellent agreement with experimental data that has been reported in the literature and obtained in our laboratory for Kn as small as 0.3. We also find good agreement with the theoretical results from numerical solution of the Boltzmann Equation for Kn > 1.


2:40 pm VT-MoA3 High Precision, Gas Analysis System Used to Analyze Grab Samples from the Space Shuttle Engine Compartment, G. M. Solomon and M. D. Boeckmann, Vacuum Technology, Inc., 1003 Alvin Weinberg Drive, Oak Ridge, TN 37830.

A system was built to analyze grab samples taken from the space shuttle engine compartment. The gas samples varied from 3 torr to 200 torr in a 210 cc sample cylinder; which uses explosive charges on a valve to "grab" the gas samples. The analysis system uses three expansion volumes to reduce the sample pressure to between 0.5 and 1 torr in a 650 cc sample vessel. Once in the sample volume, the mass peak ions signals were extrapolated to zero time. Pure gases were used to measure the system time constant, verify the molecular flow assumption and determine cracking pattern ratios for the gases of interest. The gases of interest were hydrogen, helium, carbon monoxide, methane, nitrogen, oxygen, argon and carbon dioxide. It was found that the active gases changed the partial pressure sensitivities of all gases by more than 10%, initially. An ion source conditioning step was performed using an oxygen mixture to stabilize the gas sensitivity factors with time, before the analysis was performed. Mixed gas standards of varying percentages were intercompared to test the system. Batch inlet system design criteria, calibration standards and analysis software will be reviewed.


Most vacuum processes require pressure control which is stable, quick to achieve set point, and responsive to perturbations. In common practice, a pressure control system using a proportional-integral-derivative (PID) algorithm drives a downstream throttle valve or an upstream bleed valve. As with any PID controller, the tuning of the parameters which govern the system behavior is critical to its performance. To complicate matters, vacuum systems are inherently nonlinear as a function of pressure and gas throughput and so a fixed tuning may not span the entire operating range effectively. A controller which stores multiple tuning recipes may be employed but it still requires manual tuning of each recipe for its application.

Two approaches to manually tuning a PID controller are (1) a close loop method and (2) an open loop method. Since the tuning parameters for the desired response in a given PID application are closely constrained, either method should yield similar results; however the relative ease and efficiency of the tuning processes may vary from case to case. A test where various vacuum systems are tuned using each method is used to form comparisons and recommendations.

3:40 pm VT-MoA6 Fast Fine Leak Testing of Very Large Vessels, Lyle Bergquist, Y. Tito Sasaki, Quantum Mechanics Corp., PO Box 1885, Sonoma, CA 95476.

Conventional helium leak detectors cannot detect fine leaks in vessels measuring tens of cubic meters. Because of the large amount of desorbed gases such as H₂ and H₂O relative to the amount of the tracer gas, the concentration of helium becomes extremely low. If we want the helium partial pressure to be high enough for the mass spectrometer to detect it, then the background pressure becomes too high for the spectrometer's proper functioning. Conversely, if we lower the total pressure, the partial pressure of helium becomes too low to be detectable. An added problem is the long response time that will make it uneconomical to locate the leak and fix it. A simple solution to all these problems has been developed by combining the principles of helium compression and selective accumulation. A system we designed will detect leaks in the order of 10⁻¹⁰ m³/sec in a 40 m³ vessel under 5 seconds. An additional minute will establish the exact leak rate. This design uses a turbomolecular pump of a high pumping speed and high helium compression ratio. It compresses the residual gas in the vessel—helium in the helium and the desorbed gases—into the mass spectrometer region. Then a chemical getter and a cryocondensation pump remove all but helium. In this manner the leaked-in helium is immediately concentrated in the detector region while the background pressure therein is kept low. The paper discusses this and other possible designs and applications.

4:00 pm VT-MoA7 Vacuum Insulation: The Potential for Energy Conservation, H. Alan Fine, Global Change Division, U.S. Environmental Protection Agency, Washington, DC.

A number of advanced insulation systems are currently under development. These systems generally consist of a filler material maintained under vacuum within a gas-tight barrier/container. With the exception of gas-filled panels, the improved thermal performance of these systems results from the vacuum.

Filler materials incorporated into advanced insulation systems include ceramic spacers, precipitated silica or other powders, fiberglass, aerogel tiles and open-cell foam. Barrier have generally been fabricated from laminated polymeric structures, glass or stainless steel. Vacuum requirements range from soft vacuums of approximately 50 Torr for aerogel tiles to hard vacuum of less than a micron for ceramic spacers.

The state-of-the-art of advanced insulations is reviewed. Emphasis is placed on the achieved and potential thermal performance of the insulation systems, and their potential for energy conservation when they are used to improve the energy efficiency of appliances and buildings. The barriers which must be overcome before widespread implementation of this technology will occur are outlined. The advantages and disadvantages of advanced insulations, as compared to more traditional forms of insulation, and the cost effectiveness of advanced insulations are also discussed.

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Positive processes in vacuum pump-down were studied in an aluminum ultrahigh vacuum system, using super-dry nitrogen and argon. The system is constituted of a roughing pump line, a main pump line with a residual gas analyzer, a venting gas line and a main chamber with vacuum gauges. The venting gas line can reduce other
contents (water, hydrogen, carbon monoxide etc.) of vent-gas, except argon and nitrogen, to a few ppb after two hours operation. A new elementary process (pressure proportional to approx. time \( t^{-2} \)) in vacuum pump-down was found in the system. Fig. 1 shows the curves of total pressure (Initial 760 Torr) and partial pressures in using superdry argon gas. From the data of residual gas analysis (Fig. 1), it is concluded that the change of total pressure at least till \( 2 \times 10^3 \) seconds mainly depends on the change of argon gas instead of desorption gas (water etc.). Therefore, the pump-down process approx. between 50 seconds and \( 2 \times 10^3 \) seconds is other new pump-down process than that for volume gas (P \( \propto \text{exp} \left( -\frac{S}{V} \right) \)) desorption gas (P \( \propto t^{-1} \)) and diffusion gas (P \( \propto t^{-1/2} \)); here, P, V, S and t are the pressure, the chamber volume, the pumping speed and the pumping time, respectively. It can be seen only when desorption gas from chamber surfaces is very little and pump-down time for volume gas is extremely short. We tried to explain the process using a model that main gas content (Ar) leaking extremely slow as a viscous flow from fine complicated structures in chamber surfaces.


Homoepitaxial diamond films have been grown on (100), (110), and (111) natural type IIA and type IIB diamond substrates by hot filament chemical vapor deposition (HFCVD) to thicknesses of 3 to 100 \( \mu \)m. The surface morphology, crystalline quality, stresses, and impurity content of the films were evaluated using SEM, HRXRD, Raman spectroscopy, FTIR and SIMS as a function of C/O/H ratio, (0.5–17at%), (0.5–17at%), and H (67–99at%) in the precursor gas. Variations in the C/O/H ratio were achieved by using methanol, acetone, and methane/oxygen, in a hydrogen carrier. A novel micro-Raman spectroscopic technique was used to measure stress with a \( \pm 11 \) MPa precision by calibrating the diamond Raman line to an internal krypton line. For film thicknesses > 10 \( \mu \)m, tensile stresses up to 280 MPa developed at which cracking occurred and relieved stresses down to \( \sim 100 \) MPa. No sp\(^2\) was detected for (100) and (110) films grown with \( \leq 50\% \) methanol. The FWHM of the sp\(^2\) peak varied from 2.8 cm\(^{-1}\) at low C to 4 cm\(^{-1}\) at high C. Very little broadening of the X-ray line width (22 arcsec), for films grown with low C, indicated small levels of strain and/or defects in the films. The tensile stress in the films, FWHM of the Raman line, and X-ray line widths all increased with increasing C in the precursor gas. The increase in tensile stress in the films correlated with increased concentrations of Re, O, and H in the films, as measured by SIMS and FTIR, as the C and O fraction of the gas increased. The consequences of these film properties on the use of homoepitaxial films for coating applications will be discussed.

*Research supported by BMD/IST and ONR.

5:00 pm VT-MoA10 Measurement of Metals Vapour Pressure as Function of Temperature, Vadim I. Rakovskiy.

A pressure of the saturated vapour of a chemical element as a function of temperature is a fundamental constant. Knowing it enables us to estimate the most important thermodynamic parameters of a composition, which the chemical element is a part of. The widely used Langmuir and Knudsen methods require independent measurement of the element vapour's coefficient of condensation on the cooled target.

We propose to use a "black body" trap cooled by radiation as a condensation target. If an atom gets inside such a trap it is not able to leave it. The trap could be hung by one of the shoulders of a quartz balance thus enabling to measure the vapour pressure in dynamics. With the help of an atomic beam chopper it is possible to measure separately a mass of a material collected by a trap as well as an impulse transmitted to the condensation target by the beam atoms and thus to get a real velocity of atoms in the beam. Using the methods of local chemical analysis to measure quantity of a material condensed on the condensation target and collected by the trap walls, we could determine rather accurately the condensation coefficient of the element under investigation. To determine this value more accurately, the atomic beam intensity should be monitored at the trap entrance by atomic adsorption or laser fluorescence.

The above method has been used to determine the saturated vapour pressure as a function of temperature for Ag, Mg, Zn, Pb.

THIN FILM/VACUUM METALLURGY Room A105 – Session TVFM-MoA

Diamond, Cubic Boron Nitride and Other Ultra-Hard Films I

Moderator: R. Messier, Pennsylvania State University.

2:00 pm TVFM-MoA1 Structural Control in the Vapour Growth of Oriented Diamond Films, P. Koidl, C. Wild, R. Locher, Fraunhofer-Institut für Angewandte Festkörperphysik, Tullastr. 72, D-79108 Freiburg, Germany.

We report on the growth of oriented diamond films by microwave assisted chemical vapour deposition from hydrocarbon/hydrogen gas mixtures. Both, fibre-textured films with a columnar grain alignment and hetero-epitaxially nucleated diamond films will be discussed. Starting from randomly oriented nuclei, a fibre-texture generally develops due to growth competition between differently oriented grains. We report on the growth of textured CVD diamond films with the fibre axis adjustable between (111), (110) and (100). In the latter case very smooth surfaces with almost coplanar (100)-surface facets have been realized. Using bias-assisted plasma-CVD, hetero-epitaxially oriented nucleation on large area substrates such as SiC and Si can be achieved. The overgrowth of those nuclei may lead to diamond films with preferential grain orientation both in normal and azimuthal direction.

The influence of the growth parameters on the structure and morphology will be discussed. It will be shown that twinning influences the structure of the films. The kinetics of diamond CVD and twin-formation has been studied and model calculations will be presented, describing the observed growth morphologies. Both, oriented growth and twin formation are determined by a growth parameter \( \alpha = \sqrt{3} \times V_{100}/V_{111} \), which in turn depends on the growth conditions.

The incorporation of electrically active defects like boron (acceptor) and nitrogen (deep donor) has been studied. Besides the electrical activity these dopants will be shown to cause structural changes of the diamond films. Under favourable conditions, an improvement of the orientational order of both fibre-textured and hetero-epitaxially oriented diamond films can be achieved.

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sugests that surface reactions on the diamond seed produce volatile species which diffuse to the substrate to form nucleation centers.


High quality diamond films have been synthesized in CH₄-H₂-O₂ mixtures at substrate temperatures down to 350°C, using a pulse-modulated electron cyclotron resonance (ECR) microwave discharge. The minimum temperature for diamond deposition was lowered by decreasing the gas pressure. High quality diamond films were grown at gas pressures of 0.3 to 3 Torr and substrate temperatures of 350 to 550°C by combining magnetic field discharge activation and plasma modulation. A static applied axial magnetic field established by a permanent magnet structure provided ECR enhancement, increasing gas dissociation and ionization by approximately an order of magnitude. Pulse modulation of the input microwave power provided high atomic hydrogen fluxes to the substrate without accompanying ion or electron bombardment fluxes over most of the discharage cycle. The low temperature diamond films exhibit sharp characteristic 1333 cm⁻¹ Raman phonon peaks with a small nondiamond carbon response; the linewidth is 4 cm⁻¹ at 350°C. Vertical growth rates range from 400 nm/min at 550°C to 100 nm/min at 350°C.


Diamond thin films were deposited on the various substrates (pre-treated glass, non-treated glass, pre-treated silicon, and pre-treated quartz) using a low temperature MPECVD (microwave plasma enhanced chemical vapor deposition) system. The substrate temperature can be lowered below 500°C by removing the substrate from the plasma. Among the various substrates, the diamond films form only on the pre-treated glass and the pre-treated quartz substrate. For the non-treated glass and the pre-treated silicon, the formation of graphite is an occurrence. Therefore we can confirm that the pre-treated glass and the pre-treated quartz substrate are promising substrates for the diamond formation at low temperature. In addition, we deposited the films on the pre-treated glass substrate by varying CH₄ concentration. The growth rate of the film increases with increasing CH₄ concentration. The best quality of the diamond film can be acquired at 4% CH₄. Microstructure of the films was also investigated by TEM. From these results, we will discuss the preferred formation of the diamond films on the pre-treated glass and the pre-treated quartz substrates.

4:40 pm TFVM-MoA9 Diamond Deposition on Carbon Fibers, S. Ismat Shah and M. M. Waite, E. I. du Pont de Nemours and Company, Central Research and Development, Experimental Station, P.O. Box 80356, Wilmington, DE 19880-0356 and Department of Physics and Astronomy, University of Delaware, Newark, DE 19716.

CVD diamond deposition process which involves preferential etching of non-diamond carbon phases, diamond deposition on carbon substrates is non-trivial. Thick graphite substrates can be used for deposition in which the initial etching of the substrate can be tolerated and as the diamond film forms, further etching of the substrate halts. Diamond deposition on low dimensional objects is difficult because the substrate gets etched away before the formation of a continuous protective diamond film. We have successfully deposited diamond films on carbon fibers using a pretreatment process which ensures fiber protection until a continuous layer of diamond has been formed on the fibers. Diamond films were deposited on these pretreated fibers by plasma assisted CVD. In this paper we will describe the pretreatment process and present the results of the characterization of the diamond coated carbon fibers. We will also discuss the optimum process parameters for the film deposition.

5:00 pm TFVM-MoA10 Carbon Dimer, C₂, as a Growth Species for Diamond Film Synthesis*, D. M. Gruen, C. D. Zulker, and A. R. Krauss, Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne, IL 60439.

It was shown recently that nanocrystalline diamond films can be grown using fullerene as precursors in an argon microwave plasma without hydrogen or oxygen additions [Appl. Phys. Lett. 62 (1993) 1404]. Extensive fragmentation of the C₆₀ in the microwave discharge leads to copious production of the carbon dimer molecule, C₂, as evidenced by intense Swan-band emission. We propose a mechanism for diamond film growth on the (100) surface using C₂ as the growth species. This species is known to reconstruct to the (100)-(2 × 1): H form, and at 1573K in vacuum to the (100)-(2 × 1) form, by loss of surface hydrogen. The latter reconstruction is the one most likely to obtain, under the high temperature (~850°C), argon atmosphere (100 Torr) deposition conditions characteristic of our plasma experiments. In the event, each surface carbon atom is bonded twice to carbons in the bulk and twice to a surface carbon (σ-bonding) making a “dimer” and forming a five-member ring. With C₂ as the growth species, no hydrogen abstraction reactions are required. Both of its very high energy of adsorption (813 KJ/mol C₂), the C₂ molecule can insert directly into the dimer bonds, and a second C₂ can add across the adjoining truong. The added carbons would then dimerize with their neighbors, forming a new (2 × 1) surface on a new layer, with dimer rows orthogonal to the original rows. Because the requirement for
We have shown that incorporation of NG groups increases both interfacial traps, \(D_i\) and fixed charge. In contrast, the controlled incorporation of N-atoms, which cannot participate in H-bond formation, generally decreases \(D_i\), and does not contribute to fixed charge in the dielectric near the Si-O2 interface.

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The mechanism of ultra-thin (1-5 nm) silicon oxide growth is still under intensive discussion. In this paper, we present results obtained with a novel method that sheds important new light on the controversy. Initial oxidation of Si(100) at 750-900°C. To help elucidate the mechanism of oxide growth, we have used medium energy ion scattering (MEIS) with \(^{18}\)O followed by \(^{16}\)O isotope oxidation. MEIS distinguishes \(^{18}\)O from \(^{16}\)O and gives the depth distribution for both in the thin oxide films with an accuracy of \(\sim 0.4\) nm. The isotope distribution calculated from deconvolution of an MEIS energy spectrum is found to be non-uniform, with isotopic mixing for very thin (1-4 nm) films, inconsistent with any existing model of silicon oxidation. For thicker films, sequential isotope oxidation results in an isotopic mixture near the surface, and Si\(^{18}\)O near the interface. We have also observed oxygen loss during oxidation.

*Supported in part by NSF DMR 90-19868 and 93-06899.

3:00 pm EM-MoA4 FTIR Study of Rapid Thermal Annealing of Remote Plasma Chemical Vapor Deposited a-si Nitride Films, Z. Lu, M. J. Williams, P. F. Santos-Filho and G. Lovascy, Departments of Physics, Electrical and Computer Engineering, and Electrical and Computer Engineering, North Carolina State University, Raleigh, NC 27695-8202.

Si-nitride films are widely used in the microelectronics industry. PECVD deposited silicon nitrides are known to have bonded hydrogen in the form of Si-H and Si-N-H. There is ample evidence that electrical performance of the Si-nitride films can be improved through high temperature rapid thermal annealing (RTA). This study uses FTIR to study behavior of bonded-H in Si-nitride films after RTA from room temperature to 1200°C, to establish correlations between electrical performance of Si-nitride and the amount of bonded hydrogen. The silicon nitride films were deposited using NH\(_3\) and SiH\(_4\) as source gases in a remote PECVD reactor. Different Si-nitride films were prepared ranging from Si-rich, to quasi-stoichiometric, and finally to heavily hydrogenated. The temperature dependencies for Si-H and Si-N-H reductions are different depending upon the initial bonding stoichiometry of the nitride film. A bonding model is proposed to account these results which includes the H-release with generation of molecular \(\text{H}_2\). Additional data will be presented for silicon nitride films made using N\(_2\) as N-source gas, where insights into the film and evolution chemistry can be gained. For near-stoichiometric nitrides, the Si-H bonding feature progressively decreases as the RTA temperature increases from room temperature to \(\sim 900^\circ\)C. At the same time, Si-N peak increases, indicating rebonding of nitrogen to silicon after bonded hydrogen (Si-H and Si-N-H) has been thermally released. This phenomenon is supported in Si-rich nitrides, where the initial Si-H and Si-N-H concentrations are comparable. These observations are consistent with electrical performance improvement of Si-nitride film. They indicate that the Si-H group, which is abundant in near-stoichiometric nitride films produced by low temperature PECVD is mainly responsible for the poor electrical performance. Reduction of Si-N by RTA significantly improves the quality of the PECVD nitride films grown from NH\(_3\) source gases.

3:20 pm Em-MoA5 Electrical Transport Properties of Hot Electrons at Metal, Insulator and Semi-Conductor Interfaces, R. Ludeke, IBM. T. J. Watson Research Center, P.O. Box 218, Yorktown Heights, NY 10598, and A. Bauer, Institut für Experimentelle Physik, Freie Universität Berlin, D-14195 Berlin, Germany.

Hot electron transport across interfaces is dominated by a variety of scattering processes that have been difficult to study by conventional techniques. The advent of a variant of the STM, Ballistic Electron Emission Microscopy, or BEEM allows the investigation of scattering phenomena and related issues in heterostructures over a broad electron energy range and at near-atomic resolution. A number of applications of BEEM spectroscopy over an energy range up to 10 eV will be
discussed. The technique has recently been used to show that transverse momentum is largely conserved for electron transport across the commensurate NiSi/ Si(111) interface, whereas it is not conserved for incommensurate metal-semiconductor systems. The energy dependence of elastic and inelastic mean free paths of hot electrons in Pd layers deposited on Si(100) and (111) substrates has been obtained as well. BEEM can be used to measure directly the quantum yield of electron-hole pair generation by impact ionization of injected hot electrons, which will be discussed for Si. Of current interest is the characterization of transport properties of thin insulating layers (2-6 nm) placed between the metal and the semiconductor. Transport takes place only through the electron injection into the conduction band of the insulator. The application of a bias across the insulator provides an additional variable to study the de-excitation processes of hot carriers in the conduction band of the insulator. Recent results for SiO2 interlayers will be presented.

4:00 pm EM-MoA7 Epitaxy in the Room Temperature Reaction of Ni on Si(111) Studied with Synchrotron X-ray Diffraction, P. Yang, M. Y. Lee and P. A. Bennett, Physics Dept., ASU, Tempe, AZ 85287; 1504, P. J. Eng, AT&T Bell Labs, Murray Hill, NJ 07974 and I. K. Robinson, Physics Dept., UI, Urbana, IL 61801.

We find that deposition of Ni on Si(111)-7 x 7 at room temperature produces a commensurate, epitaxial silicide at the buried metal/silicon interface. Intensity oscillations occur signifying a layerwise etching of the substrate, similar to the Pd/Si case [1]. Model calculations of the intensity profile along several crystal truncation rods allows a clear structure assignment as NiSi-theta. For the first several Å, the integrated intensities in the NiSi reflections account for all the deposited metal, i.e. no other phase forms. The structure factors show a Debye-Waller like variation against Q with an extremely large atomic displacement umax ~ 0.4 Å. Diffuse intensity appears throughout the Brillouin zone, suggesting a high density of point defects. The nature of disorder in the silicide film explains why epitaxy has not been seen at room temperature using other techniques such as MEB and LEED. Implications for low temperature epitaxial growth are discussed.


4:20 pm EM-MoA8 Phonon Scattering of BEEM Electrons at Au/Si(100) Schottky Interfaces, C. A. Ventrice, Jr., V. LaBelga, G. Ramaswamy, and L. J. Schowalter, Rensselaer Polytechnic Institute, Troy, NY 12180 U.S.A.

The relative importance of temperature dependent scattering mechanisms (i.e., acoustic phonon scattering within the metal overlayer and optical phonon scattering within the semiconductor) on ballistic electron emission microscopy (BEEM) ballistic electron transport across Au/Si(100) Schottky interfaces is not well understood. To better quantify the effects of phonon scattering, BEEM measurements on Au/Si(100) interfaces have been performed at both room temperature (RT) and 77 K for several Au overlayer thicknesses on n-type Si (ρ = 2–4 ohm-cm). The onset of the BEEM collector current, which determines the Schottky barrier height, was measured to be 0.82 eV at 77 K and 0.78 eV at RT. The observed shift in the onset of the collector current results from both a narrowing of the band gap of Si at elevated temperatures and thermal broadening of the tip's Fermi function at RT. Measurements of the average BEEM electron transmittance for energies up to 0.4 eV above the 0.8 eV Schottky threshold have revealed a reduction in BEEM electron transmittance at RT for each Au overlayer thickness. The measured relative transmittance at RT to that at 77 K ranged from 0.55 to 0.68 for Au overlayer thicknesses of 67 Å to 140 Å. Calculations of the temperature dependent attenuation length within the Au overlayer have indicated that multiple reflections of the BEEM electrons within the Au overlayer must be taken into account to explain the observed temperature dependence of the BEEM electron transmittance. (This research was supported in part by the U.S. Air Force and the Office of Naval Research).

4:40 pm EM-MoA9 Metal (CoSi2)/Insulator (CaF2) Resonant Tunneling Diodes and Transistors, M. Asada, M. Watanabe, T. Suematsu, and Y. Kohno, Tokyo Institute of Technology, Meguro-Ku, Tokyo 152, Japan.

Metal/insulator heterostructures can be considered attractive material for high-speed quantum electron devices, because high carrier density of metal and low dielectric constant of insulator are suitable for the size reduction and high-speed operation. Also, in certain material combination, strong quantum interference is expected due to very large band discontinuity at the heterointerface (~ 10 eV), which can be utilized for multiferroic and high-transconductance devices. In this paper, we report epitaxial growth of metal/insulator heterostructures and its application to quantum electron devices. CoSi2 and CaF2 were chosen for metal and insulator because of their small lattice mismatch to Si (~ 1.2 and +0.6%, respectively). CaF2 was grown at low temperature (~ 450°C) by the partially ionized beam epitaxy to suppress the thermal damage of CoSi2 underlayers. CoSi2 was grown by the two-step growth of Co and Si to 1 or 2 monolayers by MBE. By these techniques, a few nanometer-thick CoSi2/CaF2 multilayer was obtained on Si(111). Resonant tunneling diodes with two metal quantum wells showed negative differential resistance (NDR) at 77 K and room temperature. The change of the applied voltage at NDR with quantum well thickness agreed with theoretical calculation. Resonant tunneling transistors were fabricated by connecting the base electrode to one of the quantum wells of the resonant tunneling diode with high-selective wet chemical etching process. Transistor action with NDR was observed at 77 K. Depending on the choice of the two wells as the base, two different characteristics were obtained. Transfer efficiency close to unity was obtained for electrons from emitter to collector through the resonant levels in these transistors.
associated with this process are poorly understood. This paper reports simulations of collimated sputter-deposition of thin films in microscale trench using a two-dimensional molecular dynamics model (MD) and examines the effects of process parameters on film properties and step coverage. The microstructure, intrinsic stresses and step coverage are calculated as a function of working pressure, power input, and aspect ratio of the collimator. The numerical results demonstrate that the non-uniform flux coefficient strongly depends on the energetic neutral argon energy ejected from the target. The MD simulation technique is found to be helpful in optimization of the collimated sputter processes with high demands on uniformity and edge coverage. The simulation results for depositions in deep trenches are presented for a wide range of collimator aspect ratio, to explain the physics of the process.


Enormous development costs and excessive cycle times to transition new ideas into the manufacturing environment have limited semiconductor technology innovation. Through sequential and iterative process development, new process technologies require approximately 5 years to mature while process derivatives require 2-3 years to mature. This paper addresses use of advanced process and equipment models to synthesize leading edge manufacturing tools. It also presents the use of these models in an integrated environment to synthesize of new manufacturing processes. This approach, will combine a new set of programmable factory tools with a set of embedded manufacturing models and computer aided design tools. The resulting architecture will provide the capability to concurrently design a circuit along with its associated process, significantly improving the yield learning curve for new products in advanced technologies. The ability to make approximations trade-offs in performance, reliability, manufacturability, life cycle cost and cycle time will be possible with the availability of a process synthesis framework.

3:20 pm MS-MoA5 Comparison of the Sandia DSMC Molecular Flow Model to Experiment1, Paul K. Shufflebotham, Lam Research Corp., 4650 Cushing Pkwy., Fremont, CA 94538, Timothy J. Bartel, Sandia National Laboratories, Albuquerque, NM 87185-5800, Butch Berney, Lam Research Corp.

The Sandia DSMC rarefied gas dynamics model is used for the simulation of axisymmetric transition and molecular gas flow. This code has potential as a CAD tool for use in the vacuum design of low pressure plasma processing equipment, which typically operates in the transition flow regime. This assumes that the model is accurate and predictive. We have constructed an axisymmetric, transition flow test cell (VTG) to test the model through direct comparison with experiment. The VTC was equipped with a capacitance manometer, a mass flow controller and a 2000 1/s compound magnetically levitated turbopump. Spatially-resolved pressure measurements were made at dry N2 flows from 30 to 500 scm. The inputs to the model were the nozzle geometry and surface temperatures, mass flow rates and output pumping speeds. Tens of thousands of computational particles were tracked for hundreds of thousands of time steps. After a brief discussion of the known model and experimental uncertainties, the capability of the DSMC code to predict the absolute values, flow rate dependencies and spatial variations of the pressures measured in the VTC will be described in detail. The utility of the model as a vacuum system CAD tool will also be briefly discussed.

1Work supported by Lam Research, Sematech and DOE.


Plasma process non-uniformity is a major cause of yield loss during integrated circuit fabrication. Correlation of yield with chip placement on the wafer shows that yield falls off near the edge of the wafer, leading to an "edge exclusion" zone on the wafer. One possible cause of this is a non-uniformity in the ion flux. This is studied here by means of a 2-d self-consistent fluid model of a radio frequency discharge. Time-dependent electron and ion continuity, ion momentum balance, and electron energy balance equations are solved along with Poisson's equation. A semi-implicit time integration scheme and Galerkin finite elements are used to solve the equations.

The simulation domain includes electrode topography that simulates clamping and wafer edges. Results indicate that electrical property variation (e.g., conducting wafer to non-conducting clamping) causes the ion flux to fall off near the wafer edge. This may be partially responsible for the commonly observed edge exclusion on plasma processed wafers. Effect of the topography is related to plasma parameters, e.g., pressure, through the sheath thickness. Under high pressure (low sheath thickness) conditions, the ion flux is more sensitive to the topographical perturbation due to ionization rate enhancement in the sheath. At low pressure, topographical perturbation is more important. We also compare uniformity predictions obtained from the complete ion momentum balance to those from the drift-diffusion approximation (no ion inertia).

4:00 pm MS-MoA7 Simulation of a Tungsten Filled Via Process Module for Process Integration, D. S. Bang, K. Hsiao, J. P. McVitie, and K. C. Sarawat, Integrated Circuits Lab, Stanford University, CA 94305, Zoran Krivokapic, Advanced Micro Devices, P.O. Box 3453, Sunnyvale, CA 94088.

Tungsten is widely used for sub-micron VLSI via plugs because of the gap fill advantages of W-CVD over Al-PVD. A typical W-plug process contains multiple etching and deposition steps whose interactions become increasingly important in determining via characteristics as dimensions shrink. The use of computer simulation to examine equipment and process trade-offs for an integrated tungsten filled via process is demonstrated. Previous work in deposition and etching modeling has centered on developing topography models for specific process steps and equipment. This paper presents the development of modeling multistep fabrication processes for VLSI metallization. The interactions between different process steps during the fabrication of sub-micron Tungsten vias are examined. Process integration issues considered are the influence of oxide etch and glue layer deposition profiles on void formation during W-CVD, tradeoffs in collimated and non-collimated Ti/TiN PVD systems, and the effects of stop on oxide and stop TiN W-etchback profiles on subsequent metallization. Simulations are tested with experimental results.

4:20 pm MS-MoA8 Reactor- and Feature-Scale Simulation of Tungsten Chemical Vapor Deposition, A. H. Labun, Digital Equipment Corp., Hudson, MA 01749.

Models of tungsten chemical vapor deposition (WCVD) by the hydrogen reduction of tungsten hexafluoride (WF6) are presented which span both the reactor and device scales, providing the capability of predicting tungsten step coverage in features from reactor settings. Comparisons of model predictions with experiments are made at both scales. A chemical kinetic scheme consisting of 16 elementary gas-surface reactions has been adopted from the work of Arora and Pollard [1] simulating low pressure WCVD and extended to 40 Torr using SURFACE CHEMKIN software [2]. A one-dimensional, stagnation-flow model of a single wafer reactor predicts deposition rate over a wide variety of process conditions, including deviations from the desired zero-order dependence of deposition rate on WF6 partial pressure. The 'starvation' of the final elementary deposition reaction step for intermediate adsorbed WF, surface species, observed in the simulations, is evidently responsible for the resulting highly nonconformal deposition under these conditions. A simple model of an entire sub-micron feature as a single perfectly stirred reactor shows that as feature aspect ratio changes, so too does the internal chemical balance and hence the WF partial pressure at which deviations from the zero-order deposition rate begin. A submicron feature in the zero-order deposition rate regime may also be modeled as a pair of communicating perfectly-stirred reactors, the first extending approximately one mean free path from the feature opening and the second comprising the remainder of the feature, with the gas concentrations at the feature opening determined by the reactor-scale simulation. The variation in starvation conditions between the two reactors which model the feature correlates with observed step coverage, from which a sticking coefficient may be deduced. The resulting sticking coefficient-based deposition model for WCVD has been integrated into the collimated TiN emitter deposition, permitting the submicron plug-filling process to be handled by a standard topography simulator.


We present our physically based simulation package (EOLVE-
FLOW), which consists of EVOLVE [1] and a thin film thermal flow process simulation program. EVOLVE’s algorithms for simulating profile evolution during low and high pressure deposition have been discussed [2]. The thermal flow process is modeled as two-dimensional, incompressible, free-boundary flow, and has been discussed by Bornside et al. [3]. The flow simulator generates a mesh in the deposited film, and solves the Navier-Stokes equations using the penalty function finite element method. The deposition/flow process is divided into a number of time steps, and the location of the free boundary is determined as a function of time. Within each simulation time step, the local film deposition rates are determined by EVOLVE, then the thermal flow simulation program predicts the flow induced film velocity. These velocities are combined and the free surface profile is updated using the movement algorithms in EVOLVE.

As an example application of EVOLVE-FLOW, we present simulated film profiles during the simultaneous deposition and fusion flow planarization of borophosphosilicate glasses in high aspect ratio trenches. The process is based on tetraethylorthosilicate (TEOS)-silane chemistry with triethylborate and phosphine as dopant sources, and is conducted in a LPCVD reactor. We compare the planarization performance of the single step process to that of a two step process (deposition then reflow) in order to explain its superior trench filling capability.

1. EVOLVE is a deposition process simulator developed by T. S. Cale at ASU and Motorola, Inc. with funding from the SRC and NSF.

BIOMATERIAL INTERFACES
Room A106 - Session BI-MoA

Cell-Solid Surface Interactions

2:00 pm BI-MoA1 The Bone Cell/Biomaterial Interface, J. E. Davies, Centre for Biomaterials, University of Toronto, Ontario, M5S 1A1, Canada.

The future for Biomaterials lies in creating a marriage between materials science and biotechnology. Fundamental understanding of interfacial reactions between biological systems and artificial substrates will simultaneously lead to considerable advances in: (1) design of new materials for implantation in the body (Biomaterials), (2) creation of substrates for engineering of biological reactions in vitro (Biotechnology), and (3) creating substrates for diagnostic tests of biological processes in health and disease (Health Care). It has long been accepted that mechanistic explanations of reactions at solid liquid interfaces, within the biological milieu, will only emerge as a result of the development of similar events at surfaces. However, there is currently a wide gap between our understanding of the physicochemistry of surfaces and the application of this knowledge to interpretations of cell behaviour. This gap is slowly closing as a result of the considerable efforts of surface scientists who have applied themselves to biological interfacial problems, and the emergence of analytical techniques which provide information on the intact solid/liquid interface. Specific examples of the need for a surface analytical approach to understand the behaviour of both major bone cell types, osteoblast and osteoclasts, at surfaces will be provided. These examples will include studies of corrosion behaviour, as a function of passivation treatment, of metal alloys in biological systems; bone cell adhesion and cell spreading as functions of the charged, or polar, nature of a polymer substrate; changes in bone protein adsorption as a function of polymer surface modifications and the means by which bone growth can be dictated by materials surfaces.

2:40 pm BI-MoA3 Human Neutrophil Motility on Modified Surfaces, L. Harvath, N. E. Brownson, K. E. Foster, J. J. Hickman, CBER FDA, Bethesda, MD 20892, &SAIC, McLean, VA 22102.

Human neutrophil migration in the absence of a stimulus (random motility) and in the presence of various concentrations of a chemical stimulus (chemotaxis) was evaluated on modified and unmodified polycarbonate membranes. Neutrophils are the first blood cells found at an inflammatory site. The assay is routinely used to evaluate neutrophil migration through 5 µm pores in the polycarbonate membranes in response to medium alone or the chemottractant, N-formyl-methionyl-leucyl-phenylalanine (FMLP). Neutrophils that have migrated from the upper surface through the pores to the lower polycarbonate membrane surface are quantified by image analysis (Harvath et al., 1984, J. Immunol. Methods, 37: 39-45). To investigate the role of the surface in neutrophil response to a chemotactic agent, we modified the polycarbonate membranes with various self-assembled monolayers (SAMs). The silane monolayers used in this study contained various functionalities, including amines, thiols, and hydrophobic groups. The surfaces were analyzed by X-ray photoelectron spectroscopy and contact angle measurements. These modifications increased neutrophil random migration, in some cases, 3 to 4-fold over control responses. The surface modifications did not inhibit neutrophil chemotactic response to FMLP, though, amin-containing modifications substantially increased (>6-fold) the chemotactic response to suboptimal concentrations of FMLP. The significance of enhanced neutrophil migration to suboptimal concentrations of chemotactant may be directly relevant to in vivo responses to implanted materials.

3:00 pm BI-MoA4 Nanofabricated Structures for the Measurement of Signals from Single Cells, J. M. Cooper, A. Griffiths, and H. Morgan, Bioelectronics Group, Department of Electronics, University of Glasgow, Glasgow G12 8LT, U.K.

Nanofabrication is a technique which enables non-uniform electric fields generated at micro-electrodes to be used in order to guide or to position cells on the basis of their dielectric properties. By fabricating suitable geometries of such electrodes, it has proved possible to position individual cells within a two dimensional array of nano-scale amperometric biosensors. The sensors, which are based upon the immobilisation of cytochrome c on self-assembled thiyl monolayers on gold, have a potential application in the detection of superoxide for cell signalling. To this end, measurements have been made on stimulated single neutrophils.


Methods to effect specific cell adhesion to biomaterials is an area of intense investigation. In addition, recombinant protein technology has created a number of new biological drugs for diagnosing and treating disease. Combination of the products of biotechnology with biomaterials research has created an exciting new field of hybrid medical devices. A hybrid medical device can be defined as any medical device that uses a biological material, usually a recombinant protein, to effect its intended medical application. The current investigation will be a case study of a medical device used in human clinical trials to develop immune stem cells and CD4+ lymphocytes. The discussion of the methodology employed in the development of these highly specific cell adhesion devices will highlight a new type of biomaterial, surface analysis techniques such as ESCA and Attenuated Total Reflectance FT-IR and the use of monoclonal antibodies directed to cell surface antigens. Preliminary results of human clinical trials will be presented.

4:00 pm BI-MoA7 Nanofabricated Fibers for Studying the Phagocytosis of Inorganic Particulates by Macrophages, J. Gold, B. Nilsson, and B. Kaseemo, Chalmers University of Technology and University of Göteborg, 412 96 Göteborg, Sweden.

Inorganic particulates occur in the body due to, e.g., wear debris generated by articulating surfaces in orthopaedic implant devices, or by inhalation of mineral dusts and fibers. It is important to understand that these particulates can lead to failure of the implant and lung diseases, respectively. Macrophages attempt to remove particulates from the body via phagocytosis into the cell, and send out signals indicating the presence of either an inert or a toxic foreign object. Several physical and chemical characteristics of the particulates appear to play a role in determining this phagocytic response of macrophages. Typically, particulates used in the aforementioned studies are produced by grinding raw/bulk materials, spin coating, or atomization. However, these techniques generate inherent distributions in the size, shape and chemical composition of particulates even within a given production lot. In the present work we have produced fibers having controlled shapes, dimensions, sized distributions, and compositions for studying the role of these factors in the cellular response. Nanofabrication techniques, fibers can be made in a large number of materials and in a wide range of dimensions (nm to mm’s). Significant quantities for cell culture studies can be made with tight control over the fiber size distribution.

T20, SiO2, and Au fibers of dimensions 0.1, 1 and 10 µm, have been
produced and are currently being used in cell culture studies of the phagocytic response of alveolar macrophages. The preparation and characterization of these fibers will be presented. Reference will be made to the ongoing cell culture studies at the Dept. of Environmental Medicine, Univ. of Gothenburg, Sweden.


The adhesion and activation of platelets and leukocytes at blood-material interfaces was studied by fluorescence microscopy, and photometry using specific anti-CD antibodies, anti-plasma protein antibodies and the calcium ion probe Fura-2. Gold or titanium were evaporated onto glass slides and capillary blood was placed as droplets on the surfaces in a humidified chamber. The adsorption of plasma protein was monitored with FITC-labelled antibodies and the adhesion of platelets was shown by anti-CD 61 antibodies, specific for this cell type.

Adhering platelets were found after 15 seconds incubation time on both surfaces, and the number of cells increased slowly. The cells spread over the surface, aggregated and developed stress fibers in contact with the surface. The distribution of fibrinogen was heterogeneous with higher surface concentration in connection with adhering cells. Platelet-derived microvesicles were found after 8 minutes of incubation. These microvesicles showed intense staining with anti-C3c antibodies.

In other experiments, granulocytes were isolated and incubated with Fura-2. The supernatant of hirudin-treated blood, incubated with material surfaces was added to the cells and the fluorescence was recorded after emission at 340 and 380 nm. A rapid peak was shown indication influx of Ca++ into the cytoplasm. The results suggest a functional relationship between adsorbed plasma proteins, platelets and leukocytes, which may be important in the non-self recognition of foreign materials.

4:40 pm  BI-MoA9  Effect of Surface Mobility on Biointeractions, B. D. Ratner, C. D. Tidwell, Univ. of Washington, Seattle, WA 98195.

Correlations between biomaterial surface properties and biological responses involves consideration of many factors including surface chemistry, charge, and morphology. One parameter affecting biointeractions which has received limited attention is surface mobility, due in part, to difficulties in making well-defined, mobile surfaces. Molecular self-assembly provides an effective means of fabricating organic surfaces with well-defined structure and chemistry for elucidating relationships between surface structure and biointeractions. "Mixed" self-assembled monolayers (SAMs), prepared by the coadsorption of alkaneethiols of differing alkyl chain length onto gold, form monolayers comprised of a highly ordered inner region and a disordered, liquid-like outer region. Using a series of mixed SAMs with varying interfacial disorder, biointeractions can be investigated.

A series of mixed SAMs were formed by competitive adsorption of 1 mM ethanol solutions of mixtures of octadecyl mercaptan and dodecanethiol onto evaporated gold substrates. SAM surface composition, compositional depth profile, and thickness were determined by X-ray photoelectron spectroscopy (XPS). Monolayer wetting characteristics were assessed by contact angle goniometry. Biointeraction characteristics of the mixed SAMS are being investigated by albumin and fibronecin adsorption and elutability studies followed by a determination of endothelial cell growth on the SAMs.

Thicknesses of the hydrocarbon portion of the monolayers, as determined by XPS, were approximately 13-19 A. Cosines of the advancing contact angles for water and the natural logarithm of the ratio of C(1s) and Au(4f) XPS intensities varied in a sigmoidal fashion with the log of the ratio of components in solution. A solution concentration ratio of 5:1 (Dodecanethiol/octadecyl mercaptan) was required to obtain a SAM composed of an equimolar mixture of the two components.

5:00 pm  BI-MoA10  Secondary Ion Mass Spectrometry Studies of Covalently Bound Peptides to a Fluoropolymers: Imaging and Quantitation, Evan J. Bekos, Frank V. Bright, and Joseph A. Gardella, Jr., SUNY Buffalo, Chemistry Department, Buffalo, New York 14214.

We have recently reported the covalent binding of peptides, by both the N- and C-termini, to a modified fluoropolymer. The binding of a pentapeptide, YIGSR (Tyr-Ile-Gly-Ser-Arg), has been analyzed and quantified using a variety of surface sensitive techniques: secondary ion mass spectrometry (SIMS), electron spectroscopy for chemical analysis (ESCA), and attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR). In the current study, we are using SIMS to analyze and quantify a broad range of peptide sequences bound to a fluoropolymer substrate. The higher mass fragments unique to each peptide will be used to analyze mixtures of peptides. In the case of the unmodified patterned surfaces, images of peptides restricted to defined domains will be presented. Also, an established quantitative method will be used for these peptide mixtures. The use of these peptide-modified materials in neural cell tissue regeneration is currently being explored.

SURFACE SCIENCE
Room BR4 – Session SS-MoP

Aspects of Surface Science
Moderator: J. C. Hemminger, University of California, Irvine.


In situ, real-time observations of sulfur diffusion from bulk Mo(310) to the oxygen covered Mo surface with the photoelectron emission microscope show two major routes for sulfur transport on the O/Mo(310) surface. One type is characterized by annular structures centered on impurity particles. The second type is in the form of a two-dimensional front of macroscopic dimensions (several millimeters) extending over the crystal surface. Both annular and front-like features exhibit well developed, structured leading edges (see micrograph). Post-reaction Auger analysis confirms the removal of oxygen from the substrate and reveals sulfur wherever oxygen was removed. Low-energy electron diffraction shows an ordered sulfur overlay. We present data suggesting that the oxygen surface concentration regulates sulfur transport to and over the surface.

1 Field of view is 260 μm.
Supported by ONR/BMDO Grant No. N0014-91-1596.

SS-MoP2 Multi-Bounce Direct Scattering of N₂ from Cu(110), J. L. W. Siders and G. O. Sitz, Department of Physics, University of Texas, Austin, Texas 78712.

Resonantly enhanced multiphoton ionization and time resolved molecular beam techniques were used to study the rotational state and velocity distributions of N₂ scattered from Cu(110). The final rotational temperature of the scattered molecules was found to depend linearly on incident energy, Eᵣ, indicating a direct scattering process. Unlike the previous results of N₂ scattering on silver (Sitz, J. Chem. Phys. 89, 2558, tungsten and platinum (Hanisch, J. Chem. Phys. 99, 7076), the surface temperature dependence is found to be strong on copper. An anticorrelation between the final translational energy and final rotational state, J, was present for an incident energy of 0.79 eV, but was washed out at 0.09 eV. For Eᵣ = 0.09 eV the N₂ molecules scattered into the highest J states have more final rotational energy than incident energy available, which suggests that surface energy is being converted into rotational energy. The final total energy remains less than the incident energy for Eᵣ = 0.79 eV, therefore energy is being transferred to the surface. Rotational rainbows were observed at high incident energies (>0.25 eV), but were washed out at low energies and low surface temperatures (300 K). As the surface temperature is raised the rotational rainbow becomes more pronounced. The strong surface temperature dependence and absence of rotational rainbows at low Eᵣ and Tₛ is attributed to multiple bounces which appear to play a large role in the dynamics and energy transfer of the N₂/Cu system. Work supported by the Robert A. Welch Foundation, grant F-1198.

SS-MoP3 Dynamics of D₂ Recombinative Desorption from Ag(111), R. N. Carter, F. Healey, and A. Hodgson, Surface Science Research Centre, University of Liverpool, Liverpool L69 3BX, U.K.

The interaction of deuterium atoms with Ag(111) has been investigated with temperature-programmed desorption (TPD) and low energy electron diffraction (LEED). Atomic deuterium is found to adsorb to the surface at a temperature near 110 K, resulting in a TPD feature near 185 K at lowest coverages. With increasing exposure, this feature shifts up in temperature to an ultimate value of 220 K and is joined by two lower temperature peaks near 200 and 180 K. The lowest temperature feature does not saturate but grows linearly with exposure and is attributed to deuterium atoms absorbed into the subsurface. LEED shows a (2 × 2) pattern at 110 K for low coverages which is preserved only at the highest temperature peak observed in TPD. At coverages above this, a superposition of (2 × 2) and (3 × 3) patterns is observed at 110 K, which reorder to (4 × 4) after annealing at 150 K. The angular distribution of the low coverage desorption peak is sharply peaked with respect to the surface normal, with a P(0) = cosθ distribution.

The implications of these results for the D₂-Ag(111) potential energy surface as well as the kinetic analysis of the TPD will also be discussed.

SS-MoP4 Epitaxial Domains of C₆H₆ on Ag(111), J. E. Rowe, R. A. Maier, E. E. Chaban, and C.-M. Chiang, AT&T Bell Laboratories, Murray Hill, New Jersey 07974.

Epitaxial C₆H₆ films have been grown on Ag(111) substrates for thickness up to ~100 monolayers (ML). For ultrathin layers (0–2 ML) we observe growth of (111) layers of C₆H₆ which have a (2√3 × 2√3)R30° superstructure with respect to the fundamental Ag(111) 2D lattice. Low-energy electron diffraction (LEED) with a computer controlled CCD camera allowed accurate images and angular profiles to be measured. Two domains are observed at low electron energies, E ≤ 40 eV as well as multiple scattering between domains due to an anti-phase boundary structure. These thin layers do not exhibit the low temperature phase transition associated with orientation ordering in the bulk. This is likely due to the strong C₆H₆-Ag(111) interaction previously reported by Altman and Colton [1]. As growth continues above 2 ML a gradual transition occurs to a twinned (111) layer structure which interacts less strongly with the substrate. These thicker layers display the low temperature phase transition associated with orientation ordering in the bulk. However, the surface of these films show antiphase domain effects as well as the twinning with respect to the Ag(111) substrate. Domain sizes of the twinned commensurate structure are in the range 40–50 Å which is more than a factor of two smaller than the best epitaxial films grown on Cu(111) substrates. Soft X-ray photoemission of the C-1s core level shows an asymmetric shape similar to that of C₆H₆ on Cu(100) which we interpret as evidence of local orientation ordering as reported for epitaxial films grown on Cu(111).

The shift with respect to the bulk C-1s position can be interpreted as a charge transfer of ~1 electron per C₆H₆. This local ordering may be the cause of the anti-phase domains observed with LEED.


Spin coating is a well known technique for the preparation of thin polymeric photoresist layers on silicon wafers. Recently, the method has attracted attention as a highly useful procedure for the deposition of thin layers (between one and several monolayers) of inorganic salts on flat supports, of interest as models for catalysts. Here we show that a slightly modified theoretical description of the spin coating process predicts the amount of deposited material within 15% accuracy, on the basis of the following parameters: evaporation time, density, viscosity and concentration of the solution, and rotor speed.

The method has been applied to deposit submonolayers of molybdenum and chromium on flat oxides of silicon and aluminium. Rutherford Backscattering Spectrometry (RBS) and X-ray photoelectron spectroscopy (XPS) have been used to determine the amount of deposited material.
SS-MoP7 Interaction of Fullerene with the Cu(111) and Ag(111) Surfaces, T. Sakurai, X.-D. Wang, T. Hashizume, J. Kishimoto, S. Yamazaki, Y. Uray, H. Shinohara, and H. W. Pickering, Institute for Materials Research (IMR), Tohoku University, Sendai 980-77, Japan, *Faculty of Science, Nagoya University, Nagoya 464-01, Japan, *Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA 16802, USA.

Adsortion and the monolayer film formation of fullerenes (C_{60}, C_{70}, C_{60}C_{60},C_{70},SC_{60}, etc.) on the Cu(111) 1 X 1 and Ag(111) 1 X 1 surfaces have been studied by the field ion-scanning tunneling microscopy (FISTM). The fullerene molecules are mobile on the terrace at room temperature, segregate to the steps and form two-dimensional islands with a close-packed configuration. Monolayers of the C_{60} and C_{60}C_{60},C_{70},(x>30%) on the Cu(111) surface form a highly ordered 4 X 4 commensurate phases and the intramolecular structures are conserved. The interaction between fullerene and the Cu substrate to suppress fullerene's internal rotation. The intramolecular structures are interpreted in terms of the local density of the states of the C_{60} and C_{70} molecules.

For the case of the C_{60}Ag monolayer film on the Ag(111) surface, we observe eight different phases having a similar nearest neighbor distance but distinctly different rotation angles with respect to the substrate, similar to the case of the C_{60} adsorption on the Ag(111) surface. These indicate that the interaction between fullerene and the Ag(111) surface is weak comparing with that among fullerene molecules. The rotation angle is dictated by the adsorption configurations which produce higher number coincidence lattice sites.


We have used a UHV Scanning Tunneling Microscope (STM) to study the atomic scale structure of TiO_{2}(110) (|1 X 1|) and the nature of defects on this surface. Samples have been prepared which are atomically flat over large areas, and on which the (1 X 1) structure is resolved with STM. On some samples, we also find a highly elongated defect structure in addition to the (1 X 1) structure. These rowlike structures (2 A in height and 12 A FHWM) are oriented along the [001] direction in registry with the (1 X 1) structure and can extend for up to 50 A when intersecting a step edge. We have also studied the morphology of Cu overlayers on this surface. For deposition at 300 K, Cu is found to form three dimensional islands in the low coverage regime. Upon annealing, a coarsening process is observed in which the size of an average cluster increases. The clusters are found to be weakly bound to the substrate and are easily moved with the STM tip.

**Supported by NSF DMR 90-19868 and 93-06899.


These experiments examine the effect of low energy atomic and molecular ion beam exposure upon self-assembled monolayers of alkanethiols. Methane and hexanethiolate adsorbates on Cu(111) are exposed to low energy (<100 eV) molecular ions of pyridine, furan, and thiophene. The degree of ion beam damage of the adsorbate is then measured by infrared reflection absorption spectroscopy. Ion beam damage of the adsorbate ranges up to 50% for exposures of ~10^{12} ions/cm^{2}. More recent experiments utilize x-ray photoelectron spectroscopy to probe the effect of low energy Ar", O", and N" ions on alkanethiolates adsorbed on polycrystalline gold surfaces. These results indicate that low energy ion beams can be utilized in lithographic applications via modification of self-assembled monolayers for selective electronless metal deposition.


The bonding and reactivity of methanol and methyl iodide adsorbed on Mo(110) and benzyl alcohol and benzyl iodide adsorbed on sulfided Mo(110) were investigated using temperature programmed reaction, Fourier transform infrared reflection adsorption and electron energy loss spectroscopies in combination with isotopic labelling studies. At low coverages, methanol adsors in a pseudo C_{3v} geometry, based on the observation of a single C-H stretch at 2930 cm^{-1} in the infrared spectrum of CF{sub 3}D{sub 2}O. At high coverage two methoxy species are apparent, both with C_{3v} symmetry, based on the appearance of two distinct symmetric C-H stretching modes as well as two asymmetric C-H stretching modes in the infrared spectra. The average tilt angle of these species is estimated to be ~30 ± 10°, from the relative intensity of the symmetric and asymmetric deformation modes. The C-O bond is found to be extremely anharmonic, based on the observation of the overtone of the C-O stretch with an anharmonicity of ~50 ± 10 cm^{-1}. We estimate a C-O bond energy of ~20 kcal/mol, assuming a Morse potential. Surface methylene exhibits considerable C-H bond softening on adsorption on Mo(110). The molecular symmetry is lower than C_{3v} over the entire coverage range, based on the dipole character of both the symmetric and asymmetric C-H stretching modes in the electron energy loss spectrum. Facile conversion to surface methylene occurs below 250 K. We propose that the methylene groups bridge along the [001] direction by strong monodentate hydrogen bonding, which rules operative in electron energy loss spectroscopy. Phenyl disulfide is formed via S-H bond scission and S-S bond formation in benzothiophen on 0.35 ML Mo(110) at 100 K. The S-S separation is oriented perpendicular and the phenyl ring parallel to the surface, based on electron energy loss spectroscopy. The disulfide subsequently forms an upright phenyl thiolate species, prior to the onset of gaseous benzene and phenyl formation.

SS-MoP11 Hydrogenation of Cyclohexene by Subsurface Hydrogen on Ni Surfaces, Gyeong-A Son, Manos Mavrikakis, John L. Gland, Department of Chemistry, University of Michigan, Ann Arbor, MI 48109.

Subsurface hydrogen can be formed at low pressure by absorption of pre desorbed cyclohexene in Ni(100) and Ni(111) samples. Surface hydrocarbon selectively hydrogenates cyclohexene to benzene (T epithermal) at surfaces at about 176K. In contrast, no hydrogenation by surface hydrogen is observed. Desorption of molecular cyclohexene dominates on both Ni(100) and Ni(111) surfaces in the presence of surface hydrogen. Dehydrogenation of cyclohexene to benzene is a dominant thermal path way on both surfaces in the absence of hydrogen. Re- action mechanism of cyclohexene hydrogenation was suggested by TPD, EELS and isotope experiments. We have demonstrated that absorbed hydrogen is quite reactive even at low temperature. Temperature programmed desorption and high resolution electron energy loss spectra were combined to explore the dynamics of surface and bulk phenomena involved.

SS-MoP12 FT-IRAS of Adsorbed Alkoxides: Methoxide and Ethoxide on Cu(111), Shane C. Street, University of Illinois-Urbana-Champaign 61801, and Andrew J. Geiman, Carnegie Mellon University, Pittsburgh, PA 15213.

The surface chemistry and structure of methoxide and ethoxide adsorbed on Cu(111) has been studied by FT-IRAS and TPD. The structure of methoxide on Cu(111) has been a matter of debate. Previous IRAS results were interpreted to show that the C_{3v} axis of methoxy at saturation coverage lies perpendicular to the surface. Subsequent X-ray diffraction and backscattering photoelectron diffraction experiments support this orientation. However recent work with methoxy cluster compounds has led to the proposal that at low coverages methoxy is tilted, and that sterically crowding reorients the molecular axis towards the surface normal at saturation. We present high resolution spectra for methoxy at a range of coverages. For the more complex ethoxy adsorbate we present the FT-IRAS spectra for a set of seven deuterated and isotopically (13C) labelled ethoxides. This is by far the most detailed labelling study to assign vibrational modes of an adsorbate. The C_{3v} axis of the methyl group of ethoxy is found to lie roughly parallel with the surface.


Temperature programmed static secondary ion mass spectrometry (TPP(SIMS)) and temperature programmed desorption (TPD) have been used to study the kinetics of adsorption, dissociation, and desorption of NO on Rh(111). At 100 K, NO absorbs molecularly via mobile precursor state kinetics. SSPSIMS suggests threefold dissociation at low coverages with increasing dissociation of NO as the high coverage. Three characteristic coverage regimes appear with respect to NO dissociation. At coverages below 0.25 ML, NO dissociates completely at temperatures between 275 and 340 K, with an activation energy of 39 ± 6 kJ/mol, and a preexponential factor of 10^{13} s^{-1}. Nitrogen adsorb as N=, with kinetics that is strongly influenced by the presence...
of coadsorbed oxygen. In the medium coverage range, 0.25 < θ_{NO} < 0.50 ML, part of the NO desorbs molecularly, with a desorption barrier of 116 ± 10 kJ/mol and a preexponential of 10^11 s^{-1} cm^{-1}, suggesting a mobile transition state. Desorption of NO becomes progressively inhibited in the fast NO dissociation channel, which yields 273 K at 0.25 ML to 400 K, the NO desorption temperature, at a coverage of 0.50 ML. On these highly covered surfaces nitrogen atoms become destabilized, resulting in an additional low temperature N_2 desorption step. For initial NO coverages higher than 0.50 ML, NO dissociation is completely self-inhibited, indicating that all sites required for dissociation are blocked. The NO desorption of the most highly covered Br Industries NO does not generate the sites required for dissociation; these become only available after the desorption of triplet coordinated NO.

SS-MoP14 Two-Dimensional Surface Interactions and Dynamics of Benzene on Cu(111), M. M. Kannan, S. J. Stranick, and P. S. Weiss, Department of Chemistry, The Pennsylvania State University, University Park, PA 16802.

We seek to understand the interactions that determine the chemistry, structure, and dynamics of surface adsorbates. We have used ultrahigh vacuum scanning tunneling microscopy to study the low temperature behavior of benzene adsorbed on Cu(111). Electron standing waves are apparent in STM images of Cu(111) at low temperature due to scattering of surface states from step edges, defects, and benzene molecules. The resulting charge density modulations influence the binding of the benzene molecules. We observe that at low coverages benzene adsorbs along Cu step edges and at other high charge density sites. The molecules form a tightly bound 2-D solid at the step edges while benzene molecules on terraces move across the surface as a 2-D molecular gas. At the interface between the solid and gas we observe 2-D adsorption and desorption in real time. We discuss the relative strengths of the interactions at various step and terrace sites due to these nanometer scale variations in the surface electronic structure.

This work was supported by BRDC, NSF, and ONR.

SS-MoP15 RAIRS of Rh Single Crystal Surfaces During NO Reduction at Moderate Pressures, D. N. Belton, H. Pernman, S. J. Schmieg, General Motors R&D, Warren, MI 48090-9055, and K. Y. Simon Ng, Wayne State University, Detroit, MI 48202.

Although CO and HC emissions from passenger cars are primarily determined by the amount of time required to heat up the catalyst, NO emissions tend to occur primarily under conditions where the catalyst is fully warmed-up. This simple observation means that NO reduction kinetics play an important role in determining the amount of NO that will escape from the catalytic converter of a vehicle. Our research has focused on obtaining a detailed kinetic mechanism that can quantitatively describe NO reduction kinetics. To achieve this goal we have measured reaction kinetics at pressures realistic for automotive exhaust and modeled those kinetics with a mechanism consisting of elementary steps and UHV-measured rate constants. Currently we reproduce the observed reaction rates using reasonable rate constants that can be justified on the basis of UHV measurements; however, we have not been able to measure the rates of every elementary step at all applicable coverages. In order to constrain the permissible surface coverages in our model, we have recently used reflection-absorption infrared spectroscopy to measure CO and NO coverages during the NO + CO + NO + CO + O_2 reactions over Rh single crystals. Our results show that there is both NO and CO on the surface under most reaction conditions. Further, as the reaction temperature or the CO pressure is raised from the dissociation pressure, there is a strong tendency for CO to displace NO from the surface. These measurements will be put in context of the reaction mechanism and the effect of these experimental measurements on our modeling efforts will be discussed.

SS-MoP16 Vibrational and Electron Spectroscopic Study of the Interaction of Nitric Oxide with the NH(110) Surface, D. H. Hollowell, C. M. Affentrautz, and J. E. Crowell, Department of Chemistry, University of California at San Diego, La Jolla, CA 92039-0314.

The understanding of NO reactions with metal surfaces is of fundamental importance to the development of efficient catalytic systems for automobile exhaust pollution control. In an effort to understand these processes we have studied the reaction of NO with clean, as well as oxygen and hydrogen-prepared, NH(110) surfaces. At saturation NO coverage, reflection-absorption Fourier transform infrared spectroscopy (RT-RAIRS) shows two distinct NO stretching vibrations: a sharp, intense peak at 1867 cm^{-1}, assigned to atop site adsorption, and a broad medium strength peak at 1678 cm^{-1}, which we have assigned to bridge site adsorption. Additionally, a weak, broad band at 1717 cm^{-1} is observed; this feature may be a result of NO adsorption at defect sites. The coadsorption of hydrogen suppresses the higher frequency (1867 cm^{-1}) peak, thus stabilizing the bridge site, while O_2 preadsorption favors NO adsorption at atop sites. X-ray photoelectron spectroscopy (XPS) analysis of the above surfaces suggests that at least two types of adsorption occur. This data, together with the RT-RAIRS data, indicates that NO desorption depends upon initial NO exposure, with NO desorbing at 125 K, with molecular adsorption possible with increasing exposure after a critical coverage of N and O atoms at the surface is reached. Also, it is found that NO decomposes at the surface with heating, leading to little or no NO molecular species at the surface above 400 K. With further heating, the O atoms remain on the surface, while the N atoms combine to desorb as N_2 by ~350 K. The implications of the above results will be discussed.

SS-MoP17 The Role of C in O Removal from Ultra-Thin Layers of Oxidized La on Pd(100), G. W. Graham, B. U. M. Rao, and M. Ahmad,* Ford Motor Company, MD 3179, Bldg. SRL, P.O. Box 2053, Dearborn, MI 48121, Wakey State University, Department of Physics and Astronomy, Detroit, MI 48202.

The complete removal of O from ultra-thin layers of oxidized La on Pd(100), effected by heating to temperatures as low as 625 K in UHV (1,2), was found to be influenced by C present at levels not detectable by Auger electron spectroscopy due to interference with Pd. Measurements in which Pd was replaced by Rh, where correspondingly small levels of C could be detected, show that the Rh is able to reduce oxidized La under such conditions. The elimination of C from the Pd(100) surface by means of heat treatments in O_2 (tested by temperature programmed desorption of CO) only causes a modest increase (100 K) in thermal stability of the oxidized La, however, and the thermal stability of ultra-thin layers of oxidized Al was essentially unchanged. A difference in sensitivity to C of the thermal stability of oxidized La versus Al layers on Pd could be indicative of a difference in the interfacial structure which might also account for other known differences in chemisorption and sintering behavior.


SS-MoP18 A Study of Oxygen Chemisorption Phases on Pt(111), C. Pagila, A. Nilsson, B. Hernnndez, K. O. Karis, P. Bennich and M. Martensson, Department of Physics, Uppsala University, Box 530, S-751 21 Uppsala, Sweden.

O_2/Pt(111) is a system that has been object of a great number of studies during a long period of time. We now present new results concerning the identification and characterization of three different chemisorption states studied by means of different spectroscopic techniques such as XPS, XAS and DES. From our data we can conclude that these oxygen chemisorbs on Pt(111) in two different molecular phases which are precursors for the thermal activated dissociation process. A first molecular state is obtained at a temperature of about 90 K. It is weakly chemisorbed and we have identified it as a superoxo-like configuration of the oxygen molecule (O_2^-). At around 135 K a second molecular phase, more strongly involved in the hybridization with the substrate, has been characterized as a peroxy-like configuration (O_2^-). The atomic phase, with the characteristic (2 × 2) LEED patterns, occurs at a substrate temperature higher than 150 K. For this phase we will discuss the results according to a model which describes the density of states induced by the hybridization of the oxygen 2p orbitals with the 6sp-states and 5d-band of the metal.

SS-MoP20 Selective Oxidation Reactions on Rh(111)-(p2 × 1)-O: C. W. J. Bol and C. M. Friend, Department of Chemistry, Harvard University, Cambridge, MA 02138.

Oxygen addition reactions on Rh(111)-(p2 × 1)-O were investigated using temperature programmed reaction and high resolution electron energy loss spectroscopy. Ketones are formed in the oxidation of secondary thiols and halides. Olefin elimination and decomposition are competing pathways. The selectivities strongly depend on the initial oxygen coverage. At low oxygen coverage no ketone production is observed and olefin elimination and decomposition are the major pathways. Small amounts of the corresponding alkanes are also formed.

Vibrational studies show that the, at low temperature formed, thiols and the alkyl halides stay intact up till the reaction temperature. Carbon-sulfur (carbon-halide) bond cleavage is proposed to be the
rate determining step in the formation of both ketones and olefins. The reaction temperature correlates well with the C-X bond strength in different secondary thioles and alkylhalides. Further studies will address the nature of the C-X bond cleavage; homolytic, resulting in a radical, or heterolytic, forming a carbocation.

The proposed mechanism is different than those observed in the oxidation of secondary alkoxydes on Rh(111)-(p)2 x 1)-O. Here β-hydrogen abstraction leads to the formation of ketones and reaction with surface oxygen at higher temperature gives rise to a surface carboxylate, which subsequently reacts to form a ketone. These mechanisms are not feasible in the oxidation of secondary thiocarbanion and halides because of geometric reasons.

SS-MoP21 The Early Stages of Ruthenium Oxidation, J. Hrubek, D. van Campen and I. Mail*, Chemistry Department 555, Brookhaven National Laboratory, Upton, NY 11973-5000; *MEMC Electronic Materials, P.O. Box 8, St. Peters, MO 63376.

Although Ru is known to be an excellent catalyst for several processes, it is not used by the industry because it forms volatile oxides. In order to understand the initial stages of the process, we studied the interaction of atomic oxygen with a Ru(001) surface by X-ray photoelectron spectroscopy, thermal desorption and electron diffraction. Atomic oxygen reacts with ruthenium in three different stages: chemisorption is followed by subsurface diffusion and oxidation. Large concentration of oxygen can be dissolved in the subsurface region without a noticeable change of the Ru core levels. We observe the onset of reaction of dissolved oxygen with the Ru host only for concentrations corresponding to equivalent coverages of more than ~50 ML. A new Ru 3d core level shifted by 0.50 eV toward higher binding energy, grows with increasing oxygen exposure. An additional weak feature 2.6 eV below the metallic Ru 3d binding energy appears for higher oxygen coverages. The asymmetric O 1s peak shows a shoulder 2.0 eV higher than the main oxygen level. At the highest coverage studied (~870 ML) almost all the Ru metal within the probing depth of the photoelectrons is oxidized; the binding energy of the main component agrees well with previous measurements on RuO2. Completely diffused LEED patterns show that the surface is disordered for coverages higher than 3 ML. The oxidized Ru surface is thermally stable up to 950 K with the majority of oxygen desorbing between 950 and 1150 K. Heating the Ru crystal to 1560 K removes all detectable oxygen and restores the perfect structural order of Ru(001).

This research was carried out at Brookhaven National Laboratory under contract DE-AC02-76CH00016 with the U.S. Department of Energy and supported by its Division of Chemical Sciences, Office of Basic Energy Sciences.


XPS shows that the surface properties of Co/SiO2 catalysts exhibit a strong dependence on catalyst pretreatment conditions with the formation of cobalt oxide, cobalt silicate, cobalt silicide, and metallic Co determined by the activation environment. Heating a Co(NO3)2*6H2O precursor on a silica support at 400°C in air, after drying at 100°C in either air or vacuum, produces CoO that is easily reduced in H2. In contrast, samples dried at 100°C and then annealed in UHV to 400°C exhibit drastically different characteristics. Initially, the cobalt phase on the dried samples is a surface silicate. Annealing air dried samples in UHV converts the surface silicate to an irreducible bulk cobalt silicide. Remarkably, UHV annealing of vacuum dried samples promotes the formation of a cobalt silicide. The nature of the Co species formed depends upon the concentration of nitrate, H2O, and O2 species at the surface during the decomposition of the precursor. For example, conversion of the intermediate surface silicate to silicide in UHV entails reduction by NO generated from nitrate decomposition. The implications of these results for the CO hydrogenation activity of Co/SiO2 catalysts will be discussed.

This work was performed at Sandia National Laboratories for the U.S. Department of Energy under Contract DE-AC04-76DP0000789.

SS-MoP23 Disorder-Effects at the Al(110) Surface, W. Schommers, C. Mayer, H. Göbel and P. von Blankenhehn, Kernforschungszentrum Karlsruhe, Institut für Materialforschung I, P.O. Box 3640, 76012 Karlsruhe, Germany.

We have investigated the temperature dependence of the structure and dynamics of the Al(110) surface by molecular dynamics. The following model has been used: The Al particles were arranged as a slab-shaped face cubic with two (110) planes as free surfaces. The slab consisted of 11 layers, and its total number of particles in the calculations was N = 528. For the interaction between the particles we have used a realistic pseudopotential (in the long range part of the potential interactions of van der Waals-type were considered).

The behaviour of the following quantities have been studied: Pair correlation function, generalized phonon density of states, and the mean-square displacement as a function of time. Significant effects concerning disorder and diffusion were found for temperatures T > 600 K (melting temperature: 933 K). These results will be compared with experimental structure factors measured by low energy electron diffraction for momentum transfers between 0.2 and 2.2 Å-1 and at temperatures between 800 and 900 K.


Several researchers, including the authors, have recently used high resolution electron energy loss spectroscopy (HREELS) to study structure and bonding on diamond surfaces. However, the selection rules which apply must be known so that one can be correct to interpret these spectra. The selection rules in effect are determined by the mechanism for vibrational excitation. For this reason, we have investigated the mechanism for vibrational excitation during the scattering of low energy electrons from hydrogen terminated C(110):2 x 1. We find that the scattering mechanism is dependent on surface smoothness which we characterize using low energy electron diffraction (LEED). For surfaces prepared with standard techniques, impact scattering dominates the vibrational loss spectra. In addition, we have developed a method to create extremely smooth hydrogen terminated C(110) as demonstrated by the observation of half order LEED spots at an incident electron energies as low as 13 eV. Only for these extremely smooth surfaces is dipole scattering seen to contribute significantly in the region of C-H vibrations. We will discuss reasons for the dominance of impact scattering and implications for the interpretation of HREEL spectra from nonmetals.

SS-MoP25 Scattering of 3He from the Nio (001) Surface, Jeff Baker, G. G. Bishop, E. S. Gillman, S. A. Srafon, J. G. Skofronick, Florida State University, Tallahassee, FL 32306.

An experiment scattering 3He from the Nio (001) surface has been performed in an attempt to measure the magnetic structure of the Nio surface. A nozzle beam of 5% 3He in a carrier gas of He was used to produce a narrow velocity spread of the incident 3He atoms. Δν/ ν = 5.6%. This beam was scattered from the surface and the 3He in the beam was detected using a quadrupole mass spectrometer. Measurements of the scattered intensity versus incident angle produced narrow Bragg peaks, indicating that a high quality surface had been produced by cleaning in situ. The diffraction pattern obtained with 3He is slightly different from that obtained with 4He. A careful study of the region in which the magnetic Bragg peaks were expected set the maximum intensity of the magnetic peak at 4 x 10^-7 times smaller than the intensity of the specular peak. These results are discussed in terms of a comparison of the standard helium-surface interaction potential with the interaction between the nuclear magnetic dipole of the 3He and the electronic spins of the Ni2+ ions. The narrow velocity distribution obtained for 3He by this technique shows that high resolution experiments measuring the dynamics of crystal surfaces can be performed with both 3He and 4He, which should prove useful for other surfaces, both magnetic and non-magnetic.

SS-MoP26 The Chemisorption of F2 and O2 on Si(100)-(2 x 1) and Si(111)-(7 x 7): An Inverse Temperature Effect, H. C. Flamm, E. R. Behringer, D. J. D. Sullivan, and A. C. Kummel, Chemistry Department, University of California at San Diego, La Jolla, CA 92093 U.S.A.

We have measured the initial sticking probability (S0) of O2 and F2 as a function of incident translational energy (0.03eV < E < 1.4eV) and surface temperature (300K < T < 900K) on both Si(111)-(7 x 7) and Si(100)-(2 x 1). For all four systems, at higher translational energy (Ei < 0.3 eV), the sticking probability increases with surface temperature ("inverse temperature effect"), but at the highest incident translational energies (Ei > 1.0 eV) the sticking probability is (S0 ~ 1.0 for 300K < T < 900K) independent of the surface temperature ("normal temperature effect"). The inverse temperature effect for O2/Si(100)-(2 x 1) was first observed by D'Evelyn, Nelson, and Engel. The appearance and disappearance of the inverse temper-
nature effect as a function of incident translational energy for both F₂ and O₂ suggests that thermal carriers are important in the activated chemisorption process.

SS-MoP28  Isothermal H₂ Desorption Kinetics from Si(100) 2 × 1: Dependence on Disilane and Atomic Hydrogen Precursors, Lynne A. Okada, Michael L. Wise, and Steven M. George, Dept. of Chemistry and Biochemistry, Univ. of Colorado, Boulder, CO 80309-0215, USA.

The isothermal H₂ desorption kinetics from the Si(100) 2 × 1 surface were studied using laser induced thermal desorption (LITD) techniques. Disilane (Si₂H₆) and atomic hydrogen were used as the hydrogen precursors. Atomic hydrogen desorbed only hydrogen adatoms and H₂ subsequently desorbs from an atomically-flat Si(100) 2 × 1 surface. Disilane deposits both hydrogen and silicon adatoms which may produce an atomically-rough Si(100) surface. This surface roughening with silicon adatoms simulates silicon chemical vapor deposition (CVD) and may affect the H₂ desorption kinetics. The isothermal LITD studies revealed first-order H₂ desorption kinetics for both precursors. An activation barrier of E_a = 57.2 ± 2.6 kcal/mol and a pre-exponential factor of A = 2.21 × 10¹⁴ s⁻¹, were measured for the atomic hydrogen precursor. An activation barrier of E_a = 54.3 ± 2.3 kcal/mol and a pre-exponential factor of A = 2.32 × 10¹⁴ s⁻¹ were determined for the disilane precursor. Within the experimental error, the isothermal H₂ desorption kinetics were not significantly affected by the hydrogen source. The similar desorption kinetics are attributed to the surface coverage of the silicon adatoms deposited with disilane. H₂ presumably does not desorb from an atomically-rough surface because the silicon adatoms can diffuse to step edges or form Si(100) 2 × 1 islands on the underlying Si(100) 2 × 1 surface. The first-order H₂ desorption kinetics are explained by the concerted desorption of H₂ from two hydrogen adatoms paired on the same silicon dimer on the Si(100) 2 × 1 surface.

SS-MoP32  Molecular Beam Reaction Studies of Trimethylgallium (TMGa) on Ga-rich GaAs(100), Barbara A. Bensanen and Christine E. Nelson, University of Wisconsin-Eau Claire, Eau Claire, WI 54702-2036, and J. Randy Creighton, Sandia National Laboratories, Albuquerque, NM 87185.

We have used molecular beam experiments to probe the reactivity of TMGa (an important organometallic precursor used in GaAs and GaNMBE) on the Ga-rich surface of GaAs(100) at low temperatures, where adsorption and desorption are the major reaction processes, and at high temperatures, where TMGa decomposition is the dominant surface process. We have found that the TMGa saturation coverage ranges from 1.7 × 10¹⁴ to 6.0 × 10¹⁴ molecules/cm² at 173 and 613 K, respectively. Also, the shape of the sticking coefficient vs. coverage plot suggests that precursor-mediated desorption kinetics are operative. Kinetic modeling of the molecular beam data confirms that precursor mediated desorption kinetics are active and uses a Kiflik expression for the sticking coefficient. Using arhennius parameters from thermal desorption and SIMS work, we have modeled the sticking coefficients vs. coverage curves using a multi-step mechanism that includes adsorption, desorption, and TMGa decomposition into DMGa and MMGa.

SS-MoP30  Inner-Shell Promotions in Low Energy Li⁺-Al Collisions at Clean and Alkali-Covered Al(100), K. A. H. German, C. B. Weare, and J. A. Yarmoff, Department of Physics, University of California, Riverside, CA 92521 and Materials Sciences Division, Lawrence Berkeley Laboratory, Berkeley, CA 94720.

The success of low energy ion scattering (LEIS) in determining surface elemental compositions on the use of accurate models for the energy loss and neutralization experienced by scattered ions. The present study reveals that the inelastic loss and charge exchange that occur during low energy, i.e., 0.4 to 5.0 keV, Li⁺ scattering from clean and alkali-covered Al(100) are greatly affected by inner-shell excitations, a mechanism that is not usually considered in the interpretation of LEIS data. Spectra of backscattered Li⁺ ions exhibit discrete loss features associated with single scattering from Al target atoms. The loss features are resistant to the usual increase in neutralization produced by the deposition of alkali on the surface. Spectra of ion-induced electron emissions show that Li I s promotion initiates the formation of autoionizing Li I 1s2S states. It is the production of ions away from the surface via autoionization that is responsible for the lack of response of low-loss features to alkali-adsorption. The results also show that, while Li I is promoted during hard Li-Al collisions, they are not promoted during Li-K or Li-Na collisions. Because Li I s electron promotion is dependent on the target atom species, the inelastic loss and neutralization processes for Li I⁺ ions scattered from alkali-covered Al are also site-dependent.

SS-MoP31  Evolution of Surface Morphology of Si(100)-(2 × 1) During Oxygen Adsorption at Elevated Temperatures, Y. Hong, K. Wurmm, Y. Wei and I. S. T. Tsong, Arizona State University, Tempe, AZ 85278, R. Klise, B. Rötger and H. Niedermeyer, Ruhr-Universität Bochum, 44780 Bochum, Germany.

We used scanning tunneling microscopy (STM) and low-energy electron microscopy (LEEM) to study the evolution of the Si(100)-(2 × 1) surface by oxygen at low pressures and elevated temperatures. At 5 × 10⁻¹⁰ torr partial pressure of oxygen, the transition from random etching of terraces to step-etching occurs at ~380°C, while at a higher partial pressure of 5 × 10⁻⁹ torr, the transition temperature is ~625°C. The diffusing species is a dimer vacancy, formed by the deposition of two O₂ molecules. Anisotropic diffusion of the vacancies is observed, with a preferred direction along the dimer rows. The reaction probability, defined as the number of desorbed SiO molecules to the number of incident O₂ molecules, is evaluated to be 0.010 ± 0.005, almost exactly twice that of the sticking probability of O₂ on Si(100) reported previously. This suggests that virtually all adsorbed O₂ molecules were converted to volatile SiO giving rise to etching at the pressure and temperature ranges studied.

SS-MoP33  Analysis of Chemisorption Sites: Cl on Si(111)-7 × 7, C. Yan, J. A. Jensen, A. C. Kummel, U. C. San Diego Chemistry, La Jolla, CA 92093.

Using scanning tunneling microscopy, we have analyzed the different types of dimer/pair adatom chemisorption sites which result from the chemisorption of molecular chlorine as function of their incident translational energy, E_{trans}. On the Si(111)-7 × 7 reconstructed surface, there are five different types of adjacent adatom pairs (ignoring the stacking fault of one half of the unit cell): corner-corner adatoms, corner-center adatoms within one half of a unit cell, corner-center adatoms from adjacent half unit cells. It should be noted that this pair has the lowest degeneracy of all possible nearest-neighbor pairs (3 sets in one unit cell vs. 6 or 12). A possible explanation of this preference is that this is the closest set of adatom centers; another is the fact that these two adatoms are electronically decoupled across the stacking fault. At low E_{trans} (0.44 eV) where dimer/pair adatom adsorption is dominant, one type of adatom pair accounts for a majority (60%) of all double adatom adsorption: two center adatoms from adjacent half unit cells. In addition, at low E_{trans} there is no double adsorbate sites preference. This lack of adatom pair selectivity and the appearance of many single adatom sites suggests that either adatom-rest atom dissociative chemisorption, or as suggested by Boland, et al., abstraction of one Cl atom onto an adatom, is the dominant non-precursor mediated chemisorption mechanism at low E_{trans}.

SS-MoP34  Resonantly Enhanced Multiphoton Ionization of SiO Desorbing from Si(111) in Reaction with O₂, K. G. Nakamura and M. Kitaçima, National Research Institute for Metals, Tsukuba, Ibaraki 305, Japan.

Inelastic state distribution of SiO desorbing from Si(111) surface in reaction with O₂ molecular beam have been studied using resonantly enhanced multiphoton ionization mass spectroscopy (REMPI). The F²⁺ → X²⁺ transition is used for (2 + 1) REMPI with a wavelength of 290-295 nm. The experiments were performed in an ultrahigh vacuum chamber (base pressure < 10⁻¹¹ Pa). Si(111) surface is exposed to pure O₂ molecular beam (~1 ms of FWHM) at substrate temperature of ~1250 K. The SiO molecule produced by the Si surface oxidation reaction Si(0) + O₂(g) → SiO(g) is observed and its waveform is almost same as that of the incident O₂ beam. Well separated vibrational bands (FX(0,0), FX(1,1), and FX(2,2)) have been observed in the (2 + 1) REMPI spectrum. Intensities of vibrational bands are comparable to the vibrational distribution of SiO with a Boltzmann distribution at the surface temperature. The rotational distribution of SiO will be also discussed.
Charge transfer process from Cs-covered Cu(111) surfaces has been studied using low energy Li\textsuperscript+ ion scattering. Energy distributions and polar angle dependences of the scattered both Li\textsuperscript+ and Li\textsuperscript- ion intensities are collected as a function of Cs coverage. The results indicate that the Li\textsuperscript+ single scattering intensity from Cs is almost completely neutralized, while the Li\textsuperscript- intensity behaves totally differently, showing maximum at certain Cs coverage near the work function minimum (Δφ = -3.5 eV). Quantitative agreement is achieved with the observed work function dependence. The experimental results can be well interpreted by a resonant charge transfer theory which includes electron excitation, negative ion formation and level crossings. The effects of oxygen adsorption on various pre-coverages of Cs by observing the variations of the Li\textsuperscript+ -Cs and Li\textsuperscript- -Cs single scattering intensities are also investigated. These results are discussed in connection with the electron structure of alkalis on metal surfaces.

**SS-MoP35** The Site-Specific Neutralization Behavior of 2.0 keV Li\textsuperscript+ Ions Scattered from Alkali/Al(100) Surfaces, C. B. Weare, K. A. H. German and J. A. Yarmoff, Department of Physics, University of California, Riverside, CA 92521 and Materials Sciences Division, Lawrence Berkeley Laboratory, Berkeley, CA 94720.

The effects of alkali adsorption on metal surfaces is of fundamental importance in many processes. Numerous experiments have investigated these effects by studying the neutralization behavior of alkali ions scattered from alkali-covered metallic surfaces. However, previous experiments were unable to separate the neutralization probabilities, or neutral fractions, of alkali atoms scattered from substrate and adsorbate sites. Site-specific sensitivity of the neutral fraction is made possible in the present study by comparing a large scattering angle (168°) with time-of-flight (TOF) detection of Li scattered near the surface normal. This yields spectra in which the single scattering signals from substrate and adsorbate sites are well separated, thus allowing independent measurements of the neutral fractions. For low K coverages, Li\textsuperscript+ ions scattering from K sites experience essentially complete neutralization, while the neutral fraction for Li\textsuperscript+ scattering from Al sites is on the order of 50%. For K coverages approaching 0.4 ML, however, the measured neutral fractions for Li scattering from K and Al become nearly equal (at about 80%). The data support a picture in which, at low K coverages, K adsorbs behave as independent dipoles, creating an inhomogeneous surface electrostatic potential. At higher coverages, on the other hand, K forms a uniform dipole layer. Data for Na and Cs adsorbates are also presented.

**SS-MoP36** Isotope Effects in Electro- Stimulated Desorption from Physiosorbed Monolayers of H\textsubscript{2}, HD, and D\textsubscript{2} on Graphite, Bin Xia and S. C. Fain, Jr., Department of Physics, FM-15, Univ. of Washington, Seattle, WA 98195.

Time-of-flight analysis regarding field measurements using positive ions desorbed by 62 eV electrons from physiosorbed H\textsubscript{2}, HD, and D\textsubscript{2} monolayers on graphite have been used to estimate cross sections as most probable energies. The monatomic ion cross sections are 10\textsuperscript{-2} to 10\textsuperscript{-5} of the gas phase value; the diatomic ion cross sections are 10\textsuperscript{-3} to 10\textsuperscript{-6} of the gas phase value. The reduction of the ion cross sections relative to the gas phase is qualitatively consistent with greater quenching by the surface electrons for ions with lower kinetic energy and higher mass. In gas phase measurements, conservation of linear momentum in electron-induced dissociation causes the ratio of H\textsuperscript+ energy to D\textsuperscript+ energy from HD to be 2.0. The larger energy ratio observed here may be due to the quenching by the surface affecting dissociation via the antibonding HD\textsuperscript+- state differently than dissociation via autoionizing states. By lowering the electron energy to 32 eV, dissociation via the antibonding HD\textsuperscript+- state is strongly suppressed. New and analyzed time-of-flight data indicate a decrease from 3 to 2 for the ratio of the number of H\textsuperscript+ ions to the number of D\textsuperscript+ ions as the incident electron energy is lowered from 62 eV to 32 eV. Ion energy distributions from the HD time-of-flight data as a function of incident electron energy will be presented.

Supported by NSF grant DMR-91-19701.

**SS-MoP37** Internal and Translational State Distributions and Alignment of a Photodesorbed Molecule: CO/Si(100) 2 x 1, Frank M. Zimmermann, Paul L. Houston, and W. Ho, Materials Science Center, Cornell University, Ithaca, NY 14853.

We have studied photochemical (308 nm) desorption of CO from Si(100) 2 x 1 at 82 K, using state resolved detection by laser induced fluorescence. The process has two remarkably large cross sections (∼1 × 10\textsuperscript{-16} and 7 × 10\textsuperscript{-18} cm\textsuperscript{2}), which correlate with two thermal desorption peaks. Internal and translational energy distributions provide insight into the dynamics of the photodesorption process. Most notable is a very high degree of vibrational excitation, which, together with the large cross sections, may indicate relatively weak electronic quenching. The rotational as well as the translational energy distributions are bimodal, with the hotter component of the translational distribution correlating with the colder part of the rotational distribution. Both of these components individually exhibit positive rotational-translational correlations, however, in qualitative agreement with a simple dynamical model. We report the first angular momentum measurements of a photodesorbed molecule. The results indicate that the colder component of the rotational distribution is not strongly aligned, whereas the J-vectors of the rotationally hotter component show preferential alignment parallel to the surface (cartwheeling motion).

**SS-MoP38** Kinetic to Internal Energy Transfer during Polycrystalline Ion-Surface Collisions, J. A. Burroughs, S. W. Weinhaus, and L. Hanley, University of Illinois at Chicago, Chicago, IL 60607-7061.

Low energy polycrystalline ion-surface collisions occur in a wide variety of mass spectrometry, plasma, and ion beam etching processes. The transfer of kinetic to internal energy can lead to the surface-induced dissociation of the primary ions, an effect which has begun to be utilized in mass spectrometry to obtain ionic structural information. 20 to 120 eV Cr(CO)\textsubscript{5} ions are collided with monolayers of hexanethiol and heptfluorobutyric acid adsorbed on Ag(111) and the fragment ions formed in the surface collision are monitored by a mass spectrometer. Cr(CO)\textsubscript{5} fragmentation patterns from surface-induced dissociation are fit to an impulsive collision model originally used to describe gas phase collision-induced dissociation. This simple model can quantitatively describe the relative contribution of kinetic to internal energy transfer between the hydrogenated and fluorinated surfaces. Additional data on the secondary ion kinetic energy distributions of the Cr(CO)\textsubscript{5} fragments is used to obtain a more detailed description of the ion-surface collision.

**SS-MoP39** Application of Hyperthermal Beams to Achieve Adsorption and/or Absorption of Adsorbates on Diamond Surfaces, D. H. Harrity, C. Bandis, B. R. Pate, Department of Physics, Washington State University, Pullman, WA 99164-2814.

Hydrogen stabilization of the diamond surface against reconstruction is thought to be a key element enabling low pressure diamond growth. However, if every surface atom was terminated by hydrogen, carbon deposition (diamond growth) would be hindered due to a lack of chemically active sites. In a typical diamond CVD apparatus, the steady state fraction of active (hydrogen-free) sites is thought to be directly related to growth rate. The production of active sites during growth is attributed to the abstraction of surface-bound hydrogen by gas-phase atomic hydrogen (activation energy ca. 7 kcal/mole). Due to activation barriers and relaxation phenomena, adsorption and abstraction phenomena is likely to be dependent upon the excitation state (eg. kinetic energy, ionization, etc.) of the gas phase species (such as hydrogen).

In this experimental study, we employ a mass-selected low kinetic energy (10-50 eV) hydrogen source to interact species at controlled impact kinetic energy with the diamond surface. Our objective is to better understand the role of hydrogen kinetic energy in the hydrogen adsorption/abstraction mechanisms which are key to understanding low pressure diamond growth. We report the effects of hyperthermal hydrogen exposure upon both hydrogen coverage and surface structure of clean and hydrogenated diamond surfaces.

Work supported by NSF and Washington State University.

**SS-MoP40** The Effect of Preadsorbed Oxygen on the Dissociation Dynamics of Ethane on Ir(110)*, W. Hago, D. Kelly, and W. H. Weinberg, Department of Chemical Engineering, University of California Santa Barbara, California 93106.

As part of an ongoing effort to understand the basic mechanisms of alkane activation, and to find new methods of oxidizing hydrocarbons, we have examined the effect of chemisorbed oxygen on the dissociation dynamics of ethane on Ir(110). In particular, we have examined, using a supersonic molecular beam, the effect of preadsorbed oxygen on the initial probability of ethane activation on this surface at various coverages. Preadsorbed oxygen prevented a saturated monolayer, and found that oxygen, while not affecting the initial trapping probability, significantly affects the initial probability of dissociation chemisorption for both trapping-mediated and direct dissociation. For trapping-mediated dissociation, 0.5 monolayer of oxygen is sufficient to significantly inhibit any ethane activation. Furthermore, by following changes in surface carbon and oxygen with Auger spec-
troscopy, we have monitored the reaction of the dissociation products of the ethane (or direct dissociation) with the preadsorbed oxygen as a function of surface temperature.

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NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room BR4 – Session NS-MoP

Instrumentation and Metrology

Moderator: E. C. Teague, National Institute of Standards and Technology.

NS-MoP1 Quantum Friction Observed with Two-Dimensional Frictional Force Microscope, S. Fujisawa, E. Kashi, Y. Sugawara and S. Morita, Department of Physics, Faculty of Science, Hiroshima University, 1-3-1 Kagamiyama, Higashihiroshima 724 Japan.

We investigated nature of the atomic scale friction between a single asperity of SiN$_4$ and an atomically flat surface of MoS$_2$, where the frictional force becomes two-dimensional vector. Using the two-dimensional frictional force microscope (2D-FFM) which detects both the components separately and simultaneously [1, 2], we found that the single asperity shows the two-dimensional quantized jump with the lattice periodicity of the MoS$_2$, namely “two-dimensional stick-slip” phenomenon [1]. We also found that this quantized behavior shows fluctuation [2]. This phenomenon shows that on an atomic scale the frictional force is quantized two-dimensionally and acts not only along the scan direction but also across the scan direction, although there is contradiction to the principle of the classical friction. Further, we will discuss the nature of the quantized friction, using the simple two-dimensional stick-slip model with effective adhesive radius.


NS-MoP2 Microtribological Study of Lubricants Using Friction Force Microscopy, Bharat Bhushan and Vilas N. Koinkar, Computer Microtribology and Contamination Laboratory, Department of Mechanical Engineering, The Ohio State University, Columbus, Ohio 43210-1107.

Atomic force microscopy (AFM)/Friction force microscopy (FFM) is used for microtribological studies on the unlubricated and lubricated single-crystal silicon. The single-crystal silicon is coated with few nanometer thick poly- and non polar lubricants. Friction dependence upon the normal load, shear velocity and different environments are studied in detail. The friction of lubricated and unlubricated samples sliding against silicon, silicon nitride and diamond tip is also investigated. It is observed that lubricant depletion/degradation on a microscopic scale can easily be detected with FFM. Normal load and shear velocity significantly affect the lubricant performance. Some friction studies on unlubricated and lubricated thin-film magnetic rigid disks have been performed and are discussed briefly. FFM appears to be an useful tool to study boundary lubrication properties on a nanoscale.


A scanning tunneling microscope (STM) has been modified to detect surface acoustic waves (SAW) across the surface of bulk materials and thin films with a lateral resolution on the nanometer length scale. The surface acoustic waves are excited using interdigital transducers configured to produce a SAW with a frequency of 79 MHz. An rf signal of comparable frequency is also applied to the STM tip. With this configuration, the tip acts as a mixer, and the difference term in the frequency components of the tunnel current is detected with conventional STM electronics. The amplitude and phase of the SAW are directly observed on an oscilloscope, and are related to the local mechanical properties of the substrate. By imaging the surface with the STM, surface acoustic wave measurements can be performed at selected positions on the surface. The effects of surface morphology and defects on the local mechanical properties of the material will be discussed.

NS-MoP4 Modeling of the Nanoindentation of Thin SiO$_2$ Layers on Si Using Finite Element Analysis, Charles F. Draper, David M. Schafer, Richard J. Colton, and Steven M. Hues, Department of Mechanical Engineering, Vanderbilt University, Nashville, TN, 37205 USA, Code 6170, Naval Research Laboratory, Washington, D.C., 20375-5342 USA.

A main force of the atomic force microscope (AFM) as a nanoindenter is its ability to measure mechanical properties of thin films. The properties measured, however, are composite properties that reflect not only the intrinsic properties of the film, but also the underlying substrate. Upon tip/surface contact, a stress field is generated which penetrates into the sample. The magnitude of the composite modulus is determined by the relative contribution of the stress and strain fields between the layer and substrate. We have studied this effect experimentally using thermally-grown oxide layers (2-30 nm) on (100) Si wafers. Finite element analysis (FEA) is used to model the stress and strain fields within the overlayer/substrate system and quantitatively describe the nanoindentation behavior of the film/substrate system as a function of oxide thickness. Details of the FEA modeling and comparison with experimental results will be presented and discussed.


The adhesion force between 5, 15 and 30 nm colloidal and poly-L-lysine treated mica (pml) was measured using atomic force microscopy. Through incremental increases in the normal force, the lateral force needed to break the presumed weak electrostatic bond between the gold particles and pml was determined. Key to accurate measurement of these forces is accurate knowledge of the tip geometry$^{1,2}$, cantilever spring constant$^3$, and monitoring of the piezo-translator drift. The results were compared with a model of a charged sphere in close proximity to countercharged infinite plane. Measurements of both macroscopic properties (coefficient of friction) and microscopic properties (force between opposite paired charges) are discussed.


NS-MoP6 Ultrasound Influence on the Tribological Properties of CVD Diamond, V. Stitka, Research Institute “Vibrotechnika”, Kaunas, 3000 LITHUANIA, V. Baranauskas, State University of Campusinas, 13081-970 Campusinas SP BRASIL.

The friction and wear behavior of CVD diamond film sliding against aluminum, iron, steel and itself has been investigated under strong ultrasound conditions at the friction interface. Experiments were performed on an alternating “pin-on-plate” tribometer constructed as an ultrasonic motor. The bimodal mechanical vibrations were in the frequency range (25-80) kHz. The diamond samples were deposited on the molybdenum by the Hot-film assisted CVD from CF$_4$/CH$_4$ gas mixture with typical 5-10 μm lateral grain size and film thickness up to 100 μm. AFM and SEM were used for the characterization of the surface morphology. The experimental results have shown that ultrasound makes strong influence on the alumina and diamond wearing, friction and surface deformation. XPS analysis of the worn diamond crystallites indicated the occurrence of tribochemical reactions between ceramics, iron and diamond. Fast friction coefficient decrease in time was observed in the case of sliding CVD diamond film. Presence of ultrasound during the sliding decrease the friction coefficient to zero. The diamond wearing in the lower hardness materials friction pair case can be explained by high shear stresses and high oxidation rate of diamond in presence of ultrasound. Raman scattering have shown low level of induced defects during ultrasound assisted friction. The diamond films were mechanically polished from...
initial surface roughness 3 μm to 20 nm, as measured by the AFM height surface histograms. The high quality and speed of ultrasonic polishing of CVD diamond suggests that this technique is interesting for a high number of diamond applications were almost atomic flat surface is needed.

The more common availability of monochromatic, well-collimated beams of X rays, from sources such as electron storage rings, has motivated a study of the optical elements used at these short wavelengths. One of the important parameters governing the performance of x-ray optical systems is surface roughness. Surface roughness measurements have been achieved by the stylus method and many kinds of optical interferometric methods. However, it is difficult to measure the lateral period of the surface roughness of less than about 1000 Å by these methods. We use the STM to investigate the surface roughness statistics characterization of some kinds of soft x-ray multilayer films, such as Mo/Si, Mo/C, and W/Si films, on the nanometer scale. The experiment results show that the surface roughness will be larger after sputtering films, for Mo/Si (61 layers): 0.50 nm rms ~ 1.37 nm rms, for Mo/C (45 layers): 0.50 nm rms ~ 1.41 nm rms, which is depended on the kind of film system, layer numbers and thickness of every layer. In addition, we will give the correlated-roughness analysis.

NS-MoP8 Step Height Measurement Using a Scanning Tunneling Microscope Equipped with a Crystalline Lattice Scale Reference and Interferometer, Toru Fujii and Masatoshi Suzuki, Nikkon Corporation, 1-10-1, Asamizoidai, Sagamihara, Kanagawa 228 JAPAN, Hiroshi Kougam and Hideki Kawakatsu, Institute of Industrial Science, University of Tokyo, 7-22-1, Minato-Ku, Tokyo 106 JAPAN, Toshiro Higuchi, Faculty of Engineering, University of Tokyo, 7-3-1, Hongo, Bunkyo-Ku, Tokyo 113 JAPAN.
Scanning tunneling microscope (STM) has been well known for its high resolution and non-destructive surface profiling capability but its potential as a profilor for step height measurement has been rarely exploited to its maximum extent because of its unreliable vertical accuracy. Recently, an STM equipped with three-dimensional interferometer was developed and step height and pitch measurement with nanometer accuracy was achieved. However, all the features of the interferometer are not always required in step height measurement. An STM equipped with scale reference crystal for X and Y directions was simple and compact compared to the STM with interferometer, and offers sub-nanometer resolution using lattice image which consumes large memory area so that high resolution short range measurement is effective. We have successfully combined reference crystal technique and high accuracy mechanisms, and developed new method which can measure both scanning direction and feedback direction simultaneously. Interferometry was also applied to verify measured step height using reference crystal.

NS-MoP9 Comparison of Magnetic Force Microscopy Techniques, Paul Rice and John Moreland, National Institute of Standards and Technology, Boulder, CO 80303 USA.
We compare magnetic force microscopy (MFM) images of a computer hard disk obtained with four MFM techniques. MFM is becoming a widely used tool for studying the data written on magnetic storage disks. Interpreting the information obtained from various MFM techniques is paramount to understanding the magnetization of the disk. The four MFM techniques include two types of ac MFM, that detect either changes in amplitude or changes in frequency of a vibrating cantilever. The third is a dc MFM technique that detects the deflection of a non-vibrating cantilever. Finally we compare tunneling stabilized magnetic force microscopy (TSMFM) which detects the deflection of a non-vibrating cantilever using a tunneling current to stabilize the tip-sample contact. The sample imaged in this comparison has bit densities and magnetic properties typical of most currently available hard disks. These MFM techniques measure the force derivative between the tip and sample. In contrast, dc MFM and TSMFM techniques measure the force between the tip and sample. We have tried, from this comparison, to emphasize the differences of these various techniques in a current industrial application. We hope to provide more quantitative MFM measurements so that magnetic fields can be extrapolated from the images.

NS-MoP10 Scanning Thermal Microscopy with a Resistive Probe, R. B. Dinwiddie, High Temperature Materials Laboratory, Oak Ridge National Laboratory, Oak Ridge, TN, 37831; R. J. Pytkki and P. E. West, Topomatrix, Santa Clara, CA 95054.
A unique thermal probe has been developed utilizing a resistive element integrated with an atomic force microscope cantilever. Two operating modes of the thermal probe will be described. In one mode, surface temperatures are mapped by monitoring the resistive of the thermal probe. In the second operating mode, the heat flow from the self-heating thermal probe to the sample is monitored. Variations in the thermal properties of the sample (heat capacity, thermal diffusivity, thermal conductivity, etc.), as well as temperature, affect this flow of heat, changing the power necessary to keep the probe at a constant temperature. Integration of the thermal probe with a scanning probe microscope allows simultaneous acquisition of topography with the thermal data. Applications include the investigation of heat generating component or defect structures in electronic devices. The self-heating probe could be used to study the thermal properties of microscopic structures such as particles, fibers and intergranular phases. Examples of both operating modes will be shown.

NS-MoP11 An Integrated Lithography and Analysis Instrument, Grahame Rosson* and Robert Clark*, Leica Cambridge Ltd., Clifton Road, Cambridge, CB1 3QH, England, University of New South Wales, P.O. Box 1, Kensington, 2033, Australia.
We have developed a PC based integrated electron beam lithography and microscopy instrument for device research and development. This instrument has been used to expose and examine structures ranging from curved optical devices down to nanostructures used as precursors for tunnelling devices. To minimise scattering in the resist material the electron optics is designed to provide a finely focused beam of energy up to 100 kV suitable for fabricating nanostructures. The pattern data may be prepared using either PC CAD packages or a text based input, which is subsequently processed for writing utilising a Digital Signal Processor (DSP) interfaced to the PC. The DSP combines the flexibility of software control in programming the DSP, with the considerable processing capability of the DSP. The pattern data is further processed by dedicated low noise analogue lithography hardware to enhance the stability and resolution of the electron optics. In addition the instrument may be operated as a high resolution electron microscope for examining the results of the lithography. This is particularly important for examining structures in the nanometre scale. The design methodology enables the instrument to be used as an integrated tool for pattern design, conversion, exposure and subsequent investigation.

A cryogenic scanning tunneling microscope (STM) was constructed. It was designed to characterize superconducting materials under cryogenic (4.2 K) and magnetic (8 T) conditions. An attention was paid to reduce electrical noises, especially digital switching noise and ground loop noise. The preamplifier is battery-operated and is electrically isolated from the main control circuit by analog optical couplers. The digital portion of the main control circuit is static and no system clock is generated. The main control circuit is interfaced with a personal computer via 52 optical couplers. Dual electrical isolation eases the ground loop problem and shows good atomic resolution.

NS-MoP13 Are Atomic Emission Microscopy, P. G. Van Patter, J. D. Noll, and M. L. Myrick, Department of Chemistry and Biochemistry, University of South Carolina.
The scanning tunneling microscope is extremely useful in determining the topology of conductive surfaces. However, little if any other information is available to complement the topographical image obtained. Such information could eliminate doubt concerning the con-
stition of an imaged surface and could lend great insight into the observation of various surface phenomena. This work has resulted in the development of hardware and software additions for the STM, which allow the instrument to provide chemical information about the substrate via nano-scale arc atomic emission spectroscopy which takes advantage of the convenient geometry of the STM experiment. This experiment is performed by arcing across the tunneling gap with short-lived, large-amplitude (up to 150 V) pulses of the bias voltage. Previous results have shown that this arc reaches temperatures in excess of 6500 K and emits intense light. Collection of this light into fiber optics and detection of the light into a spectrometer should allow elemental analysis of the surface directly under the tip.


We demonstrate a new technique for selective metallization combining photoresist technology with self-assembled monolayers (SAMs) and electroless (EL) metallization. A substrate coated with a ligand functional SAM is coated with photoresist and then exposed according to standard photoresist protocols. Upon development, the resist is removed, exposing the underlying SAM. The EL catalyst is attached to the exposed SAM regions, followed by EL Ni metallization to produce channel constrained metal patterns at high resolution. The high etch resistance of even extremely thin Ni films towards RIE means that the patterned Ni may be employed as a highly selective etch barrier for pattern transfer into the underlying substrate. The application of this technique for sub-half micron patterning at DUW wavelengths and ±0.1 micron patterning by e-beam will be demonstrated.

NS-MoP15 AFM Tip-Sample/Sample-Tip Deconvolution, Peter C. Markiewicz, M. Cynthia Goh, Department of Chemistry, University of Toronto, Toronto, Ontario, Canada, M5S 1A1.

A simple algorithm has been implemented to directly extract information on the probe tip geometry used in the Atomic Force Microscope. The program uses known sample geometries in the deconvolution. These can be man-made, e.g., calibration standards can which can be co-deposited with samples. Being able to resolve the tip geometry in such a manner provides an effective means of inspecting the tips for manufacturing defects or the adhesion of debris. The tip information can then be used to deconvolute the subsequent AFM images to improve their quality. Examples of the deconvolution and their uses in various applications will be presented.

NS-MoP16 On the Recovery of Spectroscopic Image in Scanning Force Microscopy, Igor Yu. Sokolov, Dept. of Physics, University of Toronto, ON M5S 1A7, Canada.

A principal opportunity of detection of the sort of materials a surface consists of (spectroscopic image of the surface) is discussed for the scanning force microscopy method. It is shown that the problem can be reduced to the Fredholm integral equation of the first kind with the kernel of Hilbert-Schmidt type. Such a problem is demonstrated has a unique solution here provided we consider the case of exact measurements. Moreover, we suggest that the surface topology is known in advance. The possibility of spectroscopic image recovery for the case of the measurements with errors is discussed.

As an example, we demonstrate the obtaining of the Hamaker constant for unknown longitudinal plasma-rotation from SFM scanning.


Photoresist line widths are presently controlled by inspecting wafers in plan view with a 'metrology SEM'. Proprietary algorithms convert signal intensity to a pseudo-topographic profile and place markers at the inferred feature tops and photoresist. No standard reference materials exist for checking this measurement. This report shows the feasibility of creating an in-house standard for this purpose. Three specimens of 0.5μm wide photoresist line test patterns with a range of defocus conditions, in order to produce both aberrated and good line shapes. The specimens were examined in cross-section using the AFM and in plan view using the SEM. The AFM images had crisp edges defining the line profile. Pitch, line width, and slope angles were measured from the AFM images and compared with the pitches and line widths reported by the SEM. Differences are noted and discussed. The basis for edge contrast in the AFM is explored and a method is proposed for mathematical analysis. This work has shown that the AFM can directly examine resist line profiles and provide images of useful precision, without adding a conductive coating as is commonly done in high resolution SEM. (A conductive coating would modify both the physical width of the structure and its electron scattering characteristics.)

The author thanks Dennis Schrope, for the initial concept of this research and for supplying the specimens and SEM images, and AT&T Bell Laboratories for financial support.

NS-MoP18 PZT Thin Film Force Sensor for Atomic Force Microscope, Toru Fujii, Shunji Watanabe, Masatoshi Suzuki and Takamitsu Fujii, Nikon Corporation, 1-10-1, Asaminodai, Sagamihara, Kanagawa 228 JAPAN.

Atomic force microscope (AFM) is a powerful tool for observing not only conducting surfaces but also nonconductive surfaces with nanometer resolution. AC mode operation AFM which has the potential to profile surfaces with non-contact mode becomes important for non-destructive surface measurements, especially for measuring semiconductor surfaces covered with soft resist. The configuration of AFM is severely restricted by the detector used for measuring the deflection of the cantilever probe. This configuration which requires sample scanning and feedback sample positioning is inconvenient especially for large sample measurement such as semiconductor devices and optics. A piezoresistive cantilever capable of lever scanning and positioning was developed, however, it did not have the potential to oscillate itself for AC operation. ZnO film has been widely used in electronic appliances such as saw devices. This thin film forming technique was applied in fabricating cantilevers with thickness of 1/10-1/100 μm which was used as third actuator in STM as well as thin film sensor in AFM. PZT is one of the promising piezolectric materials for both sensing distortion and oscillating the cantilever because it has high piezoelectric constant compared to other piezoelectric materials. We have successfully achieved combining the process for fabricating Si cantilever and forming thin PZT films without deteriorating the cantilever characteristics. Preliminary images were taken using this cantilever to which electrochemically etched tungsten stylus was manually glued. Details around the pits of a surface of compact disk and textures of sputtered platinum were clearly observed.

NS-MoP19 The Design of An Atomic Force Microscope for Metrology, J. Schmied, Thomas McWaid, J. Alexander, and B. Wilfle*, 1National Institute of Science and Technology, Precision Engineering Division, Gaithersburg, MD 20899, 2Park Scientific Instruments, 1171 Borregas Ave., Sunnyvale, CA 94089, 3We have initiated a project to develop and calibrate artifacts which can be imaged on a commercial AFM. These images can then be used to calibrate the commercial AFM so that subsequent AFM measurements will be accurate and traceable back to the wavelength of light. To avoid systematic errors between different types of metrology tools we plan to calibrate our artifacts using a specially designed AFM system which we call the Calibrated AFM. This system has been constructed as much as possible out of commercially available components. We use a flexure stage driven by piezoelectrics for scanning; a heterodyne interferometer system to measure the X-Y position of the sample; a capacitance sensor to measure the Z position of the sample; an integrated piezoresistive force sensor; and a commercially available AFM control system. The control system has two feedback loops which read from the X and Y interferometers and adjust the piezoelectric voltages to keep the X-Y scan position accurate. This feedback loop can hold the X and Y interferometer readings constant to within 1.2 nm rms. The critical electromechanical and metrology issues involved in the construction and operation of such a system will be discussed in detail.


Perfect nanostructures obviously should be created in controlled conditions of ultra high vacuum. Here we would like to represent a UHV setup in which STM is combined with analytical modules equipped with AES, TDS, SIMS, LEED and controlled chlorination of sample. There are also abilities of sample surface preparation: ion etching, annealing in UHV up to 1300 K, and cooling to 100 K, etc. The major tool of this UHV combine is original STM with following features: a) no mechanical drives from outside for coarse tip movement, b) topography, spectroscopy & lithography modes, c) construction is extremely opened that allows to adsorb gases and treat the
sample surface by radiation or particle beams in situ during scanning, d) tips & samples transfer system easily allows to use the STM in combination with other surface science techniques.

For correct measurements of interatomic distances on atomic structures we used original algorithm of STM calibration and reconstruction of real STM image in presence of noticeable drift of the STM tip. Using this algorithm we measured piezoelectricities sensitiveness along all three axis and also followed their changes after annealing of UHV chamber.

Preliminary study of chlorine adsorption on copper surface confirms ability to create nanostructures (copper clusters inside copper chloride islands) with very interesting properties for nanoelectronics by STM tip.


One of the first variations of scanning tunneling microscopy (STM) was scanning tunneling potentiometry (STP). STP has nanoscopic spatial resolution and submicrowatt voltage resolution. It works well on conductive samples where a reasonable tunneling contact can be maintained during scanning. The atomic force microscope (AFM) has also been adapted for scanning potentiometry. The advantage of AFM is that the sample being scanned can be partially insulating. The likely superiority of a AFM potentiometry over STP becomes evident when one considers imaging of submicrometer structures. In such cases it will be difficult to land a STM tip on small conducting areas on the sample without crashing into insulating regions. We are presently optimizing AFM potentiometry for simultaneous topographic and surface potential measurements of patterned YBCO step-edge junctions and junction arrays. In addition, we are developing STP for low temperature measurements of non-patterned step edge.


We are investigating several techniques for improving the electrical conductivity of standard silicon cantilevers for atomic force microscopy. One easy technique we have tried is to evaporate layers of gold, platinum or indium onto the lower surface of the cantilever to create a conducting layer. Such coated cantilevers suffer from the problem that the metal covering the apex of the tip may be worn away during the process of scanning the tip. A more promising method is to dope silicon cantilevers with boron or phosphorus. This is done by baking the cantilevers at temperatures above 900 °C in proximity to industry standard planar diffusion sources in an atmosphere of flowing nitrogen. This is followed by a short etch in dilute hydrofluoric acid. These cantilevers have a sheet resistance of less than 100 Ω/ square. Because of the formation of an oxide layer, the contact resistance to the cantilevers slowly increases over a period of several days. We are currently using these cantilevers for a variety of applications including scanning potentiometry, simultaneous STM and AFM, and making electrical contact to submicrometer thin-film multilayer dots for vertical giant magnetoresistance measurements.


In this paper we discuss the effects of K and Li deposition on the emission characteristics of Spindt-type thin film field emission microcathode arrays (TFMEM) with Mo tips. We report the amount by which each alkali metal lowered the effective work function of a TFEEM, and compare this data to results of similar measurements performed with single-crystal (100)Mo surfaces. We discuss both short- and long-term fluctuations in the emission current and how these fluctuations are affected by the alkali metal adsorbates. In addition, we show that the emission current produced at a given gate voltage during alkali metal deposition is significantly larger than the emission current collected when the deposition source is turned off. We discuss the emission current fluctuations and the increase in emission during deposition using a model based on adsorbate diffusion.


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The microcolumn is a complete electron optical column consisting of a field emission source, electrostatic lenses and deflector with a total column length of only 3.5 mm for operation at 1 keV electron energy in ultra high vacuum. The electron source is a scanning tunneling microscope (STM) aligned field emission (SAFE) source, which uses a 111 W field emitter with a tip radius of about 50 nm for a low extraction voltage. A selectively coated dual electrode accelerating lens forms a source lens with low aberrations. The beam is imaged onto a sample at 1 mm working distance by a symmetrical einzel lens. All lenses are assembled from silicon membrane chips and insulating pyrex spacers held together by anodic bonding. With the present microcolumn we have measured a beam diameter of 20-25 nm. This paper will discuss the microcolumn design and give a comparison of the electron optical modelling and experimental results obtained, such as the scanning transmission imaging of grid samples.

A portion of this work has been sponsored by ARPA under an AFOSR contract.

PLASMA SCIENCE
Room BR4 – Session PS-MoP

Plasma Etching and Deposition
Moderator: D. B. Graves, University of California, Berkeley.

PS-MoP1 Deposition of a-C:H Layers Using an Expanding Ar/CxHy/CF4 Plasma, J. W. A. Gielen, M. C. M. van de Sanden, A. J. M. Biurrun and D. C. Schram, Department of Physics, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands.

Amorphous hydrogenated carbon layers are used as optical, corrosion and wear resistant layers. These properties depend critically on, for example, $sp^3/sp^2$ ratio, density and bandgap. In this study an expanding thermal arc in argon is used ($U_{arc} = 20-90$ A, $Q_{arc} = 10-100$ scc/s, $p_{arc} = 100-500$ mbar) in which the monomer ($Q_{CH}_2$) is 1-3 scc/s) is added downstream ($p_{react} = 0.2$ mbar). To influence the layer properties, an etching agent CF4 ($Q_{CF}_4 = 0-3$ scc/s) is injected. As in this method ion bombardment is absent, the ion etching agent is to manipulate bandgap, $sp^3/sp^2$ ratio by increasing CF4 flow, as determined using FTIR. No CF4 and CF4-bonds are identified in the deposited layers whereas CF is dominantly present. From in situ ellipsometry it is shown that the growth rate depends linearly on the CH2 flow and is unaffected by the etching gas CF4. The growth rates are typically 10 nm/s but larger rates can be obtained by increasing the arc power and monomer flow. The microstructure is investigated using an atomic force microscope. The dimensions of the structures observed seem to decrease with increasing CF4-flow, indicating the influence of etching during deposition. In this paper the role of the etching agent will be addressed with respect to the influence on plasma radicalisation and deposition mechanism. Relation with other work on a-C:H:F deposition will be discussed.

PS-MoP2 Silicon Oxide Deposition in an ECR Plasma with Microwave Spectroscopy as a Diagnostic*, Kok Heng Cheow, Jian Chen, R. Claude Woods, and J. Leon Shohet, Engineering Research Center for Plasma-Aided Manufacturing, University of Wisconsin-Madison, 1410 Johnson Dr. Room 101, Madison, WI 53706.

A 2.45 GHz electron cyclotron resonance (ECR) reactor was used to deposit silicon oxide films using both tetraethylsilane (TEOS) + O2 and silane (SiH4) + O2. This reactor is also equipped with an in situ microwave spectrometer in the frequency range of 75-110 GHz, which is used to monitor the gas phase chemical species in this environment. The $J = 1 \rightarrow 2$ rotational transition of the vibrational ground state of silicon monoxide (SiO) was detected and monitored in ECR plasmas of TEOS + O2 and SiH4 + O2. The integrated intensity of this microwave transition was used to obtain absolute line-integrated densities of SiO in both of these plasma chemistries. The deposition is done on a silicon wafer clamped onto a water-cooled wafer stage located 20-40 cm away from the ECR zone. Silicon oxide films were
deposited at a pressure of a few mTorr and at low wafer temperature. The chemical composition of the deposited films was obtained using infrared absorption and electron spectroscopy for chemical analysis (ESCA). The refractive indices of the films are estimated to be 1.45 by a single color ellipsometry. Dry and wet etch rates for plasma deposited silicon oxide films were compared to those for thermal oxide films and deposition rates were determined as a function of TEOS/oxygen or silane/oxygen flow rate.

*This work is supported by the National Science Foundation under Grant No. ECD-8721545. Most of the equipment for constructing the microwave spectrometer was donated by Hewlett-Packard Co. and the absolute temperature controller and TEOS were donated by Schumacher Co.

PS-MoP3 Plasma Enhanced Chemical Vapor Deposition of Silicon Dioxide from Organosilane, K. H. A. Bogert and Ellen R. Fish, Department of Chemistry, Colorado State University, Fort Collins, CO 80523.

Plasma enhanced chemical vapor deposition (PECVD) of thin films is used extensively in the semiconductor industry. Understanding the process by which deposition of silicon dioxide, SiO₂, occurs from organosilanes is important for the development of superior dielectric materials for microelectronics. Several studies of the deposition of SiO₂ from tetraethoxysilane (TEOS) by PECVD have been published, although investigations of novel organosilane precursors have been few. Using an inductively coupled rf PECVD reactor, we have investigated the deposition conditions and the resulting films from a 100% TEOS plasma. FTIR analysis of SiO₂ films from our reactor indicated hydrocarbon incorporation in the film. Results from FTIR, SEM, and ellipsometry analysis will be presented for TEOS as well as for triethoxysilane, tetramethoxysilane, trimethoxysilane, and triethoxychlorosilane. Additions of oxidants, such as O₂, to the feed gases will also be investigated. Trends in film compositions produced by varying plasma conditions such as rf input power, starting materials, and substrate temperature will also be presented. Optimization of deposition conditions for SiO₂ films will allow further elucidation of deposition mechanisms using the imaging of Radicals Interacting with Surfaces (IRIS) technique.


Plasma enhanced chemical vapor deposition (PECVD) of thin films has found extensive use in the semiconductor industry. Additionally, plasma deposited polymers can be used for membranes, sensors, and protective coatings. The chemical mechanisms for plasma deposition, however, are not well understood. We are fundamentally interested in elucidating these mechanisms. We have recently investigated the deposition of thin films from inductively coupled rf plasmas. Plasma environments are characterized by optical emission spectroscopy and deposition. Films are characterized using ellipsometry, FTIR spectroscopy, and scanning electron microscopy (SEM). Results from deposition of “diamond-like” carbon and telethon-like fluorocarbon polymer films from fluorocarbon feed (C₂F₆/H₂, C₂F₆/H₂ and CF₄/H₂) will be presented. We will also present results from plasma deposition of silicon nitride films from various silicon (SiH₄, SiCl₂, SiBr₂) and nitrogen (N₂, N₂) source. Further, films with distinctive compositions deposited from organosilane/N₂ have also been produced. Trends in film compositions produced from varying plasma conditions such as input power, plasma pressure, starting materials, and substrate temperature are presented. Optimization of deposition conditions in these systems will allow further elucidation of deposition mechanisms using the imaging of radicals interacting with surfaces (IRIS) technique.

PS-MoP5 The Ion Energy Distribution at the Substrate Surface in a RF Induction Plasma Source, J. B. O. Caughman, Oak Ridge National Laboratory.

The energy of the ions bombarding the substrate can have an effect on the etching or deposition processes occurring on the surface, such as etch selectivities, etching/deposition rates, and thin film quality. The ion energies incident on the surface of the substrate in a high density radio-frequency induction source has been measured. The plasma source consists of a planar spiral induction coil and a multipole magnet bucket, with an i.d. of 20 cm. A gridded ion energy analyzer is embedded in a grounded substrate and can be located 10–25 cm from the coil. Plasmas of argon, helium, and hydrogen have been studied, with rf powers up to 2 kW and operating pressures of 0.5–10 mTorr. Measurements indicate that the ion energy distribution is initially broad (>50 eV) at low rf powers and narrows (<10 eV) as the discharge transforms from a low-density capacitive mode to a high-density inductive mode. The rf power range where the transition occurs changes dramatically with different gases and operating pressures. For example, at a power of 2 mTorr, the transition occurs at a power of ~100 W for argon and at a power of ~1000 W for hydrogen. In general, more rf power is needed to stay in the inductive mode as the pressure is reduced. The relationship between the ion energy distribution, the rf potential of the plasma, and the rf potential of the induction coil will be presented, along with processing implications.

*Research managed by the U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.


One of the problems found in the SiN, ECR deposition is the oxygen contamination of the films in some of the deposition conditions, but the origin of the oxygen has not been clarified. In this paper, the oxygen source and the deposition conditions for the oxygen incorporation into the SiN films, deposited at different microwave powers, P (50–250 W) and N₂/SiH₄ gases flow rates, R (1.6–9) are determined. Refractive index, infrared absorption spectra and film composition measured by AES are used to investigate the presence of oxygen. Oxygen content (%) is detected in the films deposited at R ≥ 5 and P ≥ 150 W. At R ≥ 5, the oxygen content is lower (3%) for the films deposited at 200 W and no oxygen is found in the films deposited at others powers. According to the presence of oxygen, the Si-N vibration mode is located at high wavenumbers (880 cm⁻¹) for the films deposited at R ≥ 5 and P ≥ 150 W. Films with lower oxygen content or without it show the Si-N mode at 830–850 cm⁻¹, depending on the Si/N ratio. Refractive index between 1.72–1.77 are obtained for the films deposited at R ≥ 5 and P ≥ 150 W and between 1.82–2.71 for the rest of the films. The analysis clearly shows a threshold in the deposition conditions (R = 5 and P = 150 W) for the oxygen incorporation into the films. Beyond this threshold value, the oxygen content remains constant. These results, and the fact that the oxygen only appears in the films deposited at high deposition rates, indicate that the source can not be the oxygen background pressure, but the spattering of the quartz liner located into the ECR source, more effective at high microwave powers and N₂/SiH₄ gases flow ratios.

PS-MoP7 Comparison of Measured and Computed Magnetron Sputtering Tracks for Alloys of Differing Magnetic Susceptibility, Daniel R. Juliano, David N. Ruzic, University of Illinois, 103 S. Goodwin, Urbana, IL 61801, Bill Manning, Phil Frausto, John Poole, Tosoh SMD, Grove City, OH 43123.

An electron tracking code was developed in order to predict ionizations inside a magnetron. The sputtering track is then predicted from the ionization profile. The code was modeled after T. E. Sheridan et al. The code requires as inputs the cathode composition and voltage, the geometry of the device, the magnetic field configuration inside the device, the gas composition, pressure, and density, and data on sputtering yields. The magnetic field configuration was calculated with a finite element analysis code. The sputtering yields depend on the energy and weight of the impacting ion (and hence the gas type) and are calculated using VFTTR. Two types of cathodes were studied—a non-magnetic Chromium and a magnetic Cobalt alloy. Differences in alloy magnetic susceptibility cause significant perturbations in the sputtering track utilization. Spattering yields were calculated with the code compared favorably with those measured in the magnetron. The code also compiles data for collision processes (elastic, excitation, and ionization) and outputs calculated spatial distribution of radiation for comparison to optical diagnostics in addition to predicting the sputtering track.

2ANSYS 5.0A, Swanson Analysis Systems.
PS-MoP8 Synergetic Sputtering Effects During Ion Bombardment With Two Ion Species, S. Berg and I. V. Katardjiev, Inst. Technology, Uppsala University, Box 534, S-75121 Sweden.

The modification of the sputtering yield during ion bombardment with two ion species of different masses is studied with the dynamic Monte Carlo code T-DYN. Specifically, the sputtering yield of low mass targets with low to medium mass primary ions is substantially enhanced when a second ion species of higher mass is introduced. This enhancement is due to the incorporation of the heavier ion species into the target material thus changing significantly the collision cascade behaviour of the primary ions. As a result, the energy deposited at the surface is increased and hence the sputtering yield is also increased. This sputter yield enhancement is found to be a function of the ion energy, angle of incidence, masses and atomic densities of the two ion species, steady state concentration of the secondary ion species at the surface (primary to secondary ion ratio), etc. The effect is found to be most pronounced at low energies where the sputter yield enhancement is predicted to increase by a factor of two or more. The mechanisms of the sputter yield enhancement as well as the modification of the energy deposition function are discussed in detail. These computer simulations have been verified experimentally in a number of situations.

PS-MoP9 High Aspect Ratio Si Etching for Microsensor Fabrication, W. H. Juan and S. W. Pang, Solid State Electronics Laboratory, Department of Electrical Engineering and Computer Science, The University of Michigan, Ann Arbor, MI 48109-2122.

To improve the sensitivity of micro-electromechanical devices, high aspect ratio Si etching for bulk micromachining is developed. Si is etched in a Cl₂ plasma generated by an electron cyclotron resonance (ECR) source. The ECR source provides fast etch rate with high selectivity since the reactive species density and ion energy can be controlled independently. Si etch rate increases with microwave and rf power but decreases with Ar and CFₓ power in Cl₂. Among different etch masks used for Si etching, Ni provides the highest selectivity (≈ 50) compared to thermal SiO₂ (≈ 7) and polyimide/novolac resist (≈ 1-2). Etch profile becomes more isotropic with increasing microwave power and temperature, but shows no significant dependence on pressure for the conditions used. Figure 1 shows Si structures etched with 10:1 aspect ratio. With 700 nm electroplated Ni as a mask, 1 μm wide features in Si were etched down to 10 μm deep with vertical profile. This high aspect ratio Si etching was carried out with 100 W microwave power, 100 W rf power, and 2000 sccm Cl₂ at 3 mTorr. The influence of etch mask on etch profile and surface morphology will be presented. Resonant based microsensors with high aspect ratio sensing elements will be fabricated.

PS-MoP10 Plasma Etching of Pt and Ferroelectric Thin Films, R. L. Rhoades and D. B. Poker, Oak Ridge National Laboratory*, Oak Ridge, TN; and B. S. Mercer, PlasmaQuest, Richardson, TX.

Ferroelectric materials such as PZT (lead zirconate titanate) and BST (barium strontium titanate) are of growing interest for ULSI memories, optical shutters, piezoelectric microtransducers, and a wide variety of sensors and detectors. Much progress has been made on depositing these materials as thin films; however, patterning and etching remains as a major obstacle to their use in devices and circuits. Etch rates of PZT, BST, and Pt with Ar/Cl₂ plasmas have been studied using an electron cyclotron resonance plasma etching system. Plasma parameters were varied over a broad range to determine near optimal values for microwave power, RF sample bias, Ar/Cl₂ ratio, substrate carrier temperature, and total pressure. Etch rates for these films were measured in the following ranges: PZT (275-1075 Å/min), BST (105-473 Å/min), and Pt (205-800 Å/min). Etch rate data was then used to determine a preliminary process for patterning of Pt/PZT/BST layers typical of integrated circuit requirements. Selectivity of Pt/PZT/BST was controllable in the range of 1:1 to 5:1.

SEM cross section of Pt/PZT/BST layers etched by Ar/Cl₂ plasma. (Sloped wall due to transfer of pattern from original photoresist.)


Two rf inductively coupled plasma sources are studied: a cylindrical source with a 12-turn coil of 0.3 m diameter powered by a 0.3 kW, 0.46 MHz generator, and a planar source with a 3-turn spiral coil of 0.25 m outer diameter powered by a 1 kW, 13.56 MHz generator. These sources generate pure oxygen plasmas in a 0.4 m diameter, 1.2 m length, vacuum vessel with gas fills of 1-10 mTorr. The unknown (O⁺, O₂⁺, and O³⁻) plasma composition is of particular interest to our oxide growth studies which take place 0.18 m inside the vacuum vessel. Hence, plasma composition at that location is investigated with several measurements: (1) a combination of Langmuir probe radial scans and line-integrated microwave interferometry measurements, (2) optical emission spectroscopy, including Argon actinometry of selected oxygen lines, and (3) mass spectrometry with a compact instrument built for these studies. The results of these measurements will be presented for the two inductive sources and compared to the plasma compositions predicted by a global source model.

PS-MoP12 Steady-State Helicon Plasma Experiments, J. Gilland, R. Brown, N. Hershkowitz, R. Breun, University of Wisconsin, Department of Nuclear Engineering and Engineering Physics, 1500 Johnson Drive, Madison, WI 53706-1687.

Helicon waves are studied in steady-state argon and helium helicon plasma created in a solenoidal magnetic field. Maximum plasma densities are the order of 10¹³ cm⁻³. Three types of antenna structures (Nagoya Type III, Dual-Half-Turn and Spiral) are investigated. Helicon waves at 13.56 MHz are excited and identified by the wavelengths of standing waves detected by B-dot probes mounted external to and inside the plasma. Plasma parameters are measured with Langmuir probes. Both radial temperature and density profiles are measured. A microwave interferometer provides a non-intrusive measurement of plasma density. A power balance between ambipolar diffusion losses and rf power deposition is found to agree with the experimental scaling with power, B field and neutral gas. Studies of the power balance are being extended to establish the self-consistent nature of the plasma column radius.

Supported by NSF Grant No. ECS-9120326.


A large area surface modification of membranes for medical applications is an important field of plasma processing [1]. A rf hollow cathode discharge is known as an efficient method to generate large area plasmas [2]. A slotted hollow cathode discharge (SHCD) plasma source [3] has been developed for large area treatment of fast moving membranes with a width of 35 cm. A membrane is moved between two symmetrical, capacitively coupled active rf electrodes (cathodes)
with slots perpendicular to the direction of membrane motion. The slots are open at both ends to establish an electrical contact between the plasma and the rf grounded electrodes (codes). The dependence between the plasma density and the geometrical dimensions of the hollow cathode slots were examined. For measurements of the ion density and its distribution along the slots a double Langmuir probe system has been used. A dense plasma with ion densities up to $1 \times 10^{12}$ cm$^{-3}$ is produced with up to 1000 W power of 13.56 MHz excitation for a pressure range from $5 \times 10^{-10}$ to 1 mbar with argon, oxygen and nitrogen as working gases. An rapid surface modification of the membranes allows a membrane processing speed of 20 m/min. An important advantage of the SHCD concept is its up scale ability. A plasma module for membranes with a width of 1200 cm is currently under development and will be described elsewhere.


PS-MoP14 Remote Source Characteristics of Helicon Discharges, Isaac D. Sudki and Francis F. Chen, EE Department, University of California, Los Angeles, 90024-1594.

Etching of silicon in helicon sources has been shown to decrease with increasing magnetic field, even though the plasma density rises. Presumably this is because at high fields the active neutral species is depleted by the extremely high ionization rate. To increase the etch rate, one has to decrease the electron temperature while maintaining high plasma density. We have made measurements of the downstream behavior of a helicon discharge several decimeters away from the antenna. The density is observed to rise and the temperature to fall, just as desired. Using the helicon discharge as a truly "remote" source not only makes the ion flux more uniform, but also increases the ion flux and the population of the etchant species. This feature distinguishes helicon sources from inductively coupled RFI sources, where the density is highest in the near field of the antenna.


PS-MoP15 High Density, Low Temperature Dry Etching in GaAs and InP Device Technology, S. J. Pearton, C. R. Abbarnethy and F. Ren, Department of Materials Science and Engineering, University of Florida, Gainesville, FL 32611, AT&T Bell Laboratories, Murray Hill, NJ 07974.

ECR etching of GaAs, AlGaAs and GaSb in Cl$_2$/Ar or BCl$_3$/Ar and of InP in CH$_3$Br/Ar discharges is performed down to $-30^\circ$C substrate temperature and characterized in terms of rate, anisotropy, damage introduction and near-surface stoichiometry. Below $-10^\circ$C the vertical etch rates of all these semiconductors decrease due to reduced volatility of the products, and there is suppression of lateral etching with chlorine-based mixtures, but no change in anisotropy of CH$_3$Br/Ar-etched features. Etching of typical dielectrics (SiN, Si$_3$N$_4$) and metals (W, WSi$_2$) used in bipolar or FET devices in SF$_6$/Ar discharges was also characterized, and for substrate temperatures below $-20^\circ$C increased concentration of fluorine-containing residues are found on the exposed semiconductor surfaces after removal of the dielectric or metal. These can be removed by a 500 W microwave ECR H$_2$ plasma with low additional ($-25$V) dc bias on the sample. The effect of additional outer collimating magnets on the etch anisotropy was also examined, and found to have a significant improvement at low ion energies. Condensation of the etch gas occurs at $-30^\circ$C for BCl$_3$, but is absent with Cl$_2$ or CH$_3$Br. The question of sampling during high density etching or deposition will also be addressed. The uniformity of etching was measured both with and without the lower collimating magnetic and no significant change was found.


The fabrication of ultra-large-scale integrated (ULSI) circuits requires increasingly tighter control of wall profiles in order to achieve high circuit densities. Low pressure, high plasma density sources are becoming the accepted technology in many development laboratories and will be installed in significant numbers in production lines in the near future. The advantages of these sources include more anisotropic etching, high etching rate uniformity, high etching rates, and independent control of the ion energy through wafer biasing.

In order to evaluate the etching performance of these new source technologies, high aspect ratio etching needs to be studied since ULSI devices impose even greater demands with respect to aspect ratio. In this paper, we report results for the etching of silicon trenches with HBr and Cl$_2$ chemistry using a low pressure, high density helicon plasma source. We explore the effect of trench depths from 0.5 to $1.5 \mu$m oxide mask thicknesses of 0.5, 1.0, and 2.0 $\mu$m, and rf bias of 150 W and 300 W on the resulting profiles of 0.35 $\mu$m and 0.60 $\mu$m wide trenches and a sidewall next to an open field. The presence of microetching, mask undercutting, mask faceting, and wall angle are quantified. For wafers with the 0.5 $\mu$m thick masks, we do not observe any aspect ratio dependent etching; however, the etching rate does decrease with etching time.


We etched high-aspect-ratio sub-half-micro contacts in SiO$_2$ using a high-density C$_4$F$_8$ plasma generated by an inductively coupled, low pressure reactor. As expected, oxide and resist etch rates increased with increased vertical sidewall angles as observed increasingly vertically sidewall angled layers and increasing microetching depth for higher rf bias conditions on contacts, trenches, and steps. Topography modeling shows that the microetch profiles are consistent with specular ion reflection from sidewalls and the increase in their depths with rf bias is consistent with the energy-dependent etch yields in high density, fluorocarbon plasma etch chemistries reported by Oehrlein, et al.


In plasma etching processes, the demand for the directionality of energetic ions on the wafer surface increases as feature sizes shrink. It was believed that sheet collisions were the main cause of the ion angular spread at the water surface. Therefore, by moving from conventional plasma sources to high density plasma sources, where the sheaths are basically collisionless, we would expect highly directional (beam-like) ions at the water surface. However, with the use of our overhang test structures, we have found a significant amount of ion angular spread in these systems, especially in those with low bias powers.

The possible causes for this ion angular spread are (1) the ion temperature of the bulk plasma, which can be much higher than what was believed (kT$_e$ > 0.2 eV); (2) the higher ion plasma frequency due to its higher density, and the shorter transit time in the sheath due to the smaller sheath thickness, which can make the ions responding to an AC field instead of a DC field in the sheath; and (3) distortion of the electric field in the vicinity of the feature owing to its topography and static surface charging. All these factors can broaden the ion angular distribution but in different ways. The ion angular spread caused by charging is feature geometry dependent, and that caused by AC sheath field is always accompanied by a much wider ion energy distribution.

We have used silicon wafers with overhang test structures to obtain experimental data. The most important feature of these structures is to let all ions including those very off-normal angled ones to arrive on a flat surface through apertures of different sizes. With the assistance of our etching and deposition profile simulation, SPEEDIE, which also includes a Monte Carlo sheath simulator, we are able to identify the causes and effects of the ion angular spread in various etching processes.

PS-MoP19 Modelling and Simulation of Neutral Beam Processing Using a Plasma Source, M. D. Kilgore and D. B. Graves, Dept. of Chemical Engineering, Univ. of California, Berkeley, CA 94720-1462.

Charge-free processing has been proposed as a means of avoiding damage to substrates that may occur as a result of plasma processing. We use simulation to investigate a system in which a high density inductively coupled plasma is used as a source of energetic, directed
neutrals for surface processing. The beaming neutrals are produced by charge exchange collisions as well as by surface ionization on a grid which separates a plasma source chamber from a wafer processing chamber. We apply a coupled plasma and neutral transport simulation to study the properties of the energetic neutrals at the wafer surface as a function of operating conditions. The inductively coupled plasma simulation includes fluid treatments of ion and electron transport coupled with Poisson’s equation for the self-consistent potential as well as solving Maxwell’s equations for the power input from the external coil. The neutral simulation is a kinetic treatment using the direct simulation Monte Carlo method. Our simulation results show the effects of varying plasma power, operating pressures and geometries of the two chambers, as well as gas flowrate and pumping speed. We model the interchamber grid to investigate how its design influences the beaming neutrals’ angular and energy distributions. We show results which illustrate trade-offs which must be considered from the downstream chamber operating pressure. Low pressure is desirable for beam directionality while high pressure is desirable for process rate and uniformity.


We present results of the influence of the ponderomotive force on the density and electrostatic potential profiles in inductive discharges. The ponderomotive force can be written as a gradient of a potential, and the resulting electron force scales with the discharge power and inversely with the discharge pressure. The effect of the force on density profiles is demonstrated using a one-dimensional analytic theory and a two-dimensional fluid simulation. For a cylindrical discharge with an inductive coil on one axial face, this force results in a reduced particle outflow toward the coil, and increases the density peak in the discharge chamber. The smaller flow toward the coil can reduce erosion on a dielectric window between the coil and the plasma. The higher overall density increases the density at the opposite axial face by a multiplicative factor which gives a higher etch rate but minimal change in radial uniformity for the top-coil configuration. For the side-coil configuration, the ion density at the wafer increases, the ion flux to the side-wall (and assumed dielectric window) is reduced, and the radial uniformity at the wafer improves.

**PS-MoP21** Modeling Of Oxide Etching In A High Density ICP Tool, R. A. Stewart, J. D. Bokowski, P. Vitello*, and D. B. Graves, Department of Chemical Engineering, University of California, Berkeley, CA 94720.

We present results from a two dimensional (r, z) model of an inductively coupled plasma tool used for etching of SiO₂. Both inductive and capacitive coupling from the coils are included in the model. Capacitive coupling in the r-z plane is also included. The discharge physics model is based on fluid equations for electrons, positive and negative ions. Neutral species transport and chemistry are coupled to the equations for charged species, including the creation and deposition of film precursors. We use a variety of fluorocarbon mixtures, similar to those used industrially. Results are compared to experiment where possible.

*Lawrence Livermore National Laboratory, Livermore, CA 94551.

**PS-MoP22** Spatially Averaged (Global) Model of Time Modulated High Density Electron研究生 Plasmas, S. Ashida*, C. Lee, and M. A. Lieberman, Univ. of California, Berkeley, Berkeley, California 94720.

The operation of plasma sources utilizing time modulated input power has attracted much attention recently because the technique yields another discharge parameter. In this study, a spatially averaged (global) model of time modulated plasmas has been developed and used to investigate argon and C₃H₄ gas discharges. The advantage of this model is that it requires much smaller computational resources than such models as Fluid or PIC simulations. It has been found that higher plasma densities can be obtained by selecting an appropriate pulse period, for the same average power as in the case of continuous wave operation. Other parameters, such as the duty ratio and the shape of the modulation waveform also affect the plasma density. In addition, we discuss the dependence of the radical density and the electron negativity of the C₃ plasma as a function of the duty ratio and the pulse period.

This work is performed with support in part from NSF Grant ECS-9217500, DOE Grant DE-FG03-87ER13727, and LLNL Contract W-7405-ENG-48.

*On leave from R&D Center, Toshiba Corp., Japan.

**PS-MoP23** 2D RF Sheath Structure due to Geometrical Discontinuities Outside the Wafer Edge, B. Lane, Plasma Dynamics.

2D simulation results of the time dependent RF sheath in the vicinity of the wafer edge are presented. Geometrical obstacles such as steps in the underlying electrode, dielectric focus rings and grooves affect the shape of the RF sheath and hence the flux and directionality of ions impinging on the wafer edge. The simulation follows fluid ions under the assumption that the directed velocity is much greater than the random thermal velocity; electrons are assumed to have a Maxwell-Boltzmann distribution and thus have an exponential dependence on the potential. The electrostatic potential is obtained by direct solution of the Poisson equation. All material surfaces are taken to be covered by insulators and the surface charge density is self-consistently integrated. The simulation volume focuses on the wafer edge and the effects of the core plasma enter through boundary conditions. This provides a detailed look at this critical region in a computationally feasible fashion at the expense of simplifying global processes which sustain the plasma. It is found that the sheath curvature near the wafer edge can be either concave towards or away from the wafer with consequent focussing or defocussing of the ion flux to the wafer edge; the curvature depends on the details of the geometry outside the wafer edge.

Work supported by SEMATECH.

**PS-MoP24** 2D Modeling of Time Modulated Inductive Discharges, V. Vahedi*, M. A. Lieberman*, G. DiPeso*, and D. W. Hewett*, *Lawrence Livermore National Laboratory, Livermore, CA 94550; IEES Department, UC Berkeley, CA 94720.

Recent measurements and reports of plasma behavior in time modulated discharges have increased the general interest in these types of discharges. The current belief is that a very short time (< 1 μsec) after the power is turned off, the fast electrons in the system, on the tail of the electron energy distribution, are depleted. As a result, ionization, and most other electron impact dissociative mechanisms with high energy thresholds, will be greatly reduced after this short time, and the time-average concentration of certain free radicals can be significantly modified.

We will present results from 2D fluid simulations of time modulated inductive discharges showing the dependence of the time-averaged electron temperature and plasma density on the duty-cycle and duration of the RF power signal. We will also show modifications to the plasma density profile due to the modulation.

**VACUUM TECHNOLOGY**

**Room BR4 – Session VT-MoP**

**Vacuum Science and Technology**

**Moderator:** L. D. Hinkle, MKS Instruments Inc.


The rotating disc gauge measures high and potentially ultra-high vacuum by sensing the torque transferred by molecular drag between a disc rotating at high speed ~ 1000 Hz and a close coaxial disc which intercepts molecules leaving the rotor. In an early version of the device (J Vac Soc 92, 331) in which the disc is made by the process of the disc was measured by a classical torsion balance method we showed that it is capable of absolute measurements at 2 x 10⁻⁷ mbar with accuracy of order ±3%. The achievement of better sensitivities—closer to the expected theoretical limit ~ 10⁻¹⁰ mbar—was limited by vibrational problems.

In a new version of the RDG the sensor disc is freely supported by
a high temperature superconductor/magnet suspension and a dynamic technique used to measure its acceleration from rest. A principal aim of the work is to achieve a torque-free suspension system so that all the induced motion can be attributed to molecular drag, thus eliminating the term which corresponds to "residual drag" in the spinning rotor gauge, and thus limits the sensitivity of that device.

Progress towards this aim will be reported with particular emphasis placed on the development of a novel UHV compatible HTS-magnet suspension.

**VT-MoP3** Measurement of Xe Pressure by Photoelectron Counting with the Imaging Counter, Shigeyuki Sekine and Shingo Ichimura, Electrotechnical Laboratory, 1-1-4 Umezono, Tsukuba, Ibaraki 305, JAPAN, e-mail: sekine@el.go.jp, FAX: +81-298-58-5733.

Measurement of xenon (Xe) pressure was carried out by using the photoelectron-counting method adopting a laser ionization technique. For the non-resonant multiphoton ionization of Xe atoms, produced photoelectrons were extracted by a electrostatic lens and were detected by a combination of a microchannel plate and a fluorescence screen. Each bright spot on the screen corresponded to a photoelectron. The spot image was photographed by a CCD camera with a repetition rate of the laser, and the number of spots on the screen was counted by an image processor for each laser shot.

The number was measured as a function of Xe pressure in the ultrahigh vacuum (UHV) region, and a simple proportion was successfully observed. The number of photoions was also counted with the same apparatus by changing a polarity of the extraction voltage. The overall detection efficiency for both results was evaluated.


Continuous Electron Beam Accelerator Facility (CEBAF) and MKS Instruments have recently signed a Cooperative Research and Development Agreement (CRADA) for developing an ultrahigh sensitivity He leak detector. This new leak detector is based on the technique of dynamic desorption and detection of He, which was developed at CEBAF for the production of ultra leak tight Superconducting Radio Frequency Cavity pairs operating at 2K. A closed cycle He refrigerator operating at ~8 K and a low mass range quadrupole electron multiplier RGA are required for this leak detector development program. A low He detection limit (~10^-11 Torr) in the presence of a large hydrogen (~10^-10 Torr) background is an essential requisite for the RGA with an electron multiplier detector. In this paper we present the results of our investigation of high frequency operation of RGA’s for achieving the above goals. The long term stability of the RGA’s will also be presented in this paper.

*This work supported by U.S. DOE Contract No. DE-AC05-84ER40105.


The authors have been engaged in efforts to reduce gas desorption of UHV materials, especially on aluminum alloys. The special extraction technique succeeded in making thermal desorption the dominant desorption process at lower temperatures. An oxygen plasma treatment improved the dynamic gas desorption about one order. In both treatments however, carbon remains on the surface and in sputter-profiled layer. Carbon deteriorates dynamic gas desorption coefficient. To eliminate carbon, ozone treatment was applied on metal surfaces. The surfaces of aluminum, copper, and stainless steel without any treatments were exposed to ozone using an ozone pump with flowing rate of 1/min for 24 hours. The samples were shown to become less carbon, especially on Cu and Al surfaces, by AES with a turbomolecular and a dry backing pump. In addition, no carbon was detected in the sputter-profiled layer. The detectability of AES on the order of 10^-3 and number of carbon atoms could be 3 x 10^11 cm^-2. The thickness of the oxidized layer was 6.5, 8.1 and 3.0 times of the native oxide layer for Cu (120 Å), Al (170 Å) and stainless steel (60 Å), respectively. X-ray photoelectron spectroscopy shows: 1) The ratio (O/Al) is 1.5 for the treated and 1.3 for the non treated. 2) Cu^+ is included in the treated but Cu^2+ is dominant for the non treated. The surfaces are further etched with low carbon adsorption characteristics because no carbon detected by AES on the surfaces stored in a covered glass dish more than four days. The photo-desorption yield of similarly treated aluminum surface was 1.7 x 10^-11 molecules per photon at a dose of 1.4 x 10^9 photons cm^-2 using TRISTAN accumulation ring synchrotron radiation facility. This is two times of the yield of copper, obtained with the same system at a same dose. The layer, in conjunction with the simple ozone treatment, makes an ideal contamination material for storage rings and other XUV systems and components.

**VT-MoP6** Outgassing Reduction of Type 304 Stainless Steel by Surface Oxidation in Air, K. Odaaka, S. Ueda, Mechanical Engineering Research Laboratory, Hitachi Ltd., 502 Kandatsu, Tsuichiura-shi, Ibaraki 300, Japan.

The effect of surface oxidation in air on the outgassing reduction from stainless steel were investigated. The outgassing measurement system of throughput method was fabricated from type 304 commercial-grade stainless steel and was buff-and-electro-polished before the prebaking at 450°C for 30 h. The test chamber had surface area of 1.5 m² and was evacuated with a TMP. The orifice was 5.6 mm in diameter. The two extractor gauges were calibrated using a spinning rotor gauge. An evacuation pump including the baking at 200°C was repeated for four times. An outgassing rate of 6 x 10^-11 Pam’s^-1 was obtained after 200°C baking. Then, the system at room temperature was exposed to air and was heated and kept at 200°C for 3 h to oxidize the surface. Oxidation in air resulted in remarkable reduction of the outgassing rate after baking. During the first evacuation after oxidation in air, 2 x 10^-11 Pam’s^-1 was obtained. In the following 3 evacuation cycles, outgassing rate reached 3 x 10^-11 Pam’s^-1 repeatedly. Residual gas analysis showed a significant reduction in the amount of hydrogen after oxidation in air. On the other hand, oxidation in air did not affect the outgassing characteristics before baking. This may be due to an oxide layer on the stainless steel surface preventing hydrogen from diffusing out of the metal and escaping into the vacuum.


The vacuum system of the SRRC 1.3 GeV electron storage ring is designed as a low contamination, high brightness system. In addition to the standard treatments for the UHV components, several contamination reduction methods were adopted in the manufacturing processes. Each of these processes such as oil-free machining processes, completely oil-free pumping system and duct controlled installation processes. The profits of the low contaminated treatments were proved from the results of beam cleanup, low dust trapping effect and low carbonaceous residual gases in the vacuum system.

a) also at Institute of Nuclear Science, National Tsing-Hua University.


Electron storage rings tend to be operated for high beam current, especially for a beam pipe in B-Factory which is constructed by KEK-Japan and SLAC-US. Copper has been currently used the beam pipe materials under high current, however it costs higher than an aluminum alloy which is inferior under heat load for photon. Actually it is necessary to consider both cost and heat load limits. We have considered three models. 1) Cu, 2) Cu/Al hybrid, and 3) Al. We have analyzed three dimensionally by super-computer on time distribution, heat deformation, and heat stress in the heat load 5 kW/m, 7.5 kW/m and 10 kW/m. Particularly in model 2 Cu/Al hybrid method, we proposed a new configuration mentioned below. a) Material of a photon absorber is used copper bar/water cooled aluminum alloy clad. The beam chamber is an extruded aluminum alloy clad. b) Cu/Al clad is an explosion bonding method. c) Structure of weld parts is simplified to enable automatic TIG. d) Many slits of the absorber can reduce heat stress and heat strain. e) The particle free can be kept for
the cycle heat strain between the absorber and the beam pipe. Highest temperature of the absorber is 190°C for 10 kW/m of heat load in aluminum alloy, 160°C in Cu/Al hybrid systems, and 145°C in copper. In the analysis of heat stress, the hybrid system can be used in wide range heat load with slits of the absorber.


In order to investigate the dependence of outgassing rate on pumping speed in a vacuum chamber which is pumped at room temperature, a small test chamber made of 304 stainless steel which has a volume of 20 l and a surface area of 7200 cm² has been constructed. The chamber has been pumped after air exposure for 3 days with six kinds of pumping speeds in a range from 200 l/s to 0.36 l/s through orifices and the outgassing rate of the chamber has been determined from the measured final pressure at the pumping time of 72 h. As a result of the experiment, it is shown that the observed outgassing rates q are expressed with two sets of power functions of \( q = q_0 S A^{-n_1} \) and \( q = q_0 S A^{n_2} \) where S/A is a pumping parameter defined as the ratio of pumping speed S to surface area A, exponents of \( n_1 \) and \( n_2 \) are \( n_1 = 0.8282 \) for S/A \( \geq 10^{-2} \) l/sec and \( n_2 = 0.4655 \) for S/A \( \leq 10^{-3} \) l/sec respectively.


We propose a novel evacuation system with fluorine being injected into the discharge port of a turbomolecular pump (TMP) to improve effective hydrogen compression ratio. The mechanism of this improvement is to convert hydrogen into heavy weight molecule, such as HF, which has high compression ratio, by the reaction between fluorine and hydrogen in the discharge port. It was confirmed that the effective compression ratio increases to \( 10^{4}-10^{6} \) from \( 10^{3} \) in the range where the hydrogen partial pressure in the evacuated chamber was more than \( 10^{-2} \) torr by the TMP system.


In order to optimize the design of the distributed ion pumps (DIPs) to be used for a muon storage ring currently under construction at Brookhaven, the pumping speeds of several DIPs were studied in various magnetic fields. The DIPs were subjected to magnetic field B, ranging from several hundred Gauss up to 15 Kilogauss, and misalignment angles \( \delta \) (between the direction of B and the anode axis) from 0 to 13 degrees. The pumping speeds of the DIPs with cell radii of 9 and 12 mm peaked at the transition magnetic field, \( \delta = 1 \), then dropped off rapidly with an increasing magnetic field. The pumping speeds of these DIPs were not as sensitive to misalignment with respect to B as the DIPs with smaller radii. The pumping speeds of DIPs with cell radii of 5 and 6 mm remained relatively constant with an increasing magnetic field, but decreased rapidly when subjected to misalignment. The results of this study are in reasonable agreement when compared to the calculated pumping speeds using Suetugu's empirical formula.


Claw rotor compressors are used in a wide variety of vacuum pumping applications. However, the published literature on these machines offers little guidance on the physical processes which occur during pump operation, their effect on pump performance or the influence of design and operating parameters. Equations describing a multistage pump mechanism are derived. The equations are based on the laws of conservation of mass and energy and use a lumped parameters approach. The equations describe pressure and temperature changes due to volume variation, gas flux between stages, leakage within and between stages, gas mixing, and heat exchange. Expressions for the pumping speed and power consumption are also provided. Predictions based on the model are compared with experimental data.


Characteristics of H2O water pumping by glow discharge with LaB6 cold cathode are studied. For the purpose of development of a new type of sputter ion pumps, Lanthanum Hexaboride (LaB6) is applied for the cathode material on the basis of the preliminary study. Use of LaB6 for the cathode of a sputter ion pump was proposed to develop a low outgassing wall for vacuum chamber. The study showed that the vacuum chamber with the LaB6 sputter coating reached a low ultimate pressure than that without coating. The QMS analysis showed that the pressure of residual water vapor was remarkably reduced before coating. The experiment showed that LaB6 coating by glow discharge on stainless steel wall is effective to reduce the H2O outgassing rate of a vacuum wall. And the pumping speed of H2O is also estimated.


It is true that little attention has been paid to the metals other than stainless steels and aluminum alloys for the materials of vacuum chambers in UHV (Ultra High Vacuum). Titanium is another candidate new material for UHV and XHV (Extreme High Vacuum). It has such advantages as light weight, high corrosion resistivity, and small thermal deformation. To obtain basic data for the application of titanium to UHV and XHV, various samples made of stainless steel and titanium with specific fabrications and surface treatments were prepared. The outgassing rates of those samples in the bake out process at 2000°C have been measured. It was found that titanium has low outgassing rate and can be used for the material in UHV. Coating with TiN is also found to lower the outgassing rate. The gases outgassed from those samples were analyzed by quadrupole mass analyzer. Interesting results were obtained in the change of outgassing rates of hydrogen of those samples. Taking those results into consideration, a vacuum chamber made of electro-polished stainless steel and a vacuum chamber made of titanium coated with TiN were prepared and pumped down into XHV region. The pump down curves were compared and discussed.

VT-MoP15 Recombination Limited Outgassing of Stainless Steel, Bowle, C. Moore, Consultant.

It is common practice to bake stainless steel vacuum chambers and equipment in order to reduce hydrogen outgassing. The removal of atomic hydrogen from the bulk metal follows classic diffusion theory, at least at the start of bake. When low levels of outgassing are sought, experiment diverges radically from these expectations. Observed hydrogen outgassing of vacuum baked 304 stainless steel can be as much as a factor of \( 10^3 \) larger than predicted by diffusion theory. This theory assumes that all of the atomic hydrogen diffusing to the surface recombin into molecular hydrogen and escapes into the vacuum; none returns into the bulk metal. However as the surface coverage of atomic hydrogen is reduced, the recombinaton rate falls as the square of the coverage. It has been suggested that a recombinaton limit could be the cause of the high outgassing rates. It is the objective of this paper to test this suggestion. Recombination rates for atomic deuterium have been reported. These are used as a rough approximation to hydrogen rates. A numeric analysis is used to estimate the outgassing rates and the internal concentration profiles for specific bake procedures. The results are much closer to experiment than the simple diffusion theory. Adding the recombinaton limit is a useful step towards the reliable prediction of postbake outgassing rates.

1Work performed under the auspices of the U.S. Department of Energy.


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BIOMATERIAL INTERFACES
Room BR4 – Session BI-MoP

Biomaterial Interfaces
Moderator: M. J. Tarlov, National Institute of Standards and Technology.

BI-MoP1 Nanobiology: The Action of Picoscnd Nanometer-Dr. Pulse on DNA, Gong Lisan, Wu Junhan and Gong Weisi (South China Normal University, Guangzhou, GD 510631, China). EXPERIMENT SAMPLE DNA (Biological Informational Source) was treated with YAG (Ultrafast Source). PURPOSES: 1. to study the action of ultrafast nanometer-scale pulse on DNA; 2. to explore the possibility of cleavage of DNA. MATERIALS: 1. Sample DNA (bird-DNA, rDNA-HindIII); 2. YAG 266 nm, 30 ps, 50 uJ and then examined by spectrophotometry and electrophoresis. RESULTS: 1. the spectra of the tested was DNA’s typical spectra; 2. the forms of the DNA spectra did not change after the treatment of ultrafast pulses, and their peaks were still at 260 nm, but their peak values increased, showing hyperchromic effect; 3. the result of agarose gel electrophoresis revealed tailing of the DNA treated by pulse. DISCUSSION: 1. the hyperchromic effect and the tailing indicated that the structural bonds of DNA were cleaved by ultrafast pulses; 2. DNA was broken into structural units at different levels; 3. DNA photolytic products (photodimer, photohydrate, photosensitizer and transiive species, etc.) were liberated. Obviously, ultrafast pulses can change DNA molecular structure and cleave DNA. Unfortunately, under the condition of this experiment, we did not observe the cleavage on the predesignated site of DNA by YAG. As to the prospect for the future, it is possible that the combination of molecular biotechnique and ultrafast optical technique may eventually achieve cleavage on predesignated site and splicing of DNA in order to create new species.


Double-stranded DNA is a weak conducting structure. Besides, even when using PAO3 DNA the known length (50 nm), we were always able to distinguish unambiguously the sticklike structure on HOPG surface and DNA real molecules. Therefore to simplify STM image interpretation, we used single-stranded oligonucleotide of 20 nucleotide length. At low ionic strength corresponding to the melted state of DNA, single-stranded oligonucleotide with length of 20 nucleotide pairs is in the helical state in solution at room temperature. The oligonucleotide length measured at T = 0.1 was 14-15 nm. Our value for DNA length (17-18 nm) obtained at low ionic strength indicates the more flexible bonds of DNA sugar-phosphate backbone.

BI-MoP3 Fabrication of Patterned DNA Surfaces Using Lithographic Modification of Self-Assembled Organic Monolayers, Linda A. Chierici1, C. Elizabeth O’Ferrall2, Paul M. Roberts2, Walter Dressick1, Charles S. Dulcy1, Douglas B. Chrisey3 and Jeffrey M. Caiverti1, Naval Research Laboratory, Center for Bio/Molecular Science and Engineering (Code 6000), Washington, DC 20375-5348, and 2Geo-Centers, Inc, Fort Washington, MD 20744.

Self-assembled monolayer (SAM) films of various organosilanes have been characterized as surfaces for the spatially-controlled, selective attachment of single-stranded synthetic DNA. Patterns of DNA on silica substrates have been created by photochemical modification of the SAMs through lithographic masks to produce regions which promote or resist DNA attachment. The attachment of DNA to SAM-modified surfaces was followed using UV spectroscopy, radioassay, ellipsometry, and enzyme-linked immunosassay. We have also demonstrated that the immobilized DNA is capable of selective hybridization to a complementary oligomer, thus this attachment format has potential for the development of multiplexed DNA-based biosensors.

BI-MoP4 Contact versus Tapping Mode™ AFM Imaging of Native Chromatin, J. Vesena, I. D. Martin, R. Miller*, E. Henderson, and D. D. Larson, Dept. of Zoology and Genetics and Dept. of Mathematics, Iowa State University, Ames, IA 50011. Associated and dissociated chromatin structures1 are examined in both contact and TappingMode™ atomic force microscopy (AFM). Probe apices are characterized using colloidal gold standards2, AFM probe of nominally same curvature of the surface can significantly different lateral structural details of the chromatin, though similar vertical height, in these two imaging modes. These comparisons provide more evidence for lateral broadening of specimens due to shear forces. The consequence of this broadening mechanism on image reconstruction, the process of removing the image distortion due to finite tip radius, is discussed.

2. TappingMode™ is a registered trademark of Digital Instruments Inc., Santa Barbara, CA.


Absorption of water, (hydrated) ions and biomolecules constitute important early events at the interface between materials and tissue, such as in medical implants. Model experiments, using surface science methods (UHV techniques, single crystals, ...), are potentially useful for obtaining information about the molecular interactions taking place at such interfaces. In this work we are studying the adsorption and coadsorption of water and different amino acids on a Pt(111) surface using TDS and XPS as the main analytical techniques.

High resolution TDS of water adsorption shows that water starts to form multilayers and clusters after 80% saturation of the first monolayer. The sticking coefficient is slightly higher on clusters/multilayers than on the clean Pt surface. Under certain conditions bilayer formation can be observed, as evidenced by an additional peak that appears before clusters and multilayers start to form.

Depending on their side-chains and physical properties, different amino acids offer wide spectrum of possible adsorbate-surface and adsorbate-adsorbate interactions, for example different binding energies, adsorbate orientations, proton exchange reactions, and surface stability. Platinum is a particularly interesting substrate in this respect, in view of its catalytic activity for many reactions involving H2, O2, H2O, CO, ... Results from experiments exploring the effects mentioned above will be reported.

BI-MoP6 XPS and In vitro Studies of TiNi Surfaces Modified Using Various Sterilization Procedures, S. A. Shabatovskaya, J. W. Anderegg, J. E. Cunnick, Army DAAMID, D.O.E., and Department of Microbiology, Immunology, and Preventive Medicine, Iowa State University, Ames, IA 50011.

TiNi has emerged as space memory implant material with unexpectedly good biocompatibility. Despite widespread applications of these alloys in Russia, China, and Germany, nothing is known about their surface chemistry. Since nickel is a well-known antagonist of living tissues, there is a question regarding the role the alloy components play in the surface formation. The results of the first XPS surface studies of nearly equiatomic TiNi alloys chemically etched, autoclaved in steam and in water, exposed to hydrogen peroxide solution are presented. The type of oxide layer, its thickness as well as depth distribution of elements critically depend on the layer formation conditions. In general, TiNi alloys revealed a tendency to be covered by mono- and multilayer oxides with Ni atoms incorporated in the oxide lattice. Chemical etching selectively removes nickel from the surface. Hydrogen peroxide produces oxide with the highest nickel concentration (~20-30 at. %). The increase of the duration of autoclaving affects the surface nickel concentration in opposite ways depending on whether
environment is water or steam. The appropriate choice of treatment allows us to reduce the Ni concentration down to 5 at. % in surface layers as deep as 5-15 μm. Surface treatment procedures allowing to completely eliminate nickel from the surface have been developed. Variation of the alloy composition in the range 49-51 at. % Ni accompanied by structural change does not affect the surface state.

XPS investigations was followed by in situ studies of rat spleen cells behavior. Exposure of cells to autoclaved TiNi samples induced a slight increase in plating efficiency compared with cells exposed to autoclaved Ti samples and "cells alone". Samples exposed to hydrogen peroxide reveal a severe toxic effect comparable with that of pure nickel. Different biological response to TiNi surfaces requires further studies.

**Bi-MoP7** Nano-Scale Mechanics and Morphology of Laser Ablated Tooth Enamel, C. A. Dikabulo*, T. Tanganyong*, J. E. Houston**, T. A. Michaelis*, L. L. Warren** and P. Wilder-Smith**; *, **Department of Chemistry and Interface Science Department, Sandia National Laboratories, Albuquerque, NM; *Department of Chemistry, University of Western Ontario, London, Ontario, Canada; ** Balcomb Laser Institute & Medical Clinic, University of California, Irvine, CA.

It has been observed that human tooth enamel which has been surface treated with an excimer laser (λ = 308 nm) prior to fluoridation is more decay resistant than enamel which has been fluoridated alone. We have studied the surface of tooth enamel which has undergone this pre-treatment using interfacial force microscopy (IFM) to probe the mechanical properties of the surface, and both IFM and atomic force microscopy (AFM) to probe the surface morphology. The enamel consists of apatite crystals imbedded in a protein rich organic matrix that makes up less than 1% of the enamel mass. Preliminary results indicate that the laser preferentially ablates the organic material between the crystals, resulting in a rougher enamel surface. In addition, the irradiated enamel can be permanently deformed in IFM loading experiments, whereas the non-irradiated enamel exhibits an elastic response to loading, i.e., there is a large hysteresis in the force vs. displacement curve, but no permanent deformation. We will discuss possible models for the enhanced decay resistance of the laser pre-treated enamel in light of these results.


This work performed at Sandia National Laboratories is supported by the U.S. Department of Energy under contract DE-AC04-94AL85000.

**Bi-MoP8** Surface Characterization of Titanium Implants, G. N. Rakkar and J. C. Gregory, Surface Science Labs, University of Alabama, Huntsville, AL 35899; J. L. Ong and L. C. Lucas, Dept. of Biomedical Engineering, University of Alabama, Birmingham, AL 35294; D. Kawahara and M. Nakamura, Dept. of Biomaterials, Osaka Dental University, Osaka 540, Japan.

The initial biocompatibility of titanium (Ti) implants is associated with surface and bulk properties; hence surface characterization of these implants is critical for their clinical success. A goal of this study was to characterize the surface composition of Ti oxides after different surface treatments. In this paper, we present the results of XPS, AES, FTIR, and Raman spectroscopy performed on surface modified Ti (ASTM F67) samples which were immersed in α-MEM solution. Initial surface studies revealed an amorphous oxide layer on all the samples similar in composition to TiO2. After exposure to α-MEM medium, deposition of elements such as Ca and P increased, and exhibit a chemistry similar to brushite. The Ca/P ratio increased gradually, approaching 1.2 after 12 days of immersion in solution. FTIR analyses indicated an amorphous/finely crystalline Ca-P layer after immersion in α-MEM solution. In summary, an amorphous/finely crystalline Ca-P layer, having a chemistry similar to brushite was observed on all immersed samples.

**Bi-MoP9** SMS Characterization of Adsorbed Protein Films, B. Hagenhoff, A. Benninghoven, Univ. Munster, Munster, FRD and D. G. Castner, B. D. Ratner, Univ. of Washington, Seattle, WA.

One of the first events that occurs when a material is placed in the biological environment is the adsorption of proteins. The structure of the adsorbed protein film can have a significant effect on further biological events (e.g., cell attachment) and the biocompatibility of the material. This study uses ToF SIMS and XPS to investigate the structure of albumin and fibrinogen films adsorbed from buffer onto PTFE and Ti substrates. Protein concentrations from 0.0005 to 0.5 mg/ml were exposed to the substrates for 2 hrs, then the samples were rinsed and dried. Characteristic secondary ion fragments from the constituent amino acids (glutamic, alanine, serine, proline, valine, cysteine, leucine, tyrosine, etc.) of the proteins were detected by SIMS. Cation PTFE samples, both protein coated and PTFE films not adsorbed at all protein concentrations studied. Since the thickness of the protein molecules is larger than the SIMS sampling depth of ~20 Å, this indicates that PTFE surface is not fully covered by protein, in agreement with previous XPS studies. The intensities of the amino acid fragments correlated directly with the amount of N detected by XPS, and thereby the amount of protein adsorbed to the surfaces. The intensities did vary as a function of the protein, substrate, and protein concentration. These differences indicate that SIMS intensities of the constituent amino acid fragment can be used to identify both the type and conformation of adsorbed proteins. This will require correlating SIMS intensities to biochemical measurements of protein conformation.

**Bi-MoP10** Extraction of Quantitative Surface Information from Static SIMS Using Partial Least Squares, B. D. Ratner, V. H. Perez-Luna and P. Favia, Department of Chemical Engineering, University of Washington, Seattle, WA 98195.

Plasma deposited films (PDS) of fluorinated precursors exhibit low ionomogeneity which correlates with the high retention of the proteins adsorbed on them. This high retention has been ascribed to the presence of CF3 groups at their surface, which promote strong hydrophobic interactions between the substrate and adsorbed proteins. Because of the complex surface chemistry of PDS, other groups may also be implicated in the high protein retention observed. To ascertain the role of other functional groups in protein adsorption and retention, a set of fluorinated substrates were prepared by plasma deposition of CF3/H2 and CF3/CH3 mixtures. Surface characterization of these films was performed by ESCA (elemental composition and C1s spectra), static SIMS (positive and negative ions), and wettability measurements. 125I-Fibrinogen (FB) was adsorbed from a 1% baboon plasma solution and the adsorbed FB was eluted with a 1% sodium dodecyl sulfate solution. The percentage of the initially adsorbed FB remaining on the surface after the elution step is referred to as the retention. FB adsorption and surface properties were calculated by using partial least squares (PLS) regression. Preliminary results show that wettability (cos θ), CF and C-CF2 groups correlate positively with the amounts of FB adsorbed. F/C ratio, CF2 and CH3 groups correlated negatively with the amounts of FB adsorbed. F/C ratio, and CF2 groups correlated positively with retention, C-CH2, and CH3 groups correlated negatively with retention. In addition, several SIMS peaks were also identified that correlated with the retention or the amounts of adsorbed FB, specifically, certain sulfur contaminants were found to have a significant effect in protein sorption even at trace levels. This work illustrates the application of multivariate calibration methods to build quantitative models and the extraction of qualitative information from them.


Bones are considered piezoelectric, but inverse effect has not been properly checked yet, because of the predicted elongation, << 1 Å when 100V is applied to the sample.

We used two resonant microwave cavities. One acts as a reference. The length of the other is controlled by the deformation of a bone sample, when a square-wave voltage is applied. A klystron provides a periodic sweep in frequency. The resonance peaks for the two cavities are electronically compared. This results in a measure of displacements, after averaging over 300 ~ 400 cycles.

Such a device measures displacements down to 0.05 Å. When no averaging procedure is used, the system is still capable to detect 50 Å displacements. Reproducibility is 10% and improvements are shown to be achievable. The output is a linear function of displacements. Measurements up to 1 ~ 10 μm are possible in a single run. We could therefore measure at the same time both the "piezoelectric" sensor's coefficients and the thermal capacity of the bone sample.

In conclusion, our device is suitable to measure displacements on a sub-Angstrom scale. The detection is linear over a wide range, while other methods are known to be strongly nonlinear. No similar microwave device is at present commercially available.

Our numerical results support doubts on the true piezoelectric nature of the electromechanical effect in bones, and on some procedures currently in estimating bones' piezoelectricity. This will be of help in the search of a satisfactory model for bone growth and remodelling.

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Bi-MoP12 Interactions of Cells and Proteins with Self-Assembled Monolayers (SAMs), K. E. Foster, J. J. Hickman, K. E. Foster, D. A. Steger, A. E. Shaffner, and J. L. Barker; Science Applications International Corporation, McLean, VA 22102; Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, DC 20375; Laboratory for Neurophysiology, BNP, DIR, NINDA, National Institutes of Health, Bethesda, MD 20892.

We have been using silane SAMs to modify surfaces and then studying how neurons interact with these modified surfaces. We have surveyed over 20 SAMs with two different cell types, rat spinal cord neurons and hippocampal neurons, and with different growth media. The growth media and cell preparation has also been varied in certain experiments. We find that the protein adsorption during cell culture is variable on different types of surfaces and has profound effects on neuronal survivability and viability. We have characterized the surfaces before culture by X-ray photoelectron spectroscopy (XPS) and contact angle and after culture by XPS, contact angle, and ellipsometry. We will report on some interesting correlations of cellular responses to initial surface characteristics.

Bi-MoP13 Amino Acid Adsorption on Polyfunctional SAMs of Alkylthiols, K. Uvdal, S. V. Atre*, D. Allara*, and B. D. Ratner, University of Washington, Seattle, WA 98195, Penn State University*, University Park, PA 16802.

Amino acid adsorption on alkylthiol monolayers is investigated by high resolution X-ray photoelectron spectroscopy (XPS). Alkylthiol, (HS(CH$_2$)$_x$X), monolayers with four different terminating groups are studied. The terminating groups are -CH$_3$, -OH, -COOH and -COOCH$_3$.

The properties of the alkyl monolayer modified gold surface is strongly correlated to the functionality of the terminating groups of the thiol monolayer. Glycine adsorption on to these alkylthiol monolayers is studied and the pH for the amino acid adsorption solution is varied as well as the adsorption time. Alkylthiol monolayers prior to adsorption are used as references to the alkylthiol monolayers immersed into glycine solution.

High resolution XPS spectra for alkylthiol monolayers immersed into glycine solution are presented. The amino acid adsorption on to the alkylthiol monolayers is detected in the C1s and the N1s core level spectra. The peak position of the N1s peak indicates the type of surface interaction. The affinity for glycine on these alkylthiols is strongly dependent of the choice of the terminating groups.

Bi-MoP14 One-Step Immobilization of Biomolecules and Application in Biosensors, A. Hartmann, D. Bock, and S. Seeger.

Immobilization of biomolecules has advanced to become a major topic in biosensor research. So far, classical immobilization techniques have the disadvantage of the high number of preparative steps in combination with long incubation times. The Langmuir-Blodgett (LB) technique allows a one-step transfer of biofilms from the air-water interface to the substrate. Therefore stable transferable monolayers of e.g. antibodies—incorporated in a photopolymerizable matrix—are prepared. Film stabilization can be achieved by irradiation of mixed monolayers of phthalocyaninato-polyisoxiane-derivatives and the biomolecules. Photochemical crosslinking is monitored by the film area decrease with irradiation time and significantly enhanced density and stability of active immobilized antibodies. Using an evanescent wave biosensor, laser diode induced fluorescence emission of dye-linked antigens is observed so that antigens can be detected. The presented method allows the preparation of films of high stability in only a few minutes. Furthermore, this new procedure can be highly paralleled and automatized.

Bi-MoP15 Polymeric Microcapsule Arrays—A Novel Method of Bioencapsulation, Ranjani V. Parthasarathy, Charles R. Martin, Department of Chemistry, Colorado State University, Fort Collins, CO 80523.

We describe a novel method for encapsulating enzymes in conductive polymer microcapsules. Microporous polycarbonate filtration membranes were used as "templates" to prepare microcapsules of polypyrrole. The microcapsule arrays are prepared via a combination of electrochemical and chemical polymerizations. These polymeric microcapsules are hollow and typically have uniform diameters of 400 nm and lengths of 10 μm. They are arranged in a high density array in which the individual capsules protrude from a surface like the bristles of a brush. Five enzymes—catalase, glucose oxidase, subtilisin, trypsin and alcohol dehydrogenase have been encapsulated using this method. Standard array methods were used to show that the encapsulated enzymes are catalytically active. The enzyme-loaded microcapsule arrays can function as enzymatic bioreactors in both aqueous solution and organic solvents. A scanning electron microscopic image (See attached figure) revealed the unique brush-like arrangement of these capsules. This general route to enzyme immobilization should find applications in both bioreactors and biosensors.

Bi-MoP16 Extreme Hydrophobicity Affecting the Adsorption of IgG on Methylated Surfaces, Bengt Walissa, Peter Warcken, Jan Hemmingson, and Pentti Tengvall, Linkoping University, IFM, Laboratory of Applied Physics, S-58183 Linkoping, Sweden.

Adsorption of IgG onto methylated surfaces in static protein solutions was studied using sapping mode AFM in air.

Incubation of DDS (dichloro dimethyl silane)-treated silicon surfaces in IgG (1-15 μg/ml) was found to induce dendrite-like structures of IgG on the surfaces. These aggregates were highly dependent on the degree of methylation (surface energy) and on the concentration of IgG in solution. Surfaces were required to have water contact angles above 91° in order to induce dendrite formation. Similar patterns were also observed on SH(CH$_3$)$_2$CH$_3$ modified gold surfaces (water contact angle = 100°) after incubation in IgG suggesting the outermost chemical properties were responsible for the behaviour.
Nucleation and Growth: Homoeptaxy

Moderator: M. E. Kordesch, Ohio University.

8:20 am SS1-TuM1 Dynamics of the Si(100) Surface, Chris Pearson, Rob Curtis, Xin Shi and Eric Ganz, Department of Physics, University of Minnesota, Minneapolis, MN 55455.

We have used a hot STM to create movies of the Si (100)-2×1 reconstructed surface at sample temperatures between 200°C and 300°C with a 30 second frame rate. By measuring the attachment and detachment rates of groups of Si atoms we determine the activation and binding energies for the predominant processes on this time scale for various step and kink configurations. In addition to attachment or detachment of pairs of dimers, we also observe blocks of multiple pairs of dimers moving in concert.


8:40 am SS1-TuM2 STMS Studies of Vacancy-Step Interaction Kinetics During Low-Energy Ion Bombardment of the Si(001) Surface*, B. S. Swartzentruber1, C. M. Matske1, J. E. Houston1, and D. L. Kendall1, Sandia National Laboratories, and 1University of New Mexico, Albuquerque, NM.

Low-energy ion bombardment has been suggested as a nonthermal means for tailoring surface morphology during thin-film growth. In this work, we explore a specific aspect of such a scheme by studying the kinetics of Si(001) surfaces sputtered by low-energy Xe ions (~ 300 eV) at substrate temperatures between 350 and 525 C. Just as adatoms preferentially stick to the ends of substrate dimer rows on Si(001), vacancies are found to preferentially annihilate at the ends of dimer rows. Because the surface is comprised of two types of steps, those running parallel (SA) or perpendicular (SB) to the upper terrace dimer rows, ion bombardment tends to retract the SB step from its equilibrium position midway between neighboring SA steps, leading to a domain asymmetry. The resulting step morphology is determined by the competition between vacancy annihilation events which retrace the SB steps and the intrinsic rate at which the SB steps return to their equilibrium position. At temperatures where the vacancy diffusion is large enough so that no vacancy islands are formed on the terraces (step flow), the surface reaches a steady state asymmetry as the competing kinetic contributions balance. At lower temperatures vacancy islands form on the terraces leading to a layer-by-layer removal. We analyze the overall recovery kinetics of the surface with LEED and investigate the detailed surface morphologies as a function of ion dose and substrate temperature with STM. The recovery of the two-dimensional surface upon annealing is hindered by the presence of anti-phase domain boundaries which form during ion bombardment as vacancy islands coarsen. We discuss these results in terms of their possible implications on evolving morphologies during surface preparation by growth and ion bombardment.

*Supported by NSF, Materials Research Group Grant (#DMR91-21074), and by NSF, Electronic Materials Program Grant (#DMR92-01856).

9:20 am SS1-TuM4 Spatial Correlations in Surface Roughness During Low Temperature Epitaxial Growth of Ge(001), Joseph E. Van Nostrand, S. Jay Chey, David G. Cahill, M. A. Hasan, J. E. Greene, University of Illinois, Urbana, IL.

For many materials, molecular beam epitaxy (MBE) can be used to grow single crystalline layers at temperatures below any other technique. To better understand the limits to the capabilities of MBE growth, we measure the surface morphology of Ge(001) grown at low temperatures. After growing a Ge buffer layer on a Ge(001) substrate, we deposit epitaxial layers over a wide range of thicknesses, 5 to 1000 Å, and temperatures, 60 to 275°C; the samples are transferred to a scanning tunneling microscope without leaving the ultrahigh vacuum environment. The surface morphology shows spatial correlations of the surface roughness in the plane of the film. The length scale of the correlations increases from less than 100 Å at 60°C to approximately 1000 Å at 275°C. The surface roughness becomes more pronounced as the film thickness approaches the critical thickness for the transition from crystalline to amorphous growth.

9:40 am SS1-TuM5 Kinetic Smoothing of Vicinal Si(111)*, H.-C. Kao, R. J. Phaneuf and E. D. Williams, Department of Physics, University of Maryland, College Park, MD 20742-4111 U.S.A.

Processes such as growth can push surface structure far from equilibrium. One example of this is the formation of kinetically stabilized step structures, which can often remain after growth as metastable structures with low lifetimes for decay to equilibrium. We report on an unusual example of such a process, the formation of a metastable uniformly stepped surface from an equilibrium faceted surface during homo-epitaxy. The initial equilibrium structure is the faceted [1] vicinal Si(111) surface misoriented by 6° toward [211]. We have used low energy electron diffraction (LEED) to study the evolution of the step structure in situ as a function of Si flux and sample temperature. We find that growth reverses the thermodynamically induced faceting at sufficiently high flux. This smoothing can be interpreted as due to an anisotropic step velocity function such as the Schwoebel effect [2,3]. The transition to a uniform step train requires growth of hundreds of layers, suggesting the anisotropy is weak. We compare the time dependent diffraction profiles with the results of models of growth on a faceted surface, including the Schwoebel effect, and generalized models [4].

*Work supported by DOD.

10:00 am SS1-TuM6 Fractal Growth on Metal Surfaces*, Zhenyu Zhang, Xun Chen, and Max G. Lagally, University of Wisconsin-Madison, Madison, WI 53706.

In recent scanning tunneling microscopy (STM) studies of metal-on-metal growth at monolayer coverages, fractal-like islands have frequently been observed on substrates of triangular or hexagonal geometry, but to date only compact islands have been observed on substrates with square geometry. To understand this striking phenomenon, we have investigated theoretically the formation mechanisms of fractal islands by taking specific atomic-bonding geometry into proper consideration [1]. We found that three growth regimes can be defined as the surface temperature is increased: the standard diffusion limited aggregation regime; the extended fractal regime; and the compact regime. The extended fractal regime is characterized by significant local relaxation by edge diffusion. The temperature range defining this regime is relatively wide for triangular lattices, but vanishingly
small for square lattices, explaining why fractal growth has been observed only on triangular lattices. By comparing experimental and theoretical results, we also give estimates of the activation barriers against edge diffusion for several systems.


*Supported by NSF, Materials Research Group Grant (#DMR91-21074), and by NSF, Solid State Chemistry Grant (#DMR93-04912).

10:20 am SS1-TuM7 Computer Simulations of Diffusion and Island Growth, G. Vidali, O. Biham, R. K. Martin, and R. Pennett-Fox, a. Physics Department, Syracuse University; b. Hebrew University; c. Indiana University of Pennsylvania.

Motivated by recent experiments of epitaxial growth, we have performed computer simulations of submonolayer island growth of Cu on Cu(001) using realistic deposition rates and energy barriers. The key and novel aspect of our work is that in calculating the hopping rate of an adatom we take into account the environment in which it moves. We have calculated the energy barriers using the Embedded Atom Method supplemented with a simple empirical potential.

10:40 am SS1-TuM8 On the Interlayer Atomic Diffusion Mechanism in Metal-on-Metal Epitaxy, Yinggang Li and Andrew E. DePristo, Chemistry Department and Ames Laboratory, USDOE, Iowa State University, Ames, IA 50011.

From an atomistic point of view, low-temperature metal-on-metal growth (on both Fe(111) and (100) surfaces) is not well understood. To help elucidate the elementary processes in growth, we have determined the potential energy barriers (PEB) for interlayer mass transport by performing minimum-energy-path calculations. The systems studied were transition metals with filled or nearly filled d-bands while the interactions were described by the Corrected Effective Medium Theory. The dependence of the PEB on the size and shape of the 2D islands on the surfaces was investigated. As a function of the size, the PEB showed non-monotonic behaviors that differed considerably from metal to metal. We will explain the implications of these results for experimental observations (e.g., reentrant and surfactant-induced layer-by-layer growth) and predict that the reentrant growth could be observed experimentally.

Incorporation of small clusters (dimers and trimers) into the islands at the island edges will also be discussed.

*Work supported by NSF grant CHE-9224884.

11:20 am SS1-TuM10 Configurational Stability and Surface Diffusion of Rhodium Clusters on Rh(100), G. L. Kellogg, Sandia National Labs., Albuquerque, NM 87185-0344.

The properties of small clusters adsorbed on single-crystal terraces play a key role in defining the nature of epitaxial growth processes. I have used field ion microscopy to examine the stability and mobility of Rh clusters containing two to twelve atoms adsorbed on the Rh(100) plane. In contrast to Pt on Rh(100), where stable chain configurations are observed for clusters up to pentamers, all Rh clusters larger than trimers are stable as two-dimensional islands. The activation energy of surface diffusion exhibits an interesting, oscillatory behavior as a function of cluster size. Compact geometric structures (squares and rectangles) have a consistently higher activation energy than structures with extra atoms at the periphery. For example, a 9-atom cluster (square configuration) is immobile up to 440 K, whereas a 10-atom cluster becomes mobile at 380 K. Apparently, the periphery atoms destabilize the cluster leading to a lower diffusion barrier. Possible mechanisms for the diffusion process and the relationship of surface diffusion to cluster stabilities are discussed.

*Work supported by the U.S. Department of Energy under contract DE-AC04-94AL85000.

11:40 am SS1-TuM11 Scaling Properties of Fe Growth on Fe(001) Whiskers, Joseph A. Strosnider and D. T. Pierce, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Central to recent growth theories describing molecular beam epitaxy have been the scaling of various surface distributions describing the evolution of film growth. Our work on the homoepitaxial growth of Fe on Fe(001) whiskers elucidates many of the scaling properties of island distributions predicted in the early stages of growth, but shows characteristic length scales in subsequent thin film growth not found in conventional stochastic growth theories. In this presentation, we describe the evolution of Fe film growth from the submonolayer regime through thin films using scanning tunneling microscopy (STM) and reflection-high-energy-electron-diffraction (RHEED) measurements. Scaling of island size and separation distributions as a function of the ratio of surface diffusion to deposition rate is observed. The measured scaling functions for the island size distributions indicate a critical nucleus size of 1 atom for growth at 300 K, and show evidence of an increase in critical size to ~3 atoms for growth above 550 K. In the case of thin film growth, we observe a characteristic length, l, in real space images and height correlation functions obtained from STM measurements and also in the splitting of RHEED diffraction features. For growth at room temperature, this length scale is initially ~5 nm, set by the average island separation at submonolayer coverage, and is observed to increase slowly with increasing film thickness, for example, l ~ 10 nm for a 600 layer thick film.

*This work is supported in part by the Office of Naval Research.

SURFACE SCIENCE
Room A201 – Session SS2-TuM

Non-Thermal Surface Dynamics
Moderator: J. P. Cowin, Pacific Northwest Laboratories.


Recently, several investigators have applied femtosecond laser techniques to examine the dynamics of desorption at a metal surface in the time domain. In these papers it has been demonstrated that femtosecond laser light can result in highly efficient desorption. This effect has been associated with a novel mechanism for activation occurring at the high substrate electronic temperatures induced by the
ultrashort laser pulses. In this talk we present time-resolved correlation results for $O_2/Pd(111)$ in which the desorption yield is monitored as a function of delay time between a pair of subpicosecond laser pulses. This technique provides information on the lifetime of the excitation responsible for desorption. The correlation results are dominated by a subpicosecond feature, consistent with a mechanism of strong coupling of the adsorbate to the substrate electronic excitation. However, a weaker feature lasting $\approx 10$ ps is also observed. The presence of such a long-time feature is interpreted as an enhancement of the electronic mechanism through vibrational excitation of the adatom resulting from the first laser pulse. A detailed analysis of the data in terms of models for the vibrational excitation and the effect of such excitation on electronic desorption mechanisms will be presented.


8:40 am SS2-TuM2 Dynamics of Surface Photochemistry: CO + $Q_2$ on Pt(111), Darryl G. Busch and W. Ho, Department of Physics, Cornell University, Ithaca, NY 14853.

Photo-induced desorption of CO$_2$ and O$_2$ from CO and O$_2$ coadsorbed on Pt(111), and O$_2$ on Pt(111) at 80 K, has been studied using nanosecond and femtosecond laser pulses, and time-of-flight detection with a quadrupole mass spectrometer. The CO$_2$ TOF distribution obtained with 10 ns, 355 nm pulses can be fit to a modified Maxwell-Boltzmann with mean kinetic energy 2800 K and reduced width 0.89. The O$_2$ TOF distributions are bimodal Maxwell-Boltzmanns with temperatures of 600 K and 85 K for O$_2$ alone, and 950 K and 30 K for the coadsorbed system. The low energy O$_2$ peaks vary with fluence and are attributed to collisional detachment from physically adsorbed O$_2$ followed by thermal desorption. Desorption of O$_2$ from O$_2$/Pt(111) using femtosecond pulses produces a nearly Maxwell-Boltzmann TOF distribution with temperature 1400 K, which corresponds to the calculated peak electronic temperature for the fluence used. This gives evidence, along with the nonlinear dependence of yield on fluence and two pulse correlation results, that desorption by femtosecond pulses is mediated through the hot, nonequilibrium electronic distribution which results from an intense, ultra-short laser pulse.

9:00 am SS2-TwM3 Reactions of Hyperthermal Energy (5-100 eV), Molecular Ions with Metal and Oxide Surfaces, D. C. Jacobs, Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN 46556.

Often, reactions performed under thermal conditions only access a restricted range of product channels. However, hyperthermal energy collisions activate a variety of chemical processes which are important to ion etching applications, low-earth orbit spacecraft environments, and mass-spectrometric instrumentation. Studying the dynamics of hyperthermal molecular reactions on metal and oxide surfaces provides detailed mechanistic information for this important class of systems.

Our lab has conducted experiments which probe novel reaction mechanisms involving hyperthermal energy molecules with Ag, O/Ag, and SiO$_2$ surfaces. Multiphoton laser ionization is employed to prepare molecular ions in a particular quantum state, i.e. with a controlled amount of electronic, vibrational, and rotational energies, as well as a restricted distribution of reagent orientations. These ions are accelerated to a collision energy ranging from 5-100 eV and strike a well-characterized adsorbed or UHV covered surface. Dissociation, electron, and abstraction reactions are studied with attention to the specific roles that energetic and steric effects play in determining the outcome of a reactive collision.

10:00 am SS2-TuM6 Models for Positive Ion Emission by Photon Excitation at the (001) Surface of MgO, A. Gibson, J. P. LaFemina, Molecular Science Research Center, Pacific Northwest Laboratories, Richland, WA, and J. T. Dickinson, Department of Physics, Washington State University, Pullman, WA.

Positive ions, principally Mg$^+$, from MgO surfaces during sub-bandgap 248 nm irradiation has been shown to be accompanied by photoelectron emission. The ion emission is highly collimated along the surface normal, and exhibits a complicated energy dependence. Anion vacancy defects (F centers) in the surface region are necessary for this emission. To understand the desorption process, the relative energetics of Mg emission from different defect sites at the (001) surface have been computed using density functional theory, and in the local density approximation. In particular, a comparison is made between two models which describe cation emission from the (001) surface. The first model, which has also been used to explain Na ion emission from NaCl (001), involves the emission of a cation from a site adjacent to a surface F center, thereby forming a surface F center. The second model involves the emission of the cation from its initial configuration adsorbed atop the surface F center. In both cases the initial and final products are considered as a function of their charge state, and compared to the experimentally observed ion kinetic energy and angular distributions.

Pacific Northwest Laboratories are operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO 1830.


Non-thermal or “stimulated” surface chemistry has been of great interest over the years, both in fundamental surface science and in its potential for selective area processing. Recent activity includes the stimulated chemistry of adsorbed ammonia, which have applications in substrate nitridation. We have studied experimentally and theoretically the electronic excitations and the subsequent dynamics for both desorption and dissociation of chemisorbed NH$_3$ and ND$_3$ on Pt(111). We have shown that the two processes are due to distinct adsorbate electronic excitations that have identifiable thresholds and naturally produce very different dynamics.

In this presentation we discuss the ammonia dissociation channel, where gas-phase H and D products are detected by 2 + 1 resonance-enhanced multiphoton ionization. Whereas desorption correlates with the 8 eV excitation of the lone-pair 3a$_u$ electrons, dissociation correlates with the deeper 14 eV excitation of the 1e N-H bonding electrons. The H/D yield ratio varies with translational energy in the range (0.01 - 0.5 eV). It increases from near unity at energies <0.05 eV to approximately 3 at 0.5 eV. This effect can be qualitatively understood in terms of simple one-dimensional potentials, the lighter H atoms acquire more kinetic energy during the excited state lifetime and thus leave the surface with greater probability. This is supported by calculations. The relatively low H/D ratio implies that the lifetime
of the dissociative excitation is relatively long, consistent with the high dissociation yields noted in previous studies.

*This work performed at Sandia National Laboratories is supported by the U.S. Department of Energy under contract DE-AC04-94AL8500.

The hydrogen desorption mechanism from Si(100) surface has been under intensive investigation recently. Most of the previous studies were performed by thermal desorption. We will present the results of hydrogen desorption induced by the electron beam from a STM probe. The STM probe can deliver a very high current density (about 10^7 A/cm^2) locally. Combining this with the atomic scale resolution provides a unique tool to study the electron stimulated desorption (ESD) process. Hydrogen pressure on Si(100) 2 × 1 and 3 × 1 surfaces were prepared in an UHV chamber. Atomic scale desorption was achieved by bombarding the surface with electrons at STM probe biases as small as negative 4 V relative to the sample. Our observation agrees with previous reports about the recombivative nature of the hydrogen desorption, however, we find in some cases that the newly formed clean Si atoms can re-dimerize themselves with a shift relative to the original dimer. The presence of these shifted dimers suggests a possible mechanism in which the Σ-bonds between Si atoms are broken during desorption. We also have obtained electron-hydrogen conversion efficiency as a function of electron kinetic energy. The relation of desorption area to electron dosage, and threshold voltage will be discussed. These ESD results could be complementary to those obtained from thermal desorption and shed some light on the much disputed hydrogen recombivative desorption mechanism from this surface.

11:00 am SS2-TuM9 TOF and Internal State Distribution of Photo-desorbed Species from N2O/Pt(111) by 193 nm Light, D. P. Masson, E. J. LERczendorf and A. C. Kummel, Department of Chemistry, University of California at San Diego, La Jolla, CA 92039.
Polarized ultraviolet light from an excimer laser (193 nm) was used to photodesorb and photodesorbi1 N2O adsorbed on a cold Pt(111) surface. The desorbed species and their time-of-flight (TOF) were monitored by resonantly enhanced Multi Photon Ionization (MEI) spectroscopy. We have identified three major channels. The photodesorption of molecular N2 was observed only in the slowest channel where N2 produced by fragmenting the N2O is thermalized on the surface before desorption. Evidence for this behavior includes both a low (~90 K) rotational and translational temperature of the N2 fragments as well as a lack of correlation between rotational and translational energy. In the next fastest channel, only ground electronic state N2O (exit kinetic energy; 0.4 ± 0.1 eV) was seen to photodesorb. The analysis of the N2O photofragments O(2D) and N2(Δ) produced by the probe laser suggests a substantial degree of rotational and vibrational excitation upon leaving the surface. The angular distribution of the thermal N2O is also more peaked toward the surface normal than the angular distribution of the thermal N2. The desorption yield of the above two channels decreases when the polarization of the desorption laser is changed from p-polarized to s-polarized in agreement with hot carrier mediated chemistry. In the fastest channel, we have detected O(1P) which, we believe, originates from dissociation of electronically excited N2O(Π1) or (Π2) with an exit kinetic energy of 0.90 ± 0.15 eV. This may be the first experimental evidence that electronically excited species photodesorb from a metal surface.

11:20 am SS2-TuM10 UV Irradiation of Physorbsorbed Overlays: CdJ/MgO(100): Probing Photofragmentation Dynamics, Adsorbate Orientation and Overlayer Morphology, B. Howard Fairbrother, K. A. Briggs, P. C. Stair and Eric West, Department of Chemistry, Northwestern University, Evanston, Illinois 60208.
The 257 nm photodissociation dynamics of CdJ monolayers adsorbed on an MgO(100) substrate have been studied using resonantly enhanced multiphoton ionization time-of-flight mass spectrometry (REMPI-TOFMS) to detect both Cd and I photofragments. The photofragments result from direct dissociation and photodissociation of methyl iodide chromophores present in the near surface region, leading to the production of both neutral methyl and iodine fragments. Data on the morphology of the evolving film was obtained by monitoring the variation in photofragment intensity with adsorbate coverage. Methyl produced with velocities as fast as those found in gas-phase dissociation were observed with a narrow angular distribution (~cos^θ) consistent with a preferential orientation of the C-I bond along the surface normal. Collisionally slowed fragments, characterized by a temperature close to that of the overlayer, were also observed with a much broader dependance upon the angle of ejection (~cos^θ). Iodine fragments with velocities in excess of the gas-phase limit result from energy transfer with faster moving methyl fragments. A fraction of the iodine photofragments are trapped within the adlayers and give rise to photoduced chemistry.

11:40 am SS2-TuM11 Molecular Desorption of Methyl Halides from GaAs (110), P. H. Lu, P. J. Lasky, Q. Y. Yang, and R. M. Osgood, Jr., Columbia Radiation Laboratory, Columbia University, New York, NY 10027.
Temperature Programmed Desorption (TPD) has been used to study the molecular desorption of methyl halides from the GaAs (110) surface. The TDP spectra are strongly coverage dependent. For example, the peak position shifts from 155K to 125K for CH3Cl when the coverage is changed from 0.1 to 1 monolayer. A dipole-dipole interaction model has been used successfully to explain and analyze the TPD spectra. The fitting results for CH3Cl show that the effective dipole moment is 1.3D, the effective polarizability is 3.0Å^3, and the zero-coverage desorption energy is 40.0 KJ/mol. The gas phase dipole moment of CH3Cl is 1.86D. The decrease may be due to the fact that the molecules are inclined towards the surface, such that their dipole axes form an angle of 43° with the surface normal. The orientation of the molecules on the surface has been confirmed by NEXAFS and angular-resolved measurements of photodissociated methyl radicals. The angular distribution of photodissociated CH3Cl molecules, about (cosθ) for 0.5 monolayer coverage, has also been determined. The results show that even though the molecules are oriented at an angle on the surface, the thermally desorbed molecules are collected along the surface normal. Similar results are also obtained for both CH3Br and CH3I. We discuss the implications of these findings for the understanding of desorption, and likewise adsorption, dynamics of physisorbed molecules, particularly on strongly covalently bonded semiconductor surfaces. This work is supported by DOE.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A209 - Session NS1-TuM

Nanostructured Materials
Moderator: D. Bonnell, University of Pennsylvania.


Using the local density approximation (LDA) and massively-parallel computing, we examine from first principles some structural and adsorbate properties of Cu, Ru, Pd, and Ag clusters with up to ~33 atoms, with a goal of uncovering basic principles. Our results include the following:

a) Structure: Preferred structures vary considerably. For example, we find that Ag_{11} prefers the octet structure proposed by McAdon and Goddard for lithium (J. Phys. Chem. 91 (1987) 2607). Pd_{33}, however, prefers theicosahedral structure over either fcc or the octet. This result supports the polyicosahedral structures proposed by Parks et al. (JCP 96 (1992) 8267) for clusters betweenicosahedral shell closings of 13 and 55 atoms. These trends are understood based on the local bonding structure.

b) "Electrostatically" Bound Adsorbates: Binding to a cluster may be quite different than to extended surfaces. For example, ammonium prefers an atop site on Pd_{33}, a site shown experimentally not to be favored on extended Pd(111) (Burns et al., PRL, in press); in addition, NH_3 is bound almost 50% more strongly than on the extended surface.

Furthermore, we observe very slow convergence to the preferred extended-surface site with increasing cluster size. The binding energy of ammonia is well given by standard LDA theory, i.e., without a gradient correction.
c) Covalently Bound Adsorbates: Adsorbates with strong covalence, such as CH₃, bind at similar sites on finite clusters and extended surfaces. They are also well-described by standard LDA. However, adsorbates with weak covalence, such as NO, have a large error in adsorption energy without the gradient correction to LDA, while site preference, geometry, and vibrational frequencies are well-given without the gradient correction. For example, NO is found to prefer the hollow site on a 19-atom Pd cluster which mimics Pd(111), in agreement with recent experimental results (Materer, et al., Surf. Sci. 303 (1994) 319).

*Work supported by US DOE under Contract No. DE-AC04-94AL8500.

8:40 am NS1-TuM2 Kinetics and Thermodynamics of Organic Nanocluster Formation, P. V. Shilov*, K. Schaumburg**, K. Branfeldt***, **119899, Moscow State University, Physical Electronics Dept., Russia; **CISMF, Fruebejergvej 3, 2100 Copenhagen, Denmark.

The theory of oriented growth of organic clusters and films during vacuum deposition is presented. Theory is based on the consideration of thermodynamic of the system and generalization of the kinetical rate equations to the spatially anisotropic rigid molecules. Molecules and clusters are considered to have two possible orientations of long molecular axis parallel (P) and normal (N) with respect to the surface substrate. Concentrations of clusters with definite structure are determined for the equilibrium conditions. Transformations of cluster structure are investigated in terms of transition state theory (TST). Applicability of this theory is discussed. Basing on the data obtained the set of kinetical equations for the P- and N-oriented clusters is derived. To estimate adsorption energies and diffusion coefficients the atom-atom potential calculations of interaction energies between some covalent substrates and linear molecules were performed.

The developed theory predicts concentrations of N- and P-clusters appeared on the substrate in different deposition conditions. It was found that for the fixed substrate temperature and molecular length there is a critical flux of molecules onto the substrate for which the rate of P-cluster formation becomes higher than for N-clusters. The higher the temperature the less is the critical flux of molecules. These results are in a good agreement with recently obtained experimental ones which are also intensively discussed.

9:00 am NS1-TuM3 Nanocrystalline Solids as Interface-Determined Systems, H.-E. Schafer, Stuttgart University, Institut für Theoretische und Angewandte Physik, 70550 Stuttgart, Germany.

Nanocrystalline solids are characterized by ultrafine crystallites with a typical diameter of ca. 10 nm and therefore contain a high number density of structurally disordered interfaces which comprise a substantial fraction of the atoms. Due to the high number of these interfaces the properties of a nanocrystalline solid are profoundly modified compared to the chemically identical solid with a long-range periodic crystalline order. The structure of the interfaces depends on the technique of preparation as well as on the thermal relaxation and a wide variety of preparation routes as gas-phase condensation of crystallites and compaction, chemical synthesis, severe plastic deformation, crystal growth from amorphous glasses etc. are available. In order to study the structure of the interfaces on a local atomistic level nuclear probes as, e.g., positrons were used. By means of positron lifetime spectroscopy a distribution of atomic free volumes from the size of vacancies (1V) in the interfaces to nanovoids of about 10 missing atoms (10V) in triple junctions can be detected. These free volumes exhibit a high compressibility in the comparatively "soft" interlayer and tend to agglomerate upon annealing. The high diffusivities of substitutional atoms in nanocrystalline metals and the high desorption rates of gases will be correlated to these free volumes. The magnetic properties as the Curie temperature, T_C, the magnetic moment per atom, µ, and the coercive field, H_C, are taken as examples to demonstrate the modification of the macroscopic properties in nanocrystalline solids.

INVITED

9:40 am NS1-TuM5 Diffusion Controlled Growth of Metallic Nanoclusters at Selected Surface Sites, G. M. Francis, R. E. Palmer and J. R. A. Cleaver, Cavendish Laboratory, Madingley Road, Cambridge CB3 0HE, UK.

The nucleation and growth of atomic clusters at artificially created defects on a substrate is an attractive route to producing low-dimensional quantum structures [1]. We have therefore investigated the phenomenon of three dimensional particle growth at an atomic step due to the diffusion of silver adatoms across the surface of Highly Oriented Pyrolytic Graphite (HOPG) in a high vacuum (10⁻¹⁰ mbar). By controlling the growth conditions (temperature, deposition rate), cluster growth has been confined to steps on the surface avoiding terrace nucleation. This leads to the approximately 10 nm diameter as determined with a scanning electron microscope (SEM). The results suggest the viability of an important new route to the creation of controlled nanoscale quantum structures.

In order to explore in more detail the kinetics of the nanocluster growth process, we have also performed a quantitative analysis of the spatial distribution of the clusters on the surface steps. Using a recently published theory [2] we have been able to calculate atomic diffusion parameters along the steps and determine that the graphite surface has several types of straight surface steps, with different diffusion barriers varying over a range of about 1 eV. For substrate temperatures above 50°C we find that the adatoms are able to diffuse to the steps, across defect-free terraces up to 140 nm wide without terrace nucleation and in this regime the steps act as perfect adatom sinks. By contrast, at room temperature we find that terrace nucleation competes with step nucleation down to the lowest deposition rates available.


10:00 am NS1-TuM6 The Surface Composition of Semiconductor Nanocrystals, J. E. Bowen Kartar, V. L. Colvin and A. P. Alivisatos, Department of Chemistry, University of California, Berkeley, CA 94720.

The surface of a semiconductor nanocrystal plays an important role in determining its electronic structure because of the large ratio of surface atoms to interior atoms. However, because of low symmetry and absence of long range order on the crystal surface, many traditional principles of surface structure are not applicable, and thus little is known about the surface of nanocrystals.

We have studied the surface composition of chemically synthesized CdSe nanocrystals using x-ray photoelectron spectroscopy (XPS). The samples as prepared are not suitable for XPS, as they will charge nonuniformly, causing shifts and broadening of peaks. Thus, the crystals were covalently bound to gold, resulting in submonolayer coverage of nanocrystals placed ~ 10 A above the conducting surface.

Using the position and relative areas of the relevant core level peaks, we have determined that the nanocrystal surface initially contains as many capping molecules as are sterically allowed. As the radius of the nanocrystal increases from 9 Å to 30 Å, the surface gets flatter, allowing less capping molecules to fit on the surface and decreasing the surface coverage. We have also demonstrated that it is possible to displace the organic surface ligand, creating a bare surface.

This method for determining surface composition, combining a unique sample preparation with XPS, is easily extendible to a variety of surface ligands and to other chemically synthesized nanosystem systems. In combination with other techniques, such as valence band photoemission, this work will allow us to quantify the effect of the nanocrystal surface on its electronic properties.

10:20 am NS1-TuM7 Preparation and Characterization of Thiophenyl Silver Nanocrystals, M. M. Alvarez, Srilatha Murthy, R. L. Whetten and J. M. Hampikian, School of Physics, Georgia Institute of Technology, Atlanta, GA 30332-0430, School of Chemistry and Biochemistry, Georgia Institute of Technology, Atlanta, GA 30332-0400, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA 30332-0245.

Silver nanocrystals (8-40 nm) are prepared in a flowing aerosol device and solubilized by bubbling through appropriate thiol solutions. They remain in solution for days and they can be dried, handled in air and resolubilized without noticeable change in observable properties. By contrast, when silver nanocrystals are prepared in the flowing aerosol device and bubbled through neat liquids, they settle out of solution. Transmission electron microscopic studies and energy-dispersive X-ray analysis of nanocrystals prepared by these methods will be used to discuss the effectiveness of thiols in acting as capping and solubilizing agents for silver nanocrystals. Atomic force microscopic studies of arrays of nanocrystals prepared on various substrates will be presented and the applicability of atomic force microscopy as a routine technique for the determination of the sizes and shapes of nanocrystals will be discussed.

10:40 am NS1-TuM9 Polymer-Nanocrystal Composites—Integrated Materials for Electroporobolical and Electronic Devices, J. P. Spatz, A. Roesser, M. Müller, Organishe Chemie III, Universitäat Ulm, D-89069 Ulm.
We report a new method of producing nanostructured metal and semiconductor clusters which can be used to improve the properties of nanoscaled devices and therefore to investigate effects which are hidden behind the limit of conventional methods. The chemical structure and properties of the used polymer in which the crystallite is embedded can be tailored for optimisation of physical properties and applications.

Functional block copolymers with ionic endgroups are employed to bind different metal ions or coordination complexes in ordered nanostructures and in defined concentration within a polymer matrix. Subsequently, the transition metal ions are converted to small crystallites (active component) by reduction. The size of the micro domains in which the transition metal complexes are concentrated (crystallite substrate) can be rigorously controlled between a few and up to 1000 nanometers. Also the distance between the micro domains can be controlled within the same range. The maximal size of the crystallites can be controlled by the absolute amount of the transition metal per domain. Crystallite dimensions can be reduced behind the 1 nm scale.

Molecular mobility and thus crystal growth can be controlled by making use of the glass transition. These new samples are characterised by X-RAY scattering, TEM, UV-VIS absorption and photoluminescence measurements, which determine the size, size distributions and electron confinement effects. Applications (devices and quantum lasers) which could be improved or new developed will be discussed.

11:00 am NSI-TuM10 Characterization and Nanomanipulation of ZnS Nanocrystals via Atomic Force Microscopy, J. E. Coury, E. C. Pitts, L. A. Bottomley, R. Shorrosh and R. H. Felton, School of Chemistry & Biochemistry, Georgia Institute of Technology, Atlanta, GA 30332-0400 USA.

Semiconductor clusters, also known as nanoclusters or quantum dots, have recently been the object of intensive study because of their potential use in high-speed electronics applications. We have prepared and characterized ZnS nanoclusters utilizing x-ray absorption fine structure techniques, x-ray diffraction (XRD), structure simulations, and chemical analysis. The nanoclusters were “capped” with alkanethiols possessing chemically-polarizable end-groups enabling the electrostatic immobility of the particles. The transport of these nanoclusters via the probing tip between different substrates and their subsequent electrostatic deposition into well-defined arrays has been achieved. The “striking behavior” of these clusters upon repeated AFM scanning will be noted. This report describes the reliable and reproducible AFM imaging of semiconductor nanoclusters as well as their controlled nanomanipulation.


Enhancement of the mechanical properties of nanoscale materials has been observed in a number of multilayer structures. Specifically, metal-metal systems have shown increased yield and fracture strength. One potential application of the nanolayers is for high temperature coatings. The layer stability and its effect on the mechanical properties are of main concern. In this study, the layer structure evolution and its influence on the mechanical properties as a function of temperature is investigated in two MoS2-based composites, namely, MoS2-SiC and MoS2-MoSi2N2. Nanoindentation is employed to measure the hardness and modulus of the nanolayers. Cross-sectional transmission electron microscopy (XTEM) is used to examine the microstructure and the layer structure evolution when exposing to different temperatures. An amorphous structure is observed in the as-sputtered nanolayers in both types of composites. For MoS2-SiC, crystallization of MoSi2 and SiC occurs at 500°C and 700°C respectively. Figure 1 shows a XTEM image of a 500°C-1h annealed multilayer. Significant increase in both hardness and modulus is observed accompanied the crystallization process. A 900°C-2h anneal causes the complete layer breakdown to form a nanocrystalline equiaxed microstructure. For MoS2-MoSi2N2, MoSi2 crystalize at 500°C while MoSi2N2 layers remain amorphous up to ~900°C; the layered structure is preserved after annealing at 900°C for 1h. The difference in the layer stability at temperature above the two composites will be discussed. The results on the influence of micro-structure and reinforcing geometry on the mechanical properties of the nano-layer composites will be presented.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A207 – Session NS2-TuM

Nanoelectronics
Moderator: T. Higman, University of Minnesota.

8:20 am NS2-TuM1 Application of Nanoelectronic Devices, John N. Randall, Texas Instruments, PO Box 653936, MS 134, Dallas, TX 75265.

Resonant tunneling devices are finding applications in small scale integrated circuits with specialized applications. These devices are scaled to nanometer dimensions only in their epitaxial structure. A vision for the future of ultra-scaled integrated circuits described by Bate [1] includes resonant tunneling through heterostructure confined quantum dots in a limited interconnect massively parallel architecture. A universal quantum dot cell that could be used in a functionally complete edge-fed cellular cascade will be described. There is a current effort to produce lateral heterostructure resonant tunneling devices that will be the basis for the universal quantum dot cell. The critical fabrication processes that will be used to fabricate the lateral resonant tunneling devices will be presented: Lateral patterning techniques using e-beam lithography and sidewall processing will be utilized to produce 15 nm features. Ion beam assisted etching that uses separate ion and molecular halogen beams, will etch InP and related compounds. Epitaxial regrowth of InP by Chemical Beam Epitaxy or MOCVD will be employed to create lateral tunnel barriers.

INVITED


9:00 am NS2-TuM3 Quantum Cellular Automata, *P. Douglas Tougaw, Craig S. Lent, and Wolfgang Porod, Department of Electrical Engineering, University of Notre Dame, Notre Dame, IN 46556.

We discuss a new paradigm for computing based on connecting quantum dot devices in a cellular automata architecture—quantum cellular automata (QCA). The architecture is designed so that the ground state of the array, subject to the boundary conditions determined by the inputs, yields a useful computational result.

One realization of these ideas uses two-electron cells composed of quantum dots and is within the reach of current fabrication technology. The charge density is highly aligned along one of two cell axes, making this cell ideal for a two-state CA. The polarization of one cell induces a polarization in a neighboring cell in a very nonlinear way, and this interaction is used to make QCA wires which can transmit the binary information encoded in the quantum state of the cells. The different behavior of these arrays by solving the time-independent Schrödinger equation to find the many-particle ground state, and show how useful computing structures may be built from a set of logical primitives. These primitives include wires, coplanar wire crossings, inverters, and a flexible three-input structure. The three-input device can be configured as an AND gate, an OR gate, or a majority logic device. Devices as complicated as a full adder have been
simulated. We will also discuss recent work on time-dependent calculations of the switching of this class of devices.

*Work supported by ARPA/ONR.

9:20 am **NS2-TuM4** Computational Behavior in Coulomb Blockade Arrays, M. G. Ancona and R. W. Rendell, Code 6813, Naval Research Laboratory, Washington, DC 20375 USA.

The limitations on device scaling due to interconnects has led to an interest in locally-interconnected architectures composed of quantum devices. To better understand the potential of such architectures in a "realistic" context, we have studied models of locally-interconnected one-dimensional tunneling arrays operating in the Coulomb blockade regime from a computational point-of-view.Bits of information are represented by single electrons on the islands between junctions and we find that computational behaviors can be obtained if the size of an electron plus its polarization cloud is sufficiently small. We show that linear tunneling arrays can be made to act as "charge-coupled devices" for single electrons. Arbitrary patterns of electrons can be transferred by applying a regular 3+ phase biasing cycle to a set of phase gates in a way similar to that in a single electron pump [1]. This behavior, which could be used for array input/output, is depicted in the figure. We further demonstrate that other regular gate biasings can produce "useful" computations, e.g., simulation of diffusion. These preliminary results suggest that such arrays may be more broadly useful in a technology that should be scalable to molecular dimensions. We discuss these prospects, the fabrication issues, and various error sources such as cotunneling and polarization charge which can impact performance.


10:00 am **NS2-TuM6** A Novel Method for Producing Nanostuctures in Silicon Inversion Layers, B. Campbell, G. H. Bernstein, X. Huang, Department of Electrical Engineering, University of Notre Dame, Notre Dame, IN 46556.

A novel method for fabricating nanostructures based on the interaction of the MOS material system with an e-beam has been investigated. A positive charge located at the SiO2-Si interface which is both positionally and temporally stable results from the interaction of the beam with the sample. The trapped charge at the SiO2-Si interface modulates the surface potential creating a Q-1D channel. There then exists a voltage that when applied to the gate inverts the region underneath the induced oxide charge while leaving the remaining area in depletion. The transport properties of the exposed devices were measured at 1.8 K. The width of the induced channel and the carrier concentration are modulated by varying the gate voltage. Electrical transport characteristics of the post-irradiated silicon MOSFET structures reveal quantized conductance with a step size less than e^2/h. A point contact in series with a large resistance would account for the noninteger quantization value. The transport characteristics of the exposed transistors are comparable to work published by Tang et al. who used a long and narrow wire gate defined by EBL and RIE to study reduced dimensional transport. We believe that this novel technique could be applied to create a wide range of quantum structures with relatively simple processing requirements.


To be viable, nanolithography must be performed in a manner which permits transfer of the lithographically defined pattern to the desired device of nanostructured material. In this context, low voltage e-beam lithography with the STM and high voltage e-beam lithography with commercial and experimental resists are compared. The key issues are resolution, process latitude, sample preparation, and transfer of a pattern from resist to substrate. With commercial resists, we have achieved 12 nm lines in a 60 nm period grating in a 60 nm thick layer of PMMA, with a 50 kV e-beam. In contrast, in 50 nm of SAL-601 (Shipley, Corp.), the best 50 kV results were 50 nm lines on a 100 nm line-to-line spacing; whereas with STM lithography 20 nm lines were achieved with a 45 nm line-to-line spacing. Sample preparation is critical for STM lithography. It can only operate with certain resists, of limited resist thicknesses, and on clean passivated substrate surfaces. To achieve the finest resolution (≤ 10 nm), crystallographically smooth surfaces are required. The advantages of STM lithography are the smaller scattering lengths of low voltage electrons. The resulting localized high contrast exposure allows for very high resolution, no proximity effects, and a large resist process latitude. To move beyond a research technique, STM lithography requires the development of ultra thin resist films, which accentuates the problems of pattern transfer. The alternatives such as atomic manipulation or surface modification cannot be applied to a wide range of materials. Our results with self assembling imaging layers indicate that a viable STM lithographic technique can be developed which can be extended to a production nanolithography once the limited throughput is overcome.


We have demonstrated the use of self-assembled monolayers as high-resolution resists by modifying them on a small scale by the use of electron beams and transferring patterns with standard chemical etch. Focused electron beams of energies 1-50 keV have been used to pattern monolayers of octadecylisoxanones on SiO2 and TiO2 and octadecylihol on GaAs. Line widths down to 25 nm have been formed. Currently, resolution of this patterning is limited by the size of the exposing beam, but we are exploring the ultimate resolution of these materials. The critical dose for modification have been determined—both by direct observation of the material after patterning (using an atomic force microscope) and after wet chemical etching. Also, lithography based on scanned probe microscopy with energies
of ~10 eV has been used to modify the chemical properties of these monolayers down to 15 nm feature sizes.

Particles have been attached to silicon films deposited via 100eV Argon ion beam sputtering to a depth of >120 nm using octadecylsiloxane as a resist. The etch resistance to many common etchants has been measured and we are exploring other etches that may take advantage of the chemical properties of the monolayer.

The exact mechanism of the electron beam damaged is being explored using infrared and x-ray spectroscopy. An understanding of the damage mechanism should allow for better preparation of the monolayers and methods to improve their use in pattern transfer.

This work is supported by the Advanced Research Projects Agency ULTRA project.

11:00 am NS2-TuM9 Manufacturing Considerations for Quantum-Effect Devices, Martin Peckerar, US Naval Research Laboratory, Surface and Interface Sciences Branch, Washington, D.C. 20375.

As a result of evolutionary trends in microelectronic device development, we are currently at the threshold of large-scale deployment of quantum effect devices. These are devices making use of quantum interference and/or tunneling in their basic functioning. Heterostucture bipolar are in use in the microwave industry. Texas Instruments has announced a logic device, operating at room temperature, which makes use of a resonant tunneling transistor. The increased number of output states of this device allows reduced part-count on chip. Reduced dimensionality transport and single electron transport structures may see utilization by the end of the decade. Each of these novel structures presents a particular fabrication challenge, either due to the resolution requirements of the lithography employed or the requirements for minimally damaging etch processes. Laboratory demonstrations prove concept feasibility. But the barriers to scale-up to manufacturing are formidable. This talk reviews progress in movement toward a manufacturing base for quantum devices.

11:40 am NS2-TuM11 Results of NANO-II and Development of Nanotechnology in Russia, V. Alfeev, Academy of Technological Sciences of the Russian Federation, Moscow, Russia.

The overview presents the main achievements on nanotechnology, obtained by Russian scientific bodies, participated at the Second International Conference on Nanotechnology NANO-II and Exhibition in Moscow, in 1993. Success of NANO-II, in which took part well-known scientists from the USA, UK, Switzerland, Germany, Italy, France, Japan, China, etc. promoted the further development of nanotechnology in many countries especially in Russia. The attainment results served the base for forming of Nanotechnology Program, including parts devoted to nanomechanical processing, atom assembling, multifunctional nanocomplexes and basic elements of nanocomputer. The Program is authorized by the Russian Government. The more success were attained on subprograms such as: “Diamond nanotechnology”, “Superhigh terabit memory” and “Nanotron”. The first results of nanodiamond bio testing have attracted attention due especially to the behaviour of diamond particles, which have sizes proper to protein molecules. The study of thin structure of modified nanoparticles concerning their electronic properties allows new approaches to carbon nanoelectronics to be defined. Within the framework of the Program a device for storage of information volume up to 1 Tbit on the base of STM was created. Range of data cell is near 10 nm. Density of data recording exceeds laser disk one. The one-year realization of the Program also allowed to construct new samples of nanotechnological equipment for studying of surface and biological objects and for atom assembling of nanoelectronics elements. The Program is opened for participation of all interested international bodies and now we have already contacted Chinese and Ukrainian scientists as attendees of this Program. There is the Nanometer Science and Technology Division under the Russian Academy of Technological Sciences in Russia, which co-ordinates this Program.

APPLIED SURFACE SCIENCE
Room A101 – Session AS-TuM

Surface Chemistry and Contamination
Moderator: J. E. Fulghum, Kent State University.

8:20 am AS-TuM1 Overview of Particle Detection on Silicon Wafers, Terry Francis.

Contamination Reduction is the battleground of the ninety's on Silicon Wafers. Detection of the particle on the wafers is a key issue but is not sufficient to meet the criteria for contamination reduction. To meet this criteria it is necessary to identify the location of the contamination in a precise manner and then to identify the specific contaminant back to its initial root cause. This overview will cover application of Contamination/Particle Analysis by SEM, EDAX, Auger, ESCA, ICPMS, GCMS, FTIR and Surface Photoacoustic Measurement and their correlation to root cause.

9:00 am AS-TuM3 Limitations of Surface Analytical Techniques for Determining the Surface Composition of Bimetallic Particles, Allen G. Sault, Fuel Science Department, Sandia National Laboratories, MS 0710, Albuquerque, NM 87185-5800.

Calculations of expected X-ray photoelectron spectroscopy intensity ratios for both homogeneous and segregated bimetallic particles demonstrate that a minimum particle size exists, below which the relative detection sensitivity between homogeneous alloy particles and particles in which one component has segregated to the surface cannot be made. This minimum particle size varies between 3 and 8 nm, depending on the specific metal combination in question. In cases of partial segregation of one component, the minimum particle size is even larger. The implications of these results for the study of surface segregation in supported bimetallic catalyst particles is discussed.

This work was performed at Sandia National Laboratories for the U.S. Department of Energy under contract DE-AC04-94AL85000.


The emission characteristics of atomic oxygen generated by electron stimulated desorption (ESD) from the downstream surface of polycrystalline Ag membrane have been studied. The oxygen is supplied by permeation through the membrane where it emerges into an atomically bound state at the down stream ultrahigh vacuum surface and thus desorbed by ESD. The flux levels of atomic oxygen were linear with electron bombardment current and measured up to approximately 1 x 10^13 cm^-2 s^-1 (electron bombardment flux of 5 mA cm^-2 at 2.8 keV). The emission pattern appeared to be highly directed approaching a cos^2θ angular dependence. The oxygen atom to ion ratio emitted was determined to be in excess 1 x 10^6, but the specific ESD mechanism in operation is still under study. The kinetic energy of the oxygen ions was measured by TOF methods and found to have a mean energy of approximately 5 eV with a FWHM of 4 eV for large grain polycrystalline Ag. The addition of a small amount of Zr (0.5 wt. %) substantially broadened the distribution to a FWHM of 6 eV, presumably because of the increase in the adatom bond energy distribution on the alloy surface. Neutral atom energy measurements indicate that the energies are in excess of 2 eV, but the distribution has not yet been determined.

10:00 am AS-TuM6 Oxidation of Gold by UV and Ozone at 25 °C, David E. King, NREL, 1617 Cole Blvd., Golden, CO 80401.*

Gold surfaces have been found to be hydrophilic only after exhaustive preparation and with the ultimate care in sample preparation and treatment. The use of a combination of UV and ozone has been described as a viable method of producing a clean, hydrophilic, gold surface. We have found that gold surfaces stored in the laboratory after vacuum deposition and purchased high purity standards, are oxidized by a combination of UV light and ozone generated from a mercury lamp. The samples were characterized with XPS and ISS prior to and after exposure to UV/ozone in a stainless steel box in laboratory air. After the cleaning process gold surfaces were found by XPS to
contain less carbon, to be enriched in oxygen, and often contain detectable residual nitrogen. The OIs on the cleaned surface, which was not present on the untreated surface, consisted of two peaks that are attributed to gold oxide and water, presumably adsorbed onto the oxide. The oxide layer was found to be 17 ± 4 Å thick by variable angle XPS depth profiling with an initial stoichiometry of Au₂O₅. The oxide was found to be stable to extended exposure to UHV and water rinse. ISS compositional depth profiles confirmed the oxide layer thickness and that the hydrated surface layer is removed in the initial sputtering of the oxidized gold. Implications of these results about the mechanism of self-assembly of thiols on Au will be discussed.

*Performed under DOE contract DE-AC02-83CH10993.


Many hydron inorganic compounds are technologically important, yet little investigated due to their complex, often multiphase, structures and their sensitivity to many processing parameters. An example is aluminum hydroxide, often called aluminum trihydrate, which is used as a fire retardant filler in polymer matrices. Adhesive bonding properties of such composite materials are strongly affected by differences in aluminum hydroxide feedstock materials. These differences could not be detected by XRD, IR, or low energy resolution XPS. High energy resolution XPS had to contend with differential surface potentials, which paradoxically proved effective in separating the multiple hydration states of the aluminum hydroxide materials. Two materials each had two charging potentials associated with two phases of quantifiable H₂O and CO₂ content for a total of four unique phases. Despite these differences, the average oxygen to aluminum ratios of each material were identical and the photoelectron binding energies were virtually identical after corrections for the respective surface potentials. The aluminum hydroxide phases exhibited multiple hydration and carbonation levels. Examples of this quantification technique with magnesium oxides and ferric oxides will also be presented.

10:40 am AS-TuM8 The Influence of Hydrogen on LEIS Signals, R. H. Bergmans, M. J. H. Vanhommeg, A. W. Denier van de Gon, and H. H. Brouwer, Eindhoven University of Technology, Faculty of Physics, P.O. Box 513, 5600 MB Eindhoven, The Netherlands.

Hydrogen is often used as a pretreatment of samples to remove oxygen, when sputtering is not allowed. This leaves a hydrogen covered surface which may affect the surface composition or the determination thereof. In Low Energy Ion Scattering (LEIS) the composition of the topmost atomic layer of a sample is deduced from the energy distribution of the scattered ions. Because of its low mass, hydrogen can not be detected in most LEIS-geometries nor with many other surface sensitive techniques. It is known that hydrogen can obscure other elements in LEIS [1], but most of this knowledge is limited to He⁺ scattering from single elements. In this work the influence of hydrogen on LEIS signals is studied using 4 keV Ne⁺. The samples investigated consisted of Pt, Pd and Cu or combinations thereof. It was found that the presence of hydrogen can cause a decrease in the intensity of LEIS signals of nearly 100%, due to neutralization. The hydrogen did not affect the surface composition of the samples investigated, nor did it obscure one element in the alloys more effectively than another. The removal of hydrogen by sputtering was fitted to a model. From the results of the fitting sputter yields for hydrogen were determined.


Copper implanted in silica exhibits a large nonlinear index of refraction, i.e. a large third order optical nonlinearity. The nonlinear response is thought to arise from the formation of metal colloids. Other highly polarizable metal ions, e.g. Pb implanted in silica also gives rise to a third order optical nonlinearity, however lead incorporates into the silica phase. Nonlinearities may also occur from ion induced damage in the host substrate. The formation of metallic colloids or a product resulting from a reaction of the glass with the implanted ion depends on the chemistry between the host glass and the implanted ion. In order to characterize these effects we have measured the infrared and XPS spectra of Cu implanted in optical grade silica and a borosilicate glass. These results are discussed in terms of ion induced damage and the chemistry of the Cu ion in these glass hosts.

The research at ORNL was sponsored by the Division of Materials Sciences, under contract DE-AC05-84OR22400 with Martin Marietta Systems Inc.

11:20 am AS-TuM10 Investigations of the Surface Chemistry of Pathogenic Silicates, Sudipta Seal, Stephen Hardcastle, Tery L. Barr, University of Wisconsin-Milwaukee, Milwaukee, WI 53201, and Heyong He, Jacek Klinowski and Peter Evans, University of Cambridge, Cambridge, U.K.

The following is a continuation of our extensive investigations of the chemistry of complex silicate systems employing a combination of MAS-NMR and ESCA. In this case, we have extended the study to include the pathogenic asbestos, including the amphibole, riebeckite, and the serpentine, chrysotile. The materials have been investigated both before and following alterations of their physical conditions (e.g., crushing) and also before and after contact with select in-vitro tissue samples. In our studies to date, we have been able to track the "in-lattice" chemistry of the constituents of these, and related, silicates, differentiating such features as the aluminum in the tetrahedral and octahedral sites and the iron in M-4, as opposed to M-3 or M-1 octahedral positions (of the amphiboles). We are comparing our results for the present fibrous silicates with those achieved with related non-fibrous forms. In this manner, we are trying to confirm (or deny) the views of the biomedical community regarding the structural and chemical features of the fibrous silicates that are suspected to be primarily responsible for their pathogenic behavior.


SIC materials are of great interest as advanced structural ceramics, protective coatings, and as reinforcements in composites for high temperature applications. The presence of excess carbon in the microstructure has a pronounced effect on the observed thermomechanical properties; i.e. grain growth, creep resistance and oxidation behavior. The ability to quantitatively measure excess carbon in SIC materials could provide valuable insight into predicting and/or explaining the thermomechanical behavior of SIC. Secondary Ion Mass Spectrometry (Cs⁺ primary beam, negative SIMS mode) of single crystal SIC and highly oriented pyrolitic graphite (HOPG) has revealed "fingertips" for graphitic carbon in the higher mass carbon clusters. Specifically, the useful yield of the Cs⁺ cluster ion signal was a factor of 2 higher in the HOPG. However, the signal intensity ratio C6⁺/C2⁺ was strongly dependent on instrument conditions, such as contrast aperture size and field aperture position. Depth profiles of SIC coatings deposited by CVD on SCS-6 fibers best illustrated the usefulness of the Cs⁺ molecular in identifying the presence of free carbon. The Cs⁺ signal intensity levels matched perfectly in the SIC coating and SIC core fiber. Within the outer coatings of the SCS-6 fiber, which are comprised of carbon reinforced with SIC particles, the Cs⁺ signal intensity increased dramatically. In addition, the Cs⁺ signal increased sharply within the 100 nm turbostratic carbon layer between the carbon-rich coatings. This work has confirmed the ability to use the Cs⁺ cluster ion to identify and quantify free carbon in the presence of SIC.

PLASMA SCIENCE

Room A109 – Session PS-TuM

Plasma Process & Reactor Modeling
Moderator: L. A. Berry, Oak Ridge National Laboratories.

Inductively coupled plasma (ICP) reactors are being developed for high plasma density, low gas pressure etching of semiconductors and metals. As a result of experimental development and computer modeling, design rules to optimize the uniformity of ion and neutral etching precursors in these reactors have recently been proposed. For example, modeling and experiments have shown that at pressures $< 10^{-5}$ mTorr ion sources located at intermediate or large radii produce a more uniform ion flux to the wafer. We have developed a 2-dimensional hybrid model to describe ICP reactors ($< 10^{-5}$ mTorr, $\gg 10^{11}$ cm$^{-3}$ plasma density) with which we are investigating these design issues. Our goals are to optimize the uniformity of etching precursors to the wafer in metal and p-Si etching gas mixtures. The validation of the model will be discussed using electron densities measured with microwave interferometry. We will discuss results for radially dependent ion and radical fluxes in chlorine gas mixtures (rare gas /Cl$_2$, rare gas /Br$_2$) for reactor design using alternate configurations and "triode" structures. The effect of substrate topography (sacrificial surfaces, focus rings, discontinuities in material properties) on the directional orientation of the ion flux and uniformity of the radical flux will be demonstrated.

This work supported by Sandia/SEMATECH, SRC, NSF and Univ. of Wisconsin ERC for Plasma Aided Manufacturing.


We have developed a computational model to optimize the design of a surface reflection neutralization source of hyperthermal neutrals for charge-free processing. For the deployment of this technique to large scale ($\geq 8$ wafer) processing, a system design study has been completed. The model calculates particle transport of ions from a single RF ion source and multiple sources through the neutral background to a reﬂector plate. The resulting hyperthermal neutral beam density at the wafer is calculated for various plasma source density proﬁles. Angular dependence of the beam density and surface reﬂection distribution are modeled using observations from previous experiments. The model assumes specular or diffuse (or both) reﬂection of the neutrons, followed by an off-line Monte Carlo simulation of elastic and inelastic collisions with the neutral beam. Charge exchange between the plasma ions and hyperthermal neutrals is considered and compared to the effect of Frank-Condon and charge exchange neutrals from the plasma on the above proﬁles. Fine line ($< 0.5 \mu$) feature evolution under this hyperthermal neutral bombardment is calculated and presented as a function of the source neutral density.

Work supported by SRC, NSF, Sandia/SEMATECH and Univ. of Wisconsin ERC for Plasma Aided Manufacturing.

9:00 am PS-TuM3 Modeling the Electromagnetic Field Excitation of Low-Pressure, High-Density Plasma Sources, T. A. Grotjohn, Dept. of Electrical Engineering, Michigan State University, East Lansing, MI 48824.

Low-pressure/high-density plasma sources are finding increased use in a variety of materials processing applications. Several design variations have been developed using microwave and RF excitation of the plasma discharges. The modeling of these sources requires the solution of the electromagnetic fields, plasma/hard discharge physics and chemistry, and plasma-surface interactions. This paper focuses on the self-consistent solution of the electromagnetic fields and plasma discharge physics in microwave and RF plasma sources. Previous work has concentrated on solving the electromagnetic fields in unmagnetized microwave plasma sources [1], whereas, the plasma sources studied in this paper include multipolar ECR plasma sources, divergent field electromagnetics ECR plasma sources, and RF inductively coupled plasma sources. The electromagnetic fields are solved by a finite-difference time-domain solution of Maxwell's equations in two or three dimensions. The plasma discharge behavior is modeled using particle and fluid descriptions. The techniques for coupling the electromagnetic field solutions and plasma discharge behavior to a solution together self-consistently will be examined. Additionally, example power absorption profiles, electric field strengths, and plasma uniformities will be presented. The sensitivity of the spatial power absorption profile across an input power and pressure parameter space will be presented and its implications for uniformity will be discussed. Lastly, the electromagnetic field/plasma discharge simulations of the various sources will be examined with respect to identifying dominant plasma heating mechanisms.


9:20 am PS-TuM4 The Effect of Time Varying Sheaths on Radially Dependent Ion Energy Distributions in Inductively Coupled Plasmas, Robert J. Hoekstra, and Mark J. Kushner, University of Illinois, Dept. of Electrical and Computer Engineering, Urbana, IL 61801.

In moderate pressure (10$^{-1}$-10$^{-2}$ mTorr), low plasma density (10$^{10}$-10$^{12}$ cm$^{-3}$), reactive ion etching (RIE) tools, the ion transient time is often long enough that ions "see" an average sheath potential. Ion transit across the sheath may also be collisional. High plasma density (10$^{12}$ cm$^{-3}$), low pressure ($< 10$ mTorr) etching tools, such as inductively coupled plasmas (ICPs), use rf biasing of the substrate. The thinner rf sheath allows ions to transit the substrate. The ions may then "see" the instantaneous sheath potential resulting in the ion energy distribution (IED) being broadened. We have developed a 2-dimensional Plasma Chemistry Monte Carlo Simulation program (PCMCs) in which we are investigating spatially dependent IEDs in high plasma density ICP etching tools. The PCMCs uses 2-d time dependent electric fields and source functions as input. These quantities are produced by a 1-dimensional hybrid equivalent model of ICPs. The PCMCs tracks the trajectories of ions and radicals while accounting for ion-neutral and ion-ion collisions. IEDs as a function of position on the wafer will be discussed for multicomponent chlorine gas mixtures for p-Si and metal etching (Ar/Cl$_2$) and fluorocarbon gas mixtures for oxide etching (CF$_4$/H$_2$). The effect of electrode topography on ion angular distributions will also be discussed.

10:20 am PS-TuM7 Modeling the Chemistry in Chlorine Plasmas Using a Well Stirred Reactor Model with Comparison to Experimental Measurements, E. Meeks and J. W. Shon, Sandia National Laboratories, Livermore, California, Y. Ra and P. Jones, Lam Research Corporation, Fremont, California.

We present results from a plasma chemistry model that incorporates well-stirred reactor approximations to provide predictions of spatially and temporally averaged plasma properties. The solution of species, mass, and electron-energy balance equations determines steady-state electron energy and species compositions for ions, electrons, and neutral species. The electron energy equation includes a detailed power balance with losses to ions and electrons through the sheath, as well as inelastic and elastic collision losses in the plasma bulk. Model
predictions compare and test reaction-rate coefficients that are generated from solution of the Boltzmann equation and from a Maxwellian electron energy distribution function (EEDF). The model is then applied to chlorine-etch process conditions typical of high plasma density inductively coupled plasma (ICP) reactors. Model results show the dependence of species concentrations on the atomic chlorine recombination rate at both wafer and reactor-wall surfaces, as well as the effects of assumptions in the EEDF. The effect of inelastic collisions on the 'tail' of the EEDF is to reduce the ionization and excitation rates in the plasma. The dominance of surface reaction rates in determining plasma properties is expected to be equally important in higher-dimensional ICP models, due to the highly diffuse nature of these low-pressure reactors. Finally, we compare model predictions to the concentrations of ions, electrons, and radicals measured by a Langmuir probe and optical spectroscopic methods in the transformer coupled plasma (TCP) source.

10:40 pm PS-TuM8 Role of Etch Products in Si Etching by Cl2 in High Density Plasma Sources, C. Lee, M. A. Lieberman**, D. B. Graves, Dept. of Chem. Eng., Univ. of California, Berkeley, CA 94720 (**Dept. of Elect. Eng.).

For low pressure, high density plasma systems, etch products can play a significant role in affecting the plasma parameters such as the density of etchant species and electron temperature, and therefore overall processing parameters. We used a spatially-averaged global model to include both gas phase and surface chemistry in the study of silicon etching by chlorine. In this process, the etch products leaving the wafer surface are assumed to be composed of SiClx species (x = 1-4). These species, upon colliding with energetic electrons in the plasma, will undergo fragmentation and ionization. In addition, the formation of Cl⁻ from SiClx is also possible, which affects the electrostatic potential of the wafer when etch products are important neutral components.

The relatively high degree of dissociation in high density plasmas leads to the formation of elemental Si, which can deposit on the chamber walls and wafer surface. We have included surface models for both the wafer and the wafer in an attempt to better understand the role of etch products as a function of flowrate, pressure, input power, and species residence time. The phenomenological model for surface chemistry in the plasma was developed based on the available experimental data.

11:00 am PS-TuM9 Molecular Dynamics Simulations of Si Etching, H. Fell, Philips Research Laboratories, Eindhoven, The Netherlands.

Chemical sputtering of Si in a chlorine environment has been examined with molecular dynamics simulations. Two important processes are identified. Ion bombardment causes a rough surface and the chlorine passivation of the Si surface prevents the flattening of the surface due to the high activation barrier for surface diffusion. The rough surface contains reactive intermediates which can be desorbed onto the gas phase when, after an ion impact, the region has a large energy content. Secondly, the desorption of SiClx molecules is facilitated by the strong decrease of the free energy of activation with increasing temperature.

11:40 am PS-TuM11 Ion-Neutral Synergism in Ar-Enhanced Fluorine Etching of Silicon: a Molecular Dynamics Simulation, M. E. Barone and D. B. Graves, University of California, Berkeley, CA 94720.

We explore the role of low energy Ar ion bombardment (100, 50 and 20 eV) in enhancing fluorine etching of silicon, using molecular dynamics simulations. The key results from the simulations can be compared to various experimental measurements to help put together a more complete picture of the ion-neutral synergism, and the role it plays in etching. The simulations suggest that the source of the ion-neutral synergism is the creation of weakly bound species (wbs) during the short but intense collision cascade. These wbs are created throughout the SiFx layer and build up to an appreciable concentration during steady state etching. The formation of wbs occurs on short timescales, but the rate-limiting steps in etching often proceed on much longer timescales. Analysis of long timescale phenomena such as direct thermal desorption of wbs from the surface, and chemical reactions between surface wbs and thermal F impacting the surface, suggests that most of the experimental observations can be accounted for with this model. The overall yield is found to follow a square root dependence on ion energy. Kinetic energy distributions of etched species, yield as well as product distributions obtained from the simulation are compared with experiment.

Video illustrations of events occurring during ion-assisted etching will be presented.

VACUUM TECHNOLOGY
Room A102 – Session VT-TuM

Vacuum Systems for Accelerators and Fusion
Moderator: M. A. Benapfl, Lawrence Livermore National Laboratory.

8:20 am VT-TuM1 Theoretical Submonolayer Adsorption Isotherms for Hydrogen on a Heterogeneous Surface, J. P. Hobson, National Vacuum Technologies Inc., Box 4160, Postal Station E, Ottawa, Canada K1S 5B2.

Theoretical submonolayer adsorption isotherms for Argon, Nitrogen, and Helium on a heterogeneous surface, over wide ranges of pressure and temperature, were calculated by the author many years ago (1). These calculations showed good agreement with available experimental data. Since then the widespread development of large accelerators with cryogenically cooled tubes (cold bore) has made the analogous calculation of hydrogen isotherms of relevance. This paper presents such a calculation over the pressure range 10 exp -2 < P < 10 exp 3 (torr), and at temperatures of 300, 420, 600, 1000, 1500, 2000, and 3000 K. The values of relative coverage, Θ, span the range 0.1 < Θ < 1.0. The form of the isotherm used was lnΘ = \(-BP(R/mo /P_b)^2 + B = 0.0025 Cal/Mole, R = 1.9865 Cal/Mole Degree, and P_b = Vapor pressure of hydrogen at the chosen temperature. In regions where this isotherm is not quantitatively applicable the results have been rounded off to give the correct limiting values, i.e. P = P_b for Θ >> 1, and 0 = kP (i.e. Henry's Law with k constant) for Temperatures of 30 and 400 K. The results should be useful for comparison with the experimental data being assembled by Rao et al. (2). In particular the calculations which predict only relative coverage provide a useful way of evaluating the surface "roughness factor" or an experimental substrate.


Residual background pressures of helium and hydrogen are the main competing factors to the total pressure in beam line tubes of many cryogenically cooled particle accelerators and colliders. In the absence of He leaks and high H2 outgassing from room temperature components, the required base pressure of less than 10^-6 torr is easily maintained in these systems by cryosorption pumping of all residual gas species. In reality, additional pumping needs to be provided for keeping the system pressure at the required low levels. Cryosorption pumping of He by molecular sieves and charcoal has been investigated for
this purpose at 4.2 K. These studies indicate that high adsorption capacities could not be reached under dynamic conditions either due to the plugging of the pores and/or by the reduced mobility of hydrogen at this low operating temperature. Further, the low pressure adsorption data do not follow the Polanyi’s potential theory. Large surface area clean metal oxides are likely to provide the enhanced pumping speeds at low pressures with high adsorption capacities because of their open micropore structure. We have been investigating metal oxides for dynamic adsorption and desorption of hydrogen and helium. In this paper we present the theoretical analysis of the adsorption isotherm data of the molecular sieves, charcoal and metal oxides with respect to their suitability for cryogenic accelerator systems. We also discuss Polanyi’s potential theory with reference to low pressure adsorption isotherm data on metal oxides.

*This work supported by U.S. DOE Contract No. DE-AC05-84ER40150.

9:00 am VT-TuM3 D-T Experiments in TFFTR, P. H. La Marche and the TFFTR Team, Princeton University, Princeton, NJ 08543.

The first weeks of December 1993 witnessed the realization of the first deuterium-tritium (D-T) fueled plasma discharges in the Tokamak Fusion Test Reactor (TFFTR). This exciting time saw world records in fusion power output broken on an hourly basis, with output exceeding 6 megawatts. In the intervening time, although D-T high power plasma discharges are not yet commonplace, multi-megawatt discharge operation is reproducible to the point of on-demand-performance and the operation of TFFTR with tritium has become routine. These accomplishments represent the steady improvement in power output over the years by a number of fusion research devices from the microwave range of the sixties to the megawatt range of today, with a doubling time of less than one year. This steady progress is matched with a coupled improvement in several disciplines, particularly in vacuum pumping and vessel wall conditioning techniques. One technological objective for these experiments is to develop experience and expertise with safe handling and processing of tritium and tritium contaminated waste. Another objective is to understand and manage the changes in operation of the facility due to the increased levels of regulatory and safety controls brought by the use of tritium. We shall review results of D-T operation in TFFTR along with tritium processing, vacuum pumping and vessel conditioning in support of this operation.

INVITED

*This work supported by USDOE Contract DE-AC02-76-CH03073.


The He ash that will result from DT burn in a fusion reactor must be exhausted from the plasma to avoid serious fuel dilution effects. In view of this, transport and exhaust studies of He in fusion plasmas are getting increasing attention in recent years. To date, most of the He pumping investigations were conducted with pure He. In fusion plasmas, though, the exhaust gas will be a mixture of D2 and He, with He forming only a small fraction. D2 condensation on the pumping surface is shown to have an adverse effect on He cryosorption, although systematic data on this topic could not be found. We have investigated the pumping of pure He and a mixture of D2 and He (90% D2) using layers of Ar gas condensed at 4.35 K as the sorbent. The experiment revealed that: 1) cryosorption pumping speed of pure He drops precipitously if the Ar/He ratio falls below about 20, 2) the pumping speed for He in a D2/He mixture decreases in an exponential manner with the amount of D2 pumped, 3) increasing the thickness of Ar in the range of 1-20 μm showed no clear trend in the pumping speed for He in a D2/He mixture, and 4) with the pumping surface coated with a 2 μm layer of Ar, surrounded by a radiation shield having a transparency of about 17%, a He pumping speed of about 12,000 s^-1 could be obtained with 1 millibar 1 of D2 condensed on 1 m² of the pumping surface.

*Work supported by the U.S. Department of Energy, under Contract No. DEAC05-84OR24100 with Martin Marietta Energy Systems, Inc., and DEAC03-84ER51114 with General Atomics.


The 4 GeV recirculated linac at CEBAF uses 338 superconducting RF cavities to accelerate a cw electron beam for basic nuclear physics research. Pairs of superconducting RF cavities, fabricated from high purity niobium, are carefully chemically cleaned, rinsed, and assembled into hermetic units, prior to final assembly within cryostats for low temperature operation at 2K. Each cavity pair assembly contains 22 flanged joints and several meters of electron beam welds which must maintain leak tightness to UHV standards while submerged in a super fluid helium environment. Particular attention was given to the design, surface preparation, and assembly techniques for the niobium wire-sealed niobium and stainless steel flanges employed in the cavity assemblies. Finite element models of the flange geometry, and flange test assemblies that were temperature cycled to ensure vacuum integrity of the critical seals before the production cavities were assembled. After assembly of each production cavity pair, total integrated leak rates were measured in a 2K environment using an ultra sensitive (<10^-15 atm cc/s) leak detection method.

*This work supported by U.S. DOE Contract No. DE-AC05-84ER40150.


The quality factor Qe of Superconducting Radio Frequency (SRF) cavities is inversely proportional to the surface resistance of the Nb cavity walls. The residual resistance is a combination of many contributions including that of the physiosorbed hydrocarbons, residual gases and physiosorbed and/or chemisorbed hydrogen. In this paper we will present the results of our controlled studies on the effect of the quantity of the physiosorbed moist air and hydrogen on the Qe of a single cell Nb cavity. In these studies an extractor gauge is used at 2 K to monitor the pressure and quantity of the residual gases adsorbed. A monolayer of hydrogen is introduced onto the cavity at 4.3 K. The cavity is warmed to 100 K and held there for few hours, to form a hydride and the cavity is cooled back to 2 K. The quality factor of the cavity is measured as a function of the accelerating gradient. These studies are conducted with different physical and chemical treatments of the Nb cavity wall surface. The results of these investigations will be presented in this paper.

*This work supported by U.S. DOE Contract No. DE-AC05-84ER40150.


Ion pumps are widely utilized and recognized as the best pumps for UHV conditions. Despite their actual use, for historical reasons, the pumping speed in the UHV range is far from the maximum value. To analyze the pumping behaviour in UHV conditions a new pumping model is proposed, based on the direct evaluation of the sputtering rate of the getter material from the cathode. Different from former models, the present one uses a restricted set of empirical parameters; these have been evaluated fitting the theoretical results to the actual pumping speed and ion current measured on different ion pump configurations. The model allows to estimate the pumping speed as a function of the geometrical, physical and electrical parameters that define an ion pump. Introducing the correlations between all these parameters (e.g. the magnetic field depends on the element height), the pumping performance has been optimized in the low pressure range. In particular, the ratio between pumping speed and total volume (element plus magnets) has been maximized as function of cell radius, element height and anode to cathode gap. This model shows a maximum of pumping efficiency for a flat element configuration operated at high magnetic field values.

11:00 am VT-TuM9 NIF Vacuum Chamber, by R. W. Wawrik, Sandia National Laboratories, Dennis A. Muirhead, Rockwell Power Systems, Albuquerque, NM, and V. P. Karpenko, Lawrence Livermore Laboratory, Livermore, CA.

The National Ignition Facility (NIF) will be a facility capable of
generating about 20 MJ of fusion yield. Laser beams, 240 in number, will be directed at a fusion target located at the center of a 10 m spherical aluminum vacuum chamber. The required pump down time of 5 × 10^{-2} Torr in 2 hours of the 18,800 cf chamber, coupled with tritium contaminated debris, outgassing of diagnostics, neutronic activation of the materials used in the chambers construction, as well as the dimensional stability of the chamber result in a unique design for both the chamber, decontamination systems, and vacuum pumping systems. The additional requirements of oil free and full sized stand-by equipment pose additional design constraints for the vacuum system. One 36" cryopump is capable of meeting the requirements if .2 Torr can be reached in 75 minutes however, a 48" pump is less expensive and offers more safety factor in design. Three pumps will be required as a minimum, one running, one on stand by, and one in regeneration. Steam ejectors, which are used in most other large volume high altitude chambers, are not permitted due to the tritium's affinity for water. The tritium constraint also eliminates liquid ring pumps. The large Roots style blowers being used for second stage pumping don't like to be started at atmospheric pressure. The oil-free requirement may eliminate the use of straight vane type mechanical fore pumps, although when used with inert gas back-streaming or LN2 cold traps the fore pumps are usually of small capacity by design, therefore gas back-streaming is not recommended. These constraints and the conceptual design for meeting them will be discussed in the presentation.

INVITED

11:40 am VT-TuM1 A Non-circular UHV Seal That Functions Between ±200°C, R. A. Childs, J. E. Rice, Massachusetts Institute of Technology, Plasma Fusion Center, Cambridge, MA 02139

The Alcator C-MOD tokamak fusion experiment has over 40 large and small smoothed faced flanged ports that require reliable all metal seals to interface with the various diagnostic experiments and pumps that make up its 4000 liters volume. In at least 10 of these ports the environment is such that the flanges seal can experience temperature excursions between ±200°C. These particular flanges are large racetrack shaped flanges that have an I.D. of 8" wide by 24.75" tall. They are located inside the massive confinement vessel which is cooled to LN2 temperatures. Because of their location in the tokamak they can inadvertently have LN2 applied to parts of the flanges while the rest of the flange has heat being applied to maintain the vacuum vessel near room temperature or occasionally at bakeout temperatures. This can create local differentials of over 100° from one end to the other on a single flange. We will report on the development of the seal under extensive testing and after more than a year of actual use.

THIN FILM

Room A105 – Session TF-TuM

Thin Films for Sensors

Moderator: S. Semancik, National Institute of Standards and Technology.

8:20 am TF-TuM1 Thin Film Sensors for Automobile, Y. Taga, Toyota Central Research and Development Laboratories, Nagakute, Aichi 480-11, JAPAN.

A great amount of effort has been devoted to the constant improvement of such basic automobile performance as drivability, safety and environmental protection. As a result, the total combination of various technologies has made it possible to produce safer and more comfortable automobiles. Among these technologies, surface and thin film techniques are mainly concerned with sensors, optics and electronics and surface modifications.

This review paper first describes a new concept of thin film process in materials synthesis on the basis particle-surface interaction during deposition. Some examples of practical applications of thin films for sensors are then given. These include (1) a poly(vinylidene fluoride) thin film for integrated piezoelectric infrared sensors, (2) a new piezoelectric thin films of Cr-O-X system for pressure sensors and (3) a niobium pentoxide films for oxygen sensors.

Based on the recent progress of thin film technology, it has now become possible to control thin film nanostructure and to guarantee the durability for automobile applications.

Finally, future challenges of thin film sensors for automobile are discussed.

9:00 am TF-TuM3 Thin-Film-Based SAW Chemical Sensor Arrays, A. J. Rico, Microsensor R&D, Sandia National Laboratories, Albuquerque, NM 87185-0351; C. Xu, R. M. Crooks, Dept. of Chemistry, Texas A&M University, College Station, TX 77843-2355; R. E. Alred, Adherent Technologies, Inc., Albuquerque, NM 87123.

Chemical sensor arrays offer the promise of selective chemical detection without perfectly selective thin films, but the chosen films must have a degree of chemical orthogonality (preferential response to different classes of chemicals by each film). Using a six-device array of 97-MHz, ST-quartz surface-acoustic wave (SAW) delay lines, we have studied the chemical sensor response of two classes of thin film; self-assembling monolayers (SAMs) and plasma-grafted polymer films (PGFs). The sensitivity of the SAW to very small surface mass changes (<100 pg/cm²) and a range of other perturbations is necessary for characterization of analyte behavior, where SAMs and PGFs thickness range in thickness from a single molecular layer to 100 nm. SAMs were terminated by methyl, cyano, amino, and carboxylic acid functionalities, and by carboxylate-coordinated Cu++, Ni²⁺, Fe²⁺, and Zr++. PGFs were derived from acrylic acid, vinylphosphonic acid, and egulon (a phenol derivative). Arrays of SAW devices were exposed to 14 analytes representing saturated, aromatic and chlorinated hydrocarbons; alcohols; ketones; and carboxylic acids (and water sorption/desorption isotherms were obtained for each analyte over the zero to 50%-saturation vapor pressure range. Analysis of array data using multidimensional cluster analysis techniques under development in Sandia’s Vision Science Dept. results in very accurate identification of individual analytes. This work supported by the U.S. DOE under contract DE-AC04-94AL-8500.

INVITED


Zirconium carboxylate polymer-in-glass thin films have been prepared by hydrolysis of zirconium(V) acetate in excess valeric or acrylic acid. Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) have been used to determine the structure of these films. Results of spectroscopic analysis show that the polymer film consists of a Zr-O backbone coordinated with bridging and chelating bidentate carboxylate ligands. The mass sensing properties of the films for simple alcohols and amines have been determined using a quartz crystal microbalance (QCM). Our results show that altering surface structure by changing carboxylate ligands affects the mass sensing properties of the films. The zirconium acetate film selectively responds to alcohols and rejects amines. The selective alcohol response has been attributed to hydrogen bonding interactions between the alcohol hydroxyl group and O atoms (Zr-O backbone and acetate ligands) in the zirconium acetate film. The zirconium acetate film exhibits a higher response for amines, but shows only limited selectivity for the alcohols. Recent efforts to control the chemical affinity of zirconium carboxylate films using various carboxylic acids will be presented.


Surface acoustic wave (SAW) gas microsensors utilizing a gas sensitive doped metal oxide semiconducting thin film deposited between the input and output transducers of a SAW delay line oscillator provide high sensitivity than chemi-resistive sensors. Surface acoustic waves (SAW) sensor response to a target gas is maximized when the electrical conductivity of the film is within a critical range which depends on the piezoelectric substrate properties. The sensor selectivity, sensitivity, long term stability and reliability are directly related to the film microstructure. Reactively RF sputtered WO₃ and Au-doped WO₃ films which show high selectivity and sensitivity to H₂S have been deposited on Si and LiNO₃ substrates and characterized using two point probe conductivity measurements and atomic force microscopy (AFM). Film stability is improved by annealing the initially amorphous films to form polycrystalline WO₃. Conductivity is increased by decreasing the RF power or total sputter pressure during deposition. The changes in conductivity are correlated with changes in microstructure observed with the AFM. The results show that manipulation of the film mi-

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erestructure by variation of the deposition conditions can be used to optimize the performance of SAW gas microsensors.

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*NSF-REU student supported by NSF grant EEC-9000004.

10:20 am TF-TuM7 Increasing the Selectivity of Commercially Available Tin-Oxide Based Sensors for Process Environments, Roger M. Hawk and Arund Narayanasamy, University of Arkansas at Little Rock, Department of Electronics and Instrumentation, 2001 S. University, Little Rock, AR 72204.
The use of semiconductor sensors to detect changes in gas composition was discovered in the early 1950’s. The dominant device available now is the Taquchi sensor, which uses a sintered form of the n-type semiconductor, tin-oxide. This sensor is sensitive to alcohols and combustible gases and is used widely as a detector to monitor gas leakage.
However, there are limits in many applications due to the lack of selectivity. Presently, a sensor may be turned on when several gases are to be detected. This paper presents data on the enhanced specificity of commercially available gas sensors using a combination of temperature cycling and multi-dimensional signal processing techniques. Sensor parameters include: 1) surface current and voltage, and 2) complex impedance. Our investigations generated a data bank with I-V/temperature and complex impedance data from oxide temperature surfaces for known mixtures of reducing gases. The application of neural networks as a pattern recognition technique allowed trace detection of the different gases chemisorbed on the surface of the semiconductor gas sensor. This approach allows the use of a single sensor instead of multi-sensor arrays for the detection of different reducing gases. This reduces agents, eliminates problem calibration, and results in enhancement of sensor selectivity. Consequently, the cost of sensor operation in industrial multi-gas environments will be reduced.

A novel CVD method for localized thin film growth on a microscale with an immediate application in chemical sensing is reported. Previously, films used in monolithic microelectronic gas sensors were fabricated by vacuum deposition. A drawback of this method is that many promising sensing film materials are incompatible with the final etch used in micromachining of the device. The new method overcomes this limitation by depositing films on completely fabricated devices lacking only the transducing film. Thermally-activated CVD is achieved on the heated, stress-relieved, micromachined dielectric window. The window has an area of 350 μm x 350 μm and is 1.3 μm thick. It is composed of layers of SiO₂ (3000 Å), Si₃N₄ (2000 Å) and SiO₂ (7000 Å). A 5 μm thick meandering boron-diffused heater under the window permits the window temperature to be varied between ambient and over 1000°C. This structure represents a micro-heatpac with excellent temperature uniformity and rapid thermal response. The device has four-point probe electrodes to measure resistance changes. These electrodes are already in place and operational prior to thin film deposition.
Film growth occurs only on the heated region of the window and is monitored in situ using resistance measurements. End-point recognition is obtained by removing the heater current once a desired film resistance is reached. We illustrate microCVD of platinum (Pt) from tetrakisfluorophosphate platinum, P(PP₃)₄, and titanium oxide (TiO₂) from titanium isopropoxide, Ti[(CH₃)₃O]₂. The deposited films are extensively characterized using situ exposed using optical microscopy, spectroscopy, ESEM/SEM and XPS.

11:00 am TF-TuM9 Magnetoresistive Sensors for Ultra-High Density Magnetic Recording, J. Kent Howard, IBM Storage Systems Division, San Jose, CA 95193.
Magnetic recording technology has been the dominant technology for information storage since the invention of the computer. Computer disk drives have doubled in areal density every 2.5 years for the past 40 years. The areal density in the state of the art disk drives approaches 1 gigabit/in² (1 billion bits/in²) since the introduction of the magnetoresistive read head sensor by IBM in 1991. This presentation will review the concept of magnetic recording, magnetoresistive head materials and issues and the challenges to extend the magnetoresistive head to even higher areal densities. Recently, the discovery of a giant magnetoresistive effect in magnetic multilayer structures was announced. It is expected that the giant magnetoresistive effect can extend the read head sensor technology to areal densities of 10 gigahits/in² (10 billion bits/in²). We will discuss the giant magnetoresistive effect and the different options for fabrication as well as the challenges for implementation.

11:40 am TF-TuM11 Object Imaging with a Piezoelectric Tactile Integrated Circuit Sensor, Edward S. Kolesar and Craig S. Duson, Texas Christian University, Department of Engineering, Electrical Engineering Laboratory, Fort Worth, TX 76129.
The IC incorporates 64 sensor electrodes arranged in a symmetrical 8 x 8 matrix. A corresponding set of in situ high-input impedance metal-oxide-semiconductor field-effect transistor amplifiers are used to capture each trap’s pseudo steady-state response. With an electrical multiplexing circuit, the sensor’s unprocessed data can currently be measured in approximately 100 microseconds. Off-line computational assets are used to generate a tactile object image. Each electrode occupies a 400 x 400 μm square area, and they are separated from each other by 300 μm; this taxel density is consistent with the tactile nerve density of a human adult. A 40-μm thick piezoelectric PVDF polymer film was attached to the electrode array with an electrically non-conductive urethane adhesive. The response of the tactile sensor is linear for loads spanning 0.8 to 135 grams-force (gfs). The response bandwidth is 25 Hz, the hysteresis level is minimal, and, for operation in the sensor’s linear range, taxel crosstalk is negligible. The historically persistent stability and response reproducibility limitation associated with piezoelectric-based tactile sensors has been resolved by implementing a novel pre-charge voltage bias technique to establish consistent pre- and post-load sensor responses. The pre- and post-load stabilization bias responses are used in concert with the sensor’s response to a load, to generate a three-dimensional tactile object image. A rudimentary tactile object image measurement procedure for all applied loads has been devised to recognize the silhouette of a sharp edge, square, trapezoid, isosceles triangle, circle, toroid, slotted screw, and cross-slotted screw.

ELECTRONIC MATERIALS/MANUFACTURING SCIENCE AND TECHNOLOGY
Room A108 – Session EMMS-TuM

Surface Preparation and Passivation
Moderator: S. M. George, University of Colorado, Boulder.

8:20 am EMMS-TuM1 Very High Quality Thin Gate Oxide Film Formation Technology, Tadahiro OHMII, Tohoku University, Sendai, 980 JAPAN.
To realize highly reliable ULSI fabrication, the wafer surface should be free from: 1) Particle, 2) Organic contamination, 3) Metallic contamination, 4) Native oxide and 5) Surface micro-roughness. Qualities of ultrapure DI water, liquid chemicals and wafer cleaning technology have been drastically improved for last several years under the leadership of Tohoku University. Recently we found that once wafers are exposed to clean room air, organic materials adsorb on the wafer surface. This organic contamination degrade thin gate oxide integrity. However, organic contaminants on the wafer surface can be partly eliminated during a temperature ramp-up process by desorption or reaction with a little O₂ in inert gas, which is commonly used to avoid pitting of wafer surface. However, oxide growth during a ramp-up process can not be neglected for deep submicron processes which require ultra-thin gate oxide. Also a slower ramp-up rate is required for larger size wafers to reduce thermal stress that causes slip formation may enhance this problem. Therefore, the approach using a little O₂ during ramp-up to protect wafer surface may not be applicable to deep submicron devices. In order to solve these problems, we propose the following approach. First, wafers are cleaned and ozone treated ultra pure water and dried using a newly developed spin cleaner in a N₂ ambient. This process provides a high quality pre-oxide without organic contamination. Then, the wafers are transported into an oxidation furnace in an N₂ ambient. Finally, the wafers are ramped-up to an oxidation temperature in inert gases and oxidation is carried out...
in a reductive ambient with H radicals. This approach can completely eliminate organic contamination and uncontrolled oxide growth and provide very high quality thin gate oxide.

**INVITED**

9:00 am **EMMS-TuM3** STM/XPS Investigation on the Chemical Oxidation of Hydrogen Terminated Si(111) Surfaces, U. Neuwald, U. Memmert and R. J. Behm, Abt. Oberflächenchemie und Katalyse, Universität Ulm, D-89069 Ulm, Germany.

We used STM and XPS to investigate the chemical oxidation of hydrogen-passivated Si(111) surfaces in a variety of oxidizing media (air, 30% H₂O₂, conc. HNO₃). Prior to oxidation, we prepared smooth and well ordered Si(111) (1 × 1)-H surfaces by etching in 40% NH₄F solution. These samples were exposed to the different oxidizing media at room temperature for durations of up to one year. The thicknesses of the resulting thin oxide layers were determined by XPS. The geometrical interface structure was imaged by STM after selectively removing the newly grown oxide by a short dip in conc. HF.

Initially, oxide growth proceeds for all oxidizing media used in this study exclusively within the topmost Si(111) bilayer, via nucleation of homogeneously distributed 10-20 Å wide oxide patches. We found the oxidation of the first bilayer to be completed after 20 s in conc. HNO₃, after 20 min in 30% H₂O₂ and after 1 month in the case of humid air. In dry air the first layer was only partly oxidized even after 1/2 year.

The second Si(111) bilayer reacts much slower than the first layer. Interestingly, we found that for the second layer the oxidation rates are independent of the nature of the oxidant.

The oxide thickness vs. time curves, determined from both the XPS and the STM data, can be fitted assuming simple Langmuir adsorption kinetics for the first layer and field assisted oxidation according to the Mott-Cabrera mechanism for the subsequent growth of the second oxide layer.


The question of whether the initial oxidation of silicon proceeds in a layer-by-layer fashion or through 3D islands is a matter of intensive debate. For example, recent STM experiments have interpreted as supporting in-depth island oxidation at 600°C, starting at monolayer coverages. We have used high resolution medium energy ion scattering (MEIS) with approximately monolayer depth resolution to investigate the growth mode. The width of the oxygen energy spectra during oxidation at 600-700°C is found to be nearly constant up to an oxygen coverage of ~3 ML (3 atoms per Si(100) unit cell). This fact suggests that the surface oxide does not grow deeper until the entire surface is covered by this surface oxide. This conclusion is also supported by results on Si atom displacements, as deduced from Si angular distribution spectroscopy (ADDS). At 600°C, the oxygen energy peak gets broader, indicating oxidation in deeper layers. From our ion scattering experiments, we believe that the difference between monolayer oxidation at 250°C and 600°C, observed previously by STM, is more likely due to differences in local stoichiometry rather than simply the oxygen depth distribution.

*Supported in part by NSF DMR 90-19868 and 93-06899.

9:40 am **EMMS-TuM5** Controlled Nitrogen-Atom Incorporation at Si-SiO₂ Interfaces by a Low-Temperature (300°C) Pre-Deposition Remote Plasma Oxidation Using N₂O, D. R. Lee, and G. Lucovsky, Departments of Materials Science and Engineering, Electrical and Computer Engineering, and Physics, North Carolina State University, Raleigh, NC 27695-8202.

Device-quality Si-SiO₂ structures can be formed by a two-step low-temperature (300°C) process that includes (i) formation of the Si-SiO₂ interface on a H-terminated Si surface by remote plasma-assisted oxidation, and (ii) deposition of an SiO₂ layer by remote PECVD [1]. The source gas for the oxidation step is O₂ for the PECVD, they are N₂O and SiH₄. The oxidation step (i) removes residual C atom contamination to a level of ~10¹² cm⁻² (SIMS), (ii) promotes formation of a low defect density Si-SiO₂ interface (Dₐ ~ 1-3 × 10¹⁵ cm⁻² eV⁻¹) that can withstand processing temperatures to ~900°C, (ii) forms ~0.5 to 0.6 nm of SiO₂ which serves as a platform for the oxide depositions, and (iv) prevents uncontrolled incorporation of N-atoms at the Si-SiO₂ interface during oxide deposition from N₂O, thereby preventing increases in Dₐ. A similar process has been used with other remote PECVD dielectrics: silicon oxynitride alloys and SiOₓ/AlₓNₙ composites. In this paper we describe a new process that replaces N₂ with N₂O for the pre-deposition oxidation. The use of N₂O does not degrade the effectiveness of this process in reducing C-atom contamination, but increases the interfacial N-content in a controllable way from ~1.5 × 10¹⁵ cm⁻² to ~8.6 × 10¹⁴ cm⁻². C-V measurements on MOS capacitors using the O₂ and N₂O processes showed that Dₐ, and fixed oxide charge (Qₓ) near the Si-SiO₂ interface were not degraded by the > five-fold increase of N-atoms, in fact Dₑ levels decreased from ~1.5 × 10¹⁰ cm⁻² eV⁻¹ to ~1.0 × 10¹⁰ cm⁻² eV⁻¹. Performance data will be presented for MOS devices with 100 Å thick SiO₂ dielectrics fabricated on Si-SiO₂ interfaces formed by changing the O₂ to N₂O ratio in the oxidation step, and thereby controlling the level of N-concentration at the Si-SiO₂ interface. Values of Dₑ and Qₓ and their stress dependences will be reported as functions of the N-atom concentrations (by SIMS) at the Si-SiO₂ interface.


10:00 am **EMMS-TuM6** Silicon Oxide Etching using Gas Phase HF/solvent Mixtures, Anthony J. Mucat, A. Scott Lawing, Han Xu, and Horbert H. Sawin, Department of Chemical Engineering, Bldg. 66-225, Massachusetts Institute of Technology, Cambridge, MA 02139.

Gas phase mixtures of anhydrous HF and solvent have the potential to replace liquid phase solutions for silicon oxide removal in wafer cleaning applications. Both water and alcohols have been shown to be effective solvents in the gas phase but their role in the etching reaction is incompletely understood. We have conducted high vacuum compatible apparatus with a base pressure of 10⁻⁸ Torr to study the contribution of various solvents in the etching of silicon oxide with anhydrous HF under well-defined conditions. Solvents were vaporized and introduced into the apparatus along with anhydrous HF in regulated amounts. The etching rate monitored in situ by ellipsometry was studied as a function of total pressure from 0.01 to 400 Torr and process temperature from 25 to 100 °C. A water or alcohol partial pressure of approximately five percent of its vapor pressure at the process temperature was found to be necessary to obtain good etch uniformity and control at etch rates in the range of 300 Å/min. Results will be presented showing the importance of the solvent in the etching of silicon oxide using anhydrous HF.

10:20 am **EMMS-TuM7** Ion Beam Characterization of Si(100) Surfaces During Wet Chemical Cleaning, V. Altieri, N. Herborts, P. Ye, and R. J. Culbertson, Deps. of Physics and Astronomy, Arizona State University, Tempe, AZ.

This study is motivated by the development of low-temperature surface cleaning methods for Si. Epitaxial growth requires an initial surface that is ordered and as free as possible of contaminants such as C and O, or metallic impurities. Wet etching of Si by a solution of HF in alcohol after a modified RCA clean was found to produce a (1 × 1) H-terminated hydrophobic Si surfaces that are ordered at room temperature. Our purpose is to measure in a quantitative fashion the removal of C and O, the H-coverage as well as residual disorder at the surface. Hydrogen is detected by the elastic recoil at 2.8 MeV, whereas, C and O are detected by nuclear resonance analysis (NRA) at 3.05 MeV and 4.265 MeV respectively. NRA is combined with ion channeling along the Si(111) direction to increase sensitivity to C and O as well as measure the surface peak to correlate it to surface disorder. Our results demonstrate that NRA for C and O combined with ion channeling can measure in a quantitative fashion, the change in C and O and H-coverage at the different steps of wet cleaning. For instance the total amount of C on Si(100) sample decreases by a factor of 1.5 after the first step in the surface cleaning, by immersing in a solution of H₂O₂:H₂O:OH:O₂ (4:1:1) at 80°C for 10 minutes, that is intended to remove hydrocarbon from the surface. The total amount of native oxide is found to increase by a factor of 2, while the H content of the oxide does not change.


10:40 am **EMMS-TuM8** AFM Characterization of Pillar Structures on RCA Cleaned Silicon Surfaces, B. K. Furman, D. A. Neurogschi, S. L. Cohen, R. Tsai, K. Pope, M. Lither, IBM T. J. Watson Research Center, P.O. Box 218, Yorktown Heights, NY 10598, S. Basiliero,
11:04 am EMMS-TuM11 Thermal Effects on GaAs(001) Surface Prepared by Deoxygenated and Deionized Water Treatment, Y. Hirata, T. Oigo, Y. Watanabe, and M. Oshima, NTT Basic Research Labs, 3-1, Morinosato Wakamiya, Atsugi-shi, Kanagawa 243-01, Japan. NTT Interdisciplinary Research Labs, 3-9-11 Midori-cho, Musashino-shi, Tokyo 180, Japan.

Surface defects of GaAs degrade optical and electrical device characteristics such as efficiency of luminosity intensity and electron mobility. To prevent surface defects during cleaning process, we have recently proposed a new method based on electrochemical reaction. Deoxygenated and deionized water (DDODI) treatment, in extremely low concentration of dissolved oxygen (1 ppb), completely removes surface oxides and produces a passivation layer of elemental As and/or hydrogenated As on the surface due to the liberation of Ga atoms after removal of oxides. After heating the DDODI treated-GaAs(001) and (111)B surfaces in high vacuum, RHEED observation shows spotty 2 x 4 and 3 x 6 surface reconstruction patterns, and STM observation shows 2 x 2 and v/19 x v/19 structures, respectively.

In the present study, we investigate the temperature dependence of surface Fermi level (E_F) by using synchrotron-radiation photoelectron spectroscopy (SRPS) in ultra-high vacuum. SRPS reveals that the E_F for the DDODI treated n-type (8 x 10^17/cm^3) GaAs(001) surface lies at E_F = 0.9 eV at room temperature, moves toward E_F = 0.6 eV on heating to 500°C, and returns to near-flat-band position (E_F = 1.0 eV) after cooling to room temperature. These results suggest that the changes in E_F is due to the reduction in density of surface state, which is achieved with the DDODI treatment. This is because the DDODI treatment is essentially a low energy process and the DDODI treated surface is initially passivated with excess elemental As and/or hydrogenated As layer.


MANUFACTURING SCIENCE AND TECHNOLOGY
Room A110 – Session MS-TuM

Manufacturing Overview and Environmental Issues
Moderator: G. W. Rubloff, North Carolina State University.

8:20 am MS-TuM1 Environmental Consciousness: A Strategic Competitiveness Issue for the Microelectronics Industry, Gregory E. Pitts, Director, Environmental Programs, Microelectronics and Computer Technology Corp. (MCC), 3500 W. Balcones Center Dr., Austin, TX 78759-5398, (512) 238-3800, (512) 338-3814, pitts@mcc.com

The electronics industry has become increasingly aware of the need to develop a strategic approach to identify cost-effective, long-term solutions for environmental issues. Many organizations have invested resources to address environmental issues and business opportunities. As these efforts have evolved, it has become apparent that a strategic, coordinated approach for addressing environmental issues and business opportunities will help the electronics industries maintain a competitive edge in the international market and keep up with competitors who have long-range planning partnerships in the industry and government for addressing environmental issues. This paper will discuss several activities to develop a broad environmental strategy and solutions for priority environmental issues.

INVITED

9:00 am MS-TuM2 Integrating Regulatory Policy & Science—Can It Be Done? Bruce C. Jordan, Director, Emission Standards Division, MD-13, U.S. EPA, Research Triangle Park, NC 27711.

Regulatory activity within the United States is placing a heavy burden on industry. The economic consequences of government regulations have increased manyfold in the last few years. Too often, industry has left in the hands of lawyers, and non-scientific oriented employees, or professional lobbyists its role in regulatory activity. Yet, within most industries lies the knowledge and capability to achieve the most cost-effective approach to meeting environmental objectives.

This presentation will review how industry and the academic community can take a more active role in regulation development. A review of the regulatory process for the Clean Air Act will be presented.
Examples where industry can and have become actively involved in regulation development will be discussed and results reviewed. Finally, the future of the relationship between the industry and regulator is outlined and options illustrated.

9:40 am MS-Tu5 Environment, Safety and Health Issues in Manufacturing.

One of the roadmaps in the National Technology Roadmap for semiconductor technology is the 15 year strategy for Environment, Safety and Health improvements in semiconductor manufacturing. Materials and process changes are a significant part of this strategy which will be discussed in this paper.

SEMA TECH, a consortium of leading United States semiconductor manufacturers and ARPA, is a major implementor/facilitator of the national roadmap. The SEMATECH ESH program trends in semiconductor manufacturing and their relation to the national roadmap will be presented.

INVITED

10:20 am MS-Tu7 Factory of the Future: The “Whole Factory” View, Dr. Sam Harrell, Chief Strategy Officer, SEMATECH.

The primary task of a semiconductor factory is to produce integrated circuits of outstanding performance at a steeply declining cost per electronic function delivered. The semiconductor industry over the next decade will need to continue to drive costs down in order to maximize manufacturing productivity in the climate of rising product complexity, equipment cost, and risk. Historically we have concentrated on aggressive yield improvement and declining wafer fab cost per cm² as the dominant factors for increasing productivity. To continue to be competitive, SEMATECH is extending its focus beyond cost per cm² to the more total view of cost per function. We are working toward a combination of solutions to meet our customers’ challenges recognizing that no single solution will provide enough benefit to achieve the productivity gains end users expect. The “Whole Factory” View is a useful strategic planning tool for defining the future of manufacturing in the semiconductor industry.

INVITED


This talk describes several opportunities for long-range research at the academic-industrial interface that could significantly impact future generations of semiconductor manufacturing technology. A vision for success is emerging within the semiconductor industry that calls for increased cooperation and collaboration in tackling critical competitive issues. This vision is driven by the need for leading-edge, high-performance, semiconductor-based electronics. It comprehends the importance of balancing the escalating cost, complexity, and sophistication of the research and development processes required to bring new science and technology to an environmentally conscious marketplace. The presentation uses continuously shorter product and process research and development cycle times is engendering effective and innovative working partnerships between independent companies, government, and university R&D programs. These pioneering efforts have led to industry consensus on identifying several critical gaps and potential showstoppers on the path to future technologies. These recognized critical issues provide opportunities for long-term research that will increase fundamental understanding of chemical and physical processes, enable high-performance and robust technologies, and strengthen the supporting infrastructure of trained scientists and engineers.

INVITED

11:40 am MS-Tu11 Photocatalytic Oxidation for Point-of-Use VOC Abatement in Microelectronics Manufacturing, Mahbub Ameen, Ronnie Yaragheneh, John Nico, and Gregory B. Raupp, Department of Chemical, Bio & Materials Engineering, Arizona State University, Tempe, AZ 85287-6006.

In response to the Montreal Protocol of 1989, many microelectronics manufacturing firms are replacing chlorofluorocarbon hydrocarbons (CFCs) used in cleaning processes with volatile organic compounds (VOCs) such as acetone, isopropanol and glycol ethers. Although these solvents provide acceptable cleaning capabilities, these, as well as other VOCs, are on the 1990 Clean Air Act Amendments list of hazardous compounds. As a result, a threshold has been established for VOC emissions, forcing the industry to consider VOC abatement equipment to limit their emissions. We have recently initiated a research and development program aimed at establishing the commercial viability of gas-solid heterogeneous photocatalytic oxidation (PCO) for point-of-use abatement of VOCs in air streams. In PCO, VOCs present in process or air vents can be rapidly and completely oxidized to innocuous byproducts over near-UV illuminated titanium dioxide thin film catalyst at room temperature. Photocatalytic oxidation appears to be well-suited to the special requirements of the semiconductor processing industry. In this paper we review these requirements in the context of the recently-published SIA roadmap. The specific requirements for VOC abatement from (1) a typical photolithography track, and (2) solvent cleaning stations are presented, as are bench scale PCO kinetics for target VOCs.

INVITED

BIMATERIAL INTERFACES
Room A106 - Session BI-TuM

Protein-Solid Surface Interactions
Moderator: B. D. Ratner, University of Washington.

8:20 am BI-Tu1 Molecular Recognition at Protein-Biological Composite Interfaces, P. S. Stayton, R. Clark, C. L. Long, L. Klumb, A. Chilkoti, A. A. Campbell, G. Drohoby, University of Washington, Seattle, WA 98195 and Battelle Pacific Northwest Laboratories, Richland, WA 99352.

The Materials Science community has focused much recent attention on the way in which biological composites and ceramics are formed and processed. Biology provides many examples of exquisitely constructed hard materials, ranging from bone to seashell nacre, and "lessons from nature" may prove valuable in attempts to improve ceramic/composite processing strategies. In most known examples, proteins provide the molecular control necessary to control the hierarchical microstructure of composites with unparalleled specificity and structural resolution. Despite their well established importance, however, there is little known of the direct molecular recognition mechanisms used by protein surfaces to control the nucleation and growth of biological composites such as calcium oxalate and hydroxyapatite.

We have utilized a combination of site-directed mutagenesis, functional characterization of protein-crystal interactions, and high-resolution structural analysis to elucidate molecular recognition processes at the protein-biological composite interface. These studies are providing a detailed molecular picture of the mechanisms utilized by proteins to control composite structure. The principles and rules defining biological composite engineering should be generally useful to materials scientists interested in improving ceramic engineering technologies.

INVITED

9:00 am BI-Tu3 Surface Plasmon Imaging of Biotin-Streptavidin Binding on UV-Photopatterned Alkanethiol Monolayers Self-Assembled on Gold, D. P. Piscenec, M. Tarlov*, and W. Knoll, Max-Planck Institute for Polymer Research, Ackermannweg 10, D-55099 Mainz, Germany and *National Institute of Standards and Technology, Gaithersburg, MD 20899.

We report the surface plasmon imaging of UV-photopatterned alkanethiol self-assembled monolayers (SAMs) on gold and subsequent biomolecular recognition reactions on these surfaces. To photopattern the monolayers, a SAM of desired surface functionality is formed, then irradiated with UV light through a mask, and then immersed in a solution containing a second alkanethiol molecule of different terminal functionality. Using this procedure, patterned monolayers are formed containing two regions of different surface properties with micro-scale features. Surface plasmon resonance was used to ascertain the effectiveness of the UV-photopatternning process by measuring changes in SAM thickness following UV exposure and immersion in a second alkanethiol solution. The binding of the protein streptavidin to biotin-terminated alkanethiols was used as the model biomolecular recognition system. The specific binding of streptavidin to the surface was maximized by using a mixed monolayer consisting of the biotinylated thiol diluted into a hydroxy-terminated SAM. A pure hydroxy-terminated monolayer was used to retard the non-specific binding of streptavidin to selected regions of the surface. In this talk the use of surface plasmon microscopy to detect and image in real time the binding of streptavidin to the patterned biotinylated SAM surfaces will be demonstrated. In addition, the potential use of surface plasmon microscopy for parallel addressing of biosensing and diagnostic arrays will be discussed.
9:20 am **BiTuM4** Proteins at Surfaces Studied by Scanning Probe Microscopy, C. J. Roberts, M. C. Davies, D. E. Jackson, K. M. Shakesheff, S. J. B. Tendler and P. M. Williams, The Laboratory of Biophysics and Surface Analysis, Department of Pharmacology and Pharmacaceutics, The University of Nottingham, Nottingham, NG7 2RD, UK.

The application of scanning probe microscopy (SPM) to the analysis of biomolecular structure and function is a goal sought by a number of laboratories. We have developed a number of widely applicable immobilization techniques to allow reproducible SPM imaging of proteins on a variety of substrates, including gold, mica and polyethylene terephthalate. Here we will present molecular resolution data of biotinylated antibody-antigen complexes bound to polyethylene terephthalate via a streptavidin link. Specifically the SPM data will compare a passively adsorbed anti-streptavidin antibody to a biotinylated antibody, both for surface coverage and functionality to the antigen, ferritin. To validate SPM data and to aid in its interpretation we commonly employ complementary biophysical techniques, and in this case we have used surface plasmon resonance to supply data on antibody and antigen adsorption rates to a polystyrene surface. The biological and biomolecular information which may be retrieved from such data will be demonstrated by the observation of an approximate ten-fold increase in functionality of the biotinylated antibody bound via streptavidin to polystyrene as opposed to the antibody passively adsorbed to the polystyrene surface.

9:40 am **BiTuM5** Plasma Protein Interactions with Solid Surfaces, R. E. Marchant, C. Siedlecki and S. J. Eppell, Department of Biomedical Engineering, Case Western Reserve University, Cleveland, Ohio, 44106.

This presentation will review our recent studies directed towards understanding the nature of protein interactions with synthetic surfaces. We describe a molecular-level study by atomic force microscopy (AFM) of von Willebrand Factor (vWF) on hydrophilic and hydrophobic solid surfaces. vWF is large multi-meric plasma glycoprotein, which is an important biopolymer involved in thrombus formation, particularly in areas of the vascular circulation where high shear stresses are prevalent. vWF is composed of extended linear chains, with a distribution of molecular weights ranging up to 20 x 10²⁰, making it the largest globular protein in plasma. In our early experiments on hydrated vWF deposited on mica and imaged by AFM, the protein molecules were easily swept across the surface by the lateral force applied by the probe tip. Removal of interfacial water by drying increased protein-surface adhesion and permitted molecular level imaging in air, in which the distinctive globular domains of vWF were resolved and analyzed. When vWF was studied by AFM on a hydrophobic surface of octadecylchiroicosane (OTS) self-assembled monolayer under phosphate buffered saline, strong protein-surface adhesion was observed. The self-assembled monolayer deposited on gold was sufficiently smooth to permit identification of the adsorbed protein and the surface was sufficient to withstand an estimated applied lateral force of 19 nN, showing that vWF experiences strong interaction with a hydrophobic surface in aqueous media. This allowed repeated scanning by the AFM probe, and images of vWF on a submolecular scale were obtained. Statistical analysis of adsorbed vWF images and flow, that the protein is composed of large globular domains with elliptical cross sections of average dimensions 56 nm (major axis) 26 nm (minor axis) and 2.8 nm (height).

On the basis of the analysis of the globular domains, a model describing the three-dimensional structure of vWF dimer adsorbed on a hydrophobic surface in a physiological solution will be presented.

**INVITED**


There is an established need in industry for surface coatings which resist fouling by organic macromolecules and bacteria. Poly(ethylene oxide) (PEO) films have been recognized in the biomaterials literature as having desirable protein- and cell-resistant properties. Radio frequency plasma deposition of oligo(ethylene oxide) monomer onto a molecularly smooth PEO substrate was used in this study to vary the number of substrates to study the effects of the gas phase on the plasma deposited longer oligo(ethyleneoxide) chains improve the protein resistance of the films. Films were prepared on glass substrates by radio frequency plasma deposition of monomers containing from 2 to 12 ethyleneoxide units in a tubular, capacitively-coupled reactor. Depositions were performed under controlled pressure and flow, dissipating the deposition power to enhance film adhesion. Micron thickness, roughness, composition and surface energy were subsequently characterized by AFM, XPS, SSIMS, and contact angle goniometry. The protein-resistance of the film was determined by I-125 radiolabeled fibrinogen adsorption. Partial least squares modelling was performed to determine whether protein resistance could be correlated to oligo(ethylene oxide) fragment length in the mass spectra of the films.

10:40 am **BiTuM8** Biodegradability of Titanium Implant Alloys, Susan J. Kerber, Material Interface Inc., Sussex, WI 53089-2244.

A study was conducted regarding the adsorption of peptides on cp-Ti and Ti-6Al-4V. The peptides used were arginine-glycine-aspartic acid-serine (RGDA), arginine-glycine-aspartic acid (RGD), and arginine-phenylalanine-aspartic acid-serine (RDAS). The tripeptide RGD is known to be important for specifically specific adhesion reactions. This research was conducted to investigate the reason for a tendency to thrombus formation with Ti-6Al-4V that is not observed with cp-Ti. After argon plasma cleaning, coupons of the titanium alloys were inserted into solutions with variable concentrations (0.0625 mg/ml to 2 mg/ml) of an individual peptide group under constant temperature and pH conditions. The samples were rinsed, dried, and analyzed with x-ray photoelectron spectroscopy (XPS). Adsorption isotherms were observed by obtaining the relative amount of peptide adsorption as a function of solution concentration. It was determined with XPS that the major adhesion mechanism for the peptides to the titanium alloys was hydrogen bonding. CP titanium and Ti-6Al-4V were hypothesized to react differently as implants because Ti-6Al-4V has a more electropositive surface which allows fewer hydrogen bonds to form. Hydrophilic reactions were of secondary importance during bioadhesion, influencing the structure of the second layer adsorbed. There was no correlation found between the net charge of the peptide groups and their adhesion to the alloys.

11:00 am **BiTuM9** Electrochemically and Glow Discharge Modified Titanium Surfaces Used for Biological Evaluation, B. O. Arons, J. Lasnmaa, M. Rodahl, and B. Kasemo, Chalmers University of Technology and University of Goteborg, 412 96 Goteborg, Sweden.

Titanium is a suitable model material for studying how different surface properties influence biological response since: (i) it is a commonly and successfully used biomaterial, and (ii) its surface properties can be varied over a wide range. In this work we have used differently prepared titanium surfaces for studying how protein adsorption in vitro and bone response in vivo is influenced by different structural and chemical properties.

Electrochemical methods and glow discharge plasma methods were used for preparing Ti surfaces with varied surface structures, oxide thicknesses, and surface compositions. Characterisation of the prepared samples was done by AES, XPS, AFM, SEM, and FT Raman spectroscopy. Enzymelinked immunosorbent assay (ELISA) was used to measure the amount of proteins (fibrinogen and albumin) adsorbed onto the surfaces. Screw-shaped Ti samples were implanted in rabbit bone and, after different time periods, retrieved and sectioned for bone histomorphometry analysis.

Electrochemical preparation results in a titanium dioxide (TiO₂) surface layer of different thickness (4-200 nm). Glow discharge treatment results in either a TiO₂ (thickness 2-300 nm) or a nitride TiₙOₙNₙ (thickness 2-7 nm) surface layer depending on the gas (O₂ or N₂) and other discharge parameters. On top of the surface there were always a contamination layer, in most cases consisting of mainly hydrocarbons.

The results of the protein adsorption and bone response studies will also be summarized and discussed.

11:20 am **BiTuM10** Molecular Machining by Enzymatic Modification of a Chemisorbed Lipid Monolayer, Brian M. Peek, Thomas E. Kurtz, David C. Turner, Douglas D. Archibald, Deborah Leach-Scampavia, and Bruce Paul Gaber, Center for Bio/Molecular Science and Engineering, Code 6900, Naval Research Laboratory, Washington, DC 20375 and National ESA and Surface Analysis Center for Biomedical Problems, University of Washington, Seattle, Washington, WA 98195. We wish to combine the monolayer-scale control of the atomic force microscopy (AFM) with the specificity of enzyme chemistry. An enzyme on the tip of an AFM cantilever could be used to write a pattern on a surface modified with the enzyme's substrate. Ideally, this could allow the creation of patterns with molecular resolution. Thus, we have modified silicon wafers with an antigen-terminal silane (EDA), and attached a carboxylic acid derivative of dimyristoylphosphatidylcholine (DMPC) to create an immobilized lipid monolayer. The resulting lipid layer, characterized by AFM, electron microscopy for chemical analysis (ESCA), and ellipsometry, was also treated with phospholipase C (PLC), the enzyme which catalyzes the cleavage of...
the glycerol-phosphate ester bond of the lipid headgroup. Analysis of
the resulting surface, by the above methods, shows that the enzyme
modified the immobilized lipid layer. We are currently examining
different methods to immobilize PLC on a surface in an oriented
fashion while retaining full enzymatic activity.

11:40 am  BI-TuM11 Static Secondary Ion Mass Spectrometry Study
of Amino Acids on Acidic and Basic Polymer Surfaces, S. Kovatch,
Y. Kim, E. Bekos, J. A. Gardella Jr., State University of New York
at Buffalo, Buffalo, NY 14214.

Characterization of adsorbed proteins on polymer surfaces is an
important step in the development of new biomaterials. Static SIMS
is one tool used to gain information on the adsorbed chemical species,
and has successfully been employed for analysis of polymers on metal
substrates. In order to apply SSIMS to protein analysis on polymer
surfaces, an understanding of the secondary ion emission of proteins
is essential. In developing a protocol to study these systems, we have
chosen to investigate a more fundamental system: the adsorption of
amino acids on polymer surfaces. Adsorption on chemically modified
polystyrene sulfonic acid and polyaminostyrene is used to investigate
the effect of surface acidity or basicity on the secondary ion yields of
phenylalanine and tyrosine. These results are compared to previous
work on polycrystalline metals.1

A. Benninghoven, D. Jaspers and W. Sichtermann, Appl. Phys. 11
(1976) 35.
SURFACE SCIENCE
Room A205 – Session SS1-TuA

Dynamics and Kinetics of Surface Processes
Moderator: D. C. Jacobs, University of Notre Dame.

2:00 pm SS1-TuA1 Dynamic of the Interaction of O Atoms with O2/Pt(111), C. T. Rettner, J. Lee, and D. J. Auerbach, IBM Res. Division, 650 Harry Road, K33/801, San Jose, CA 95120.

Exposing an 18O2-covered Pt(111) surface to a beam of 18O atoms at a surface temperature, T₀, of 80 K yields a prompt 18O18O product. In addition, 18O₂ molecules are displaced from the surface. In both cases, the nascent 18O leaves the surface with a bimodal velocity distribution that is clearly different from that obtained for laser-induced thermal desorption. We find no evidence for 18O leaving the surface. The 18O18O product appears to result from a new desorption mechanism in which the adsorbed is directly displaced by the incident species. In this latter case, the desorbed molecule must carry away part of the adsorption energy of the incident O atom. Photodesorption at 213 nm gives a velocity distribution that is identical to that for the displacement reaction, indicating that the nascent molecules are actually displaced by photofragments at this wavelength, rather than being directly photodecomposed. Our findings also provide insight into the mechanism of the thermal desorption and dissociation of 18O2 on Pt(111), which occurs at temperature T₀ ~ 150 K. The fact that the velocity distribution for thermal desorption is different from that for displacement by O₂ molecules indicates that desorption must precede dissociation in this system.

2:20 pm SS1-TuA2 O₂ Adsorption on Silver Surfaces, F. Buatier de Mongeot, M. Rocca and U. Valbusa, CFSBT-CNR and INFN, Dip. d) Fisica, via Dodecanese 33, 16146 Genova, Italy.

We have studied the interaction of O₂ with Ag(100) and Ag(111) using a supersonic molecular beam to investigate the dependence on crystal face and temperature of the sticking probability S which was measured both by using the method of King and Wells, and by evaluating the derivative of surface coverage versus exposure with the EELS measurement [1].

For Ag(111) at T₀ = 100 K the initial sticking coefficient S₀ is lower than our experimental sensitivity of 2 · 10⁻⁶. For longer exposures we find that the condensation of H₂O originating from the background of the vacuum chamber (base pressure ~ 10⁻¹¹ mbar) induces the formation of OH. We find no evidence for the presence of adsorbed superoxido oxygen, in contrast with literature [3], neither for the clean surface, nor in presence of H₂O contamination as the observed losses can be assigned to H₂O.

The reactivity of Ag(100) towards oxygen is comparable with the one of Ag(111), recorded in our laboratory [3]. For molecular adsorption we find S₀ = 0.15 at T₀ = 100 K, E₀ = 0.65 eV and normal incidence. S₀ depends strongly on surface temperature, showing an abrupt decrease above the dissociation temperature of 200 K. Preliminary data indicate that low temperature molecular adsorption follows normal energy scaling, while dissociative adsorption follows nearly total energy scaling. Such a behaviour is observed for the first time to our knowledge.


2:40 pm SS1-TuA3 CO₂ Sticking on Pt(111): The Role of Kinetic Energy and Internal Degrees of Freedom, Dmitrii Kuligov*, Mats Persson, Claes Akertloud, Igor Zoric and Bengt Kasemo, Department of Applied Physics, Chalmers University of Technology, 412 96 Gothenburg, Sweden.

The characteristic features of CO₂ adsorbed on Pt(111) include low energy intramolecular vibration (80 meV) and a probable free rotation of the adsorbed molecule. This system is suitable for studies of the role of internal degrees of freedom on the sticking process. We have carried out measurements of nondissociative sticking coefficient, S₀, of CO₂ on the Pt(111) surface as a function of incident kinetic energy, E₀ (60 meV < E₀ < 1.2 eV) at different incident angles. The observed features include a high initial sticking coefficient of 0.6 at 60 meV which declines sharply with increasing incident energy to S₀ = 0.2 at 0.4 eV followed by a slow decline towards a value of S₀ ~ 0.1 at higher measured energy independent of the incident angle. Such a behavior, unexpected for a physisorption system (the binding energy of CO₂ on Pt(111) is ~ 0.2 eV) suggests an important role of the intramolecular degrees of freedom in the sticking process. Simple modeling based on a soft cube model shows that the main features of the observed behavior may be rationalized in terms of rotationally mediated sticking, while the effect of the intramolecular vibrations is negligible. In addition, a more realistic modeling of the energy transfer to phonons via rotations will be presented based on classical trajectory studies of a rotating rigid molecule scattered from a slab of substrate atoms.

*Permanent address: Institute of Interphase Interactions, POB 1146, St. Petersburg 194291, Russia.

3:00 pm SS1-TuA4 Investigations of the Adsorption and Desorption Dynamics of D₂ at Si Surfaces, Kurt W. Kalaisinski, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, D-14195 Berlin, Germany.

Adsorption and desorption in the hydrogen/Si system have been investigated by means of supersonic molecular beam and laser techniques. The dependence of the molecular D₂ sticking coefficient, S, on surface temperature, T₀, and nozzle temperature, Tₚ₀, has been measured. The sticking coefficient increases with increasing T₀ and increasing Tₚ₀. This directly demonstrates for the first time that hydrogen adsorption on Si is activated. The increase in the effective sticking coefficient with T₀ shows that surface excitations play an important role in the reaction dynamics. An attempt is made to formulate a unified description of the reaction dynamics. The role of surface configurations in the dynamics and their interpretation within a Marcus-like theory will be discussed.

3:20 pm SS1-TuA5 Probing Reactive Deposition and Surface Dynamics Using In Situ, Real-Time Emission Microscopy, Martin E. Kordesch, Ohio University, Athens, OH 45701.

Emission microscopy is one of the oldest forms of electron microscopy. The images obtained are direct maps of the surface electron yield under illumination by various sources, including light from UV to soft x-rays, low energy electrons, ions, energetic and field emission. The image composition is a complex combination of sample topography, surface electron yield, and microscope design. Ultraviolet and soft x-ray Photoelectron Emission Microscopy (PEEM) images of surface processes during chemical vapor deposition and atomic layer epitaxy of diamond show a number of reaction phenomena that illustrate the problems suitable for observation with the emission microscope: nucleation of adsorbed layers, adsorbate controlled diffusion from the bulk, pattern formation, melting and the evolution of topographical features during "reverse growth". Neutral impact images of layers deposited using a seeded molecular beam will be presented, as well as examples of surface dynamics on Mo and Diamond surfaces.

INVITED

Work supported by NATO and the ONR-BMDO.

4:00 pm SS1-TuA7 Simulation of Complex Reaction/Desorption Kinetics Using a Simple Stochastic Method, F. A. Houle and W. D. Hinsberg, IBM Research Division, Almaden Research Center, San Jose, CA 95120.

We describe a computational approach which allows realistic, quantitative models of temperature programmed desorption kinetics to be devised from experimental data. It is based on the exact stochastic solution to the master equation described by Bunker and Gillespie, modified to simulate reactions under the influence of arbitrary time-temperature profiles. It is well-suited to modelling many types of adsorbate systems, including those with attractive and repulsive interadsorbate interactions, and those which undergo phase transitions and chemical reactions in competition with desorption. By combining stochastic simulations with experiment, it is possible to access the wealth of information on surface kinetics and instantaneous coverages con-
Nucleation and Growth: Metals
Moderator: D. M. Zehner, Oak Ridge National Laboratories.

4:40 pm SS1-TuA9 Interactions Between Adsorbed Molecules, D. C. Skelton, D.-H. Wei, S. D. Kevea, Department of Physics, University of Oregon, Eugene, OR 97403.

The energetic interactions between adsorbed atoms and molecules govern a diverse array of finite coverage surface phenomena ranging from formation of ordered overlayers to growth of thin films to determination of reaction pathways. Several groups have combined adsorption and desorption isotherms with quasiequilibrium analysis to probe the interaction potential between identical adsorbed molecules. We will report experiments which extend these efforts to coadsorption of CO with NO or NH3 on Pt(111) and Ni(111). Using time-resolved EELS coupled to pulsed molecular beam dosing, we have measured the low coverage desorption rate constant of NO, CO as a function of CO coverage. We thus can isolate the CO-Ni lateral interaction energies from the simultaneous complexicity of NO-Ni interactions. We find that some systems exhibit kinetic compensation characterized by compensation temperatures as high as ~350K, while other systems do not exhibit significant compensation. Prior results from single-adsorbate systems imply a substantially attractive lateral potential energy well in the former but not the latter type of system. We compare this to the lateral potential energy of the currently-popular transfer matrix approach to quantify this conclusion. While the interactions are statistically coupled, we find a direct and nearly linear relationship between the magnitude of the attractive lateral well and the compensation temperature. We discuss these interactions in terms of residual gas phase potential energy surfaces and through-later-surface interactions which arise from screening of the adsorbed molecules.

5:00 pm SS1-TuA10 A New Technique to Measure Surface Diffusion with the STM, M. L. Lozano, M. C. Tringides, Department of Physics and Astronomy, Iowa State University—USDOE,* Ames Laboratory, Ames, IA 50011.

We present preliminary results on a new technique using the STM to characterize surface diffusion. The time dependence of the tunneling current is analyzed through its spectral density. The spectra of clean, stepped Si(111) shows no temperature dependence. If 0.3 ML of oxygen is dosed onto the surface, broadening of the spectra with temperature is observed, indicating adatom mobility. The data agree with theoretical work which indicates that the power spectrum of the tunneling current when condensate moves on the surface takes the form \( \omega^2 \sim \ln(V/D) \cdot f^{-2.6} \), which is also shown with Monte Carlo simulations that this form is characteristic of single-diffusion processes. When single-diffusion on the surface and on the tip is simulated a different power spectrum dependence is observed, so most likely only diffusion on the Si(111) surface is probed. These measurements demonstrate that the STM can be used in “real-time” to measure diffusion coefficients on surfaces.

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1 Sametski and Kornyshev, in press.
2 Ames Laboratory is operated for U.S. Department of Energy by Iowa State University under Contract No. W-7405-ENG-82.

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SURFACE SCIENCE
Room A201 - Session SS2-TuA


We have observed mass flow on the Au(111) surface at room temperature using time-lapse sequences of STM images. In these sequences, layered nanoscale nonequilibrium structures decay in size and highly curved monatomic step edges become less curved [1]. This behavior suggests mass flow driven by curvature and mediated by adatom movement both across the terrace and along step edges. Further, this behavior is strongly dependent on conditions at the surface; for instance, the mass flow can be greatly enhanced by the presence of adsorbates.

Based on earlier works [2], we have developed a macroscopic, thermodynamic theory of mass flow driven by step edge curvature which is consistent with the temporal behavior of the observed decay. This theory suggests that adatom attachment and detachment at step edges is the controlling step in the mass flow. Ongoing Monte Carlo solid-on-solid simulations have successfully reproduced consequences of step edge curvature as predicted by the macroscopic theory. These simulations are being used to probe the connection between the thermodynamic and atomistic explanations for the experimentally observed behavior.


This work supported by F49620-93-1-0504 and the Cornell Materials Science Center (NSF-DMR-921654).

2:20 pm SS2-TuA2 Apparent Giant Applation of Small 2D Ag Islands on Ag(100), P. Bedrossian, B. Poelsema, G. Rosenfeld, L. Jorritsma, N. N. Lopikin, and G. Comsa, IGV-Forschungszentrum Julich, Germany. Permanant Addresses: 1Lawrence Livermore National Laboratory, Livermore, CA; 2University of Twente, Enschede, The Netherlands.

Rocking curves acquired with Thermal Energy He-Atom Scattering (TEAS) indicate a giant contraction of small, homoeptaxial, 2D Ag islands on Ag(100) normalized to the lattice constant of Ag(100) vanishing at room temperature, for 1/2 ML coverage. In contrast, high resolution SPA-LEED measurements reveal no corresponding normal relaxation of atoms within the islands. The apparent discrepancy between the LEED and TEAS data can be reconciled by attributing the anomaly observed in TEAS to a smoothing of the electronic corrugation (Smoluchowski effect), which is probed by the He atoms for small, densely-spaced islands. The dependence of the magnitude of the effect on growth temperature indicates that it is related to the size and separation of the Ag islands and is most pronounced for a high density of small islands, as would occur with 1/2 ML coverage at low temperatures. At 180K, SPA-LEED data indicate that these islands typically consist of 20 to 40 atoms.
P.B. and NNL were supported by the Alexander von Humboldt Foundation. PB was also supported by Lawrence Livermore National Laboratory, under the auspices of the US Dept. of Energy, under Contract W-7405-Eng-48.

2:40 pm SS2-TuA3 A RHEED Specular Diffraction Spot Intensity Study of Ag(111), Homoeptaxia, K. Roos, K. Stanley, C. Papageorgopoulos, M. C. Tringides, Bradley U.; Iowa State U.; U. of Ioannina, Greece.

The homoeptaxial growth of Ag on Ag(111) was studied as a function of substrate temperature and the flux rate using RHEED. The decay in the intensity of the specularly diffracted beam was observed. This allows probing the film morphology at low coverages (θ < 0.2), necessary to test the scaling theory of nucleation. Surprisingly, the intensity as a function of coverage, I(θ), did not noticeably depend on the flux rate, but varied significantly with temperature. Flux rates between 1/30 and 1/300 monolayers/sec and temperatures from 150 to 300K were used.

Our results are not consistent with the temperature and flux independent I(θ) predicted by the kinematic approximation which is based...
solely on the interference of the reflections from different levels. If instead, the step density is assumed to be dominantly responsible for the intensity decay, an activation energy and growth exponents x, y for the island density, N = (F/D)^y, can be extracted. The results do not agree with simple nucleation theory which for irreversible growth predicts the island density to have the same dependence on temperature as it does on the flux rate. Results from Monte Carlo simulations will also be presented supporting the scaling of the step density vs. flux and temperature in model systems.


3:00 pm SS2-TuA4 Enhanced 2-Dimensional Growth of Cu on Cu(100) by Seeding, Anna K. Swan and John F. Wendelken, Oak Ridge National Laboratory,* Oak Ridge, TN 37831-5071.

Typical growth experiments are performed at temperatures and flux rates far from equilibrium, where kinetic effects determine the growth behavior. Therefore, the diffusion rate and deposition rate determine the island density distribution in the submonolayer regime. For layer-by-layer growth to occur, atoms landing on top of islands have to be transported to the lower terrace before nucleation takes place on the top layer. This interlayer mass transport depends on both the "step down" activation barrier and the island density. In this work, the role of the island density and the step down barrier are examined independently with high-angular resolution low-energy electron diffraction. At a given temperature, the surface is seeded at a high flux, \( F_i \), producing a high-island density with an average island separation, \( S_i \). Growth is then continued at a lower rate, \( F_k \), which on the unseeded surface would produce a larger average island separation, \( S_k \). The degree of 2-dimensional growth of the first layer is determined by monitoring the specular intensity in out-of-phase condition as a function of coverage. An increase of the ratio of the intrinsic length scales, \( R = S_i / S_k \), enhances the 2-dimensional growth. Using the same length scales at different temperatures, the effect of the step down barrier is examined. The work is performed in a temperature region where the critical nucleation size is a dimer³ and where the islands retain a compact square shape.

2. H. Dür et al., submitted.

3:20 pm SS2-TuA5 Kinetic Roughening in a Multilayer Nucleation and Growth Model for M/M(100) Homoepitaxy, M. C. Bartell and J. W. Evans, Iowa State University, Ames, IA 50011.

We analyze kinetic roughening in a realistic model for metal/metal(100) homoepitaxial thin film growth, where nucleation and growth of near square islands occurs with critical size \( i = 1 \) in each layer, and where a key additional parameter is the Schwoebel barrier for downward interlayer diffusive transport. This model applies to, e.g., Fe/Cu(100) homoepitaxy, where it has been shown to reproduce full size and separation distributions observed for submonolayer growth between 250 and 500K². We obtain and elucidate the experimentally observed crossover from a split diffraction profile for ultra-thin films to the non-split scaled form of Yang et al.² for thicker films. We also examine the effective exponent, \( \beta \), describing roughening of the film. At least for lower temperatures, we argue that modeling and interpretation of roughness and \( \beta \)-values requires realistic treatment of nonuniversal features of the growth process such as crystal geometry, deposition dynamics, and island separation and structure, and illustrate this point with the example of Cu/Cu(100). Control of interface roughness through modulation of deposition flux or substrate temperature is also explored.

4This work was supported by NSF Grant CHE-9317660.

3:40 pm SS2-TuA6 First-Principles Calculations of Transition-Metal Surface Stress, Peter J. Feibelman, Sandia National Laboratories, Albuquerque, NM 87185-0344.

Despite their fundamental role in crystal growth morphology, absolute surface stress are not yet measured directly. Values are extracted from surface phonon spectra, via use of empirical elastic models.¹ To provide a reliable foundation for intuition and to help improve surface-phonon analyses, I have developed a novel first-principles method to compute surface stress, and applied it to several Group VIII metal crystal faces. For the anisotropic Pd(110) surface, I obtain (001) and (110) tensile stresses of 0.10 and 0.16 eV/\( \AA^2 \). The (001) stress is expected to be smaller because surface relaxation reduces the tension in the first to second layer bonds. This, however, disagrees with the ordering of the results for Ni(110)² based on a central-force model.² In that case, analysis of surface phonon data yields the counter-intuitive result that the stress in the (001) direction is larger by a factor of two than in the (110) direction. *Work supported by the U.S. Department of Energy under Contract DE-AC04-94AL85000.


4:00 pm SS2-TuA7 Strain Induced Alloying of Immiscible Metals in Thin Film Systems, J. L. Stevens and R. O. Hwang, Sandia National Laboratories, Livermore, CA 94550.

The influence of stress induced by lattice mismatch in thin film superlattice systems is known to influence the interface structure greatly. In multi-component overlayer systems, the different stresses acting on the film constituents alter their mutual interactions and can lead to drastic departures from their bulk phase diagrams. We have investigated these effects in the growth and structure of the thin film systems of Ag/Cu [1] and Ag/Cu on Ru(0001) using scanning tunneling microscopy. Due to the opposite signs of the lattice mismatch of the respective film components, mixing of the overlayer metals is found despite their bulk immiscibilities. New two-dimensional alloys of specific stoichiometry are stabilized. By studying the details of the morphology and composition of the resulting films, the driving mechanism for this behavior can be attributed to strain relief. Furthermore, domain formation of the various phases is observed which can also be understood in terms of strain relief. Such effects are expected to play a crucial role in the formation of interfaces in superlattices and could also lead to the formation of novel materials. Supported by DOE Contract No. DE-AC04-94AL85000.


4:20 pm SS2-TuA8 Surface Alloying and Deallowing of Au On Ni(110) and Ni(111) Studied by STM and RBS, F. Besenbacher, L. Plath Nielsen, J. Stensgaard, and E. Laugesen, Center for Atomicscale Materials Physics and Institute of Physics and Astronomy, Aarhus University, DK 8000 Aarhus C, Denmark.

The growth of Au on Ni(110) and Ni(111) have been studied at room temperature by in-situ Scanning Tunneling Microscopy (STM) and Rutherford Backscattering-Spectroscopy (RBS).

On Ni(110) we observe that incoming Au atoms are replacing surface Ni atoms up to Au coverage of 0.4 ML, i.e. forming a surface Au-Ni alloy, and that the hereby squeezed out Ni atoms agglomerate into Ni islands on the surface. Increasing the Au coverage above 0.4 ML causes an abrupt phase transition from two to three-dimensional growth through the formation of [001] directed Au chains. From a detail analysis of the amount of Au incorporated in the Au chains it can be concluded that the phase transition involves a deallowing mechanism where Au atoms are 'popping' out of the alloyed surface and nucleated into Au chains leaving behind Ni vacancies. Correlated with this concentration induced deallowing we observe a reversed Ni-mass transport from the Ni islands back into the leftover vacancies.

STM results of the growth of Au on Ni(111) reveals that even this closed packed surface shows the formation of Au-Ni surface alloy but only observed at elevated temperatures indicating that this is an activated process.

The formation of an Au/Ni surface alloys on both Ni(110) and Ni(111) and the concentration induced deallowing on Ni(110) will be discussed and supported by total energy calculations within the effective medium theory.

4:40 pm SS2-TuA9 Using STM to Understand Diffraction Oscillations: Fe Growth on Cu(100), David D. Chambliss, (IBM Research Division, Almaden Research Center, 650 Harry Road, San Jose, CA 95120 USA) and Kevin E. Johnson (Department of Chemistry, Goucher College, Baltimore, MD 21204 USA).

Intensity oscillations in surface-sensitive electron diffraction (RHEED and MEED) are widely used to analyze epitaxial growth, so
an understanding of the origin of intensity variations is needed. Direct comparisons of RHEED with scanning tunneling microscopy (STM) results have verified the qualitative correspondence between surface roughness and decreased intensity.1,2 We examine quantitatively the relationship between experimental diffraction intensity variations for room-temperature growth of Fe on Cu(100) and topography measured with the scanning tunneling microscope (STM). Variations in spot profiles and integrated intensity are predicted from STM data using Fourier transform methods to implement kinematical formulations. When the detector size is explicitly included, the agreement is good with the experimental MEED results of Thomassen et al.3 The agreement demonstrates the similarity of samples prepared in the different laboratories and the applicability of kinematical analysis to MEED oscillations. A significant conclusion is that the detector defines a characteristic length of the measurement that must be included in a quantitative interpretation of MEED data.


5:00 pm NSI-TuA10 Long Jumps in the Surface Diffusion of Adatoms: W, Ni, and Pd on W(211), D. Cowell Senft and Gert Ehrlich, University of Illinois at Urbana-Champaign.

Molecular dynamics simulations indicate that atomic jumps longer than a nearest-neighbor spacing may become important at high temperatures, but there has been little experimental evidence that this is a significant effect in the actual diffusion of isolated metal adatoms. Field ion microscopic observations have now been made for W, Pd, and Ni adatoms on W(211), for which migration is one-dimensional. The measurements reveal that for these atoms, diffusion obeys a simple Arrhenius relation, with entirely normal prefactors. Analyses of the displacement distribution function, to determine contributions from different types of jumps, show that W moves entirely by single jumps. Ni atoms make occasional double jumps, even at the lowest temperature examined, but the number of long jumps is not much affected by temperature. For Pd, the number of long jumps increases rapidly with temperature, and double as well as triple jumps contribute significantly—the first experimental indication of long jumps in one-dimensional diffusion.

*Nanotechnology: Supported by the National Science Foundation under Grant DMR 91-01429.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A209 - Session NS1-TuA
Nanostructure Properties: Chemical and Electrochemical
Moderator: M. Hara, RIKEN, Japan, R. J. Hamers, University of Wisconsin.

2:00 pm NSI-TuA1 Electron Tunneling through Water Clusters in Electrochemical STM, S. M. Lindsey, T. W. Jing, J. Pan, D. Lamper, A. Vaughn, J. P. Lewis and O. F. Sankey, Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85287.

When measured in electrochemically clean conditions, tunnel current versus distance curves in aqueous electrolytes yield decay constants bigger than 1 Å⁻¹. Interpreted in terms of a simple barrier, this corresponds to several eV. In non-bonding electrolytes, however, the decay constant depends strongly upon the voltage applied between the tip and the substrate, the bond without the water itself. The effect is independent of the conductance at which the measurement is initiated and also independent of the electrochemical potential of the substrate. We will show that, operated at G3 resistance, the gap probably contains at least 4 water molecules and the current is dominated by the electron tunneling of a chain of hydrogen bonded molecules. When the electric field in the tunnel gap is high enough to 'freeze' the cluster, the strain imposed by movement of the tip results in stretching of hydrogen bonds with a consequent rapid decay of current. If, on the other hand, rotational transitions are permitted (i.e., at low fields) the strain may be taken up by rotations of molecules about the hydrogen bonds, resulting in a slow decay of current with tip movement.

These results are consistent with a simple 1-electron calculation of the gap resistance and suggest that the ability of the STM to image wide-band-gap materials stems from (a) the fact that the decay of the wavefunction at energies well into the gap is not as rapid in a small cluster as it is in the bulk and (b) electronic interactions, even in a weakly bonded material like water, lead to significant residual conductance even far from the band edges.

This work was supported by grants 1R21 HG00818-01A1 from the National Institutes of Health and N00014-90-J-1455 from the Office of Naval Research.

2:20 pm NSI-TuA2 The Nature of Mixed Adlayers on Platinum Single Crystal Electrodes Probed by In-Situ STM and Infrared Spectroscopy, L. Villegas and M. J. Wexler, Department of Chemistry, Purdue University, West Lafayette, Indiana 47907-1393, USA.

The adsorption of carbon monoxide with metal adatoms, e.g. bismuth or lead, is of particular interest in studies on the catalytic activity of platinum electrode surfaces towards the electrooxidation of numerous organic molecules. The unprecedented real-space structural information obtained from scanning tunneling microscopy (STM) images of electro-chemical adlayers when combined with infrared reflection-absorption spectroscopy can provide further insight on coadsorbate interactions for in-situ electrode surfaces. CO containing adlayers are excellent model systems for these studies due to the sensitivity of the CO IR absorption frequency to the binding site on platinum-group transition metals. Consequently, CO binding site as well as coverage changes induced by the metallic coadsorbates can be readily deduced from IR spectra. STM images obtained from Pt(111) electrode surfaces preadsorbed with irreversibly adsorbed Bi and immersed in CO saturated acid solutions indicate that most of the surface (70%) is covered by an atomically intermixed adlayer of Bi and CO. This finding is consistent with the corresponding in-situ IR data. The rest of the surface, however, displays structures previously observed for pure CO adlayers on Pt(111). An interesting observation is that, although no complete segregation of Bi and CO domains occurs, regions free of Bi atoms are clearly distinguishable. The catalytic implication of such findings on this and other Pt single-crystal surfaces will also be noted.

2:40 pm NSI-TuA3 Scanning Probe Microscopy of Organic Materials, J. E. Frommer, IBM Almaden Research Center, 650 Harry Road, San Jose, CA 95120.

The requirements for successfully imaging organic materials in SPM experiments are at once the same and different from other classes of materials. This is because overall, the class of organic materials is characterized by diversity. Bonding within organic involves covalent, van der Waals, ionic, electrostatic, and hydrogen bonds, in many combinations. Therefore, the strongest point to bring to the imaging of organics is an awareness of the multiple modes of behavior that they can take on. This awareness is necessary in sample preparation, setting scanning parameters, and in interpreting results.

This overview will cover a broad range of organic materials, from molecular crystals to weakly anchored liquids. Intermolecular interactions will be discussed, and how the scanning instruments probe these forces. The organic samples will be discussed in the context of the broad range of environments in which they are found, e.g., friction, adhesion, membranes, manipulation.

INVESTED

3:20 pm NSI-TuA5 Molecular Scale Topographic Features and Electronic Structure in Thin Organic Films Observable by STM, R. C. White, B. Birchfield, and E. N. Schulman, Columbia University, New York, NY 10027 USA.

We have characterized the structural and electronic properties of a polymer thin film on the molecular scale using a scanning tunneling microscope (STM). The polymer studied was a polyimide (PMDA-ODA) commonly employed as an insulating layer in microelectronics. Results of this work lead to an understanding of the mechanism by which poorly conducting organic materials can be imaged by STM. Prolonged exposure to ultra-high vacuum (UHV) was employed to eliminate extraneous current sources such as mobile ions, and allow the true electronic conductivity to persist in sufficiently thin films to image the tunneling current. This resulted in an increased topographic resolution, and we have obtained the first STM images of an un-modified...
polymide surface, verified by measurement of contact between tip and film. In addition, we have distinguished nanometer scale, spatially localized regions of the polymer film which exhibit strong, voltage dependent switching between discrete conductance levels, similar to a localized two state switching previously observed in thin SiO₂ films. This process in SiO₂ is due to simple electron trapping and de-trapping, while that observed in the organic film displays more complex kinetics similar to multilevel fluctuators in metal micro bridges. Thus the conductance mechanism in the organic film is related to trapping and de-trapping, in localized "defects" which result from intermolecular intercalation. This mechanism of electron current supply through the film dominates and thus no intramolecular electronic states are accessible by STM.

3:40 pm NS1-TuA6 Atomic Ordering Within AlGaAs Alloys Studied Using Cross-Sectional Scanning Tunneling Microscopy, A. R. Smith and C. K. Shih, University of Texas at Austin, Austin, TX 78712, Y. C. Shih and B. G. Streetman, Microelectronics Research Center and Department of Electrical Engineering, University of Texas, Austin, TX 78712.

We have investigated the composition fluctuations of AlGaAs alloys with atomic resolution using X-STM. Clean cross-sectional (110) surfaces are prepared in UHV by cleaving samples in situ. Experiments are performed on p-type samples with an aluminum content of 30%. Within the AlGaAs regions, we have observed localized alloy fluctuations similar to those seen by Johnson et al. in which Al-rich regions were observed which align preferentially along the [110] and [112] directions. The alloy concentration modulation wavelength in this case is on the order of 40-50 Å. In addition to this long wavelength modulation in the alloy composition, we have also observed alternating rows of Al/GaAs within the AlGaAs alloy region which lie along the [110] and [112] directions. This observation indicates that there are long range orderings consisting of alternating AlAs/GaAs (111) planes in the bulk. The influence of the growth conditions on the observed alloy ordering will be discussed. Further investigations are currently underway to identify the atomic mechanisms behind these ordered features.

4:00 pm NS1-TuA7 Characterization of the Gallium Vacancy on GaAs (110), G. Lengel, R. Wilkins, M. Weiner, J. Gryko, and R. E. Allen, Texas A&M University, College Station, TX 77843.

Using scanning tunneling microscopy and spectroscopy, together with tight-binding molecular dynamics simulations, we previously characterized the geometry and electronic structure of the As vacancy at the (110) surface of GaAs [1]. Here we apply the same techniques to the Ga vacancy. There is an interesting symmetry between the two defects: In both cases, the neighboring atoms (Ga neighbors for the As vacancy, As neighbors for the Ga vacancy) are observed to relax upward relative to their positions on the pristine surface. Also, there are three defect levels (per spin) associated with the three new dangling bonds around the vacancy site. For an As vacancy, these can be viewed as pulled down from the Ga-derived conduction band; and for a Ga vacancy, as pushed up from the As-derived valence band. The energies of the calculated levels indicates that the As vacancy is preferred on p-type GaAs, and the Ga vacancy on n-type, in agreement with the observations. The levels also indicate that the As vacancy (on p-GaAs) should be positively charged, and the Ga vacancy (on n-type) negatively charged; these results are again consistent with the experiments. There is just one principal difference between the As and Ga surface vacancies: The Ga neighbors around an As vacancy are found to spontaneously relax upward in the molecular dynamics simulations. The As neighbors around a Ga vacancy, however, relax upward only in the presence of a modest electric field. We therefore attribute the latter relaxation to an interaction between the surface atoms and STM tip. The As neighbors can be pulled upward only easily because they have only two remaining bonds. According to both the simulations and the measurements, these atoms do not shift laterally to recombine.

5:00 pm NS1-TuA10 Direct Observations of Electric Field Gradients Near Field Emission Cathode Arrays, Y. Liang, D. Bonnell, The University of Pennsylvania, Philadelphia, PA 19104, W. Goodhue, D. Ruthman, C. Bozler, Lincoln Lab, Boston, MA 02476.

The variation of electric field gradients above arrays of field emission cathodes is characterized with atomic force microscopy (AFM) using both imaging techniques and force-displacement measurements. Cathodes have submicron diameters at the base, decreasing to nanometer scale tips. Effects of AFM tip convolution will be considered explicitly. The dependence of the spatial distribution of field gradient on applied bias and height above the array is compared to theoretical predictions. Results confirm a parabolic relationship between sample bias and field gradient and power law dependence on height.


We have previously reported a new approach for selective electrosless metal deposition utilizing chemisorbed ligated colloidal palladium films in conjunction with a tin-free, aqueous based Pd(II) catalyst. This process provides several advantages over traditional methods including patternability, a well defined interaction between catalyst and surface through ligation, and elimination of the acceleration step during processing. From recent STM images it appears that formation of the latent image in the monolayer is not the limiting factor in the resolution of this process. Rather the particle size of the electrosless metal is large enough to degrade the patterned resolution. Using a combination of AFM, SEM, and TEM we have examined how variations in the metal bath and catalyst effect the particle size of the electrosless metal. It was found that the size and distribution of catalyst particle sizes depends on the age of the catalyst solution, the deposition time, the formulation, and the nature of the ligating surface. Shifting the size distribution of bound catalyst particles to smaller sizes results in a dramatic reduction in the metal particle size. Results are discussed in terms of current electrosless metallization theory. Selective metallization with the new catalyst was used to produce features with linewidths to approximately 100 nm by high voltage e-beam lithography and features ranging from 35-100 nm using STM exposure. In both cases pattern transfer was accomplished using reactive ion etching to depths of 200-500 nm in Si.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room A207 – Session NS2-TuA

Nanomechanics and Nanotribology: I
Moderator: S. Cohen, Weizman Institute of Science.

2:00 pm NS2-TuA1 Atomic Modelling of Friction and the Interaction Between AFM Tips and Ionic Surfaces, A. L. Shluger, A. L. Rohl and D. H. Guy, The Royal Institution of Great Britain,
Interaction of nano-asperities with ionic surfaces is relevant to the micro-mechanisms of friction, nanolithography, modification of surfaces, and AFM imaging of their structure. Periodic surface structures of several alkali halides, alkali-earth fluorides and oxides have been recently obtained by Scanning Force and Friction Microscopy. Several images of steps, corners and point defects have also been obtained in our and other groups. Correct interpretation of these experiments requires theoretical studies of the basic mechanisms of the interaction of realistic nano-structures, such as surface roughness and AFM tips, with ionic surfaces. In this study we focus on the chemical nature and the interaction of different nano-asperities with ionic surfaces at long and short distances. Determination of adhesion and scanning force microscopy in particular, consider the interaction of three different types of asperities, composed of protonated SiO₂ and MgO and consisting of up to 66 atoms, with the perfect, stepped and kinked (001) surfaces of LiF, NaCl and CaO by quantum-chemical and atomistic simulation techniques. The associated surface distortions caused by this interaction were investigated. Friction and AFM images of steps, kinks and point defects at different tip-surface separations and forces were analyzed both experimentally and theoretically. From the AFM perspective, it is demonstrated that the optimal tip-surface distance for 'atomic resolution' is about 3–5 Å.

2:20 pm NS2-TuA2 Molecular Dynamics Simulations of Metal-Oxide Surfaces and Interfaces, F. H. Stelzr and J. W. Mintmire, Naval Research Laboratory, Washington, DC 20375, USA.

The metal-oxide and metal-metal oxide interfaces are ubiquitous in the adhesion of ceramics and metals. A range of technologically important materials applications are critically dependent on the properties of these interfaces: ceramic-metal composite materials, conductive metals on metal-oxide substrates in electronic devices, and anticorrosive surface oxide layers on structural metals are just a few representative examples. Atomistic-scale simulations of metal-oxide materials can be important in understanding and predicting the effect of the interface region on materials properties. Our primary objective is to model the current research effort is to simulate the atomistic-scale dynamics and energetics of technologically important metal/metal-oxide interfaces. We need an empirical method that allows the local cation valence to account for the local environment which includes the Coulombic electrostatic interaction among the anions and cations. We have investigated the use of an electronegativity-based model for direct calculation of charge transfer in metal-oxide systems and incorporated the resulting electrostatic potential into an overall model potential. We present a brief outline of our approach for modeling the electrostatic component of the potential energy of ionic systems, and how standard empirical potential techniques can be effectively merged with this approach. We then discuss our results for α-alumina, looking at both surface relaxation of low-index faces of alumina, and strain effects on an alumina/aluminum interface. Our results indicate that this approach will be able to provide physically realistic empirical potentials for future simulations on mixed metal/metal-oxide systems.

2:40 pm NS2-TuA3 Electron-Ion Dynamics: A New Technique for Simulating Both Electronic Transitions and Atomic Motion in Molecules and Materials, Roland E. Allen, Texas A&M University, College Station, TX 77843.

In 1964, A. Rahman introduced the technique of molecular dynamics, in which the motion of atoms and molecules is computed from classical potentials. Twenty years later, we introduced quantum molecular dynamics, in which the forces on atoms are computed from the electronic structure, via the Hellmann-Feynman theorem. We have now developed a much more general technique, electron-ion dynamics, which permits electronic transitions and atomic motion to be treated simultaneously. The key features of this technique are the following: (1) Use of the interaction picture in solving the time-dependent Schrödinger equation 〈dψ/dt = H₂ψ. (2) A generalized Hellmann-Feynman theorem which is valid for excited states, and even during the course of electronic transitions. (3) An Einstein detailed-balance argument for treating spontaneous emission of radiation. (Absorption and stimulated emission processes are also included directly, through the radiation field.) (4) A time-dependent self-consistent-field approach, which is equivalent to the random phase approximation. This technique is not limited to one-photon and one-photon processes; it can be applied to arbitrarily strong radiation fields and arbitrarily violent ion motion. It is suitable for both ab initio and semiempirical calculations, but our first illustrative calculations are for excited state chemistry in tight-binding models.

This work was supported by the Office of Naval Research and the Robert A. Welch Foundation.

3:00 PM NS2-TuA4 Scanning Near-Field Acoustic Microscopy: Nanoscale Material Properties, N. A. Burnham, A. J. Kulk and G. Grenaud, Department of Physics, Swiss Federal Institute of Technology, 1015 Lausanne, Switzerland.

A Scanning Near-field Acoustic Microscope (SNAM) is an atomic force microscope that detects transmitted high-frequency acoustic waves. By exciting the cantilever above its resonant frequency, its effective stiffness increases. Hence, normal-force images at low loads and SNAM images may be simultaneously acquired, eliminating the limiting compromise between force resolution and cantilever rigidity. Greater detail is evident in the SNAM images than in the normal-force images. The SNAM contrast is related to the local surface forces, adhesion and elastic properties.

The principles of this new technique will be explained, and a few examples of SNAM images of graphite and a test structure with varying elastic properties will be shown and compared to the usual low-frequency force modulation images. Data interpretation will be emphasized, and in particular the relative roles of adhesion and elastic modulus will be examined.

3:20 pm NS2-TuA5 Tribology with the Atomic Force Microscope, D. Frank Oglethorpe, Jun Hu, Xu-dong Xiao, C. Morant, and M. Salmoner, Materials Science Division, Lawrence Berkeley Laboratory, Berkeley, California 94720.

The atomic force microscope is a powerful tool to study adhesion and friction in single asperity contacts under well-defined conditions. Using the AFM, we have obtained atomic lattice resolution images in both topographic and lateral force modes on a variety of ordered surfaces, including mica, NaCl(001), LiF(001), KMMnF₆(001), Au(111) films, self-assembled organic monolayers on Au(111), and more recently, the potential has been measured as a function of load on these surfaces in ambient laboratory conditions. Additional friction vs. load measurements on some of these surfaces have been carried out under controlled humidity, in liquids and in ultra-high vacuum. Absolute calibration of normal and lateral forces in the AFM is difficult, so comparative measurements have been made with the same tip on different surfaces. We have also carried out a systematic study of friction on mica to see if this surface is a suitable '‘AFM friction standard’’. Results of friction measurements for different mica samples, for different scan directions, for different layers, as a function of humidity and in different environments will be presented. Issues involved in quantitative tribological measurements with the AFM will be discussed. INVITED

4:00 pm NS2-TuA7 Effect of Contact Area on Nanoindentation Curves Obtained Using the Atomic Force Microscope, Charles F. Daper, David M. Schaefer, Richard J. Colton, and Steven M. Hues, Materials Science Division, Lawrence Berkeley Laboratory, Berkeley, California 94720.

Nanoindentation mechanical properties may be quantitatively measured by nanoin indentation using the atomic force microscope (AFM). However, a lack of understanding of the exact size and nature of the contact area between the tip and surface severely hampers the extraction of absolute material property values from raw nanoindentation data. Commonly used AFM tips (diamond, W, Si,N) have ill-defined nanoscale tip apex geometries and consequently greatly complicated absolute mechanical property measurements. The contact of spherical indentors, however, are well-described by Hertzian contact mechanics. In order to study the effect of contact area in greater detail, we have imprinted a variety of materials with spheres of decreasing diameter (100 nm to nanometer range) and have studied the effect of the contact area changes on the nanoindentation curves and derived mechanical properties.


We have used Interfacial Force Microscopy (IFM) to quantitatively measure the mechanical properties of various surfaces of single-crystal Au in order to study the effect of crystal-face and probe-tip radius when the tip size is nanoscale, i.e., 50-500 nm. The etched W probes are analyzed by SEM profiling and the Au surfaces are passivated with self-assembling monolayers of alkanethiol molecules. The IFM tech-
tique is used because it is quantitative, mechanically stable, has a zero-compliance sensor and can readily accept characteristic probes. Contact mechanics permit a determination of the elastic modulus and the shear-stress threshold for plastic deformation. Measured values for the elastic constants follow the expected trend for the various faces. However, unexpected deviations in the measured elastic constants, the shear-stress thresholds and the plastic-strain thresholds are observed for truly nanoscale deformations. We discuss these findings in terms of a non-continuum model which takes account of the tip size relative to the substrate lattice constant.

*Work performed at Sandia National Laboratory supported by the US Department of Energy under Contract DE-AC04-94AL85000.

4:40 pm **NS2-TuA9** Surface Potential Control of Adhesion in an Atomic Force Microscope, J. Larsen, T. W. Jing, D. Lampner, and S. M. Lindsay, Dept. Physics and Astronomy, Arizona State University, Tempe, AZ 85287, Y. Q. Li, Dept. of Chemistry, Texas A&M University, College Station, TX 77843, N. J. Tao, Dept. of Physics, Florida International University, University Park Campus, Miami, FL 33199.

Control of adhesion is possible by varying the potential of the conducting surfaces of both a probe and a substrate in an electrochemical cell for an atomic force microscope. We have studied adhesion between a gold-coated force sensing probe and a gold (111) 23 x 83 surface under potential control in perchlorate electrolytes. The maximum adhesion, at the potential for zero charge (PZC), is in reasonable agreement with predictions based on the known surface energy of gold. The adhesion is reduced to zero by relatively small excursions of the surface potential each side of the PZC.

This work was supported by grants 1R21 HG000818-01A1 from the National Institutes of Health and N00014-90-J-1455 from the Office of Naval Research.

5:00 pm **NS2-TuA10** Local Modification of Mechanical Properties of Poly(styrene)-Polyethyleneoxide Blend Surfaces, H.-Y. Nie, M. Motomatsu*, W. Mizutani, and H. Tokumoto, JRCAT-NAIR and JRCAT-ATP*, Higashi 1-1-4, Tsukuba, Ibaraki 305, Japan.

We report the modification of local mechanical properties of poly(styrene) (PS)-polyethyleneoxide (PEO) blend surfaces with atomic force microscopy (AFM) and force modulation microscopy (FMM). In the course of the experiments, the slopes of force curves are quite informative to examine mechanical properties.

We used PS-PEO blend films annealed at 90°C for 1 day, exhibiting phase separations. In this particular case, it is possible to distinguish PS and PEO from topography because only PEO crystallizes. Force curves on PS and PEO measured before and after FMM scanning changed remarkably. Before scanning, the slope of force curve on PEO was smaller than that on PS, indicating PEO is softer than PS. After scanning, however, the slope of force curve on PEO became close to or larger than that on PS, depending on stiffness of cantilevers and scanning rates. This can be explained as follows: during FMM scanning, applied forces resulted in plastic deformation of PEO, causing PEO stiffer than PS. We point out possibilities of controlling elastic properties and of recognizing molecules of polymers with this technique.


**APPLIED SURFACE SCIENCE/SURFACE SCIENCE**

Room A101 – Session ASSS-TuA

Electrochemistry and Liquid/Solid Interfaces

Moderator: G. D. Davis, Martin Marietta Laboratories.

2:00 pm **ASSS-TuA1** Evidence of Specific and Non-Specific Adsorption of ClO on Ag(110), A. Kratkiopoulos and E. M. Stase, Dept. of Chem. Eng., BF-10, University of Washington, Seattle, WA 98195.

The adsorption of ClO and of HClO with H2O on Ag(110) has been studied with HRLEELS as a model of anion adsorption at the solid-liquid interface. The vibrational spectrum of adsorbed ClO shows losses at 470, 615, 905, 1020 and 1230 cm^-1, indicating a lowering of symmetry due to adsorption. A model for the adsorption is proposed. When cosorbed with H2O, the vibrational spectra resembles that of the fully solvated ClO-, suggesting the formation of a nonspecifically adsorbed anion in UHV. The vibrational structure of the water shows signs of the “structure breaking” effect of ClO in aqueous solution.

Cosorption of ClO and H2O produces adsorbed perchlorate and hydronium, as indicated by the (Cl-O) at 1260 cm^-1 and of H2O at 1080 cm^-1. Additional H2O partially leaches the perchlorate, allowing it as completely as for cosorption of H2O and ClO, due to preferential hydration of H2O. The symmetric bend of H2O shifts to 1130 cm^-1, suggesting a more aqueous environment. The combination of adsorbed and hydrated ClO with hydrated H2O represents a model of specific and non-specific adsorption. Reversing the dosing order results in formation of aqueous H2O at the acid/water interface. However, some molecular acid remains undissociated in the top layer, making this an inadequate model of non-specific adsorption.

2:20 pm **ASSS-TuA2** Surface Chemistry of Water and Hydrogen on Single Crystal Ni3Al(111), Yip-Wah Chung and Wen-Jui Chia, Northwestern University, Dept. of Materials Sci. & Eng., Evanston, IL 60208.

Intermetallic compounds such as Ni3Al have low tensile ductility when tested in normal room temperature and ambient. This is believed to be due to hydrogen embrittlement, hydrogen being produced by the interaction of water vapor in air and fresh metal surfaces exposed during tensile testing. In this talk, we present results of the interaction of water and hydrogen using TPD, Auger and X-ray photoelectron spectroscopy. The interaction depends strongly on surface orientation. On the (111) surface, water adsorbs molecularly. On the (533) stepped surface and the (100) surface, water dissociates to produce hydrogen.

Water desorption was not observed up to 700K. AES/XPS showed that adsorbed oxygen or hydroxyl remains on the surface. The absence of water evaporation during desorption suggests complete dissociation of water into hydrogen and oxygen atoms on the (533) and (100) surface. The mechanism of environmental embrittlement of intermetallic compounds by water dissociation to produce hydrogen atoms is demonstrated.

2:40 pm **ASSS-TuA3** In Situ Study of 304 Stainless Steel’s Passive Layer Exposed To HCl Acid Using A Scanning Tunneling Microscope, J. M. Galligan, T. J. McKrell, University of Connecticut, Storrs, CT, 06268.

The nature of 304 stainless steel’s passive layer and how it is altered as a function of exposure time in HCl acid has been studied using a Scanning Tunneling Microscope (STM). This device offers the distinct advantage of in situ studies, on a microscopic scale, of the electrical character of the surface in the presence of a corrosive environment. In the present paper we present evidence which shows how the surface structure of 304 stainless steel is altered by hydrochloric acid (HCl) while the surface is probed with an STM. In particular, the in situ current (I) and bias voltage (V) curves were measured as a function of etchant exposure time. The important region, concerning passivation, is the extent and the slope of the curve near where the current is zero. This region corresponds to corrosion resistance and shows a low or zero slope near where the current is equal to zero. The research method establishes a quantitative measure of the extent of protection and the kinetics of the removal of the passive layer. The findings of this research are correlated with nanoscale STM images of the 304 stainless steel’s surface.

3:00 pm **ASSS-TuA4** Tunnel Channels and Imaging Mechanisms in STM: W-, Ptl-, and Ag-Interfaces in Electrolytes, G. Reppuhn, Inst. of Physical Chemistry, University, CH-3000 Bern 9, Switzerland; J. Halbritter, KfK, IMF 1, Postfach 3640; 76021 Karlsruhe, Germany.

In scanning tunneling microscopy (STM) operation in ultrahigh vacuum and, usually also in UHV, tips and substrates are covered by adsorbed, hydrides or oxides. These coatings are difficult to identify and to control. In an electrolytic environment receipts exists for cleaning tip and substrate. In addition, the potential electrolyte-tip or electrolyte-substrate changes the interface in a well defined way.

STM charge transfer is far from being simple, like direct tunneling in vacuum given by exp (-2d/2m*e*v). With the gap width d and φ as tunnel barrier height. For example, the observed tunnel currents
often decay very slowly with \( \phi = \omega t \) by \( n \) intermediate states simulating a low tunnel barrier \( \phi_{0} = \omega t + 1 \). Thus, reported \( \phi_{0} \) values are often not a barrier height but resemble intermediate state tunneling via a new barrier distance and effective the technique distance to \( d_{0} = \omega / \omega \). We identify the imaging processes and tunnel channels via \( n \) intermediate states by voltage and distance spectroscopy and separated up to 7 intermediate tunneling states in the: oxide-, hydroxide-, and double oxide layers. We report on the intermediate states in this electrolyte interface to dipol resonances of the ordered, solid-like HOH-OH dipol layer changing with interface potential. The thickness and dipol moment of the interface layer are obtained from distance and potential dependencies of \( \phi_{0} \). The importance of the dipol layer interfaces on deposition and growth is discussed.

3:20 pm ASSS-TuA5 In-situ Measurement of Thickness Changes with Electrolytic SPM, R. Nyffenegger, H. Siegenthaler, P. H"{a}ring* and R. K"{o}tz*, Institut f"{u}r Anorganische, Analytische und Physikalische Chemie, Universit"{a}t Bern, 3012 Bern, Switzerland; *Paul Scherrer Institut, 5232 Villingen, Switzerland.

Scanning Probe Methods (SPM) not only allow a mapping of surface topography in real space but also enable the measurement of thickness changes of the sample. Assuming a constant distance between tip and sample during the entire experiment, the tip will follow the thickness change of the sample and the corresponding driving signal of the piezo can be recorded and assigned to this thickness change.

This method has been used to measure in-situ thickness changes of electrochemically formed surface films in electrolytic environment, that can be charged or discharged (e.g. conducting or redox polymers). This change in the thickness state often leads to a surface modification, e.g. formation of hydroxide or oxide layers or swelling/shrinking of polymers phases induced by ion exchange. Typical examples include nickel hydroxide films and polyaniline layers. Both are investigated in view of their practical application in novel types of batteries.

The obtained results can be assigned to the corresponding thickness changes. Experimental and principal considerations suggest a lower detection limit of ca. 1 nm. Below this limit, alterations of relevant imaging parameters (e.g. inverse depth length for STM, stiffness of the sample in the case of AFM) may affect the thickness measurements and falsify the experiment.

3:40 pm ASSS-TuA6 Electrochemical Etching of Si(111) Surfaces in Fluoride Solutions Studied by STM and AES, R. Houbert* and U. Memmert, Abt. Oberfl"{a}chenchemie, Universit"{a}t Ulm, D-89069 Ulm, Germany, *Fr 10.2, (Experimentalphysik), Universit"{a}t des Saarlandes, D-66041 Saarbr"{u}cken, Germany.

The electrochemical etching of Si(111) surfaces in conc. NH\(_{4}\)F solution in dependence of etch potential and etch time was investigated by I-V curves, AES and STM. The samples were etched under full potentiostatic control in an ex-situ electrochemical cell and transferred afterwards into a UHV system for the AES and STM measurements. Within the potential regime considered in this study we were able to characterize five distinct potential regions in the I-V curves: A cathodic range, the open circuit potential (OCP) and three anodic ranges, separated each other by two current maxima. During etching at cathodic potentials and the OCP no oxides are formed on the surfaces. STM imaging shows that an isotropic etch mechanism leads to very smooth and regular surfaces. Etching at anodic potentials yields much rougher surfaces.

To the formation of large, several Si(111) bilayer deep holes, the AES measurements display still no oxide below the first current peak, while in the second anodic region between the two peaks app. one bilayer is oxidized. The oxide thickness increases drastically for potentials above the second current peak. The anisotropic etching for cathode potentials and the OCP is explained by the local binding situation for different surface sites, while for anodic potentials long-range effects have to be considered. Anisodically in NH\(_{4}\)F etched samples show quite different topographies than samples which were treated in HF/ethanol solution. While in NH\(_{4}\)F solution the holes on the surface stay rather shallow, for the HF/ethanol solution the formation of very narrow and deep pores was found. This difference is explained by the different ability of these solutions to attack Si even without an external anodic current applied.

The solid-solution interface is the location where many environmentally important reactions take place. Results from two different approaches for examining the effects of surface structure and chemistry on surface reactivity in solution will be described. Crystals which readily dissolve and grow in ground water have significant potential for storing or releasing contamination. Atomic Force Microscopy (AFM) has been used to observe and measure the dissolution of the cleavage face of CaCO\(_{3}\) in aqueous solutions. Dissolution occurs at steps and the step velocities differ by a factor of three (or more) depending upon the step structure. Impurities, such as Mn, can alter the rate of dissolution and change the structure of a dissolving pit. Current efforts are to relate a model of relative surface reactivities for different sites to the observed dissolution patterns and rates. Although defects are known to strongly influence the reactivity of oxide surfaces, the defects observed or created in vacuum, may not be those present in solution. The reaction of water with "undamaged" and defected surfaces of TiO\(_{2}\) (110) has also been examined by transferring a specimen prepared in vacuum to solution and then returning the specimen to the spectrometer. These results are compared to the effects of low-pressure (p < 10\(^{-6}\) Torr) and higher pressure (10\(^{-6}\) < p < 10\(^{-5}\) Torr) exposure to water vapor.

4:40 pm ASSS-TuA9 AFM and STM Studies of Oxide Formation, Deposition, and Dissolution on Copper Single Crystal Surfaces, Andrew A. Gewirth, John R. LaGraff, and Mauhtui Ge, Department of Chemistry, University of Illinois, 505 S. Mathews Avenue, Urbana, IL 61801 USA.

We used the AFM to image the initial stages of oxidation on Cu(110), Cu(111), and Cu(111) electrode surfaces in dilute acid solutions. On Cu(110) images obtained in H\(_{2}\)SO\(_{4}\) and HClO\(_{4}\) solutions with a pH near 2.5 revealed oxide monolayers consisting of (100) and (001) chains. These chains represent the initial oxide layer growing on the Cu surface. After completion of this monolayer, the images revealed formation of n x 1 structures where n = 1, 2, 3, 4, which were attributed to precursor structures which develop prior to bulk oxide development. In solutions where Cl\(^{-}\) was deliberately added, a different overlay exhibiting a c(2 x 2) lattice structure was observed. On the Cu(100) face we observed that a(2 x 2) or R45\(^{\circ}\) adlattice is formed on this material at potentials negative of the rest potential. The oxide monolayer is amorphous on Cu(111), but becomes ordered at neutral pH values.

The oxide monolayer controls the initial nucleation of Cu deposits onto Cu surfaces. We found in addition that deposition could be preferentially enhanced on Cu by scanning the AFM tip over areas on the surface. The tip removes the oxide layer and forms nucleation sites on the Cu substrate.

5:00 pm ASSS-TuA10 In-situ Observation of Hg Amalgamation Formation and Stripping on Au using Atomic Force Microscopy, L. A. Nagahara*, X. Yang**, K. Tomani**, Y. Wei*, K. Hashimoto**, and T. Fujishima*, Department of Applied Chemistry, Faculty of Engineering, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113, Japan; *National Laboratory of Molecular & Biomolecular Electronics, Southeast University, Nanjing 210018, China.

We have used atomic force microscopy (AFM) to investigate in-situ the initial stages of Hg amalgamation on Au films as well as Hg stripping from the amalgamated film. The Au(111) on a glass substrate used in this study were prepared via vacuum-deposition at elevated substrate temperatures. In the case of Hg deposition, amalgamation occurred in the form of small islands. Further deposition resulted in the growth of these islands as well as the formation of new islands. Adjacent to these islands, a portion of the Au film is observed to be preferentially removed at pinhole sites and resulted in larger and deeper pits. Highly ordered regions also showed dissolution as evident by the increase in mono-atomic stepped terraces. When the Hg was removed from the amalgamated film, the entire surface became noticeably rougher indicating that the amalgamation process has occurred even in well-ordered regions. The AFM results and the mechanism for amalgam formation and stripping will be presented.

4:00 pm ASSS-TuA7 Effects of Structure and Chemistry on Surface Reactivity in Solution, D. R. Bauer, A. S. Lea, Y. Liang, Li-Qiong Wang, and M. H. Engelhard, Pacific Northwest Laboratory, Richland, WA 99352.
PLASMA SCIENCE
Room A109 – Session PS-TuA

Advanced Plasma Reactors
Moderator: T. D. Mantei, University of Cincinnati.

2:00 pm PS-TuA1 Helicon Plasma Source Excited by Flat Spiral Coil
* J. E. Stevens, Princeton Plasma Physics Laboratory, M. J. Sosnowski, Princeton University, Department of Chemical Engineering, and J. L. Cecchi, University of New Mexico, Department of Chemical and Nuclear Engineering.

We have operated and characterized a new helicon plasma source with an end-launch antenna configuration. Power at 13.56 MHz is coupled via a four-turn flat spiral coil into an m = 0 helicon mode with the application of a weak (B > 5G) axial magnetic field. Plasma parameters were measured with Langmuir probes and the structure and absorption of the helicon wave fields were determined with magnetic induction probes. Plasma densities of 10³-10⁴ cm⁻³ are produced in Ar for pressures in the 1-50 mTorr range with a 5-60 G magnetic field. RF power absorption occurs primarily via collisionless Landau and ECR damping for pressure below ~10 mTorr. Above this pressure, collisional damping dominates. Absorption is strongest for magnetic fields less than ~20 G. This source requires no separate source chamber, and thus combines the compactness of flat coil inductively coupled sources, with the advantages of remote plasma generation found in wave-supported sources. Additionally, by not constraining the helicon parallel wavelength, the source can be easily optimized for a variety of operating conditions.

* Work supported by the Semiconductor Research Corporation.


As part of research efforts to develop sub 0.35 μm design rule devices, we are etching in a dual reaction chamber cluster tool made by Lucas Labs of Sunnyvale, California. The reaction chambers are geometrically, electrically, and mechanically the same; thus, allowing direct side-by-side comparisons of different plasma sources while excluding platform dependent effects. In this study, we present the results from the continuation of our effort to compare commercially available, low pressure, high density sources. The first two sources were a multipole ECR manufactured by Wavemat of Plymouth, Michigan and an rf-inductively coupled helicon manufactured by Lucas Labs. The third source is a 12° diameter helical resonator manufactured by Prototech Research of Tempe, Arizona.

We first perform a statistical evaluation of blanket polysilicon etching rate and etching rate uniformity as a function of source power, reactor pressure (2-5m Torr) and rf-bias (25-75 W). Based on these results, operating conditions for optimum uniformity for each source are determined. The effect of process parameters on polysilicon profiles is then evaluated and compared. The etching process in each source is optimized to give vertical profiles and the optimized recipe is compared. Finally, we also present the radial Langmuir probe measurements of the plasma properties above the wafer corresponding to the etching conditions explored above.

2:40 pm PS-TuA3 Ponderomotive Effects in Helicon Plasmas, R. Brown, J. Gilland, N. Herzhkowitz, R. Brown, University of Wisconsin, Department of Nuclear Engineering and Engineering Physics, 1500 Johnson Drive, Madison, WI 53706-1687.

Computer modeling of Nagoya Type III, Dual-Half-Turn and Spiral antenna systems used to excite helicon waves, has been carried out with the code ANTENA. The code considers a cylindrical plasma with an arbitrary radial density and temperature profile and realistic antenna structures, in a uniform magnetic field. With the exception of end conditions, it provides a good model of the experimental configuration in our laboratory helicon plasma in which the helicon wavelength λ < l, the plasma length. We report investigations of the role of radial ponderomotive force in providing or reducing MHD stability of the helicon plasma with emphasis on the roles of the m = ± 1 and m = ± 1 azimuthal modes. Comparisons are presented to experimental data.

Supported by NSF Grant No. ECS-9120326.

3:00 pm PS-TuA4 A High Etch Rate, Highly Selective, Sub-half-micron Contact Etch Process in a Low Pressure, High Density TCP Oxide Etcher, P. K. Gadgil, A. Asthana, and I. Moses, Lam Research Corporation, Fremont, California 94538.

A low pressure, high density transformer coupled plasma (TCP) source with independent RF bias control has been characterized for etching sub-half-micron contact holes in silicon dioxide on 200 mm substrates. The dependence of etch selectivities of silicon dioxide to photore sist, silicon nitrides, and silicon on different process parameters has been investigated. This paper discusses the effects of feed gas chemistry, the ratio of inductive-to-capacitive nature of plasma generation, and TCP power on the etch selectivities and the tradeoffs therein. The etch selectivity of oxide-to-nitride increases with more inductive plasma generation, and with more polymerizing chemistries. A high aspect ratio (6:1) contact etch process with high oxide etch rate (>8000 Å/min), high oxide-to-nitride selectivity (>5), high oxide-to-tantalum selectivity (>20), and low RIE lag in 0.35 μm contacts has been achieved by optimizing the process parameters. The results indicate that the TCP technology is a promising candidate for future sub-half-micron contact and via etch applications.


A planar rf inductively-coupled plasma etching system has been constructed. The plasma is confined by an external multipole magnetic bucket made of strong Nd-Fe-B magnets. The system is equipped with a helium-backside-cooled electrostatic wafer chuck and a loadlock chamber. A single-sided Langmuir probe was used to measure electron temperature and electron velocity distribution function (Evdf) radially and axially for both Ar and N₂ plasmas with rf powers up to 1 kW at lower pressures (~10 mTorr). By rotating the single-sided probe we find that the Evdf's are noticeably anisotropic. The values of the plasma potential, which were highly uniform radially, were independent of the probe orientation, but those of the floating potential depended strongly on it. The radial uniformity of rf floating potentials was confirmed by measurements using an emissive probe. The Langmuir probe measurements also showed non-Maxwellian Evdf's for both Ar and N₂ plasmas. Because of the multipole confinement, the plasma densities are quite uniform axially and the radial uniformity improves as the axial distance from the quartz window increases. The system was further characterized using a capacitive probe and an electromagnetic (B-dot) probe.

*This work was supported by the National Science Foundation under Grant ECD-8721545.


Radio-frequency (rf) transformer coupled plasma (TCP) provides for a better way to generate spatially confined high density gas discharge plasmas for microelectronics processing. Commercial processing equipment using this technique is currently available; however, it is limited in size to 8-inch diameter by problems with plasma uniformity and window erosion. We have developed a new TCP design that allows for larger dimensions with good uniformity. The current equipment requires an expensive thick quartz plate vacuum window that separates the rf inductor and the plasma. The thick quartz plate reduces coupling efficiency and causes uniformity problems. Our device incorporates the rf inductor and dielectric window inside of the vacuum chamber, allowing for a thin layer of quartz or other dielectric material, as required by the process, to be placed between the coil and the excited plasma. This thin dielectric layer, as well as a newly designed rf tuning network, allow the plasma to be scaled to larger devices while maintaining good uniformity. Plasma ashing results for an 8-inch device show 2% uniformity across 6-inch wafers. Characterization of the plasma generated by a larger scale device, as well as ashing results for the device, will be presented.

This work supported by NSF grants DDM-9108531 and DDM-9111697.

4:00 pm PS-TuA7 Large Volume Electron Cyclotron Resonance Plasma Generation by Use of Slotted Antenna Microwave Applicator, J. Engemann, M. Schott, F. Werner, and D. Korsc. The generation of large area, low pressure (below 10⁻³ mbar) mi-
The etching process in ULSI fabrication demands high plasma uniformity. For high precision and uniform processing, ions must strike the wafer uniformly at a 90° angle. Achieving this requires a highly controllable plasma generation region, low-temperature ion generation, and readily diffusible electrons. It is therefore important to generate plasma at low pressure and without a magnetic field. Using a cavity resonator and slot antenna, we recently developed a compact microwave plasma reactor with virtually no magnetic field adjacent to the wafer. We have termed it the Microwave Slot Antenna (MSA) reactor. Microwaves are introduced to the cavity resonator in order to enhance the electric field. The microwaves are radiated through the slot antenna to the processing chamber. The plasma generation region can be controlled by adjusting the slot antenna. Uniform control of the plasma and RF bias distribution is readily achievable because the electrons near the wafer can easily diffuse.

We numerically simulated the distribution of the electron temperature and density and the microwave EM field. We applied the MSA reactor to the Si etching process. After achieving uniformity of <5% and an undercut of <20 nm, we confirmed that the MSA reactor is efficient for the ULSI process.

VACUUM TECHNOLOGY
Room A102 – Session VT-TuA

 Moderator: H. F. Dylla, CEBAF.

2:00 pm VT-TuA1 Water: Its Measurement and Control in Vacuum, S. A. Tison and J. P. Looney, National Institute of Standards and Technology, Gaithersburg, MD 20899.

The presence of water vapor in vacuum systems has many deleterious effects which can influence the yield of vacuum manufacturing processes, the ultimate pressure of vacuum systems, and the stability of vacuum instrumentation. Water vapor can enter the vacuum system from the process gas, can adsorb from interior surfaces or diffuse through polymer seals. Although the presence of water vapor is well known, the ability to measure its abundance with conventional gaging is not well understood. Additionally although many models exist for predicting water adsorption and desorption from surfaces, their reliability or applicability is not well established. Some recent advances in water measurement, such as using optical techniques to measure water vapor abundance, have promise to advance the understanding of water dynamics in vacuum systems. A workshop dedicated to examining the current state of water vapor measurement and control in vacuum was held at the National Institute of Standards and Technology in May of 1994. This paper will summarize the contents of the workshop and review recent progress in water measurement and control.

INVITED

2:40 pm VT-TuA3 Thin Film on Stainless Steel for Extremely High Vacuum Material, K. Saitoh, S. Inayoshi, Y. Ikeda, Y. Yang, and S. Tsukahara, Tsukuba Institute for Super Materials, ULVAC JAPAN, Ltd., Tsukuba, JAPAN 300-26.

We noticed thin films on stainless steel (TiN/SS) as one of the most promising candidate materials for extremely high vacuum (XHV) use. All the TiN films were vapor deposited at the optimum conditions...
determined previously and vacuum properties of the TiN/SS samples have been compared with those of the electrolytically polished SS samples.

The outgassing of the short samples of 20 mm² × 0.5 mm was measured by thermal desorption spectroscopy heating up to 450°C in ultrahigh vacuum. The total outgassing of TiN/SS was always lower than that of SS when they were measured in the same conditions.

Hydrogen permeation of membrane samples were measured varying the temperature and the thickness of TiN films. The TiN film works as the barrier to hydrogen desorption from stainless steel surface. The TiN/SS membranes showed about two orders less permeation rate than use of SS at 50°C and the difference of them is the higher at the lower temperature.

As the final experiments the chamber samples of 254 mm φ × 380 mm were examined. The temperature depending outgassing rates of the chambers after the bake-out at 150°C for 2 days were measured by the throughput method with special consideration to completely exclude the outgassing of the gauges and spectrometer tubes under measurements. At the ultimate upstream pressures of about 1 × 10⁻⁵ Pa and the downstream pressure at XHV range the residual gas component was only hydrogen and the specific outgassing rate of the TiN/SS and the SS chambers were 6 × 10⁻¹² (6 × 10⁻¹¹) (for hydrogen) and 9.7 × 10⁻¹⁰ Pa × m⁻¹ for nitrogen at 21.4°C, respectively. The result proves that the TiN films on stainless steel are applicable to wall material for XHV apparatus.

3:00 pm VT-Tu4A SIMS Analysis for Aluminum Surfaces Treated by Glow Discharge Cleaning, J. R. Chen*, G. Y. Hioung, Y. C. Lin*, Synchrotron Radiation Research Center, No. 1 R&D Road VI, Hsinchu Science-Based Industrial Park, Hsinchu, Taiwan 300, R.O.C., W. H. Lee and C. C. Nee, Institute of Nuclear Science, National Tsing-Hua University, Hsinchu, Taiwan 300, R.O.C.

The glow discharge cleaning treatments on aluminum surfaces were studied by the SIMS method. The cleaning effects of DC and RF glow discharge with different gases were compared. The elements or compounds of the contamination or reaction products were identified from the analyses of mass and energy distribution of the secondary ions. The secondary ion energy distribution was similar for the elements with the same group. The results and discussions are to be described in this paper.

a) also at Institute of Nuclear Science, National Tsing-Hua University.
b) also at Department of Physics, National Tsing-Hua University.


The effectiveness of glow discharge cleaning (GDC) as a means of decreasing the pump-down time of vacuum systems was measured. The measured outgassing rate of an electropolished stainless steel surface was measured. The measured outgassing rate of an electropolished stainless steel surface was measured. The test specimen was exposed to glow discharges of uniform gases (He, H₂/Ar, H₂/Ar, H₂/Ar) for a standard one hour of venting to ambient air. It is shown that the GDC is most effective in reduction of outgassing rate of the GDC. GDC is a new way to achieve a low outgassing rate and substantially the pump-down time of a factor of 13. For given glow discharge conditions, the outgassing reduction factor is linearly proportional to the glow discharge power. The reduction of post-GDC outgassing is a result of enhanced desorption of H₂, CO, and CO₂ by GDC as observed by vacuum analysis during the discharge. The GDC results are consistent with our previously proposed diffusion models for water outgassing from metal surfaces.

*This work supported by U.S. DOE Contract No. DE-AC05-
84ER40150.


The Advanced Photon Source (APS), currently under construction at Argonne National Laboratory, will be one of the most powerful synchrotron x-ray photon sources in the world. The beamline sections where the x-ray will be transported will contain high thermal load ultrahigh vacuum (UHV) compatible components, such as photon shutters, fixed masks, slits, and bellows. These components are made of OFHC copper (for photon shutters), GidCo® Al₂O₃ dispersion strengthened copper (for fixed masks and photon shutter face-plates), TZM high temperature molybdenum alloy (for slits), machinable tungsten (for safety shutters) and 304 or 316 stainless steels (for UHV component joints and bellows). Before any of these components can be assembled, they must be cleaned to remove surface contaminants so that the ultrahigh vacuum necessary for successful operation can be achieved. Fabrication of the components will involve cleaning processes, such as explosions, chemical etching, brazing and soldering, et al. Removal of surface oxides of joining materials is crucial to obtain secure bonding. Although the vacuum and mechanical properties of the cleaned surface are of prime importance, environmental and safety factors are also of great concern. Both conventional and environmentally safe, commercially available cleaning detergents were investigated, and the surface cleanliness was evaluated using X-ray photoelectron spectroscopy (XPS). Several important results have been learned in this study. Some of the commercial cleaning detergents were effective as conventional etching solutions for the materials. The effect of the material microstructure on the surface cleanliness and XPS analysis results will also be discussed in detail.


Machinable tungsten and molybdenum alloys are extensively used as safety shutters and optical slits at the Advanced Photon Source (APS) front ends. These materials may present a vacuum problem because of their porosity. Also, environmentally acceptable cleaning procedures have been used for these materials. We have chosen specially heat-treated machinable tungsten with a density of 18 g/cm³ for safety shutters and TZM (a molybdenum alloy containing 0.5% titanium and ~0.1% zirconium) for optical slits. Thermal outgassing tests have been performed for a machinable tungsten set with a total surface area of 4500 cm² and a 2.8 × 4.6 × 32.6 cm² piece of TZM. A cleaning procedure using alkaline detergent ultrasonic washes and vacuum furnace baking was used prior to the outgassing measurements. Outgasing rates 10 h after initial pump down at room temperature are 1.6 × 10⁻¹⁰ Torr l/s cm² for machinable tungsten and 8.0 × 10⁻¹⁰ Torr l/s cm² for TZM. The outgassing rates 24 h after an in situ bake at 160°C for two days decreased to 2.2 × 10⁻¹⁰ Torr l/s cm² for machinable tungsten and 1.2 × 10⁻¹⁰ Torr l/s cm² for TZM, respectively. Optical studies confirmed that the TZM sample is more porous than the machinable tungsten sample. Further studies of a denser TZM sample have been planned to study the effect of porosity on the outgassing rate.


4:40 pm VT-TuA8 Photon Stimulated Desorption (PSD) Measurements of Extruded Copper and of Welded Copper Beam Chambers for the PEP II Asymmetric B-Factor, C. L. Foerster, C. Lanni, Brookhaven National Laboratory, Upton, NY 11973, and C. Perkins, Stanford Linear Accelerator Center, Stanford, CA 94305 and M. Caudill, Lawrence Livermore National Laboratory Livermore, CA 94551.

PEP II is being built as a higher luminosity electron-positron collider, with asymmetric beams of 9 GeV and 3.1 GeV, having maximum currents of 3.0 A. Based on our previous work on the NSLS VUV beamline U10B, a copper was selected for construction of UVH beam chambers and absorbers to minimize the pressure rise from synchrotron radiation during operation. An extruded beam chamber and a welded beam chamber were fabricated from the selected copper for PSD measurements on NSLS X-ray beamline X28A. The chambers were exposed to white light with a critical energy of 5 keV, both direct and through a 0.010 inch thick Beryllium filter. Each chamber was exposed to a dose of approximately 10¹⁴ photons per meter at an incidence angle of 30 mrad, after argon glow conditioning and a 150°C vacuum bake. Desorption yields for H₂, CO, CO₂, and CH₂ are reported as a function of accumulated photon flux, critical energy, and chamber preparation. The results are compared with the previous work on beamline U10B and with those of other published work for copper.

*Work performed under the auspices of the U.S. Department of Energy, under contract DE-AC02-76CH00016.
Boron nitride based transition metal coatings are presently being considered as potential materials for coating applications requiring high hardness, high temperature and corrosion resistance. Ti-B-N layers have been produced by sputter deposition from a BN target onto which small Ti platelets have been positioned. By changing the number of platelets, the Ti-B-N composition has been varied and the films studied by AES and XPS. Quantification of the film composition by AES is discussed, considering the Ti L,M2,M3 and N KLL,L3 peak overlap. Changes in the Ti L,M2,M3 and B KLL,L3 peak regions with composition are examined and correlated with DOS calculations. Using XPS, shifts in the B 1s and N 1s peaks have been used to compare the phase composition with that predicted by the thermodynamic phase diagram. Correlations of XPS peak positions with the structural data show that films of highest hardness are obtained when a composition of approximately Ti:B:N = 4:6:6 is obtained, the phase composition being a combination of TiN and TiB2.

3:00 pm TFVM-TuA4 Electron Cyclotron Resonance Plasma Assisted Growth of Thin Carbon Nitride Films on Si(100): Film Composition and Structure, A. Bousetta, A. Bensoula, and M. Lu, Space Vacuum Epiaxy Center, University of Houston, Houston, Texas 77204-5505.

We report compositional and structural properties of thin carbon nitride films on Si(100) substrates at temperatures in the range of 100-650°C using e-beam evaporation of graphite assisted with ECR plasma generated nitrogen beam. The effects of a SiN buffer layer, a varying nitrogen gas flow into the ECR source, and the substrate temperature, on the C/N composition ratio, and C-N crystal structure were investigated using Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectroscopy (RBS), and Raman spectroscopy. From both RBS and XPS, the nitrogen concentration in the film was calculated in the range of 20-98% and varied directly with the nitrogen partial pressure and hardness in the ECR source. FTIR spectra from films with low nitrogen content exhibited a high visible to infrared-transmittance and were dominated by amine groups (NH2) with the presence of C-N stretching modes. Raman spectra however show no evidence of CN bonding and were characteristic of a graphitic carbon. In contrast, in high nitrogen content thin films, both the transmittance and NH2 absorption band decreased whereas the C-N absorption band increased. Raman spectra of these films show a well resolved peak at 1275 cm⁻¹ suggesting the formation of a C-N phase with predominately sp² bonding.


The recent announcement of the synthesis of C3N4 by plasma enhanced laser ablation has increased interest in this unique material. Carbon nitride may have several useful properties for wear and corrosion resistant coatings, electrical insulators, and optical coatings. We have produced carbon nitride coatings containing up to 40% nitrogen using ion assisted planar magnetron RF sputtering in a nitrogen atmosphere. Up to 2 μm thick coatings are produced on alumina, silicon, SiO2, and glass substrates using a graphite target. Films with greater than 95% transparency in the visible wavelength and greater than 98% reflectance at 1000°C. Compositional variabilites and oxygen presence were monitored with SIMS depth profiling and the Raman spectra are compared with those of other carbon and carbon nitride films.

3:40 pm TFVM-TuA6 Thermal Behavior of Carbon Nitride and TiN/NbN Superlattice Coatings, S. Lopez, M. S. Wong and W. D. Sprout, BRL Industrial Research Laboratory, Northwestern University, 1801 Maple Avenue, Evanston, Illinois 60201-3135, USA.

Hard coatings are commonly used today to enhance the life of numerous tools (drills, cutters) and wear parts (bearings). Carbon nitride (CNx) and TiN/NbN superlattice films are being evaluated for such tribological applications, and it was necessary to test their behavior under thermal treatment. The coatings were reactively sputtered on various substrates. Thermal annealing of both coatings were per-
formed at temperatures up to 1000°C either under high vacuum (10⁻⁷ Torr) or in air. Several properties of the coatings before and after annealing were evaluated including hardness (nanoindentation), surface profilometry (AFM), structure (XRD), and composition (AES).

In vacuum, the carbon nitride coatings lost part of their nitrogen as the annealing temperature was increased and a critical temperature for stability was found to be around 600°C. No change in composition and slight changes in layer structure were observed for the TiN/NbN superlattice films annealed at temperatures up to 800°C under vacuum for 2 hours.


Carbon films were deposited on Si substrates using mass selected C⁺ ions at deposition energies of 2 to 45 KeV and substrate temperatures from 25°C to 250°C. A recent publication described the growth mechanism of similar films deposited at lower ion energy (<2keV) and the dependence of their structure and properties on the deposition parameters. The present study investigates the growth mechanisms and properties at higher energies.

The main characterized features were: (i) morphology at the nanometer scale, by Atomic Force Microscopy (AFM) (ii) density, by Rutherford Backscattering Spectroscopy (RBS) in conjunction with profilometry (iii) chemical composition and depth profile by Auger Electron Spectroscopy (AES) and (iv) bonding configuration (sp²/sp³) by Electron Energy Loss Spectroscopy (EELS).

In the low deposition energy regime a smooth surface was an indication of internal subsurface growth with a higher sp³ component, while rougher surfaces were consistent with a higher sp² component. At higher C⁺ energies, films with relatively smooth surfaces evolved. The mechanisms (different from those at the low energy regime) affecting the films growth, surface morphology and bulk properties are discussed.


4:40 pm TFVM-TuA9 Thickness-Distribution Control of Large-Area DLC Films Formed by CH₃/H₂ Supermagnetron Plasma. H. Kinoshiita, S. Honda, and M. Hosoda, Shizuoka University, 3-5-1 Johoku, Hamamatsu 432, Japan.

Supermagnetron plasma was first applied to CVD with adjusting both rf phase difference (RFPD) and rf power ratio (13.56 MHz) whose powers were supplied to two parallel cathodes. In most experiments, stationary magnetic field of 45G was applied parallel to a lower cathode, on which a 5-in. Si wafer was set. Thickness uniformities of diamondlike carbon (DLC) films were evaluated as a function of rf phase difference. When RFPD was 0°, its uniformity became poor (±9%) and at about 180° it became excellent (±3%). Based on this result, we measured the upper cathode rf power (UPRF) dependence of its thickness distribution at the fixed conditions of RFPD of 180°, electrode spacing of 45mm, lower cathode rf power (LORF) of 400W, and gas pressure of 6 mTorr. When UPRF was set to 0W, linearly graded thickness-distribution was observed along perpendicular direction to magnetic field lines, which shows a typical magnetron mode. At UPRF of 100W, almost uniform distribution of ±2.9% was obtained, and at 200W inverse distribution to that of OW was observed, i.e. inverse magnetron mode. Extremely high uniformity of ±1.4% was observed at RFPD of 180° by rotating the magnetic field. Rather high hardness of about 1400 kg/mm² was obtained in this condition. From this experiment it was confirmed that, at RFPD of about 0° secondary electrons emitted from the upper cathode drift along it and generate plasma over the upper cathode. At about 180°, on the contrary, that emitted from the upper cathode drift toward the lower cathode and generate plasma over the lower cathode. 3


5:00 pm TFVM-TuA10 Structural Analysis of Hydrogenated Carbon Films Obtained by Reactive dc Magnetron Sputtering. S. Fujiwaki, M. Kitoh, Production Engineering Research Laboratory, Hitachi, Ltd., Totsuka-ku, Yokohama 244, Japan; H. Matsumoto, Y. Kokada, Data Storage & Retrieval Systems Division, Hitachi, Ltd., Kozu, Odawara 256, Japan.

Amorphous hydrogenated carbon films were prepared by reactive dc magnetron sputtering in argon plasma containing methane as the reactant gas. The films were characterized using various spectroscopic measurements such as Raman scattering, optical absorption spectroscopy in the ultraviolet (UV)—visible (VIS) region and infrared (IR) absorption spectroscopy.

The spectral analyses indicated that changes in Raman spectral intensity occurring with increasing methane gas content were determined to be caused by changes in optically resonant components in the films. Furthermore, significant correlation was seen between the relative intensity of IR peaks and wear durability. It can be concluded that the increase of CH₃ bonds at sp³-carbon cites is assumed to restrict the network of C-C connections and thus reduce the film’s wear durability. Also, the chain-like structure of olefinic compounds, was assumed to increase the wear durability at sp²-carbon cites. From this, the films formed at a methane gas content of about 20% were found to have the highest wear resistance.

ELECTRONIC MATERIALS
Room A108 – Session EM-TuA

Moderator: K. J. Bachmann, North Carolina State University.


Silicon-based Optoelectronics: Optical interconnections are attractive from the point of view their high performance. In this work we have developed technologies to fabricate optical waveguides consisting of SiO₂XNy core and SiO₂ cladding layers at low temperatures (300°C). The propagation loss for curved waveguides has also been simulated and compared with experimental results.

Remote plasma enhanced CVD was employed for the deposition of SiO₂Ny and SiO₂ films to reduce plasma-induced damage. The source gases were SiH₄, N₂O and NH₃. The refractive index of the SiO₂Ny core layer can be precisely controlled from 1.5 to 1.95 by changing the gas flow ratio. The propagation loss of the slab waveguides with SiO₂Ny core (470 nm) and SiO₂ cladding layers (800 nm) ranges from 0.93 to 1.54 dB/cm depending on the core index used.

Simulation of the loss for curved waveguides is necessary for design of optical interconnections. We have developed a simulator for the propagation loss in the optical waveguides by means of the
beam propagation method. The loss for curved waveguides increases drastically for radii of curvature shorter than 400 µm, which is in good agreement with experiment results from a simple test chip with integrated LEDs, Si-mirrors, curved waveguides and LED-based detectors.

In summary, the fundamental technologies to realize the optoelectronic integrated circuits have been developed. \textbf{INVITED}

\textbf{2:40 pm EM-TuA3 Silicon Optical Bench Waveguide Technology, Charles H. Henry, AT&T Bell Laboratories, Murray Hill, N.J. 07974.}

This technology is based on optical waveguides similar to those in conventional optical fibers by depositing thick films of doped silica on silicon substrates, primarily by low pressure chemical vapor deposition. The films are patterned by photolithography and etching into complex optical integrated circuits. Components include taps, splitters, star couplers, broad and narrow band multiplexers and low speed switches. Commercial application of optical integrated circuits require nearly perfect optical waveguides and very low loss, polarization independent control of the absolute refractive index and control over the optical phase difference between different paths. The SIoB waveguide technology has the potential to meet all of these needs. The waveguide fabrication, properties, components of current interest and opportunities for systems applications areas will be reviewed. \textbf{INVITED}

\textbf{3:20 pm EM-TuA5 Novel LSI Memories with Optical Interconnections, M. Koyanagi, Tohoku University, Sendai, Japan.}

The performance of the microprocessor has been significantly improved owing to the progress of LSI technology. However, even if the microprocessor performance is rapidly improved, the overall performance of the computer system is not always so rapidly improved because it is eventually limited by the data transfer speed or the data bandwidth of the buses. In particular, this bottleneck in the bus becomes more serious in the interchip processing computer system. We have been interested in employing optical interconnection as an intrachip or interchip interconnection to increase the data transfer speed among processors and memories in the parallel processing computer system. In this paper, we describe new LSI memories with optical interconnection such as Optical RAM bus (ORAM bus) memory. Optical RAM (ORAM) and Optical Coupled Common (OCC) memory which have been proposed to increase the data transfer speed and the data bandwidth by transferring data among many memories through optical interconnection. These memories have three-dimensional (3D) structure which consist of several 2D memory layers with LEDs and photodetectors or photodiodes. The memory layers are connected each other through many free-space optical interconnections in the vertical direction. The basic functions and performances of these new memories are evaluated using an optical-electrical circuit simulator. In addition, the test chips are fabricated to confirm their basic functions. \textbf{INVITED}

\textbf{4:00 pm EM-TuA7 Rare Earth Doped Silicon Light Emitters, J. Michel and L. C. Kimerling, Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139.}

Recent results on the improvement and understanding of light emission from the Si:Er system are reviewed. Erbium emits light at 1.54 µm, a wavelength ideal for optical communication. While most recent studies focused on co-doping with oxygen to optically activate erbium, fluorine codoped samples show two orders of magnitude efficiency improvement at low temperatures. Room temperature performance of Si:Er based edge emitting LED's, capable of direct waveguide coupling, will be shown and discussed in the context of system requirements for integrated optical interconnection. \textbf{INVITED}

\textbf{4:40 pm EM-TuA9 SiGe/Si Quantam Well Light Emitters, Yasuhiro Shirkai and Susumu Fukatsu, Research Center for Advanced Science and Technology, The University of Tokyo, Komaba, Meguro-ku, Tokyo 153, Japan.}

Highly luminous SiGe/Si heterostructures are now fabricated by various growth techniques such as molecular beam epitaxy (MBE), UHV/CVD, rapid thermal CVD and so on. Especially, gas source MBE where hydrogenated gases, SiH₄ and GeH₄, are used as molecular beam sources can provide extremely promising results for light emitting device application. Quantum wells (QWs) grown by this method show a clear quantum confinement effect as the well width is changed and their luminescence properties are well understood in terms of effective mass approximation. The temperature dependence reflects the band alignment at the heterostructures and carrier dynamics in multi-QWs provide strategies to realize light emitting devices. These results indicate that device properties of this system are predictable from the quantum mechanical consideration, suggesting high potentials in band engineering and device designing. Some examples of LEDs including room temperature operation are demonstrated and new concepts of optical devices exploiting the nature of this material system are proposed. \textbf{INVITED}

\textbf{MANUFACTURING SCIENCE AND TECHNOLOGY}

\textbf{Room A110 – Session MS-TuA}

\textbf{Advanced Manufacturing Equipment – A Moderator: M. Liehr, IBM TJ Watson Research Center.}

\textbf{2:00 pm MS-TuA1 The Semiconductor Equipment Industry: Applied Materials Role and Growth, Robert Z. Bachrach, Applied Materials, 3050 Bowers Ave., MS 1250, Santa Clara, CA 95054.}

Applied Materials celebrated its 25th anniversary in 1993 and in 1994 became the first Semiconductor Equipment Manufacturer to achieve revenue of one billion dollars per year. An overview will be presented of some of the factors and circumstances that have allowed Applied Materials to differentiate itself in the industry both technically and in business performance. Applied Materials now provides its customers with advanced manufacturing equipment for Etch, CVD, PVD, Implant, and HTF (High Temperature Films) for 6"/8" wafer processing. Applied Materials has focused on single wafer processing with common architecture multi-chamber mainframes: Precision 5000® Endura®, and CenturaM. A major thrust is to extend these capabilities to the next wafer size of 12" and 14" silicon which is expected to be manufacturing starting in 1998. \textbf{INVITED}

\textbf{2:40 pm MS-TuA3 Advances in Semiconductor Manufacturing Equipment, Evert van de Ven, Tom Bowman, Novellus Systems, San Jose, California.}

The Semiconductor Industry is maturing rapidly and with its requirements for manufacturing equipment have been changing from process capable hardware to turnkey production systems with guaranteed performance.

This paper will review the main issues critical to high volume manufacturing and how changes, historically and in the future, are affecting the equipment requirements and design approach. With these changes, the main focus on equipment is shifting to productivity cost/performance and production reproducibility. To determine performance to these requirements, new metrics, such as equipment productivity index, value added time, and sigma six processing are being added to defect density and cost of ownership. Using these and other criteria, the advantages and limitations of various equipment concepts will be compared and examples of successful implementation presented. Finally, an overview will be given of the challenges for next generation equipment. \textbf{INVITED}

\textbf{3:20 pm MS-TuA5 MESC Cluster Tools for Advanced Metalization, P. H. Ballentine, T. Omstead, M. Moslehi, CVC Products, Inc., Rochester, NY 14603.}

Cluster tools are playing an increasingly important role in semiconductor manufacturing. Individual process steps may be integrated into a single vacuum environment to reduce surface contamination and minimize operator handling. By using a modular approach to system design, individual process modules may be taken off line for service and burn in while the rest of the tool remains in production, thus reducing qualification time. From an equipment development point of view, process modules may be developed and characterized independent of the wafer transport platform and cluster control architecture. This provides a significant reduction in time-to-market for new process equipment. The adoption of the SEMI MESC (Modular Equipment Standards Committee) standards has provided additional benefits by allowing two or more equipment suppliers to integrate "best of breed" process modules into a single cluster tool. We have used our CVC Connexion platform to rapidly develop thin film deposition tools for both the data storage and semiconductor industries. Process modules for PVD, RTP, MOCVD, and soft etch have been developed in parallel and combined for use in production of thin film recording heads and development of advanced multilevel metallization schemes. Portions of the process modules have been based on tech-
of structures. All of these mechanisms contribute to gap filling capability of BPSG on high aspect ratio structures (>2.5:1) with small openings <0.17 μm at temperatures as low as 750°C.

4:00 pm MS-TuA7 The Development of the Fast Thermal Processor (FTP), Katsuya Ohtumura, Toshiba.

Furnace operations (oxidations, anneals and LPCVD) account for a large percentage of integrated circuit fabrication cycle time and short cycle time is necessary to minimize product development time and manufacturing capital investment. Therefore, further innovation is needed in the areas of furnace tooling and processing in order to improve ULSI fab productivity.

Today, high-temperature furnaces are carried out in either conventional furnaces or newer rapid thermal processors (RTPs). Furnaces have relatively slow ramp rates (3–10°C/minute) compared to RTPs (500–1000°C/minute), but furnaces can typically process 100–150 wafers at once while RTPs are usually single-side (single-wafer) processes. Also, there is a large body of knowledge related to conventional furnaces and RTP that has yet to be duplicated in RTTs. But because of the large difference operating regimes, it is difficult to transfer learning between the two types of equipment. Moreover, there are many unresolved problems that are unique to RTP.

In order to combine the best features of furnaces and RTPs, Toshiba and TEL collaborated to develop the Fast Thermal Processor (FTP). The FTP utilizes standard vertical furnace configuration with a small batch size (50 wafers) and enhanced heating and cooling capability to achieve ramp rates of 50–100°C/minute. In addition, thermal and CVD processes can be done sequentially in the same chamber to achieve cost-effective process development and manufacturing. 🎯

4:40 pm MS-TuA9 Low Thermal Budget Gap Filling for Semiconductor Manufacturing, Kevin J. Uram, John K. Shugrue, and Nathan P. Sandler, Lam Research Corp., 49026 Milpom Dr., Fremont, CA 94538.

Advanced device designs and new material compatibility are placing greater demands on the gap filling capability of interlayer dielectric films. In this paper we present results of recent studies of the deposition of borophosphosilicate glass (BPSG) from the reaction of tetraethoxysilane (TEOS), triethylborate (TEB), phosphine (PH₃), and oxygen in the temperature range of 750° to 800°C for high aspect ratio gap fills and interlayer isolation applications. Our studies have shown that at least three different mechanism contribute to gap filling by BPSG in this temperature range. Viscous flow of the deposited glass is the predominant mechanism for gap fill at temperatures in excess of the glass transition temperature. A simple physical attraction between approaching side-walls has been observed to contribute to gap fill. Finally, some degree of chemical selectivity can be achieved from the deposition of BPSG from the above mentioned reactants. This chemical selectivity contributes to preferential gap fill from the corner structures.
Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, DC 20375; 1: Dept. of Electrical Engineering, Stanford University, Stanford, CA 94305; 2: Science Applications International Corporation, McLean, VA 22102.

We are developing a simple neural-based biosensor that detects the presence of wide ranges of toxic threats capable of interfering with many types of cellular and electrical functions. This approach uses neural cells as device components that must be integrated into a hybrid device consisting of electronic microcircuits and hardware dedicated to environmental control and sample processing. Silicon nitride-passivated iridium microelectrode arrays are surface-modified using self-assembled monolayers and used for detection of repetitive action potentials. Repetitive APs of the NG108-15 cells can be used to monitor both direct current activation/block and modulation by receptor/second messenger interaction. A key aspect of this integration involves the interfacing of cells to the surface, the nature of the electrical signals produced by the cells, and the electronics used to translate these signals into useful measurements. We will discuss our progress in modulating the physical, chemical, and electrical characteristics of this interface.

3:00 pm BI-TuA4 Micrometre Resolution Molecular Patterning at Transducer Surfaces, J. M. Cooper, H. Morgan and D. J. Fritchard, Bioelectronics Group, Department of Electronics, University of Glasgow, Glasgow, G12 8LT, UK.

Techniques for controlling the architecture of immobilized biomolecular films onto transducer surfaces have a wide range of potential applications in bioelectronics, particularly in biosensing and in the control of cell guidance. In this paper, a novel method for the site-specific attachment of biological molecules onto glass, gold and silicon is described. The technique, which is based upon the modification of the sensor surface and the activation of a bound photosensitive ligand, provides a method for two dimensional patterning of proteins onto precise areas whilst preventing non-specific binding to non-designated sites. Different molecules, including enzymes and antibodies, have been immobilized with a micrometre scale resolution whilst retaining their biochemical activity. Initial results are presented on the application of this system to multianalyte immunosensors.


The incorporation of biomolecules into optoelectronic devices to serve as molecular recognition elements is becoming an accepted methodology for the fabrication of ultrasensitive detection devices. The choice of biomolecule, its method of incorporation, and the chemical and physical environment in which it functions, are all critical parameters in determining specificity, sensitivity, and long-term use of the biosensor. While much is known about the interaction of biomolecules under solution conditions, the biochemical and physical interactions at the solvent-substrate interface in heterogeneous systems are yet to be fully defined.

Our own research effort has relied on the use of antibodies to serve as the detection component in several different biosensors systems. These devices employ a variety of substrates for antibody immobilization and operate under diverse chemical environments. In order for such sensors to function reproducibly with long-term stability, specialized immobilization methods and chemically-tailored substrates must be employed. These are required to maintain antibody activity and prevent the deleterious effects of non-specific adsorption or denaturation at the interface.

4:00 am BI-TuA7 Molecular Assemblies of Functionalized Polydiacetylenes, Deborah H. Charvey, Anke Reichert, Raymond Stevens, Geoff Kuzmiako, Jon Nagy, Center for Advanced Materials, Lawrence Berkeley Laboratory, Berkeley, CA 94720, and Department of Chemistry, University of California, Berkeley, CA 94720.

Self-organized membrane-like structures such as Langmuir-Blodgett films and liposomes provide new materials with well-defined surface chemical functionality. If the lipid molecules which comprise these structures are endowed with biochemically-active functional groups and with polymerizable groups, new multifunctional materials can be created. The applications of these materials will be discussed, particularly with respect to colorimetric sensor devices and pharmaceutical drug discovery.

4:40 pm BI-TuA9 Preparation of an Immunosensing Surface by a Mixed Self-Assembled Monolayer, P. Heiduschka, J. Ricker, W. Beck, G. Jung and W. Göpel, University of Tübingen, Institute of Physical and Theoretical Chemistry, Institute of Organic Chemistry, Auf der Morgenstelle, D-72076 Tübingen.

Thiols adsorb very strongly onto gold surfaces. When long-chained alkanethiols are used, so-called self-assembled monolayers of crystalline order may be formed which are stable over months in different media. In order to utilise these monolayers for analytical purposes in a biochemical sensor, they have to be modified chemically by incorporating molecules with appropriate recognition properties. We choose epitope peptides. Epitopes represent the sequence of amino acids of an antigen (e.g., a virus) which is recognised by the specific antibody. In our studies, the epitope of the virus of the foot-and-mouth disease was synthesised chemically and served as the antigen. The peptide was modified by coupling with hydroxyundecanethiol (HUT). The resulting derivative absorbs on gold and yields dense layers. The adsorbed layer was stabilised and optimised with respect to its surface density by coadsorption of the unmodified HUT with the HUT-peptide. The binding conditions of the specific antibody were characterised by electrochemical impedance measurement. After the molecular recognition step, the complex of the antibody with the epitope could be destroyed in 6 M urea solution. The recognition properties of the peptide layer remained after the removal of the antibodies. Therefore, reproducible detection of antibodies was possible with the same monolayer coating.

5:00 pm BI-TuA10 Hydration and Dehydration of Adsorbed Protein Monolayers on Different Surfaces, M. Rodahl, F. Höök, and B. Kasemo, Chalmers University of Technology and University of Göteborg, 412 96 Göteborg, Sweden.

An apparatus for sensitive hydration/dehydration studies based on the quartz-crystal microbalance (QCM) is described. The system is intended for studies of water equilibrium uptakes and uptake/release kinetics, respectively, at different humidities, at approximately 1 atm total pressure. The sensitivity is in the submonolayer regime, i.e. better than 3 ng/cm². The mass sensitive crystal is AT-cut and operating at 10 MHz fundamental resonant frequency.

In the present work we have applied this method to study hydration and dehydration of adsorbed monolayers of the proteins ferritin and myoglobin on (i) UV/ozone cleaned (hydrophilic) gold surfaces and (ii) gold modified by self-assembled monolayers of thiol (rendering the surface hydrophobic). For comparison, results are presented for the same surfaces without adsorbed proteins.

Information about the adsorbed layers and their water uptake is obtained by the observed frequency shift of the QCM and the simultaneously measured Q-factor of the oscillator. This provides information both about mass changes (due to hydration/dehydration) and about the viscoelastic properties of the adsorbed layers.
Tuesday Afternoon Poster Sessions

SURFACE SCIENCE
Room BR4 – Session SS-TuP

Aspects of Surface Science
Moderator: J. C. Hemminger, University of California, Irvine.

SS-TuP1 Simulation Studies of Electron Transfer at a Metal-Aqueous Electrolyte Interfaces, B. B. Smith, J. W. Halley, School of Physics and Astronomy, University of Minnesota, Minneapolis, MN 55455.

We report new simulation studies of the rate of ferrous-ferric and cuprous-cupric electron transfer at a metal electrolyte interface. In contrast with earlier work, new features in our study include a detailed account of the effects of the field associated with the charging of the electrode, inclusion of entropic effects in the calculated free energy barriers, and a study of the dependence of the relevant free energy surfaces on the distance of the ion from the electrode. The qualitative picture of the reaction mechanism which emerges is significantly more detailed than that in earlier work. The dominant factors in determining the rate and mechanisms of electron transfer are the distance dependence of the following: the work function of the metal, the concentration profile, and the electronic matrix element. Calculated free energy barriers for the ferrous-ferric reaction are consistent with experimentally measured ones. We also estimate the equilibrium potential for this reaction from the model and find a value which is within 0.1 V of the measured one. For the cuprous-cupric reaction we find a more pronounced effect of the electric field arising from the charge on the electrode. In the cuprous-cupric case, we present the limits within which a single step mechanism is relevant to the more complicated real world reaction.

SS-TuP2 Adsorption of Water on Oxidized Tungsten and Suppression of Electron Induced Oxygen Ion Emission by Molecular and Dissociated Water, M. Akbulat*, N. J. Sack and T. E. Madey, Rutgers University, Department of Physics and Astronomy, and Laboratory for Surface Modification, Piscataway, NJ 08855.

We have studied the adsorption and decomposition of water on an oxidized W(100) surface, and have measured the transmission of low energy (<10 eV) O\textsuperscript{+} ions through ultrathin films of water, H\textsubscript{2}O\textsuperscript{+}, condensed at 20 K. Our goal is to address the fundamental scattering and charge transfer processes that influence ion transport through molecular layers. O\textsuperscript{+} ions with a peak energy of 7 eV and a narrow angular distribution (FWHM ~ 15°) are generated by means of electron stimulated desorption (ESD) from the O\textsuperscript{+} oxidized W(100) surface and their yield, energy and angular distributions are measured as a function of H\textsubscript{2}O\textsuperscript{+} coverage with a digital ESDIAD (ESD ion angular distribution) detector. Thermal desorption spectroscopy (TDS) demonstrates that the majority of water adsorbed in the first monolayer remains molecular and desorbs with a peak temperature of 155 K. However, both TDS and ESD measurements indicate that a small percentage of H\textsubscript{2}O\textsuperscript{+} dissociates upon adsorption. We find complete suppression of the O\textsuperscript{+} ESD emission upon adsorption of only a fraction of a monolayer of H\textsubscript{2}O\textsuperscript{+}. We suggest that charge transfer between O\textsuperscript{+} and H\textsubscript{2}O\textsuperscript{+} is the main reason for the attenuation of O\textsuperscript{+}.

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SS-TuP3 Adsorption, Desorption, Mixing, and Solvation Kinetics of Mixed Methanol and Water Multilmayer Ices, R. Scott Smith, C. Huang, E. K. L. Wong, and Bruce D. Kay, Molecular Science Research Center, Pacific Northwest Laboratory*, Richland, WA 99352.

In an effort to understand the surface chemistry of ice and its applicability to processes occurring in liquid solutions we have studied binary mixtures of water and methanol ices grown using molecular beam deposition. It is well-known that liquid mixtures of methanol and water exhibit non-ideal solution behavior due to hydrogen bonding and hydrophobic solvation. Molecular beam deposition of both TPD (TPD and isothermal) are used to study the adsorption, desorption, mixing, and solvation kinetics of mixed multilayer (5-100 ML) ices of water and methanol. Compositionally tailored multilayers of methanol and water are grown on Ru(0001) using both sequential and simultaneous beam dosing techniques. The adsorption spectra exhibit complex features that depend strongly on both the mole fraction and the thickness of the multilayer. Analysis of the desorption spectra reveal both the extent of mixing and the details of the solvation kinetics. These kinetics depend strongly on the initial phase of the ice (amorphous or crystalline). The experimental results are analyzed using a kinetic model that describes liquid solution evaporation. From this analysis we can extract a binary phase diagram. The mixed water and methanol ices exhibit non-ideal behavior reminiscent of the liquid solution results. Details of the experimental techniques, results, and implications regarding ice solutions at low temperatures will be presented.

SS-TuP4 The Interaction of HCl With Ultrathin Ice Films: Observation of Adsorbed and Ad sorbed States, J. D. Graham and J. T. Roberts, Department of Chemistry, University of Minnesota, Minneapolis, MN 55455.

To gain understanding of heterogeneous chemistry that occurs in the polar stratosphere, the temperature programmed desorption and Fourier transform infrared reflection absorption spectra of hydrogen chloride interacting with amorphous and crystalline ultrathin (4-20 monolayers) ice films were studied. The desorption spectra reveal two distinct HCl states, at 140 and 180 K, labeled α- and β-HCl respectively. The β state, which is formed exclusively at low HCl exposures, evolves concurrently with water sublimation. This state is assigned to the sublimation of a stoichiometric HCl water phase. Infrared spectra suggest this phase is HCl·H\textsubscript{2}O. The α state is associated with HCl adsorbed on the hexahydrate surface. Experiments conducted on ice\textsubscript{d} show a much smaller amount of H-D exchange between HCl and D\textsubscript{2}O than would be expected from dissociative adsorption. Thus it is believed that HCl adsorbs molecularly. The activation energy of α-HCl desorption is 33 ± 5 kJ mol\textsuperscript{-1}, which strongly suggests hydrogen bond formation. This is supported by the infrared spectra with the disappearance of the surface O-H stretching mode upon desorption of HCl. A comparison of crystalline and amorphous films shows that the rate of HCl·H\textsubscript{2}O formation is much slower in crystalline films. This is due to a lower sticking coefficient of HCl on the crystalline films.

SS-TuP5 Differences in the Surface Chemistry of Amorphous and Crystalline Ice Thin Films, Jason E. Schoff, Jeffrey T. Roberts, University of Minnesota, Dept. of Chemistry, 207 Pleasant St. SE, Minneapolis, MN 55455.

The interactions of several small molecules with thin (50-80 ML) films of both amorphous and crystalline ice have been studied using temperature programmed desorption spectroscopy (TPD) and infrared reflection absorption spectroscopy (IRAS). Amorphous and crystalline films can be clearly distinguished using IRAS. Those adsorbate molecules which are good hydrogen bond acceptors show one more desorption state from amorphous ice than they do from crystalline ice, and also show differences in their IR spectra when adsorbed on the different two types of ice films. This is interpreted as indicating either a reduction in the number of free-OH bonds on the surface of crystalline ice, or a reduction in the accessibility of such dangling bonds to adsorbate molecules. This conclusion is supported by the significant reduction in intensity of the free-OD stretching peak which is observed upon crystallization of deuterated ice films in IRAS experiments. Molecules which do not readily form hydrogen bonds show no significant differences in behavior when adsorbed on the two types of ice films. To the best of our knowledge, this represents the first conclusive evidence for a difference in the chemical behavior of crystalline and amorphous ice surfaces.

SS-TuP6 SPA-RHEED-Spot Profile Analysis for In situ Characterization, B. Müller and M. Henner, University of Hannover, Appelstr. 2, D-30167 Hannover, Germany.

The morphology of the surface dominates the quality of the epitaxial growth. In order to tailor low dimensional artificial structures the surface must be characterized during growth. One of the most powerful methods for in situ studies is RHEED. The quantitative analysis on island sizes and step distributions, however, is mainly performed by
LEED because of the more complicated geometry of RHEED and the influence of inelastic scattering. To overcome these difficulties, a novel RHEED instrument with extremely high angular resolution (0.1 mrad) and energy resolution of about 2 eV is developed. For the first time the SPA-RHEED instrument points out the important contribution of inelastic processes to the intensity distribution of the spot profiles. Electronic losses due to plasmons and band transitions are clearly detected and quantitatively described by the dipole scattering theory. A meaningful evaluation of the profiles with respect to defects is only possible with a good energy resolution. For well prepared Si(111) with various island sizes in different azimuth the elastic and inelastic scattering is investigated throughout several Brillouin zones. Mean terrace sizes between 50 and 5000 Å are detected. Characteristic structures are correlated with the scattering vectors and are separated into surface and bulk related phenomena because of the refraction effect. The extremely high angular resolution of SPA-RHEED enables determination of island size up to the micrometer range and lattice constants with an accuracy <0.1 %. Surface damage is reduced due to the extremely low current. Therefore the features of the SPA-RHEED instrument offer new applications for RHEED.


Ice surfaces are important in ozone depletion involving heterogeneous chemistry on polar stratospheric clouds. The adsorption, desorption and diffusion of H₂O on single-crystal ice grown epitaxially on Ru(001) were measured using optical interference and laser-induced thermal desorption (LITD) techniques. Low energy electron diffraction (LEED) studies confirmed that the ice films were crystalline to thicknesses of at least 140 Bilayers (1 Bilayer defined according to Thiel et al., JCP 75 (1981) 5556). Condensation and evaporation coefficients were measured using optical interference. The condensation coefficient, α, was measured at temperatures between 95 and 170 K and decreased versus temperature from α = 1.00 ± 0.08 at 95 K to α = 0.68 ± 0.11 at 170 K. The evaporation coefficient, γ, was obtained using isothermal desorption measurements and remains constant at γ = 0.62 ± 0.08 from 160 K to 193 K. An Arrhenius analysis of the desorption rates reveals zero-order desorption with an activation barrier of E_d = 12.8 ± 0.3 kcal/mol and a preexponential of νo = 4.5 x 10^{11} s^{-1} molecules/(cm²s). Isotope experiments were performed to investigate the desorption of a single bilayer of H₂O^{18} from a multilayer of H₂O^{16} ice on Ru(001). The isothermal desorption of H₂O^{18} measured with LITD followed first-order desorption kinetics. These measurements were performed using a residence time for H₂O on ice to be determined at different temperatures. LITD studies also examined the surface diffusion of H₂O on ice. A step coverage profile of H₂O^{18} on a multilayer of H₂O^{16} ice was created using a masking technique. By following the decay of the H₂O^{18} coverage profile with LITD, an upper limit of D = 6 x 10^{-4} cm²s^{-1} was placed on the diffusion coefficient of H₂O on ice at 140 K.


Steps on copper surfaces are mobile for temperatures larger than 300K. Due to the fine scan speed the tunneling tip finds the step in different positions in consecutive scan lines. Therefore, steps in STM images display sudden jumps in the step position. For Cu(1 1 19) we have measured the probability distribution P(Δx, Δt) to find time intervals of Δx scan lines, each requiring a time Δt (see figure). A different dependence of P(Δx, Δt) is found for short and long intervals between two consecutive step jumps. Short intervals obey an e^{-0.3Δx} law which is caused by the emission of adatoms from kink sites and their subsequent return. For long intervals an exponential tail is found in P(Δx, Δt) which is due to uncorrelated kink motion. We have developed analytical models for P(Δx, Δt) in the two regimes in terms of the microscopic events of adatom generation and migration along the steps. The models were tested employing Monte-Carlo simulations. With the help of these models we have determined the activation energies for adatom generation and migration from the experimental data.

SS-TuP9 Atomically Resolved STM Study of the Interaction of Boron with Si(001), Yafun Wang, and Robert J. Hammers, Department of Chemistry, University of Wisconsin, Madison, WI 53706, USA.

The interaction of boron with Si(001) is very important for semiconductor processing. STM, tunneling spectroscopy and surface photovoltage measurement have been used to study the geometric and electronic structures of boron-induced reconstructions produced by thermal decomposition of diborane on Si(001). At T > 675 K, boron atoms organize into ordered structures on the surface. STM images reveal a number of boron-induced reconstructions which arise from the arrangements of three structural units. Boron atoms bond at substitutional sites and form a delta-doped layer. These boron-induced structures are chemically inactive, but electrically active. Over 1075K, most boron atoms diffuse into bulk and it also produces surface faceting. This is the first atomic-scale study of boron-induced reconstructions on Si(001) and it will provide a deeper understanding of semiconductors. The iteration process of new structures and their effects on further silicon film growth will be discussed. This work is supported by the U.S. Office of Naval Research.

SS-TuP10 The Role of Energy Loss Mechanisms for the Trapping Dynamics of Alkanes on Platinum Surfaces, J. A. Stinnett, R. J. Mudri, Stanford University, Stanford, CA 94305, and J. C. Tully, AT&T Bell Labs, Murray Hill, NJ 07974.

The molecular adsorption of methane, ethane, and propane on several platinum surfaces were studied by simulation to examine the relative importance of the energy loss mechanisms available for trapping on the surface. Methane was simulated as a spherical species. Ethane and propane were considered to be pseudo diatomic and triatomic molecules, respectively, interacting with a vibrating periodic solid slab. Three dimensional stochastic trajectory calculations with a Morse pairwise methyl-platinum potential were used to represent the gas-solid interaction. The potential parameters were employed for methane, ethane, and propane with other platinum surfaces. For example, the application of these potential parameters to the Pt(110)-(1 x 2) surface predicts the azimuthal dependence and the enhancement in the trapping probability for ethane seen experimentally. These calculations suggest that ethane trapping on Pt(110)-(1 x 2) is enhanced relative to Pt(111) due to increased conversion of normal momentum to parallel momentum. Methane and propane simulations were also successful at representing the incident angle and energy dependence on trapping. The wide range success of all these calculations provides confidence for the prediction of adsorption probabilities and dynamics from a very limited data set.

SS-TuP11 Development of Surface Morphology and Microstructure During Si Epitaxial Growth, D. P. Adams and S. M. Yalosso, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109-2136, D. J. Eaglesham, AT&T Bell Laboratories, Murray Hill, N.J. 07974.

The development of surface morphology and microstructure has been studied for homoepitaxial growth on both patterned, nonporous Si substrates and flat Si(100) surfaces. Growth on nonplanar substrates was useful for studying changes in morphology over a range of temperatures. First, growth at higher temperatures has been characterized in terms of a number of well developed high index surfaces (including Si(117), (115) and (113)) which form at a sidewall. Subsequent low temperature growth on each of these surfaces has also been studied. Each Si(hkl) surface was found to exhibit a crystalline to amorphous transition at low temperatures; however, the epitaxial thicknesses, θfcc, are different for each [hkl] surface (in general,
h_{001}(001) > h_{001}(117) > h_{001}(115) > h_{001}(113)$. Furthermore, $h_{001}(hkl)$ was found to follow an Arrhenius relation with substrate temperature. The “activation energies”, characteristic of this breakdown in epitaxy, were then determined for each substrate by measuring the epitaxial thickness over a range of temperatures below $-300 \degree C$. $E_{act}(hkl)$ was determined to be $0.35 eV$, $0.33 eV$, $0.30 eV$ and $0.27 eV$ for (001), (117), (115) and (113) surfaces, respectively.

The origin of this breakdown in epitaxy has also been identified. Working on flat Si(100) starting surfaces showed that surface roughening leads to the formation of amorphous material (a phenomenon intrinsic to low-temperature growth). In this investigation, the role of $h$ on changing the epitaxial thickness was also elucidated. Increased amounts of atomic $D_{h}$ introduced to the growth surface were found to lower $h_{001}$, but also increase the roughening rate within the epitaxial portion of the film and permit a larger roughness amplitude before nucleation of the amorphous phase. Interestingly, the surface roughening rates, measured here by TEM (using thin Ge marker layers to mark the profile) and RHEED, indicated that growth of $Si$ at low temperatures is characterized by a large roughening exponent, i.e. $\beta > 1$.

SS-TuP12 Nucleation Phenomena in the Chemical Vapor Deposition of Iron on Si(001), T. M. Mayer, L. L. Tadder, R. S. Zwanziger, Sandia National Laboratories, Albuquerque, NM 87185.

We report studies of the initial stages of growth of Fe films on single crystal Si(001) surfaces by chemical vapor deposition (CVD). In-situ high temperature scanning tunneling microscopy (STM) is used to image surface reactive sites, island morphology and size distributions as a function of coverage. At temperatures of 100-150 $\degree C$ we observe selective decomposition of the FeCO$_3$ precursor at Si dimer sites, with no reaction observed at step or defect sites. Isolated atom-sized features are initially observed, followed by linear chains of atoms aligned along Si dimer rows at increasing coverage. At coverage in excess of one monolayer, larger islands characteristic of three dimensional growth appear. We observe no residual C or O in complementary x-ray photoelectron spectroscopy (XPS) experiments, nor do we see evidence for C or O contamination in the STM. The shape and size distribution of nuclei are consistent with an autocatalytic growth process, in which selective decomposition of the precursor occurs at existing metal nuclei. This small sticking coefficient and short residence time of FeCO$_3$, and apparent low mobility of Fe atoms on Si at 100-150 $\degree C$ suggest that site-selective adsorption and reaction of the precursor gas molecule is responsible for the morphology of the Fe nuclei. Kinetic Monte-Carlo simulations are presented to examine the dynamic processes of nucleation. Low energy electrons from the STM tip are also effective at enhancing local area nucleation of Fe through electron induced decomposition of FeCO$_3$.

This work is supported by the U.S. Department of Energy under contract DE-AC04-94AL8500.

SS-TuP13 STM Observations of Hydrogen-Induced Ag Cluster Formation on the Si(111) $\sqrt{3} \times \sqrt{3}(R30^\circ)$-Ag Surface, Itsuo Katayama and Yasuyuki Ohba, Department of Applied Physics, Faculty of General Education, Osaka Institute of Technology, Asahiku, Osaka 555 Japan, Hideki Ohishi, Yasuji Yamamoto and Kenjiro Oura, Department of Electronic Engineering, Faculty of Engineering, Osaka University, Suita, Osaka 565 Japan.

Scanning tunneling microscope (STM) has been used to study structural transformation of the Si(111) $\sqrt{3} \times \sqrt{3}(R30^\circ)$-Ag surface caused by atomic hydrogen adsorption. When atomic hydrogen adsorbed on the $\sqrt{3}$-Ag surface, the surface structure dramatically changes. The atomic hydrogen attack the Ag-$\sqrt{3}$ surface, indicating that the domain boundaries or step edges, and the clusters are found to be formed on the $\sqrt{3}$-Ag domain. This suggest that Ag atoms break the Ag-Si bond followed by the formation of both the bonding of H-atom to the Si dangling bonds thus formed and the clusters formed by Ag free atoms released from the $\sqrt{3}$ lattice. The hexagonal shape observed and its orientation suggest the formation of Ag(111) clusters, and the size of the clusters has been confirmed to be less than 15 nm. These STM results are in agreement with our previous ion scattering results. Spatial distributions of the clusters, observed as a function of the exposure to atomic hydrogen, reveal that surface diffusion of Ag atoms over the hydrogen-covered regions is largely suppressed compared with that on the $\sqrt{3}$-Ag surface. It has been disclosed that hydrogen adsorption has two critical roles: the first is that hydrogen atoms break Ag-Si bonds and release free Ag atoms on the $\sqrt{3}$-Ag surface. The second role of hydrogen is to suppress surface migration of the Ag atoms over the hydrogen adsorption region on the specimen surface.

SS-TuP14 The Evolution of Growth Front in a Heteroepitaxial System: Pb On Cu(100)*, Hong Zeng and G. Vidali, Department of Physics and the Solid State Science and Technology Program, Syracuse University, Syracuse, NY 13244.

We have measured in real time the evolution of the growth front in the kinetically driven low temperature (150 $\degree K$) growth of Pb on Cu(100). Films from above 1 layer up to 450 layers were studied using helium beam scattering apparatus, LEED and Anger electron spectrometer. The large lateral mismatch, the difference in bonding strengths and symmetry between substrate and film produces a rather complex growth pattern. The first Pb layer is ordered with a square supercell ($5 \times 5$) rotated by $37^\circ$. The second layer grows by islands with a double step of about 4.87 $\AA$. Overall the second and third layer grow in a disordered fashion. The next layers grow in a quasi-layer-by-layer mode. The LEED patterns show the presence of a [111]Pb exposed surface with domains rotated by $90^\circ$. From about 16 equivalent monolayers (ML) until 450 ML the average terrace width changes little. The interface width grows as $t^{p}$ with $p = 0.3$ until about 40 ML. The lateral correlation length grows with deposition time as $t^{\alpha}$ with $\alpha = 1$. After that the growth saturates and a groove instability develops; the $\alpha$ value is slightly smaller than 1 due to a logarithmic correction. Manipulation of the growth of the film was also achieved by preparing a first layer with different structures or by annealing.

*Supported by NSF grant DMR 919735.

SS-TuP15 Island Growth and Energies in Submonolayer Heteroepitaxy on Cu(100), J. F. Wendelken, H. Durr, and J.-K. Zhao*, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6030 and Southwest Missouri State University, Springfield, MO 65804-0094.

Submonolayer epitaxial growth may be described in terms of a scaling relationship $L \sim t^{\Phi}$ where $L$, $D$, and $F$ are the average island separation, adatom diffusion constant, and deposition flux, respectively. The scaling exponent $\Phi$ reflects the critical nucleus size required to begin the growth of an island. High angular resolution LEED was used to observe the island morphologies and separations as a function of flux and deposition temperature $T$ in the steady state regime. Two dimensional diffraction profiles at temperatures of 220 to 305 K and STM topographic images at $300 K$ reveal square islands with close-packed [110] step edges indicating a low energy barrier for diffusion along this step direction. From measurements of the island separation as a function of flux, the critical island size is found to change from a dimer at 223 K to a tetramer at 263 K and above. An Arrhenius plot of the island separation vs $1/T$ for a fixed flux can be best fit with two line segments with a turning point at 225 K indicating the onset of dimer dissociation. The slope of this plot in the low temperature (dimer) regime indicates a diffusion energy barrier of $0.36 eV$. From the change in slope, the average binding energy of dimers and trimers is estimated to be 0.14 eV.


Adsorbate mobility is critical to the growth, stability, and structure of surface films and structures. We use the scanning tunneling microscope to obtain an atomic scale view of the motion of adsorbates on surfaces. For sufficiently slow diffusion, we follow this motion in real time. Reducing the rate of motion can be accomplished by chemically modifying the adsorbed surface under study to increase the strength of lateral interactions or by reducing the temperature at which the measurements are performed. We have used both these strategies to enable the study of adsorbate diffusion. For faster adsorbate motion we are able to observe the partial occupancy of surface sites on the time scale of our measurements. For lateral motion induced by surface processes such as adsorption or chemical reaction, we are able to analyze the position of the adsorbates or reaction products in order to measure the distances covered and to elucidate the means by which energy is accommodated to the surface. For sufficient adsorbate mobility, we observe molecules nucleating, clustering and forming ordered structures at steps and other surface defects.

This work has been supported by BRDC, NSF, and ONR.
SS-TuP17 Local Morphology Effects on Mass Transport on Sputtered Au(111): an STM Study*, T. Curcic, J. G. McLean, B. H. Cooper, LASSP, Cornell University, Ithaca, NY 14853. We have performed a UHV study of nanoscale clusters of vacancies on a clean Au(111) surface at room temperature. The vacancy clusters were formed by sputtering the surface with a low energy (500 eV) Ar ion beam. Time-lapse sequences of STM images reveal the following: (1) while vacancy clusters persist as highly stable features on Au(111) at room temperature, changes in local morphology do occur, and (2) dramatic changes in mass transport coincide with certain changes in local morphology. We discuss two microscopic mechanisms which may be responsible for the changes in mass transport: (1) onset of vacancy diffusion (intra-layer mass transport) and (2) enhanced inter-layer mass transport due to the increased adatom pressure near curved step edges.

*Supported by the Cornell Materials Science Center (DMR-9112654); additional support from F49620-93-1-0504.

SS-TuP18 Identification of metastable bcc Ni Film Growth on Al(001) Surfaces Using High-Energy Ion Scattering, X-Ray Photoelectron Diffraction, and X-Ray Photoelectron Spectroscopy, V. Shatthanandan, Aditi A. Saleh, R. J. Smith, Physics Department, Montana State University, Bozeman, MT 59717.* High-energy ion scattering spectroscopy (HEIS), x-ray photoelectron diffraction (XPD), and x-ray photoelectron spectroscopy (XPS) were used to study bcc Ni (001) films deposited on Al(001) surfaces at room temperature. The HEIS/channeling experiments were performed in the normal direction, and the surface peak areas of Al and Ni were monitored as a function of Ni coverage. Following each HEIS measurement the XPS spectra of the Al 2p and Ni 2p core levels were collected. In the XPD experiments polar-angle scans at both the Al 2p and Ni 3p emission were collected for three different coverages of Ni, namely at 5, 15 and 51 monolayers (ML), and for Ni thin films on the (001) and [110] azimuths of the substrate surface. The forward scattering peaks obtained in these experiments suggest that up to 5 ML of Ni coverage, a Ni film grows as a metastable bcc(001) structure in a Stranski-Krastanov (SK) like growth mode. That is, first a flat overlayers, followed by 1 layer thick and, not necessarily commensurate with the substrate lattice. This is followed by Ni island formation. After 5 ML of coverage, the bcc islands coalesce to cover the surface. These results are significantly different from the Ni/Al(110) results in which a strong Ni-Al interface reaction was observed.

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SS-TuP19 The Initial Growth of Ultrathin Pd Films on Cu(001), J. Yao, Y. G. Shen and D. J. O'Connor, Department of Physics, University of Newcastle, New South Wales 2308, Australia. The deposition of Pd on Cu(001) at 300 K was studied by low energy alkali ion scattering and low energy electron diffraction (LEED). The results of the azimuthal scans at grazing incidence showed evidence for surface alloying by the substitution of Cu with Pd atoms in the first layer even at very low concentrations. XPS measurements confirmed the incorporation of Pd atoms into the second layer. No variation was observed at coverages below 0.3 ML. A clear maximum in the best (2 × 2) LEED pattern intensity was seen at a 50% Pd-50% Cu in the first layer, while there was 30% Pd-70% Cu in the second layer. Further growth of the Pd beyond the surface (2 × 2) mixed layer was found to promote more Pd atoms in the top two layers, destroying the ordered arrangement of the surface (2 × 2) structure. The disordered ion scattering effect at higher coverages was evidenced by measuring the Pd-Pd pair double scattering peak using a 2 keV Pd ion scattering. The structure of the Cu(001)-(2 × 2)Pd surface phase was also probed by measuring the intensities of the (2 × 2)-Pd structure, and the variation in the intensities of the (2 × 2) peak with Pd coverage was noted. The results showed that the Pd and Cu atoms are located at the same plane within the experimental uncertainty of 0.05 Å.

SS-TuP20 Structure and Stability of Steps on the GaAs(110) Surface, J. M. McCoy*, Department of Physics, Montana State University, Bozeman, MT 59717, and John P. LaFemina, Pacific Northwest Laboratory**, Richland, WA 99352. An improved understanding of the behavior and structure of steps at semiconductor surfaces is important for device fabrication, as well as being of fundamental interest. Using a tight-binding total energy calculation procedure [1], we have calculated the atomic geometries and energies of monolayer and bilayer steps running in the [110] and [112] directions on the GaAs(110) surface. These computations provide important insight into the dependence of the relative step stability upon the direction of the step and upon the step-edge atomic configuration. We find steps in the [112] direction to be much more stable than steps in the [110] direction, in agreement with STM results which suggest such a stability difference on the basis of extreme differences in step structure [2].


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SS-TuP21 The Thermochemistry of CBr4 on GaAs(100), M. McEllistrem, D. Sloan and J. M. White, NSF Science and Technology Center, University of Texas, Austin, TX 78712. Recent GaAs film growth studies have shown carbon tetrabromide (CBr4) to be a promising precursor for carbon doping. We have investigated the thermochemistry of CBr4 on GaAs(100) using temperature programmed desorption (TPD), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SSIMS). Our TPD results, following low temperature (~100 K) adsorption of CBr4, show a physisorbed state for doses greater than 1 monolayer, from which the molecule desorbs intact at 195 K. For submonolayer coverages, desorption of molecular CBr4 is not seen; instead, bromine desorbs as GaBr3 (x = 1-3) at 650 K, leaving carbon behind on the surface. XPS studies confirm the submonolayer decomposition of CBr4, and indicate that the CBr bond has broken and bromine migrated onto the GaAs surface at temperatures as low as 400 K. The kinetics of the CBr4 decomposition have been investigated by SSIMS. The results will be related to film growth studies.

SS-TuP22 Surface Chemistry of Dimethyl Hydrazine on GaAs(100), Y.-M. Sun, D. Sloan, A. Schwanner and J. M. White, Department of Chemistry and Biochemistry, The University of Texas at Austin, Austin, Texas 78712. The thermal chemistry of dimethyl hydrazine (DMH) on GaAs(100) has been studied by X-ray photoelectron spectroscopy and temperature programmed desorption. DMH molecularly adsorbs on GaAs(100) at 150 K, and forms three dimensional islands as indicated by TPD spectra. Molecular desorption occurs at 180 K with a high temperature shoulder extending to 400 K. No evidence for complete desorption during the temperature ramp was found. Dissociation did occur during dosing at 400 K. N(is) XP spectra show that N-N bond breaking occurs. Two decomposition products, dimethyl amine and ammonia, desorbed at 490 and 580 K, respectively. GaN formation was observed when DMH was dosed at surface temperature above 600 K, and carbon contamination was below the detection limit of XPS. There was no evidence for C-N bond breaking at reaction temperatures below 500 K.

This work was supported by the NSF Science and Technology Center, Grant CHE 8920120.

SS-TuP23 The Adsorption of Si on GaAs(001)-(c4 × 4); STM Evidence for Si Induced Disruption of As Layers, T. S. Jones1,2, A. R. Avery1,2, D. M. Holmes1,2, M. R. Faksy3, B. A. Joyce3, Department of Chemistry and IRC for Semiconductor Materials, Imperial College, London, UK.

Atomic resolution scanning tunneling microscopy (STM) has been used to study the adsorption of Si onto GaAs(001) surfaces, grown in situ by molecular beam epitaxy (MBE), with a view to understanding the incorporation of Si in doped GaAs structures. For surfaces prepared at 400°C, the clean GaAs surface is characterised by a well-defined c(4 × 4) HREED pattern, a structure terminated with two layers of As. Filled states STM images of this surface indicate that the basic structural unit, when complete, consists of rectangular blocks of six As atoms, with the As-As bond in the surface layer aligned along the [110] direction. Deposition of Si onto this surface at 400°C and relatively low coverages (<0.5 ML), causes disruption of the c(4 × 4) layer and formation of dimer rows on the surface. The Si resides in what was the topmost As layer, while displaced As forms dimer rows on top of this Si. In contrast, deposition of Si at 400°C on the GaAs(001)-(2 × 4) surface, shows no evidence for such a disruption.
The importance of the underlying GaAs surface reconstruction in determining the incorporation of Si into the growing structure will be discussed, in particular relating this to the electrical behaviour in Si δ-doped GaAs.

SS-TuP24 Adsorption and Decomposition of Ammonia and Hydrazine on the GaAs(100)-(8×2) Surface. Elisabeth Apen and John L. Gland, Department of Chemistry, University of Michigan, Ann Arbor, MI 48109-1025.

Temperature programmed desorption (TPD), x-ray photoelectron spectroscopy (XPS), and high resolution electron energy loss spectroscopy (HREELS) have been used to study the adsorption and decomposition of ammonia and hydrazine on the GaAs(100)-(8×2) surface. Ammonia was found to decompose to a moderate (250K) temperature with an NH₂ deformation mode in the HREELS spectrum. Hydrazine was found to adsorb in a "side-on" (vs. "end-on") fashion by XPS. At low coverages, hydrazine dissociation is complete with ammonia the major gas phase product. At higher coverages, N₂ is the major decomposition product. The absence of a high temperature TPD recombination peak indicates the formation of N₂ occurs via an intramolecular mechanism. Both NH₃ and NH₂ are identified as reaction intermediates for hydrazine decomposition on the GaAs(100)-(8×2) surface. Above 350 K, only NH₃ species are present on the surface.

SS-TuP25 Chemical Reactions of Triethylantimony on GaAs(100). John M. Heitzinger and J. G. Ekerdt, NSF Science and Technology Center, University of Texas, Austin, TX 78712.

As many based materials are potentially important for a number of device applications where a small band gap is desirable. Materials such as GaSb and InSb have been successfully grown from organometallic sources using a number of techniques. However, several problems including relatively high growth temperatures and carbon incorporation persist. In an effort to understand the underlying reactions for these problems and evaluate the effect of various ligands on film growth, we have initiated a surface science study of antimony precursors. Our result for triethylantimony indicate that both molecular and dissociative adsorption occur for a substrate temperature of 55°C. A small amount of triethylantimony desorbs from the surface between 55 and 100°C. Upon heating the surface to 300°C, the ethyl ligands undergo a β-hydride elimination reaction to form ethylene. No evidence for ethyl radical desorption is observed indicating homolysis is not an important pathway for ligand removal. This behavior is different from that observed for triethylgallium where both homolysis and β-hydride elimination reactions occur. Most of the triethylantimony initially chemisorbed on the surface is converted to atomic Sb by 380°C. This indicates that once triethylantimony is chemisorbed, its reaction probability is high. We have also performed exposures of triethylantimony at 400°C. For equivalent exposures, more Sb is desorbed at 400°C relative to substrate temperatures of 55°C. This is the expected result of site blocking by ethyl groups which remain adsorbed on the surface at the lower adsorption temperature. These, and other results will be related to film growth results.

SS-TuP26 The Interaction of Hydrogen Atoms with Ethyl Groups on Si(100), P. A. Steiner, J. M. Heitzinger, M. McEllistrem, J. Bonser and J. M. White, NSF Science and Technology Center, University of Texas, Austin, TX 78712.

We have investigated the interaction of hydrogen atoms with ethyl groups adsorbed on Si(100) with Auger electron spectroscopy and temperature programmed desorption (TPD). Ethyl bromide was exposed to the Si(100) surface at 350 K and produced adsorbed ethyl groups and Br₂ desorption. The ethyl group desorption occurs at 350 K via a β-hydride elimination reaction. When H atoms are exposed to an ethyl + Br₂ saturated surface at 350 K, the Br₂ is removed efficiently by hydrogen atoms. However, only a small fraction of the ethyl groups are removed at this temperature. Removal of the Br₂ atoms causes the ethylene desorption temperature to increase by 100 K. This indicates that absorbed Br₂ decreases the activation energy for the β-hydride elimination reaction. The removal rate of ethyl groups by H atoms increases with increasing substrate temperature; the ethyl and Br₂ removal rate become comparable at 450 K. Arrhenius plots indicate an activation energy for ethyl group removal of 4 kcal/mol. These results, along with the hydrogen flux dependence and kinetic order will be discussed. The data are consistent with a "hot" precursor mechanism in which the majority of the activation energy required for the reaction to remove ethyl groups is acquired from the precursor, i.e., the H atom, and not the substrate.

This work was supported by the National Science Foundation, Grant CHE-8920120.

SS-TuP27 Infrared Evidence for an Electrically Conductive Diamond (110) Surface. Bob L. Mackey, John N. Rustad Jr., John E. Crowell, James E. Butler, Naval Research Laboratory, Washington, DC 20375; 2) University of California, San Diego, La Jolla, CA 92123.

The internal reflectivity of the diamond (110) surface was observed by multiple internal reflection infrared spectroscopy (MIRIRS). Surprisingly large variations in the broadband infrared reflectance were seen with annealing or hydrogen dosing. The broadband internal reflectance of the H-terminated surface is found to be substantially on external desorption of the hydrogen. The reflectivity of the surface was restored by exposure to atomic hydrogen. The change in reflectivity increased with frequency, and is greater for S-polarization.

A simple Drude electron gas model and Fresnel reflectance calculation is used to predict the reflectance of a diamond surface modified by a thin conductive layer for a range of possible materials. The model shows that the observed optical effects are consistent with electrical conductance changes in a thin surface layer. Broadband reflectance changes may provide a sensitive and generally applicable method of measuring surface electrical conductivity in situ.

*Fannie and John Hertz Foundation Fellow, additional support provided by ONR and ARPA.

SS-TuP28 Surface Chemistry of Potential CVD Precursors, Diethylsilane and Diethylgermane, on Ge(100), A. Mahajan, B. K. Kellerman, N. Russell, S. Banerjee, J. G. Ekerdt, A. Tasch, J. M. White, National Science Foundation Science and Technology Center, University of Texas at Austin, Austin, TX 78712.

The adsorption and desorption kinetics of diethylsilane (DES) and diethylgermane (DEG) on a Ge(100) (2×1) surface have been studied using temperature programmed desorption (TPD), Auger electron spectroscopy (AES) and high resolution electron energy loss spectroscopy. DES and DEG adsorb at room temperature in a self-limiting fashion. Data indicate that both precursors dissociatively chemisorb, producing a hydrogen- and ethyl-terminated surface. TPD of DES-saturated and DEG-saturated surfaces required two species, ethylene and hydrogen, desorbed from the surface. The ethylene signal evolved from the decomposition of the ethyl groups via β-hydride elimination, with the desorption peak temperature of 620 K for both precursors. The hydrogen TPD spectra were also similar for both precursors and consisted of two peaks: a low temperature peak corresponding to hydrogen desorption from the monohydride state and a high temperature peak corresponding to desorption of hydrogen rate-limited by β-hydride elimination of the ethyl groups. The area under the peak in the TPD spectra was calibrated against the areas for known hydrogen coverages. Using this, the Si/Ge atom coverage following DES/DEG saturation exposure was found to be approximately 0.3 ML, assuming complete β-hydride elimination of the ethyl groups. No carbon contamination was detected on the surface by AES following exposure and TPD cycles of either DES or DEG.

This work was supported by Science and Technology Center under NSF Grant CHE-8920120 and ONR Contract No. N00014-91-J-1213.

SS-TuP29 Surface Reactions of Ethylgermanes on Si(100), Li Chen, Lori A. Keeling, Wei Du, and C. Michael Greenleaf, Department of Chemistry, University of Missouri-Columbia, Columbia, MO 65211.

The adsorption and decomposition of mono-, di-, and triethylgermane on the Si(100)-(2×1) surface was investigated with the intent of elucidating the surface processes leading to the deposition of epi-axial Ge films from gaseous Ge-containing sources. The low temperature adsorption of each molecule was investigated, as well as, their thermal decomposition. Similarities in the thermal decomposition of these molecules was observed. H₂ and C₂H₄ are observed in temperature programmed desorption experiments. The desorption of a NH₂ group from the Si(100) surface is achieved through a β-hydride elimination reaction within the adsorbed ethyl group. The kinetics of ethyl group decomposition are also determined. The amount of Ge that can be deposited in a reaction cycle is correlated with the number of sites occupied by the ethyl groups upon dissociation.
of a given ethylenegermine. The interactions of ethylenegermes with Si(100) will be discussed in detail and the implications for Ge atomic layer epitaxy will also be explored.

SS-TuP30 An STM Study of Hydrogen Adsorption on Si(112), A. A. Baski and J. J. Whitman, Naval Research Laboratory, Washington, D.C. 20375.

An ideally terminated Si(112) surface would consist of short (111) terraces separated by monoatomic-height steps running along the [110] direction. It can be considered a (111) surface "miscut" by 19.5°. Assuming such an ordered array of steps exists, this surface has been used as a potential substrate for quantum wire growth and heteroepitaxy. However, our recent STM studies revealed that this surface undergoes a major reconstruction. The novel, 3D reconstruction structure consists of 50-100 Å wide (337) terraces separated by a variety of short downward-sloping regions [necessary to maintain the (112) plane]. In order to further elucidate the nature of this reconstruction, and investigate possible effects of processing, we have studied the effect of H adsorption on this surface.

Following room temperature adsorption of atomic hydrogen (produced by a hot filament at ~ 5 cm from the sample and H2 pressures ~ 1 x 10-7 Torr), the facet-reconstruction to (337) terraces is still observed, but the atomic-scale structure is disordered. We attribute the disordered features to the presence of SiH, SiH2, and SiH3 groups on the surface, similar to those observed on Si(001). The (337) terraces persist even if the surface is exposed to H at ~ 400°C under conditions which normally produce a monohydride-covered Si(001) surface. However, at this higher temperature the atomic-scale structure within the reconstruction is modified by the adsorbed H, producing new ordered structures. These structures and their evolution with increasing H coverage reveal new details about the atomic-scale structure of the clean surface reconstruction. Models of the H-covered surface and the implications of our observations for processing of this substrate will be discussed.

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Materials Characterization
Moderator: R. J. Hamers, University of Wisconsin, Madison.


This study reports on the chemical identification, distribution and morphology of Se nanoclusters formed during the crystal growth process of ZnSe single crystals. The ZnSe crystals were grown by the Physical Vapor Transport method from prepurified starting materials. The surface was examined using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX). The formation of nanoclusters and their relation to the crystal growth procedure was investigated. The occurrence of the nanoclusters was linked to the purity and stoichiometry of the starting materials. The characteristic surface morphology of the crystal growth facets. Acknowledgement: This work was performed through funding provided by the NASA through the Fisk Center for Photonic Materials and Devices, Grant #NAGW-2925, and partial support from NASA Lewis Research Center, Grant #NAG3-1430.

NS-TuP3 Chemistry in Nanoporous Materials: Semiconductor Particles Formed Within a Solid Template, Catherine M. Zelenski and Peter K. Dorhout, Colorado State University, Fort Collins, CO 80523.

Metal sulfide semiconductors have been synthesized within the nanometer-sized pores of an aluminum oxide template. Several solution methods for the formation of nanoparticles of semiconductors within this template have been studied. A method of dipping the film into precursor solutions produced fine particles of the semiconductors trapped within the pores near the surface of the film. A second method utilizing a U-tube apparatus produced a thin coat of metal sulfide particles along all inner surfaces of the aluminum oxide template. Primary methods of characterization include UV-Vis spectroscopy, TEM, SEM, energy dispersive X-ray microanalysis, and electron and X-ray powder diffraction. Thus, very small particles have grown to completely fill the 50-70 nm diameter pores of the template; however, several other methods for these syntheses are being studied.

NS-TuP4 Scanning Tunneling Microscopy of Thin Films of Ta2S5 Prepared by an Interrcalation-Exfoliation Method, A. Manivannan, C. R. Cabrera and A. Fujishima*, Dept. of Chem. and Materials Research Center, Univ. of Puerto Rico, P.O. Box 23546, San Juan, PR 00931 USA, *Dept. of Synthetic Chem., Faculty of Engineering, The Univ. of Tokyo, Hongo, Bunkyo-Ku, Tokyo-113, Japan.

Two dimensional atomic and electronic systems are of specific interest with respect to their phase transitions due to the formation of charge density waves (CDWs). Layered transition metal dichalcogenides (LTMDs) are the suitable candidates for two dimensional studies where they are prepared as single molecular layer film. In this work, single layer suspensions of metallic layered compounds Ta2S5 were prepared by a lithium intercalation-exfoliation procedure. These suspended layers were directly deposited on a titanium substrate and heat treated at 250°C in vacuum for 2 hrs. Scanning electron microscopy (SEM) measurements revealed a lamellar microstructure with the basal plane of Ta2S5 crystals oriented parallel to the substrate. Scanning tunneling microscopy (STM) investigations indicated the existence of CDWs having peak spacings of ~ 12 Å similar to that of single crystals. The surface atomic structure has also been imaged by STM which showed lattice distortions and deviations. The existence of CDWs in these restacked single layer(s) of Ta2S5 allows us to study the intralayer interactions and also supports the theoretical work of a two dimensional model of CDWs in one layer. Moreover, the presence of CDWs in these films contradicts the failure to observe a CDW diffraction pattern in the previous electron microscopy study. Thus, STM seems to be a powerful and unique technique to study the existence and dynamics of CDWs in these single layer(s) of Ta2S5 films.


Interest in nanostructures is fueled by the potential for finding novel properties in material structures that have small physical dimensions. Indeed, composite materials assembled from nanosized components exhibit properties very different than bulk depending on the size and characteristics of their individual constituents. A dramatic example is found in the reaction rate of mixtures of Al and MoO3 nanopowders. Powder mixtures with average diameters of 2-500 Å achieve reactive power 1000 times that of conventional thermite powders. Also, reactive power is tunable by mixing together powders with other average particle sizes. Nanostructures can be manipulated to yield changes in the behavior of the composite, e.g., partial oxidation of Al nanopowders reduces total energy released in reactions of Al and MoO3 powders. Often, properties of nanosized components can be inferred from macroscopic changes that occur in a bulk quantity of material. We have used thermogravimetric analysis (TGA) to measure changes in mass of Al powder samples annealed at a particular temperature during exposure to oxygen. Samples are characterized using transmission electron microscopy (TEM) and Rutherford backscattering spectrometry (RBS). As-prepared Al particles consist of a core of crystalline Al protected by a ~ 30 Å thick shell of amorphous alumina. Activation energy for oxidation is determined from a series of isothermal TGA measurements taken at different temperatures for powder samples with different average particle sizes. Activation energy is found to depend on the average size of the Al particles and to be much smaller than for flat Al samples.

NS-TuP6 The Origin of Universality in the Top Layer Relaxation of Zinblende (110) and Wurtzite (1010) Surfaces, G. Brown and M. Weimer, Department of Physics, Texas A&M University College Station, TX 77843-4242.

Extensive experimental and theoretical studies have confirmed the "universal" nature of the relaxation that occurs at zinblende (110) and wurtzite (1010) cleavage surfaces. This relaxation can be parameterized in terms of a rapidly rigid bond rotation of the top layer atoms, with a "universal" tilt angle for each structure that is independent of chemical composition. No precise origin for this chemical independence, or transparent model of what happens to the orientation and character of the dangling bonds as relaxation proceeds, has yet been found.

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been proposed. In addition to enriching our understanding of the perfectly periodic surface, such a picture might well provide a semi-quantitative basis for describing more complex phenomena such as defect formation and photoreaction.

To address such questions, we have examined these relaxations within the context of a simple analytical model, considering those bond length conserving reorientations that preserve the symmetries consistent with experiments and more detailed theoretical calculations. We derive a general mathematical relationship between the top layer atom hybrid orbital energies and the constraining local geometry. Our results show a relaxation that is constrained by orthogonality limits on the amount of anion orbital rehybridization, so that the minimum energy configuration is determined explicitly and obviously by the bonding geometry, and not by atomic term values. This leads to a universal tilt angle for each structure that is in good agreement with the accepted values.

A particularly noteworthy feature of the model, in addition to its simplicity, is that it shows both tinbladed and wurtzite surfaces can be treated equivalently within a single framework, where the same driving forces and limiting factors govern the relaxation. The model furthermore provides an explicit representation for the hybrid character and orientation of the dangling orbitals during all stages of the relaxation.


We have probed the nonlinear optical properties of asymmetric GaAs/AlAs multiple quantum wells in the mid-infrared spectral range using the Z-scan technique and the Vanderbilt Free-Electron Laser (FEL). The Z-scan method gives an accurate and absolute measurement of the linear index of refraction and the nonlinear absorption coefficient while not requiring sophisticated instrumentation. The FEL is a source of high intensity (15 MW) radiation with tunable wavelength (1-8 μm) and tunable pulse width (0.7-1.5 ps). In a Z-scan measurement, a thin semiconductor sample is scanned along the optical axis (z-axis) of a focusing element causing strong nonlinear effects to occur as the sample moves through the beam waist. The nonlinear coefficients are obtained by fitting the normalized transmission spectrum to well-known analytic expressions. In the bulk semiconductors, the studies focused on the near optical band gap nonlinear refractive index and the two-photon absorption coefficient. In the quantum well materials, the intensity dependence of the intersubband separation was measured. Saturation effects and free-carrier absorption are also discussed. These are the first successful Z-scan measurements performed at the Vanderbilt FEL.


Si/ZnS multiple quantum wells with closely lattice matched ZnS barriers were grown on silicon substrates by metalorganic chemical vapor deposition (MOCVD) in a horizontal-flow reactor. Photoluminescence studies revealed emissions peaks which were blue-shifted with respect to the photon-assisted emission from bulk silicon substrates. The observation of an emission shift to shorter wavelengths is consistent with our calculations of quantum confinement effects in silicon nanostuctures. A high temperature (900°C) silicon buffer layer was first grown from silicon on both (100) and (111) oriented Si substrates, followed by the epitaxial ZnS cladding layer. Silicon films with thickness ranging from 3 to 15 nm were then deposited on the ZnS at low temperatures (350-500°C) using a selenide. A ZnS capping layer completed the structures. Multiquantum well structures featuring as many as six wells were grown, and emission peaks at wavelengths as short as 830 nm were observed. The Si thicknesses were determined by transmission electron microscopy. The presence of the Si quantum wells were further substantiated by SIMS depth profiling and from emission shifts detected by micro-Raman spectroscopy.

*Work supported by the US Department of Energy.

**NS-TuP9 Luminescence of Nanostructures Induced by Carriers Injected from a Scanning Tunneling Microscope Emitter, Lars Mattill, Joakim Lindahl, Mats-Erik Pistol and Lars Samuelson, Lund University, Department of Solid State Physics and The Nanometer Structure Consortium, PO Box 118, S-221 00 LUND, SWEDEN.

In recent years the interest for devices based upon the properties of low-dimensional structures has increased substantially. In order to reveal the electrical and optical properties of such structures scanning tunneling microscopy techniques have proved to be particularly useful. The main advantage with these techniques is the possibility to locally characterize the material. One such technique is the combination of local injection of carriers using a scanning tunneling microscope (STM) and simultaneous luminescence detection of the tip sample region. The modes of operation are either a mechanism that is similar to high-energy cathodoluminescence (CL) spectroscopy, where the injected carriers generate electron hole pairs which in turn recombine radiatively, or direct radiative recombination of the injected carriers with holes (or electrons). In this paper CL excitation and STM excitation will be compared. The sample studied is an AlGaAs/GaAs quantum well and quantum wire sample making it possible to inject carriers directly into a single quantum wire using STM injection.

**NS-TuP10 Spectral and Structural Features of Porous Silicon Prepared by Chemical and Electrochemical Etching Processes, D. F. Thomas, L. A. Jones, E. Nakamichi, L. Zachowiak, Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada. N1G 2W1.**

It is known that fluorescent porous silicon can be formed by both electrochemical and chemical etching procedures. The continuing effort to understand the formation mechanisms involved and the nature of the resulting photo- and electroluminescence is complicated by the wide variety of resulting structural features with relevant dimensions that span several orders of magnitude. Porous silicon was formed from both n- and p-doped Si(111) and Si(100) using both an HF-electrochemical process and a HF/HNO, chemical etchant. Both fluorescence and excitation spectra were acquired to analyze the spectral properties of the samples. The electrochemically prepared samples generally produced fluorescence peaked around 560 nm while the chemically prepared samples peaked around 610 nm. Excitation spectra show a monotonic increase in fluorescent intensity when the excitation wavelength is scanned from 500 nm down to 275 nm (the instrumental limit). One notable exception to these trends was a chemically prepared sample which showed blue fluorescence at 440 nm in addition to the orange fluorescence. The structural properties of these samples were studied by scanning electron microscopy (SEM) and scanning force microscopy (SFM). On the scale of microns, the surface features varied widely between sample preparation techniques. Three types of surface porosity are identified with remarkably different surface roughness characteristics. The correlation between sample preparation procedures, the resulting surface roughness, and the subsequent photoluminescence will be presented.


Scanning Electron Microscopy, Scanning Force Microscopy (SFM), Raman Spectroscopy, and Photoluminescence were used to characterize samples of highly optical efficient porous silicon. Preparation conditions of the samples were as follows: p-Si(100) substrates with electrical resistivity in the 1-2 ohm-cm range were electrically etched by means of an HF:H₂O:ethanol solution, at the proportion: 1:1:2. Anodization time was 15 minutes and the current density employed were 1, 5, 25, and 40 mA/cm². The Raman and photoluminescence measurements were correlated to the mesostructure as determined by SEM. By using images both from the surface as well as from the side it was possible to construct a three-dimensional model of the mesostructure of porous silicon. It is concluded that the presence of photoluminescence in this type of samples do not necessarily have its origin in the fibrous-type structures as reported by several authors.

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**NS-TuP12 The Size Effects Study on the Vibrational Properties of the Si Nanocrystalline Materials, H. F. Zhang, T. Jin, Y. P. Li, C. S. Wang, S. D. Xiu, Department of Physics, University of Science and Technology of China, Hefei 230026, P.R. China.**

With the growing study on the nanometer material and the porous silicon, it is obvious that the predominant reason for these materials having special properties is the size effect of the crystallites. As we know, not only the electronic structure and optical properties study are important aspects for us to explain the experimental results, but also the Raman scattering and infrared absorption study are also
important techniques, and the latter ones are correlated to the vibra-
tional properties. In this paper, The Extension Hukel Tight-binding 
method (EHT), the Recursion Method as well as the Dean method 
were used to study the vibrational densities of states on the real Si 
structure with the size from 4.70A to 50.7A were systematically studied. 
The results that when the crystallite sizes are different, the densities 
of states are varied (the peaks become sharp and shift to higher ener-
gies, as the size growing) are presented and discussed, which are in 
good agreement with experiment. On the other hand, when the number 
of the atoms in one crystallite is up to 3109, that means the diameter 
of the crystallite is 21.7A, the densities of states are the same more 
or less, and all close to that of the crystalline Si, these results agree 
well with the electronic structure study on the hydrogenated Si clusters.

NS-TuP13  Atomic Force Microscopy and Scanning Tunneling Microscopy 
Study of Ge1−xFx Nanophase Materials, Da-Ming Zhu, 
Department of Physics, University of Missouri-Kansas City, Kansas 
City, MO 64110, USA; Jin Shi, Department of Physics, University 
of Illinois at Urbana-Champaign, Urbana, IL, USA. 
We have conducted a study of nanophase Ge1−xFx materials using 
atomic force and scanning tunneling microscopes. Our goals are to 
characterize the size and shape distributions of nanoparticles in the 
materials and to detect possible interfacial diffusion of atoms between 
particles. The materials were synthesized via gas-condensation in an 
ultrahigh vacuum system. The condensed particles were compressed 
in situ to form thin disks before exposure to air. We have prepared 
a series of samples with Fe concentration varying from 0% to 86%. 
The sample with 6% concentration of Fe is almost insulating. We 
have obtained images with an atomic force microscope which clearly identify 
each individual nanoparticle. The sizes of the Ge1−xFx particles are 
very uniform, being about 50 nm. For the samples with high concentration 
of Fe, we have used scanning tunneling microscope to examine the boundaries between different particles, in an attempt to study 
the atomic diffusions between particles. We will compare our results with those of transport measurements conducted on the 
same samples.

NS-TuP14  Growth, Structure and Diffusion of Small NaCl Clusters 
on the (001) Surface of MgO, A. L. Shugler and A. L. Robb, 
The Royal Institution of Great Britain, 21 Albemarle St., London W1X 
4BS, U.K. 
Study of the stability, atomic and electronic structure and mobility 
of clusters adsorbed at insulating surfaces can provide deeper insights 
into the micro-mechanisms of growth of materials by molecular beam 
epitaxy and vapor deposition, and the formation of nanostructures. 
Comparison of the structural and optical properties of adsorbed clusters 
with analogous properties of the bulk materials and free clusters is 
important for further understanding of the properties of materials 
as a function of their dimensions. In this study we have investigated 
atmospheric-pressure nitric oxide desorption, stability and mobility of NaCl, (x = 1.36) clusters adsorbed on the (100) surface of MgO using atomistic 
simulation and quantum-chemical MO LCAO calculation techniques. 
Crystall and cluster optical absorption spectra were calculated using 
the configuration interaction technique. We considered attachment 
of new NaCl molecules to existing surface structures, modelling their growth. It was demonstrated that starting from Na16Cl16 clusters they 
begin to form two-layered rectangular structures which are isotropic 
and grow by filling the kink sites. The stability of the clusters increases 
slowly with their size. Due to a large mismatch of the MgO lattice 
constant and interatomic separation within the clusters, at large cluster 
sizes (x > 24) only average cluster-surface interactions play a role. 
The mobility of large clusters is anisotropic and much higher than 
that of small ones. The optical excitation energies and other electronic 
structure parameters are calculated as a function of cluster size and 
structure.

NS-TuP15  Atomic Force Microscopy of Au Implanted in Sapphire, 
D. O. Henderson, A. M. George, A. Burger, R. Mu, S. H. Morgan, 
Fisk University, Physics Dept., Nashville, TN 37208, R. H. Magruder 
III, Dept. of Applied and Engineering Sciences, Vanderbilt University, 
Nashville, TN 37235, C. W. White and R. A. Zahr, Oak Ridge National 
Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6057. 
Ion implantation is an attractive technique for modifying the near 
surface properties of a wide variety of materials. The implantation of 
Au into Al2O3 leads to the disruption of the long range order in the 
crystal producing an amorphous layer. Annealing the implanted crystal 
at 1373 K restores the crystallinity of the sample. The annealing also 
imparts a purple color to the Au implanted sample which is associated 
to the surface plasmon resonance of gold colloids. The width of the 
surface plasmon resonance depends on the colloid size, whereas the 
resonance frequency depends on the colloid shape. In order charac-
terize the size and shape of Au colloids formed by ion implantation, 
we have performed AFM measurements on Au implanted sapphire. 
Infrared spectra for the Au implanted sapphire are also reported and 
are discussed in terms of ion induced damage.

The research at ORNL was sponsored by the Division of Materials 
Sciences, under contract DE-AC05-84OR22400 with Martin Marietta 
Systems.

NS-TuP16  An STM Study of Molecular Intermediates in the 
Dissociative Adsorption of closo-1,2-dicarbododecanoborane on Si(111), 
I. M. Carpinelli and E. W. Plummer, Oak Ridge National Labs, Oak 
Ridge, TN 37831-4057; D. Byun and R. A. Dowben, University of 
Nebraska, Lincoln, NE 68588-0111. 
Closol-1,2-dicarbododecanoborane (B12H12) is a source compound 
for the deposition of a high resistivity form of boron-carbide (B4C) 
and has been used to make Si:B/Si(111) heterojunction diodes [1]. 
The goal in employing this source compound is "direct writing" or selective 
area deposition of boron-carbide using very short wavelength (high 
resolution) radiation. For success in the direct writing technique, the 
source molecule must exhibit a molecular precursor state on Si(111). 
Using an STM, we have imaged such molecular isosahedra after ex-
posure to closo-1,2-dicarbododecanoborane. Prolonged exposure to our 
source leads to a reconstruction of the Si(111)-7×7 surface. Based 
upon Auger electron spectroscopy data, we postulate that following 
dissociation of the surface intermediate species, some boron atoms 
substitutionally occupy selvedge sites. This selvedge layer of boron 
is believed to cause the surface reconstruction.


NS-TuP17  Fabrication and Optical Characterization of Aluminum 
Nanometal/Porous Aluminum Oxide Thin Film Composites, 
C. R. Martin, G. L. Hornyk, B. P. Berggren, 
Department of Chemistry, Colorado State University, Fort Collins, CO 80523. 
an aluminum nanometal inclusion/porous aluminum oxide host 
composite thin film is predicted to be transparent in the visible range 
of the spectrum so long as the dimensions of the particles are very 
small (circa 10 nm) according to our computer simulations. The 
theoretical plasmon resonance absorption maximum derived from these 
simulations is near 190 nm wavelength (please consult the figure given 
below). We present an experimental strategy for fabrication of these 
transparent composite thin films by means of electroplating in a to-
luene/aluminum bromide/potassium bromide/triethylborohydrosilane 
solution under an inert atmosphere blanket.

For aluminum oxide composites which contain nanometals of greater 
dimensions, where scattering phenomena cannot be neglected, we em-
ploy our dynamical Maxwell-Garnett effective medium theory model 
to explain spectral characteristics. In addition to the apparent trans-
parency as described above, these composite films can be made to 
possess electrical conductivity with respect to the reverse direction 
of the film. Film structures are characterized by transmission and 
scanning electron microscopy.

NS-TuP18  Nanometer-Scale Observations of the Corroded Surfaces 
of Metallic Glasses, Y. Watanabe, Y. Nakamura and S. Hayakawa, 
National Defense Academy, Yokosuka, Kanagawa 239, Japan.
In order to study the influence of crystallization on corrosion re-

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sistance of metallic glasses, surfaces of as-cast and annealed metallic glasses, Fe$_2$Ni$_2$Mo$_6$Bi$_4$, are observed with atomic force microscopy and the change in the surface morphology before and after corrosion by nitric acid is examined on the nanometer-scale. On the surface of the as-cast specimen before corrosion, several protrusions with a height of less than 50 nm are observed on the smooth matrix, while on the surface of the as-cast specimen after corrosion the matrix becomes rough and the protrusions are found to grow although the corrosion has occurred at room temperature. This may be explained by the assumption that (1) protrusions are metal boride crystallites and they are not corroded by nitric acid because of its iron-rich composition and (2) crystallites buried under the matrix has appeared by corrosion of the matrix. On the surface of the 873 K-annealed specimen before corrosion, the surface is covered with protrusions of 100 to 200 nm height and the smooth matrix is also observed, but the surface of the annealed specimen after corrosion is almost covered with protrusions with projections of less than 100 nm and the smooth matrix has disappeared. These observations suggest that during corrosion large crystallites have been peeled from the surface because the corrosion of the matrix progressed. This peeling of crystallites seems to result in deterioration of corrosion resistance of metallic glasses after annealing.

**NS-TuP19** Dynamical Characteristics of Low Frequency Current Fluctuations from Local Areas of Semiconductor Field Emitter Surfaces, R. Z. Bakhitzon and S. S. Ghosh, Department of Electronic Engineering, Bakhitzon State University, Ufa, 4500074, RUSSIA.

Semiconductor field emitter surfaces are by no means static and surface structure changes of nanometer scale can occur spontaneously near or slightly above room temperatures especially if high electric field is applied. Such changes can be studied simultaneously both in terms of space and time using conventional Scanning Probe Methods (STM, AFM), whereas they give rise to current fluctuations. In this paper we describe the dynamical characteristics of elementary low-frequency current fluctuations which originate from local surface areas of the p-type Si field emitter. We also present the results of noise measurements providing the possibility of single atom movement observation within the local areas of 10 nm. All types of linear elementary fluctuations, i.e., oscillating, relaxing and aperiodic processes as well as non-linear oscillating (i.e., telegraph form) processes are considered.

**NS-TuP20** Many-Particle Effects in Tunneling Electrons from Metal and Semiconductor Surfaces, M. A. Kozhushner, G. K. Ivanov, Institute of Chemical Physics, Moscow 117997, RUSSIA and I. I. Olcinit, Volgograd State University, Volgograd 400087, RUSSIA.

We develop theory of tunneling processes in many-particle systems on the grounds of Lipman-Schwinger integral equation that gives us possibility to calculate the asymptotics of many-electron wave functions with accuracy provided by standard quantum-chemical methods in the main region of electron localization (MREL) [1]. In this approach, interaction between electrons and inter-electron correlations are naturally included into consideration.

Application of the theory to calculate parameters for tunneling from metal surfaces displays two main effects of interaction between electrons. First, the effective tunneling potential depends on topology of Fermi surface, quantum numbers of the tunneling electron and does not coincide with widely used image potential. Second, tunneling transition is accompanied by inter-electron exchange of energy, that results in effective change of tunneling probability energy dependence.

We obtained general expression for tunneling current in STM, that gives the possibility of solution of reverse STM problem—extrapolation of wave functions data for surface and tip atoms from tunneling current measurements. The specific examples of STM imaging in case of graphite, Cu and Au surfaces illustrate the general scheme of direct and reverse STM problems' solution.


**NS-TuP21** STM/STS Investigation of Ion-exchanged Polimer Multilayer LB Films: 2D Conductivity, Resonance Tunneling, Charge Superstructure, N. Maslova, Yu. Molskov, V. Nikanorov, V. Panov, S. Sudnov, R. Yusupov, Moscow State University, Physical Department, 119899, Moscow, Russia.

The Langmuir-Blodgett (LB) films of ion-exchanged polymers have been investigated by scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) methods.

For the ion-exchanged polymer films with odd layers number some peculiarities have been discovered. In this case the dV/dI(V) dependencies have a peak (local maximum) with the width about 250 mV near the zero bias. Surface reconstruction corresponding to charge density wave and the oscillating character of I(V) dependencies have been observed. The STM feedback loop generation was observed when the setpoint of STM is in the negative slope area of I(V) curve.

This effects are explained by the theoretical model that the overall charge density of states is determined by the superposition of effects connected with the two-dimensional conductivity character and charge density wave.

**NS-TuP22** Determination of Large Heterogeneous Rate Constants Using Nanocone Electrodes, Vhoo P. Menon and Charles R. Martin, Department of Chemistry, Colorado State University, Fort Collins, CO 80523.

A method for the determination of heterogeneous rate constants for electrochemical reactions has been described. This method involves the use of highly oriented polycrystalline carbon electrodes in conjunction with a scanning electrode to perform a series of high-precision measurements. These measurements are made in order to determine the heterogeneous rate constant for a particular reaction. The rate constant is determined from the current-voltage data obtained at the scanning electrode, which is used to provide a means of controlling the current density of the electrode. The rate constant is determined by fitting the current-voltage data to a theoretical model and determining the parameters of the model that provide the best fit to the data. The parameters of the model are then used to determine the heterogeneous rate constant. The method has been applied to the determination of heterogeneous rate constants for a number of different reactions, and the results have been compared with those obtained by other methods.

**APPLIED SURFACE SCIENCE**

Room BR4 — Session AS-TuP

Aspects of Applied Surface Science

Moderator: D. R. Baer, Battelle Pacific Northwest Laboratories.


Thin polythiophene films, prepared using a vacuum evaporation process, have been studied using x-ray photoelectron spectroscopy (XPS). Emphasis is placed on the chemical structure of the deposited films and the results are compared to films synthesized using more conventional chemical or electrochemical methods. The films were deposited, with a starting material of polythiophene powder, using a resistively heated quartz crucible under high vacuum conditions. The as-deposited films have a mirror-like quality and the XPS analysis of the film, which is compared to that of the initial powder, indicates that no chemical change occurs during the deposition process. Results are also presented concerning the oxidation of these films in an iodine environment and this analysis is related to conductivity measurements of the vapor-deposited films.

Supported by the National Science Foundation under grant No. DMR-9217526 and the NAU Organized Research Program.


Densely packed and highly ordered thin films of various phthalocyanines are obtainable by deposition from solution, provided that side chain modification of the Pcs's has been carried out to improve processability. Silicon phthalocyanines can be polymerized along the central axis, to form an O-O-Si linkage, which provides for covalent packaging of the cofacial Pe rings—processability as LB thin films is produced through various alkane side chains (4 per ring system). Divalent metal Pcs's (CuPc and ZnPc), with the appropriate side chains, will also organize during simple solution casting, and/or through the formation of LB thin films. Both types of thin films show a high
degree of organization, which impacts on both the electrochemical and optical properties of these assemblies. The degree to which electrochemical oxidation of the P circles is possible is difficult to determine—however, since these films are vacuum compatible, surface analysis techniques such as angle resolved XPS can be used to determine oxidation stoichiometry (i.e., the degree of counter ion incorporation into the near surface region of these thin films).

AS-TuP3 Metal Overlays on Self-Assembled Monolayers: 5. ISS and XPS of the Ag/COOH Interface, G. C. Herdt, and A. W. Czander, NREL, Golden, CO 80401.

The purpose of our research is to study the interactions or processes between vacuum deposited metals and self-assembled monolayers (SAMs) under controlled conditions. In this work, XPS and ISS were used to characterize mercaptoundecanoic acid (MUA, H\(\text{S(CH}_2\text{)_11COOH}\)) and mercaptohexadecanoic acid (MHA, H\(\text{S(CH}_2\text{)_16COOH}\)) SAMs before and after depositing up to 1.0 nm Ag at ca. 10\(^{-1}\) torr. The SAMs were prepared by self-assembly onto gold films on (100) silicon substrates in an ethanolic thiol solution. XPS spectra indicate that no strong interaction occurs between the deposited Ag and the COOH organic functional group (OFG). ISS compositional depth profiles (CDPs) for Ag on MUA and MHA (this work) and octadecanethiol (ODT, H\(\text{S(CH}_2\text{)_16CH}_3\)) CDPs are compared over a temperature range of 112 to 293 K. The ISS results indicate that Ag remains on the surface of MUA and MHA for up to 1 h after deposition, whereas Ag penetrates ODT in less than 5 min at 295 K. The time dependence of the slower Ag penetration through MUA and MHA will be compared with that for ODT at temperatures below 295 K.

Although Ag/OFGs are expected to have relatively weak interactions, the Ag/COOH system was anticipated to be more interactive than was found, so rapid penetration of Ag through the COOH SAM is an unexpected result.


*Performed under DOE contract DE-AC02-83CH10039.


Self-Assembled monolayer films (SAMs) allow the construction of thin organic films with highly specific properties. The effect of hydrocarbon chain length on the orientation of these films has been extensively characterized using contact angle, ellipsometry, external-reflection infrared spectroscopy, and electron spectroscopy for chemical analysis. However, the effect of hydrocarbon chain length on the composition of films formed from solutions of mixed adsorbates has been studied less extensively. In the current study, static secondary ion mass spectrometry (SSIMS) is used to address this question. Specifically, quantitative techniques that have been developed for the analysis of monolayer films will be utilized. These techniques, which involve the quantification of molecular secondary ions as a function of ion dose, have been used to determine the composition of mixed monolayer Langmuir-Blodgett films. Results from mixed monolayer Langmuir-Blodgett films are used to quantify Self-Assembled monolayer films. Surface excess of long chain acids in mixed Self-Assembled monolayer films are quantified.

AS-TuP5 Temperature Controlled TOF-SIMS Investigations of Polymer Materials, M. Deimel, B. Hagenhoff, A. Benninghoven, Physikalisches Institut der Universität, Wilhelm-Klemm-Str. 10, D-48149 Münster, Germany.

The temperature dependence of secondary ion (SI) yields can give a deeper insight into the ion formation process in SIMS. Up to now, however, such investigations were limited to relatively small molecules adsorbed on clean metal surfaces due to the use of quadrupole instruments. We added a device for temperature control to a time-of-flight (TOF) mass spectrometer such making use of the higher accessible mass range and sensitivity of TOF analyzers. We investigated the temperature dependence of SI yields for several polymers including Polystyrene (PS), Polydimethylsiloxane (PDMS), Polymethylmethacrylate (PMMA), a perfluorinated polyether (Krytox) and Polyethylene glycol (PEG) prepared as monolayers on different metal substrates. The results show that the temperature behaviour in principle is independent of the respective substrate material. Intact polymer molecules disappear from the surface at well defined temperatures when the target is heated. For PEG a common disappearance temperature \(T_D = 500\) K is observed independent of the polymer chain length caused by a thermal fragmentation process. For other polymers the observed desorption temperatures increase with the number of repeat units. From these observations we determined the activation energy for the desorption as a function of the polymer chain length according to the Polanyi-Wigner model.

AS-TuP6 Radiation Damage on Some Organic Materials During SIMS and XPS Analysis, M. Kudo*, T. Nishihara* and T. Hoshi*.

*Seikei Univ., Musashino, Tokyo 180, Japan, **ULVAC-PHI Inc., Chigasaki, Kanagawa 253, Japan.

It is generally recognized both Static-SIMS and XPS are non-destructive analytical techniques. However, in S-SIMS, the obtainable mass spectra change a great deal according to the primary ion dosage. In XPS, both monochromatic and non-monochromatic x-ray sources also cause sample damages, the latter, generally, more serious due to the higher energy Bremsstrahlung x-rays irradiation. Although some systematic analysis data have been presented for some typical materials, the detailed interpretations and standard criteria for practical analysis have not been established yet.

In this study, we investigated systematically the damages on some radiation sensitive materials such as Poly Tetrafluoroethylene (Teflon), Poly Caprolactam (Nylon 6), Poly Carbonate (PC), Poly Ethylene Terephthalate (PET) and some others. The S-SIMS mass spectra were recorded at an appropriate dosage intervals, each followed with XPS analysis. The x-ray irradiation damage was also evaluated using both XPS spectra and S-SIMS mass spectra analysis. The results are discussed in terms of the dependence of damages on the chemical bonding of the materials investigated and on the energy and beam densities of the primary probes, as well as differences in nature between the ion and photon induced damages.

AS-TuP7 XPS Characterization of Nylons, Sanakar N. Raman, Physical and Analytical Science Center, Monsanto, 730 Worcester Street, Springfield, MA 01151.

Nylon is a material of very high commercial importance, in the aviation industry, as fabrics, numerous molded parts and carpets. In this paper XPS spectra for a variety of Nylons is presented. Spectra for Carbon, Oxygen and Nitrogen and the entire energy range, is presented along with curve fit results for C 1s, O 1s and N 1s peaks. The concentrations and curve fit results are consistent with theoretical predictions. These results are extremely useful reference data in the characterization of commercially manufactured nylons. The nylons studied here are pure materials with no additives. In the real world commercial case there may be additives and other materials incorporated in nylon to improve performance, which will tend to change the chemical bonding thereby leading to slightly different peak shapes and different atomic concentrations.

AS-TuP8 Surface Characterization of Silylated Substrates by TOF-SIMS, B. Hagenhoff, M. Deimel, D. Rading, A. Benninghoven, J. Grohe, Universität Münster, D-48149 Münster, Germany.

The treatment of hydroxylated surfaces with functionalized silanes is a commonly used modification technique in technological areas ranging from biosensing to the environment. The use of silylated materials. Silylated substrates are normally analyzed by IR and NMR techniques providing information on surface near layers and the bulk material. The knowledge about the molecular structure and composition of the uppermost monolayer which governs important surface properties like wetting, adhesion, and long-term stability, however, is still sparse.

We applied time-of-flight secondary ion mass spectrometry (TOF-SIMS) which offers molecular information about the uppermost monolayer with high sensitivity to the characterization of hydroxylated surfaces modified by organosilicon compounds RSiX₃ (R = CH₃, Ph, X = OCH₃, OC₂H₅, Cl). In order to be comparable with standard application process of protective agents the silanes were allowed to react with the substrate for three weeks in ambient air without additional amounts of water. The spectra reveal that in all cases the uppermost monolayer consists of totally hydrolyzed polymeric siloxanes which do not contain the reactive group X (siloxiquoxanes of the structure (CH₃SiO)₃ X). A comparison with additionally performed IR and NMR experiments shows that the surprising occurrence of siloxanes under the reaction conditions used is definitely restricted to the uppermost monolayers.

AS-TuP9 Characterization of an Abrasion Resistant Coating of Polycarbonate, R. M. Friedman*, W. W. Linton* and M. P. Mason**, Analytical Sciences Center, Monsanto Corporate Research, St. Louis,
MO 63167-0001 and *Department of Chemistry, University of North Carolina, Chapel Hill, NC 27599-3290.

Plastics and polymers are being used increasingly in place of traditional materials in construction because of their light weight, impact resistance, and low cost. Unfortunately, plastics are generally soft and subject to abrasion, fracture, or other forms of mechanical failure. To offset these limitations, surface coatings are being used to protect against degradation. Two key properties of the coatings are hardness and clarity reflecting the chemical composition and microstructure of the material. For example, glassy polycarbonate, marketed under the trademarked name LEXAN®.MR5 with an abrasion resistant surface, MARGARID®. The material is described in an extensive array of patents, but there is little discussion in the literature concerning its molecular or microstructural characterization. A commercial sample of the material was analyzed by TOF-SIMS, angle-resolved XPS and ATR-FTIR. TOF-SIMS yielded identification of the surface siloxyl building blocks through analysis of characteristic fragment ions. ATR-FTIR helped to differentiate between molecular species with the same empirical formula, e.g., SiO₂H₂(OCH₃) and SiO₂(OH)(CH₃), as well as to indicate a gradient in composition within the surface coating as confirmed by angular resolved photoemission. Coupling the TOF-SIMS results with the quantitative elemental composition determined by XPS yielded detailed insight into the nature of the siloxane surface coating. The correlation of analytical techniques with different sampling depths (TOF-SIMS < XPS < FTIR) and information about long range molecular structure (XPS < FTIR < TOF-SIMS) provides a broadly applicable approach to characterizing abrasion resistant coatings.

*Present address: DuPont Co., Wilmington, DE.

AS-Tup10 Fundamental Characterization of Siloxane Adhesion Promoters on Metal and Metal Oxide Surfaces, Sourabh Mishra and Jeffrey J. Weimer, Joint Materials Science Ph.D. Program, University of Alabama in Huntsville, Huntsville, AL 35899.

Silane adhesion promoters have the form Xₙ-Si-Rₙ, where R is an organic functionality and X is a group that hydrolyzes to form a siloxane. They are used extensively to enhance bonding of polymers to metals, however the fundamental bonding mechanisms are not well characterized. Sililation of Cu and oxidized Cu surfaces using trimethyl-chloro- and trimethyl-methoxy-silane has been investigated to determine the mechanisms involved in bonding. Compositions and oxidation states of elements on the substrates were determined using x-ray photoelectron spectroscopy (XPS). Glancing incidence Fourier transform infrared spectroscopy (FTIR) was used to characterize vibrational frequencies at the immediate promoter-metal interface. Silane deposition was carried out using wet chemistry techniques, and the layers were cured under vacuum at high temperatures. The silane reduced oxides on the surfaces during sililation, and curing led to removal of residual chlorine and unbound chlorosilane. Surface oxides were not reduced by the methoxysilane until curing. The results signify the relative importance of physisorption and hydrogen bonding of silane prior to formation of a covalently bonded siloxane. This work is being continued with chloro-, methoxy-, and other monofunctional silanes used in bonding polymers and proteins to biocompatible and metal surfaces.

AS-Tup11 Film Growth and Surface Structure of Methylen Blue on Mica, G. Hahn, W. R. Casorri, and N. D. Spencer, Materials Dept., ETH Zurich, CH-8092 Zurich, Switzerland.

Methylene blue (MB) is often used to determine specific surface areas of high-surface-area materials. The molecule is generally considered to be a rectangular parallelepiped and to adsorb in a flat orientation. We have investigated the adsorption of MB on a mica substrate with AFM/LFM, XPS, ToF-SIMS, and NEXAFS in order to monitor film growth, film thickness, coverage, homogeneity, and the orientation of the molecules. It was found that the growth depends critically upon the preparation conditions. Under one of the experimental conditions a film that crystallizes but is not detected. These results may also have some impact on our understanding of the surface properties of layered silicates, which are of ever increasing interest in colloid science.

AS-Tup12 Nanometer Resolution Imaging of the Growth of Water Monolayers on Mica from its Vapor, Jyun Hu, Xiu Xiong Xue, D. F. Ogletree and M. Salmeron, Material Science Division, Lawrence Berkeley Laboratory.

For the first time we have been able to image with nanometer resolution the layer by layer condensation and evaporation of water films on mica as a function of humidity. This has been done using a non-contact AFM imaging method based on the polarizability of materials under applied electric fields. Using a biased AFM tip and an insulting mica surface, the polarization forces can be detected from a few hundred A's away from the surface. We found that wetting proceeds by formation and growth of 2D islands. The islands often adopt polygonal shapes with edges that are aligned with the mica crystallographic directions. This suggest ordering of the molecules beyond the first layer, as in ice. Studies of the time dependence of the polarization force due to the motions of charges and molecular dipole will be presented as well.


Atomic Force Microscopy (AFM) was used to characterize the influence of various cleaning and surface finishing processes on potassium dihydrogen phosphate (KDP) crystals used for nonlinear optical applications. Diamond turning off the surface results in a visible periodic structure, with 0.2-0.3 micron sized features distributed along the path of the diamond cutting tool. Various surface cleaning methods, including oxygen plasma cleaning, were investigated. Surface morphology and degradation (fogging) were related to process history and environmental factors.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

AS-Tup14 Counterion and Dopant Induced Effects on the Structure of Electropolymerized Polyacrylamide Thin Films, T. L. Porter, A. G. Sykes, Northern Arizona University, Flagstaff, AZ 86011.

The transport of charge in polyaniline and polyaniline based materials is highly dependent upon a number of chemical and structural factors. Polyaniline in an amorphous state may display the characteristics of a Fermi glass. Polyaniline in a semicrystalline state exhibits metallic properties on a local scale and characteristics of a granular conductor on the macroscopic scale. The structural state of polyaniline in turn is determined largely by the chemical preparation parameters. We have used the technique of scanning probe microscopy in the non-contact mode to study the dependence of electropolymerized film nanostructure on several of these parameters, including choice of counterion, dopant concentration and film thickness. We have obtained the first direct, real space evidence of a nanometer-scale island structure in HCl doped unsubstituted electropolymerized polyaniline. The effects of chemical preparation on the film structure and the resulting conductivity will be discussed in terms of models based on a granular conductor. Earlier studies on polyaniline substituted polyanilines using SPV in the contact mode will also be compared to the noncontact results presented here.

AS-Tup15 Contribution of Surface Roughness in the Nanometer Regime to the Depth Resolution in Sputter Profiling, A. Pischon, S. Hoffmann and A. Zalar, Laboratory of Processing and Heat Treatment of Materials, Vuerimientie 2 A, FIN-02150 Espoo, FINLAND

Max-Planck-Institut für Metallforschung, D-70174 Stuttgart, Germany, Institut für Elektronen und Vacuum Technik, P.O. B. 59, 61111 Ljubljana, Slovenia.

Ion bombardment induced surface roughness is one of the most important effects degrading the depth resolution in sputter profiling of thin films. Because usual depth resolution measurements at abrupt bilayer interfaces always involve many other possible contributions, particularly ion induced atomic mixing, it is important to subtract these effects from each other. Scanning force microscopy (SFM) is ideally suited to obtain a precise measurement of the surface roughness within the analyzed area, typically 10 × 10 µm², in AES depth profiling. Comparison of Ni/Cr multilayer sample roughness after sputtering with and without sample rotation showed a considerable variation of the surface roughness between 2 am and 20 nm. The nanomorphology of the Ni and Cr layers is different and can be correlated with the difference in the physical properties of Ni and Cr. Surface roughness distributions will be given and SFM tip effects on the achieved surface roughness values will be considered.

AS-Tup16 Influence of Ion Species, Ion Energy and Substrate on Sputtering-Induced Ripple Topography, John J. Vayo, Robert E. Doty and EunHee Cirlin, Hughes Research Laboratory, Malibu, CA 90265.

The influence of ion species (O²⁻, N²⁻, and Ar⁺), ion energy (1 to
10 keV) and substrate (Si, Ge, and Al,Ga). As with \( x = 0 \) to 0.8) on the sputtering-induced formation of surface ripples has been studied using changes in secondary ion yields, scanning electron microscopy and atomic force microscopy. Attention is focused on the initial stages of ripple formation, which we characterized primarily by the ripple wavelength as a function of ion-substrate combination and ion sputtering conditions. Depth profiling using 0.5 keV Ar+ together with Auger electron spectroscopy was used to determine the amount of retention and the distribution of O2 and N2 primary ions. For O2 sputtering of Si at an angle of incidence of 40° from the normal the ripple wavelength increases linearly from 1.5 to 9 keV. Sputtering of Si with N2 also produces ripples but with a shorter wavelength. This difference between O2 and N2 is interesting because the ballistic aspects of both ion species are similar. The ripple wavelength is also found to decrease with increasing Al content in Al,Ga. As alloys. Ripples do not form when sputtering any of the substrates with Ar+, or on Ge with any of the sputtering conditions used. These results are discussed in terms of several mechanisms they may initiate ripple formation such as surface diffusion, surface stress, retention of primary ions, and variations in sputtering yields.

**AS-Tup17** Use of Resonance Ionization Microprobe Analysis in Material Sciences, Heinrich F. Amlinghaus, Charles F. Joyner and Tom J. Whitaker, Atom Sciences, Inc., Oak Ridge, TN 37830.

We have built a new analytical time-of-flight instrument capable of sputter-initiated resonance ionization microprobe (SIRIMP) measurement. This instrument can obtain quantitative element concentration images with high spatial resolution and virtually no matrix effects. The SIRIMP technique is especially valuable for ultratrace element analysis in samples where the complexity of the matrix is frequently a serious source of interferences. The instrument utilizes three different ion guns for sputtering, a liquid metal gallium ion gun, a mass-filtered microbeam ion gun and a mass-filtered low energy sputtering ion gun which can be used either independently or simultaneously.

In our presentation we will describe the implementation of the new SIRIMP system to solve a number of analysis problems and illustrate its salient characteristics with data from a wide range of applications. Results presented will include a) depth profiles of dopants implanted in various matrices, b) studies of surface and bulk contaminations in semiconductor samples and c) high-resolution lateral distributions of trace elements in semiconductors. The practical capabilities of SIRIMP to determine trace elements as a function of depth and lateral position in semiconductors will be discussed.

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This work was partially supported by the Advanced Research Projects Agency.


Adhesion between polymers, or between a polymer and a more rigid solid, is difficult to study without destroying the bonded interface. Post-failure analysis of rupture surfaces is only indirectly related to the nature of the system prior to failure. We have developed a non-destructive method of extracting the vibrational signature of polymers as a function of depth in a laminated construct. Our approach is based upon Raman scattering. It employs high precision control of the evanescent field beyond a dielectric under internal reflection conditions. Through detailed angle-dependent measurements, and the use of LaPlace transforms, we are able to reconstruct the spatial distribution of molecular structure. Demonstration experiments on model systems related to polymer adhesion are presented.

*This research was supported by the Office of Naval Research, contract No. N00014-93-1-0101.

**AS-Tup19** Thickness Determination of Uniform Overlayers on Rough Substrates by Angle Dependent X-ray Photoelectron Spectroscopy, P. L. J. L. C. M. Jansen and J. W. Niemanse, Schult Institute of Catalysis, Eindhoven University of Technology, PO Box 513, 5600 MB Eindhoven, the Netherlands.

It is well known that ADXPS can be used for determining the thickness of thin overlayers. In the straight-line approximation to photoelectron transport a simple expression relates the intensity ratio of overlay and substrate signals to the thickness of a uniform overlayer on a flat substrate. This expression is an very popular one and has been used often to evaluate the thickness of all kinds of overlayers on flat as well as rather rough substrates. We have analysed the errors involved in applying the simple expression mentioned for thickness determination of overlayers on rough substrates. We have used an algorithm for simulation of fractional Brownian motion to model substrate roughness in a realistic way on the computer and then calculated signal intensities by a Monte Carlo method for electron trajectory simulation. Our calculations indicate that the local steepness of the surface determines the deviation of XPS signal intensities as compared to those of a flat surface. We have applied Atomic Force Microscopy and ADXPS on aluminium foils and roughened silicon wafers, and confirmed the calculated link between local steepness and signal intensity.

The errors made in thickness evaluation based on the simple flat substrate model can be as large as 50% or more. An important result of our work, however, is that the error is only a few percent for an off-axis angle (between analyser and surface normal) of 35°, independent of roughness, overlay thickness, and type of material.

**AS-Tup20** Angle-Resolved XPS Data Acquisition, B. J. Tielisch and J. E. Fulghum, Chemistry Department, Kent State University, Kent, OH 44242.

In order to develop standard methods for angle-resolved XPS (ARXPS), it is important to evaluate not only data analysis methods, but also the effect of data acquisition conditions. Comparisons of algorithms have shown that a careful choice of algorithm parameters is needed to obtain reasonable and reproducible results. However, practical variations in data acquisition parameters have remained largely unexamined. The effect of the range and number of angles for which data is acquired, the signal-to-noise ratio of the data set, and variation in analyzer acceptance angle will be discussed for a variety of sample types and algorithms. We will demonstrate that for some sample types, it is possible to obtain useful information from noisy data acquired at only two or three angles. Practical guidelines for ARXPS data acquisition will be outlined.

This work is partially supported by the W. M. Keck Foundation and the NSF Science and Technology Center for Advanced Liquid Crystalline Optical Materials (ALCOM) under DMR-89-20147.

**AS-Tup21** XPS Studies of Compositional Changes Induced by Ion Bombardment on LaPO4, Surface, O. P. Ivanova, A. V. Naumkin, L. A. Vasiliev, V. I. Rakovsky, Research Center for Surface and Vacuum Investigations, 6 bld.1, Kazarmenny per., 109028 Moscow, Russia.

It was previously shown that ion-induced chemical processes play an important role in formation of altered layer. We have investigated compositional and chemical changes in LaPO4 bombarded by 6 keV Ar+ ion bombardment in terms of possible chemical reactions between decomposed species during relaxation process. It is clear that ion energy is large enough to disrupt numerous chemical bonds and to form decomposed components in the near surface region. The relaxation process depends on the preferential sputtering and chemical activity of decomposed species, the formation of thermodynamically stable phases and on the structure of the undamaged layers. The chemical composition of altered layer was determined by the 01s and 2p2 lines curve fitting procedures. The intensity ratios separation and binding energies were obtained from the initial surface spectra measurements of LaPO4 and LaO. It was found that chemical composition may be presented as LaPO4, LaPO4, PO4 and LaO, with relative intensities of 0.3, 0.22, 0.09 and 0.38 respectively. The chemical aspects of preferential sputtering are discussed.


Ceria is an important component in current catalyst formulations for the removal of CO, hydrocarbons, and nitrogen oxides during the oscillatory behavior present in automotive exhaust. The amount of oxygen storage in the converter correlates with the ceria content in the catalyst. We have studied the effect of thermal aging on the oxygen storage/release and activity in a 50% Pd/10% Ce catalyst. X-ray photoelectron spectroscopy (XPS) was used to measure changes in the oxidation state of the catalyst after various oxidation and reduction treatments and rate measurements of the CO oxidation reaction were used to determine the reaction kinetics and activity. These measurements were then compared to the oxygen storage/release characteristics measured in a pulse reactor system. The catalysts were tested fresh and after cycled aging at 1000°C. To de-
termine which Ce species were present on the surface, we fit the XPS Ce(3d) data with combinations of spectra from standard materials (CeO2 and Ce2O3). Acting as Pt/Rh catalysts causes a loss of oxygen storage capacity by forming compounds which do not allow the Ce to cycle between oxidation states. For the fresh catalyst, the observed kinetics for CO oxidation reaction show all of the signatures attributable to a catalyst with a high degree of ceria/noble metal interaction. Changes in the kinetics upon aging are consistent with a loss of ceria/noble metal interaction, and the precipitation of the oxygen storage in the Pd/Co catalyst was found to be similar to the Pt/Rh/ Ce catalyst. The XPS results correlate well with the oxygen storage measurements from the pulse reactor system.


We have investigated the quantitative depth profiling of oxygen content in SrTiO3 substrates and SrTiO3 films on SrTiO3 substrates using 35O(0) and 37O(0) 0.245 MeV resonant backscattering spectrometry. SrTiO3 films were evaporated onto SrTiO3 substrates by MBE methods with an hexadecagon reaction O2 gas, at(0) and at(0) resonant backscattering was corrected and an absolute volume of oxygen concentration could be accurately estimated with other metallic elements. For SrTiO3 substrates, one SrTiO3 substrate fabricated at A company had a different stoichiometry from a chemical analysis (namely, that is Sr:Ti:O = 1:1:3) and indicated the remarkable deficiency of Sr and oxygen content, while one SrTiO3 substrate fabricated at another company had a chemical stoichiometric composition. SrTiO3 films composed strongly affected by the compositions of SrTiO3 substrates, and nearly constant depth profiling results were obtained from film surfaces to substrate surfaces in spite of O2 flux densities. These results are consistent with depth profiling results of SAL-AES performed as cross checking. Continuity of oxygen volume at the interface will be demonstrated for SrTiO3 films on SrTiO3 substrates and other superconducting films on SrTiO3 substrates.

AS-TuP24 Negative Charge Production from Bombardment of a Cu Surface by Low Energy Oxygen Atoms and Ions,* Thomas M. Stephen, R. C. Amme and Bert Van Zyl, Department of Physics, University of Denver, Denver, Colorado 80208.

At atomic beam apparatus capable of producing oxygen atoms and ions has been used to investigate negative charge production from copper, as well as stainless steel, surfaces over a range of energy of 15 eV to 500 eV. The yield in all cases rapidly approaches zero as the beam energy diminishes towards approximately 1 eV; a ten percent yield is observed for all species at beam energies in the vicinity of 150 eV. The neutral oxygen atoms are generated by electron photodetachment of O2 in an extended laser cavity producing visible radiation (ca. 450 to 530 nm wavelength). A 25W Spectra Physics argon ion laser was employed for this purpose; the cavity radiation was chopped at 40 Hz and the resulting ion currents measured to permit absolute neutral beam intensity determinations. The neutral beams are 100% ground state O(3P), since no excited states are accessible for the ions and radiation employed. A model will be discussed which provides fair agreement for the negative charge production observed.

The paper will report the effect that biasing of a sample has on the properties of a reactive etching process. When aluminum is etched using a sputter-field argon ion etcher with nitrogen as the etching gas the aluminum metal is first converted into an oxygen-nitride. The oxy-nitride is formed because of residual oxygen in the etching gas. If etching is continued past an initial two minute etch, then the surface continues with approximately the same composition. Surface composition is determined by core and valence band X-ray photoelectron spectroscopy, with the valence band data interpreted by spectra generated by multiple scattered Xo calculations.

If the aluminum sample is subjected to a 15 volt d.c. negative bias, then oxy-nitride is formed after the initial two minute etch, but continued etching leads to the formation of a surface that is almost entirely aluminum oxide. In addition the case of the biased sample a number of the XPS features are seen, including nitride, adsorbed N2, and a peak at high binding energy due to implanted N2. This latter peak is only seen when the sample is negatively biased. The paper will suggest reasons for the chemical changes that occur when the sample is biased.

This material is based upon work supported by the Air Force Office of Scientific Research under Grant No. F496620-92-J-0144.

AS-TuP25 The Effect of Copper Overlayers on the Thermal Stability of Chromium Carbide on HOPG, J. J. Bellina, Jr., Saint Mary's College, Notre Dame, IN 46556 and K. A. Briggman, Northwestern University, Evanston, IL 60208.

Recent reports indicate that the wetting of graphite by copper improves with the addition of small amounts of carbide-forming metals, such as chromium. Since there are applications for these copper-graphite materials, the formation and thermal stability of chromium carbide films on graphite with and without copper overlayers have been studied. Our results from reactions of chromium films with highly-oriented pyrolytic graphite (HOPG) indicate that chromium carbide is metastable. It only forms on an ion-damaged surface, and decomposes above 500°C. Thus, there is a question of the observed wetting of graphite by chromium-doped copper in fact due to chromium carbide formation at the interface. Experiments were performed in-situ in UHV by thermally evaporating chromium and/or copper onto the basal plane of a HOPG substrate, and observing the Auger electron spectra as a function of annealing temperature. Changes in the peak shapes and intensities of the carbon KVV and chromium and copper LMM and MVV Auger transitions are interpreted in terms of metal film structure, and carbide formation and decomposition. The results are used to evaluate the role of carbide formation in the wetting of graphite by chromium-doped copper films.

AS-TuP26 Electric States of Segregated Metal Atom on Metal Surfaces and Potential Use for Field Emitter, M. Yoshizake and K. Yokobuhara, National Research Institute for Metals, Ibaraki, JAPAN.

The segregation phenomena of substrate copper atoms onto the deposited titanium film were observed using Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). The Ti film of 1 μm thickness was deposited by r.f. magnetron sputtering on polycrystal Cu surface. The specimens were heated in XPS apparatus. After a certain time, Cu atom diffused onto the surface of Ti film from the substrate with the 60% of the activation energy of bulk diffusion. The surface concentration of Cu saturated at about 5%. When the segregated surface was destroyed, Cu-saturated surface structure was easily reformed. The binding energy of Cu 2p3/2 of the segregated Cu was 0.24eV higher than that of pure metal. It suggests that the electron belongs to Cu is attracted to Ti and the segregated Cu slightly changes positively. This direction of charge transfer was also observed when Ti was deposited on Cu substrate. The binding energy of Cu 2p3/2 of Cu, on which surface was covered with Ti was almost same as segregated Cu. It is well known that when adsorbed atom causes charge transfer and forms electric dipole, the work function changes.

In the case of Cu segregation on Ti film, the charge transfer from Cu to Ti is estimated to be 0.11e from the above binding energy. It is concluded that when this value is used for the calculation of the dipole moment, about 0.62eV decrease in work function is expected and this material has great potential for a field emitter.

AS-TuP27 The Effect of Sample Bias on the Nitrogen Etching Products of Aluminum, M. A. Roote, P. A. M. Sherwood, Department of Chemistry, Willard Hall, Kansas State University, Manhattan, KS 66506-301.

The paper will report the effect that biasing of a sample has on the properties of a reactive etching process. When aluminum is etched using a sputter-field argon ion etcher with nitrogen as the etching gas the aluminum metal is first converted into an oxy-nitride. The oxy-nitride is formed because of residual oxygen in the etching gas. If etching is continued past an initial two minute etch, then the surface continues with approximately the same composition. Surface composition is determined by core and valence band X-ray photoelectron spectroscopy, with the valence band data interpreted by spectra generated by multiple scattered Xo calculations.

If the aluminum sample is subjected to a 15 volt d.c. negative bias, then oxy-nitride is formed after the initial two minute etch, but continued etching leads to the formation of a surface that is almost entirely aluminum oxide. In addition the case of the biased sample a number of the XPS features are seen, including nitride, adsorbed N2, and a peak at high binding energy due to implanted N2. This latter peak is only seen when the sample is negatively biased. The paper will suggest reasons for the chemical changes that occur when the sample is biased.

This material is based upon work supported by the Air Force Office of Scientific Research under Grant No. F496620-92-J-0144.
must include data elements which fully describe all aspects of the record, such as the specimen, the instrument, the experiment, the calibration, the spectral data, the data analysis methods, and the results. The list of data elements making up the database and their definitions is called the data dictionary. The data dictionary for electron spectroscopy used by the American Vacuum Society’s Surface Science Spectral Database contains 188 data elements grouped into 8 information classes. In developing the data elements, consideration was given to existing surface analysis standards, existing standards and databases in other spectroscopies, and input from the user community. Several areas of complications required substantial dialogue with users and instrument manufacturers to achieve a satisfactory solution. For example, the variety of instrument designs and measurement strategies makes it difficult to define a universal set of elements that would be commonly used electron spectrometers. Also, since data records are contributed from the entire user community and peer-reviewed by the same wide community, explicit guidance must be given to describe the various quality level and completeness requirements.


Development of the Auger, XPS and SIMS data dictionaries for the AVS Surface Science Database was carried out in close coordination with parallel efforts in the broader analytical chemistry community. This collaboration between the surface science and analytical chemistry communities has been directed toward the specification of a unified data model that would permit merging and cross-referencing of data from different analytical techniques. Attempts to find a common data model for such diverse analytical techniques as XPS, IR, NMR and chromatography have been frustrated by the inherent limitations of available information management technology. We have been able to extend conventional data modeling concepts to provide a natural environment for describing analytical measurements in general. The generic data model that has emerged employs a matrix array to describe the measurement process as well as the instruments and specimens that are involved. This new data model is being proposed as a standard for both surface science and analytical chemistry information systems.

ELECTRONIC MATERIALS
Room BR4 - Session EM-TuP

Aspects of Electronic Materials and Processing 1
Moderator: F. A. Houle, IBM Almaden Research Center.

EM-TuP 1 Thermal Oxidation of Heteroepitaxial Si1-x-GeVxAlloy Thin Films, Hong Xiang, Nicole Herbors, Peihua Ye*, Sean Hearne, Department of Physics and Astronomy, *Department of Chemical and Materials Engineering, Arizona State University, Tempe, AZ 85287-1504.

Heteroepitaxial Si1-x-GeVxAlloy thin films have been found to undergo thermal oxidation in Si-based technology. However, good quality dielectric on Si1-x-GeVx are not obtained by thermal oxidation. Our motivation is to explore the effect of carbon in thermal oxidation of Si1-x-GeVx as well as its role on strain compensation in Si1-x-GeVx alloys. Pre-cleaned Si(100) wafer, MBE-processed Si1-x-GeVxAlloy thin films were oxidized at 1000°C in dry oxygen atmosphere for two hours. The thickness and the composition of all samples before and after oxidation were measured by Rutherford Backscattering Spectrometry (RBS) combined with ion channeling at 2.0 MeV and carbon nuclear resonance measurements at 4.3 MeV using 3He+. The structures were also characterized by Secondary Ion Mass Spectrometry (SIMS), High Resolution TEM (HRTEM), FTIR and Raman Spectroscopy. As expected, 2.0 MeV RBS analysis shows that a uniform layer of SiOx is grown on pure Si(100) sample during thermal oxidation. For all other samples, a layer of SiOx is also formed, while Ge segregates towards the top surface and at the SiOx/Si1-x-GeVx and SiOx/Si1-x-GeVx interfaces. However, RBS analysis also shows that carbon in Si1-x-GeVx reduces the amount of Ge segregated during thermal oxidation. This observation will be discussed in conjunction with SIMS, HRTEM, FTIR and Raman data.

EM-TuP 2 The Role of Temperature During Synthesis of Heteroepitaxial Si1-x-GeVxSi(100) Single Crystal Thin Films by Molecular Beam Deposition (CIMD), N. Nicole Herbors*, Peihua Ye, Jiong Xiang*, Sean Hearne*, Dept. of Chemical and Bio and Materials Engineering, *Dept. of Physics & Astronomy, Arizona State University, Tempe, AZ 85287-1504.

The motivation for this study is to investigate a new ternary group IV semiconductor, Si1-x-GeVx, for strain and bandgap engineering. Heteroepitaxial Si1-x-GeVxSi(100) is synthesized by Combined Ion and Molecular beam Deposition (CIMD). Characterization involves Nuclear Resonance Rutherford Backscattering Spectrometry (NRA) combined with ion channeling, Secondary Ion Mass Spectrometry (SIMS), High Resolution Transmission Electron Microscopy (HRTEM), Fourier Transformation Infrared Spectroscopy (FTIR), and Raman Spectroscopy. NRA shows that up to 2% C can be incorporated in heteroepitaxial Si1-x-GeVxSi(100) films, which is about three orders of magnitude above the 0.1% solubility in Si. TEM shows that Si1-x-GeVxSi(100) films are epitaxial with a well defined interface between films and substrate. Cross section TEM also shows that films deposited at 600°C exhibit a crystalline secondary phase coherent with the matrix and localized in small regions (2 nm in diameter), while the film deposited at 500°C exhibits a uniform phase contrast. FTIR spectra for films deposited at 500°C show a distinct absorption mode at 604 cm⁻¹, which is characteristic of substitutional carbon in Si. However, FTIR spectra for films deposited at 600°C neither exhibit silicon carbide precipitates (wave number 800 cm⁻¹) nor substitutional C (wave number 604 cm⁻¹). Raman and FTIR spectra will be discussed in light of microstructural findings by HRTEM.

Research supported by AFOSR/ARPA contract F49620-93-C-0018.


Multiple SiGe quantum wells were grown by MBE on Si(100), (110), and (111) substrates in order to determine the optimum growth conditions for these structures to be used as infrared (IR) detectors. The nominal structure was: five to fifteen 4 nm Si0.6Ge0.4 quantum wells separated by 30 nm Si, with the center 3 nm of each quantum well doped with B at concentrations ranging from 1 to 5 x 10¹⁸ cm⁻². The photoluminescence (PL) measurements at 4K showed that bandedge, phonon-resolved PL was most intense for the quantum wells grown at 710°C on the Si(100) substrates and grown at 800°C on the Si(110) and Si(111) substrates. However the quantum well IR absorption was strongest for the samples grown at 550°C. Using a single incident light technique, the strong absorption observed in doped structures between 4.0 and 5.0 μm on each of the substrate orientations has been tentatively associated with bound-to-continuum quantum well transitions. Using a waveguide structure, polarization-dependent quantum well intersubband IR absorption was observed at 8.4 μm and 10.5 μm, corresponding to HH₁ - SO₃ and HH₂ - HH₁ transitions, respectively, for the samples grown on Si(100). Both SIMS and TEM analysis of the structures, as a function of growth temperature, will be reported.


Erbium epitaxial Si films doped with Er have been grown at low substrate temperatures by plasma enhanced chemical vapor deposition. The Er gases used were sublimed organometallic precursors, iridium(III-triphenylsilyloxy) erbium (Irh), was designed specifically to perform this task.

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The deposition process in general relies on the beneficial effects of low energy ion bombardment to reduce the growth temperature. This bombardment may also lead to the enhanced incorporation of carbon into the deposited film. Processing conditions were varied to gain an understanding of the impurity incorporation mechanism. Film quality and impurity concentration were determined by Rutherford back-scattering spectrometry and secondary ion mass spectrometry.


Surface reconstruction of carriers is an important process during the operation of semiconductor devices such as heterojunction bipolar transistors and integrated circuits. This is especially true as device dimensions decrease. In particular, GaAs-based materials have very high surface reconstruction velocities and much effort has been devoted to reducing surface reconstruction through surface passivation. Sulfide passivation is an effective method, but the improvement is only temporary if the surface is exposed to oxygen. We have examined silicon nitride encapsulation of sulfide passivated surfaces as a technique to improve the stability of the passivating effect. This passivation scheme was applied to GaAs/AlGaAs microdisk lasers. There is interest in both the fundamental physics and potential applications of these lasers. From our perspective, these microdisks serve as a sensitive probe of the surface processes since the optical mode volume is of the order of the diameter at the edge of the disk. These lasers have diameters ranging from 2 to 10 µm. The microdisks did not lase without the sulfide treatment when optically pumped at 77K using 632 nm up to 10 kW/cm². With sulfide treatment cw laser was achieved but the lifetime was only a few seconds. Silicon nitride encapsulation of the sulfide passivated microdisks resulted in a dramatic increase in the laser lifetime. Furthermore, the laser output could be increased by nearly an order of magnitude by annealing at 400°C for 300 s by cw operation over a period of several seconds. Results on test structures using photoluminescence and secondary ion mass spectrometry will also be discussed.


Sulfidation of III-V semiconductor surfaces markedly improves their electronic properties, but exposure to atmosphere results in loss of passivation. Dielectric encapsulation halts atmosphere-induced decay, but passivation can be reduced through loss of sulfur and other damage during dielectric deposition. For retention of maximum passivation, improved methods of dielectric deposition must be developed. To study the effects of various passivation techniques on the electronic and chemical properties of the S/N/Si/Si/Si interface using optical (photoluminescence (PL) and Raman) and surface analysis (XPS and x-ray-excited Auger) techniques. Thin (~ 40 Å) SiN films are deposited on polycrystalline GaAs using an electron cyclotron resonance (ECR) plasma deposition system that permits variation of ion incidence angle on energy, surface illumination, temperature, and gas composition. Film thicknesses are selected to permit Auger and photoelectron escape from the buried SiN/Si/Si/Si interface for chemical characterization. For commonly employed ECR deposition conditions, approximately 50% of the Si is lost during deposition, and a second Si(111) peak joins the single peak observed on uncapped Si/GaAs. Both Raman and PL measurements reveal shallower depletion regions and improved electronic properties over the passivated surface, but at levels reduced from the original Si/Si/Si/Si interface. The effects of altered deposition conditions on surface composition and electronic properties and alternatives for improved passivation will be discussed.

This work was performed at Sandia National Laboratories and supported by the U.S. Department of Energy under Contract No. DE-AC04-94AL85000.

Em-TuP7 Correlation of Surface Morphology with Chemical Structures of Sulfur-Passivated GaAs(100) Investigated by Scanning Tunneling Microscope and X-ray Photoelectron Spectroscopy, Jeong Sook Ha, Seong-Ju Park, Sung-Bock Kim, and El-Hang Lee, Research Department, Electronics and Telecommunications Research Institute, P.O. Box 106, Taeuk Science Town, Taejon 305-606, Korea.

Nanometer-scale characterization of semiconductor surfaces and preparation of atomically flat and chemically stable surfaces are of particular interest for precise control of ultrafine device structures. In this paper, the surface morphology and the chemical structures of the n-GaAs(100) samples after etching with sulfuric acid and sulfur passivation with (NH4)2S solution were systematically investigated by scanning tunneling microscope (STM) and X-ray photoelectron spectroscopy (XPS). STM measurements were performed in a constant current mode under ambient condition. The surface morphology was observed to be quite different depending on the surface treatments. In particular, the effect of water rinse between etching and sulfur passivation on the surface morphology was examined. The sulfur passivated sample with examined after water rinse showed a roughness similar to that of the as-etched sample with a surface undulation of 20 Å. However, sulfur passivation without post-etch water rinse dramatically improved the surface flatness giving a surface undulation of 5 Å. It was also shown that the surface roughness induced by etching with sulfuric acid was improved after HCl treatment for 10 minutes. Similar effects could be explained by the preferential removal of gallium oxide and arsenic oxide by HCl and water, respectively. XPS measurements were found to support this reaction mechanism. In this work, we will discuss the correlation of the surface morphology with the chemical reaction on the surface revealed by STM and XPS investigation.

EM-TuP8 Etching of Polysilicon with a Chlorine Trifluoride Beam, Han Xu, Anthony J. Muscat, A. Scott Lawing and Herbert H. Sawin, Department of Chemical Engineering, Bldg. 66-225, Massachusetts Institute of Technology, Cambridge, MA 02139.

Thermally excited molecular beams (CIg, SF6, CIF3, etc.) have the potential to replace wet cleaning for removing metal contaminants and repairing damage on wafer surface in situ. These "dry" beams etch silicon surface rapidly without damaging the underlying silicon lattice as ion-assisted processes do.

We have studied the etching of n-doped polysilicon with a room temperature CIF beam under high vacuum conditions. Etch rates were measured at silicon temperatures between 13 and 250°C. An Arhenius analysis of the data gave an apparent activation energy of 0.4 kcal/mol. The etched surface was terminated by F atoms as shown by the intense SiF3 peak after etching. Samples on which a monolayer of copper atoms were deposited before etching showed that the etching rates at room temperature were similar to those without Cu. When the sample temperature was raised to 250°C, the etching rates after Cu deposition were an order of magnitudes higher. The apparent activation energy was 3.0 kcal/mol in the latter case, and the etched surface was terminated by Cl as well as F atoms. These results suggest that the copper atoms catalyzes the halogenation of the silicon surfaces. The correlation between the etching rate and the surface roughness will be discussed as well.

Em-TuP9 Structural and Chemical Changes During Annealing of Gas-Phase Polysilicide Passivated InP, R. W. M. Kwok, G. Jin, Dept. of Chemistry, The Chinese University of Hong Kong, Hong Kong; L. Huang, W. M. Lau, Dept. of Materials Engineering, The University of Western Ontario, London, Ontario, N6A 5B7 Canada; C. C. Hsu, Institute of Semiconductors, CAS, Beijing, PR China.

The structural and chemical changes induced by annealing InP exposed to gas-phase polysilicide, a heat treatment which is known to activate the passivation for surface state reduction (J. Vac. Sci. Technol. A11, 990 (1993)), were investigated using in situ low energy electron diffraction (LEED), thermal desorption spectrometry, and X-ray photoelectron spectroscopy (XPS). A blurred InP (1 × 1) LEED pattern was observed on InP exposed to UV/O2, and then etched by a 1:30 HF solution, which became sharper with an additional exposure to gas-phase polysilicide. Vacuum annealing of the polysilicide treated InP gave a more ordered (2 × 1) passivated InP surface as suggested by a further sharpening improvement of the LEED pattern. The sharpest pattern obtained was after annealing at 400°C. Accompanying these structural changes were the desorption of species containing H and S at 300°C, and those containing P and S at 400°C. Further, XPS showed that the P-S bonding species and weakly chemisorbed In-S species which were present in the polysilicide passivated surface, disappeared with the annealing at 400°C. Further annealing at 540°C resulted in the destruction of the surface sulfide passivation as indicated by the reduction of the surface sulfur, desorption of sulfur as well as phosphorus, and an increase of surface state densities by capacitance-voltage measurements. The resultant surface showed a blurred (2 × 1) LEED pattern.

EM-TuP10 Sputter-Cleaning and Smoothening of GaAs(001) Surfaces Using Glancing-Angle Ion Bombardment, J. G. C. Labanda, Dept. of Physics and Astronomy, Northwestern Univer., Evanston, IL 60206, L. Hultman, IFM, Linköping University, Linköping, Sweden,

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Glancing-angle argon ion bombardment has the advantages over normally incident ions of reducing ion damage and ion implantation projected ranges, reducing channeling, and preferentially removing surface asperities leading to flat surfaces. The effect of different bombardment conditions on the surface morphology and perfection of GaAs(001) substrates was studied. Initially rough surfaces become smoother with irradiation as evidenced by spotty reflection high energy electron diffraction (RHEED) patterns becoming streaky and by atomic force microscope (AFM) images. Optimal surfaces with AFM roughness < 0.4 nm and showing short bulk and reconstruction RHEED streaks were obtained at a dose of 5 x 10^13 Ar ions/cm² with an Ar ion energy of 1 keV and a dose of 2 x 10^14 Ar ions/cm² with energy E = 1000 eV incident at an angle θ = 15° from the surface plane. Lower E, θ, and dose values were not as effective in smoothing and removing contamination, while larger values yielded rougher surfaces. Cross-sectional transmission electron microscopy (XTEM) images of bombarded GaAs surfaces showed that ion damage increased with increasing dose, E, and θ. Damage was resolved for the optimal smoothing and cleaning conditions noted above. However, defects were eliminated by using θ = 6°. Defect-free GaAs and InGaAs layers were grown on the spattered surfaces by molecular beam epitaxy even when sub-surface ion damage was present. X-ray diffraction results for these films will also be reported.

EM-TuP11 Sputter Deposition of Yttria Stabilized Zirconia onto a Porous Au Substrate, A. F. Jankowski and J. P. Hayes, University of California, Lawrence Livermore National Laboratory, Livermore, CA 94551-9000.

Thin film synthesis of solid oxide fuel cells is motivated by the objective to increase power densities for applications as in the light transportation industry. Process issues key to manifesting a fuel cell stack from single cells include: i) The ability to transport gas through porous conducting electrodes, deposit a defect-free electrolyte on the porous electrodes, and provide sufficient structural integrity for stack assembly as well as temperature cycling. This study addresses the issue of electrolyte layer deposition on a substrate under conditions appropriate for stack assembly. Our initial approach is to use a porous metal substrate to permit measurement of the electrolyte performance as well as provide a pore size similar to conventionally used electrode materials, e.g. sintered compacts. The sputter deposition of Au, under proper process parameters, serves as the porous substrate for the electrolyte layer. An optimum choice for the electrolyte material is yttria stabilized zirconia (YSZ). The focus of the study is to evaluate the process parameters of rf sputtering a YSZ target to densely coat a porous substrate and provide a defect-free, 2-5 μm thick electrolyte suitable for operation at temperatures of 750°C or less. With consideration to the scattering effects of sputtered neutrals, an elevated working gas pressure of Argon facilitates filling surface voids of the porous substrate leading to the formation of a defect-free layer of cubic YSZ as examined with electron microscopy techniques. Whereas many deposition processes call for an elevated substrate temperature to produce the cubic phase, we use a room temperature substrate. This is advantageous with respect to our objective of utilizing a coefficient of thermal expansion—matched porous substrate for the planar electrodes. Electrolyte coated, anode and cathode pairs will be sintered together to form a fuel cell stack. Therefore, the electrolyte and electrodes will be subject to the same thermal history during processing and future temperature cycling.


Gas-source molecular and chemical beam epitaxial techniques are being increasingly utilized in the production of III-V optoelectronic devices. It has been demonstrated that all of the donor and acceptor impurities in GaAs can be passivated with atomic hydrogen by the formation of complexes. The "hydrogenation" of the charge carriers provides a simple means of patterning and isolating discrete components in integrated device structures such as tunnel externally modulated lasers and, more recently, alternative approaches to implantation. The hydrogen sources can easily be incorporated in growth systems for in situ processing. The passivation mechanism for the shallow- to mid-level cation-site acceptors is presumed to be similar in the phosphide based III-V's. The exception to this, to date, is the column IIa acceptor, Mg, in GaP which exhibits little or no passivation. This suggests that Be (also column IIa) may be passivated in GaP and less so in GaAs, but may also be difficult to passivate in InP. We show that the carrier concentration of Be-doped InP (100) can be compensated by more than four orders of magnitude by diffusion of atomic hydrogen generated by an electron cyclotron resonance plasma source. Chemical beam epitaxy allows doping to 3 x 10^19 cm^-3 with an effective carrier density of 4 x 10^17 cm^-3. C-V depth profiling shows effective compensation of the carriers within the first 0.4 μm from the surface. Be has a larger electron affinity than that of Mg and its compensation may be explained by its higher affinity for H [J. Weber and M. Singh, Mater. Res. Soc. Symp. 104, 325 (1988)].


The structures of a series of strained and relaxed single crystal Si1-xGe1-x, alloy thin films grown by MBE on Si(100) and Ge(100) substrates have been studied by Ge-K and Si-K x-ray absorption spectroscopy. A constrained multi-file analysis of the extended fine structure signal has been used to evaluate the Si-Si, Si-Ge and Ge-Ge bond lengths in order to investigate a controversy concerning the extent to which these bond lengths vary with composition. The results are discussed in terms of a recent "topological rigidity" model. Polarisation dependent Si-K near edge spectra of the strained alloys are related to band structure calculations in order to demonstrate the sensitivity of the Si-K spectral features to the influence of strain on the conduction band electronic structure.

Research supported by NSERC and the Ontario Centre for Materials Research. Experiments at CHESS and SRC, supported by NSF.


Si-capped SiGe samples grown by CVD were implanted with several doses of 200 keV SiB ions. In order to completely amorphize the layer, one sample was implanted with 1 x 10^16 ions/cm² of 200 keV Si ions at LN2 temperature. Rapid thermal annealing was performed for 200 seconds at both 600°C and 960°C. Since samples annealed at 600°C exhibited little epitaxial regrowth, they were subsequently annealed at 800°C for 200 seconds. Samples were studied using Rutherford backscattering spectrometry (RBS) and ion channeling. Carbon was quantified using the 12C(a, α)C14 C ion beam resonance at 4.265 MeV. It was found that the regrowth kinetics were significantly different from those of SiGe alloys. A greater thermal budget is required for regrowth. Furthermore, at the higher temperatures the carbon diffuses greatly out of the layer leaving a carbon depleted region. This has a positive impact on the doping of this material.

1Research conducted at Arizona State University was supported by AFOSR (Draper) Contract F49620-00-C-0081. Research at Oak Ridge National Laboratory is partially supported by the Division of Materials Science, U.S. Department of Energy, contract DE-AC05-84OR21400 with Martin-Marietta Energy Systems, Inc.


The strain in commensurate SiGe heteroepitaxial films grown on Si(100) substrates has been quantified using ion channeling. The films were grown by molecular beam epitaxy (MBE) and chemical vapor deposition (CVD). Rutherford backscattering spectrometry (RBS) was used to quantify the Ge concentration as well as the film thickness. Nuclear resonance elastic ion scattering was used to quantify the carbon. Channeling was done using 2MeV He⁺ ions which yielded...
angular scans in theta. The layers were etched using a 4 mol solution of HF and HNO₃ in order to obtain a reliable scan in the substrate. The shift in minima in the angular scans gave the tetragonal strain which ranged from 1 to 1.8%. This indicates that carbon is compensating for at least part of the strain caused by the high Ge content above the critical thickness of these layers.

*Research conducted at Arizona State University was supported by AFSOR (Darpa), contract F49620-93-C-0081.


Gaseous alternatives to Sb and alkyl-Sb sources are actively being sought. Trisdimethylaminoantimony (DMASb) appears to be a promising precursor for use in Metalorganic Molecular Beam Epitaxy (MOMBE), though at present little is known regarding the use of this source in ultrahigh vacuum (UHV) growth environments. This paper will present a comparison of Sb and DMASb as Sb sources for growth of InSb and AISb from trimethylindium (TMI) and trimethylalane (TMAA) respectively. The feasibility of GaSb growth from triethylgallium (TEG) and elemental Ga will also be discussed for each of the two Sb sources. In addition to their effect on growth rate and morphology, the fate of the Sb precursor on carbon incorporation will be examined as a function of V/III ratio and growth temperature.

Finally, the feasibility of carbon doping from CBR will be discussed, paying particular attention to the incorporation of other impurities such as Br and hydrogen.

EM-TuP17 Zinclende-CDSe on GaSb(110): Characterization of Epitaxial Growth and Electronic Structure, G. Neukohl, K. Horn, Fritz-Haber-Institut der MPG, 41951 Berlin, Germany, K. O. Maynor, Department of Natural Sciences, University of Karlstad, 650 09 Karlstad, Sweden, and B. A. Evans, Athrofa Golegd Defrydym Cymru (NEWI), Cwysy GB.

Substrate-stabilized pseudomorphic growth offers the chance to study the electronic structure of a particular semiconductor in different crystal structures, and to investigate the influence of structural differences on bulk and surface states. We have grown layers of CDSe which, in the bulk crystallizes in the stable wurtzite structure, in the zincblende modification on cleaved GaSb(110) surfaces by molecular beam epitaxy in an ultrahigh vacuum photoelectron spectrometer. The growth mode and structure of the overlayer were studied by means of LEED and core as well as valence level photoemission, using synchrotron radiation. The attenuation of substrate states and the development of layer intensities with the deposition increases with the growth rate. An interface reaction, the amount of which depends on growth temperature, leads to the liberation of Sb, which floats on the growth front, and the formation of a Ga-Se compound, as signaled by changes in substrate and overlayer core level line shape. The valence band offset for this lattice-matched heterojunction interface system was determined, and found to be of the staggered type. This is in agreement with predictions based on the dielectric midgap energy model. Angle-resolved valence band spectra were interpreted in terms of direct transitions, and yielded good agreement with band structure calculations for the cubic phase.


GaN is a potential candidate material for high temperature electronics and for blue-UV lasers. Though there are many reports on the Hall mobility measurement, few studies of the carrier lifetime have been reported. Since the minority carrier lifetime is an important parameter in device design and operation, we performed a photocconductivity (PC) decay study on GaN films grown by low-pressure MOCVD. The PC decay monitored by coplanar geometry was excited by a KrF excimer laser and was recorded with a storage oscilloscope. The films examined included undoped n-type GaN and a Mg-doped insulating p-type GaN. Negative PC is observed from films with high electron concentrations, while positive PC is observed from the Mg-doped p-type sample. For all the films, the PC decay obeys a power law f-7/3 with an α value in the range of 0.2 to 0.5. α depends very slightly on the laser excitation power. The implications of the results and device design considerations will be discussed.


Formation of electrical contacts to MBE grown p-type (nitrogen doped to ~3 x 10²¹ cm⁻³) ZnTe using sputter deposited Au films (1500 Å) has been studied using Current-Voltage (I-V) data, Auger electron spectroscopy (AES), secondary ion mass spectroscopy (SIMS), and optical and scanning electron microscopy. I-V measurements were performed on the samples as deposited as well as after annealing at T > 150°C in forming gas for times < 90 minutes in order to determine the optimum annealing temperature and time. Lower resistances were measured for 150°C and 200°C for 15 minutes, while the resistance increased slightly with longer anneals. At 250°C the I-V data were still linear, but higher resistances were measured for all annealing times. For an optimum anneal of 200°C for 15 minutes, a current density of 2.0 x 10²¹ mA/cm² was obtained at 5V. Reactions at the interface have been studied by AES and doping profiles have been determined by SIMS. Microscopy shows a planar interface with minor perturbations of morphology of the Au contacts. These results will be compared with previous studies of the reaction of Au and Ag with p-ZnTe with respect to current density, compound formation and surface morphology.

*This work supported by ONR/ARPA Grant N00014-92-J-1895.

EM-TuP20 High Field Ni-SiC Schottky Barrier Diodes with Single-Metal Process, J. N. Sun and A. J. Steckl, Department of Electrical and Computer Engineering, University of Cincinnati, Cincinnati, Ohio 45221-0030.

Wide-bandgap silicon carbide is receiving increased attention for high power and high temperature applications. Metal-SiC Schottky junctions are a promising approach for the development of high power SiC MESAET. We report the use of Ni as both Schottky and ohmic contact metal to fabricate a high breakdown field (~165 V/cm) Schottky barrier diode (SBD) on a thin (1.6 μm) 6H-SiC epitaxial layer (n = 2 x 10²¹ cm⁻³) grown on r-SiC substrate. Ni metallizations are carried out by sputter deposition at room temperature followed by different thermal processes to distinguish the ohmic and Schottky contact regions. I-V characteristics show the Ni/SiC SBD has a high breakdown voltage of 150V, an extremely low leakage current density and excellent thermal stability. The ideality factor of less than 1.2 is an indication of a well-behaved thermionic emission mechanism. To compare with a commonly used SiC Schottky structure, Pt/6H-SiC diodes have also been fabricated using the same process and SiC material. Although the Pt/SiC diode has a leakage current density as low as the best results reported, the Ni/SiC exhibited electrical characteristics and thermal stability superior to those of the Pt/SiC structure. For example, Ni-SiC diodes have a leakage current density of ~16 A/cm² at blocking voltage of 100V, 100 x less than that of a Pt-SiC structure. Subjected to thermal exposure, Pt-SiC structures show thermal degradation for temperatures beyond 400°C, while the Ni/6H-SiC Schottky junction exhibits a stable performance to a temperature stress as high as 600°C. Furthermore, using Ni as both Schottky and ohmic contact metal greatly simplifies the fabrication process.

EM-TuP21 Temperature-Dependent Optical Bandgap of Metastable Zinclende-Structure α-GaN, A. Lastras-Martinez, H. Navarro-Contreras, and G. Ramirez-Flores, Instituto de Investigación en Comunicación Óptica, Universidad Autónoma de San Luis Potosí, 78000, San Luis Potosí, SLP, México; R. C. Powell and J. E. Greene, Materials Science Dept., Coordinated Science Laboratory, and M-
The results obtained indicate that use of SCB as precursor for growth of 3C- on 6H-SiC is very promising for low temperature deposition of crystalline films.

**EM-TuP24** Electron Emission from Wide-Bandgap Negative Electron Affinity Materials, C. Bandis, D. Haggerty, and B. B. Pate, Department of Physics, Washington State University, Pullman, WA 99164.

The diamond (111) 1 x 1 surface was found to exhibit negative electron affinity (NEA) over a decade ago [1]. Our recent work [2] on electron emission properties of the NEA-adatom (111) surfaces find that the creation of free excitons play an important role in the high quantum yield which has been obtained with near threshold photoexcitation. We found that bound-electron-pair transport to the surface and breakup in the electric field of the surface dipole. In this report, we present evidence for the existence of such an emission mechanism in other wide bandgap materials. These results suggest that the exciton-derived emission mechanism is common, and is likely to apply to a wide variety of wide-bandgap materials.


**EM-TuP25** Conformality of SiO Films from TEOS-Sourced Remote Microwave Plasma-Enhanced Chemical Vapor Deposition, D. A. Levelukis, H. Liao, T. S. Cale and G. B. Raupp, Department of Chemical, Bio & Materials Engineering, Arizona State University, Tempe, AZ 85287.

The deposition of silicon dioxide step coverage in micron scale trenches on substrate temperature, absorbed microwave power, total pressure and O2:TEOS flow ratio were systematically investigated in a remote plasma configuration in which oxygen is excited upstream of the deposition chamber in a microwave discharge. This configuration allows us to investigate the plasma-induced deposition of SiO2 from TEOS in the absence of charged species (ions and electrons). Of the independent parameters investigated, temperature has the strongest effect on film conformality. For example, step coverage in a long rectangular trench of aspect ratio two is greater than 90% at 250°C, but less than 40% at 400°C at otherwise fixed conditions. Experimental film profiles in trenches are compared to simulations using EVOLVE, a ballistic transport and reaction simulator. In our simulations we assume that deposition proceeds through oxidative attack of TEOS or TEOS fragments adsorbed on the growing film surface by oxygen atoms. A thermally activated, heterogeneous oxygen atom recombinaction reaction consumes oxygen atoms without leading to deposition. An electrochemistry model of the excitation between the plasma source and the feature mouth. We demonstrate how physically-based modeling can be used to extract kinetic rate parameters from experimental data, and how to use physically-based simulation to predict operating windows for high step coverage and good film quality.

**MANUFACTURING SCIENCE AND TECHNOLOGY** Room BR4 – Session MS-TuP

**Manufacturing Science and Technology**

**Moderator:** G. W. Rubloff, North Carolina State University.

**MS-TuP1** Low-Cost Optical Reflectivity Temperature Measurement System, H. A. Atwater and D. S. Gardner, (a) California Institute of Technology, Pasadena, CA 91125 and (b) Intel Corporation, Santa Clara, CA 95052.

We describe the results of a project to assess the use of a low-cost ($\leq$700.00) optical reflectivity system as a practical surface temperature diagnostic for on-wafer measurements during sputter deposition. The physical processes which lead to temperature-dependent optical reflectivities are material-dependent, but are large enough for many integrated circuit materials (e.g., Al, Si, Cu) to be useful for thermometry. Correlation of thermocouple temperature measurements made in an isothermal environment with optical reflectivity allows...
system calibration via an empirical fit to the nonlinear dependence of reflectivity with temperature. Dividing the reflectivity data for a given material by the fitting function allows relative temperature measurement with precision of ±4°C at temperatures above 170°C, for Al and Si. The reflectivity measurement during sputter deposition of Cu indicated a large temperature rise during film deposition. Limits to optical reflectivity thermometry, as well as extensions to rapid thermal annealing and chemical vapor deposition will be discussed.


A Dipole-ring Magnet (DRM) has a capability of producing high fields up to 6000G with excellent uniformity. Its low stray magnetic field outside of the ring eliminates interference between chambers, and makes it suitable for compact new mainframe systems. We have developed new processes for quartet micro-etching employing magnetron RIE system using the DRM.

Si etch rate was found to increase with magnetic field strength due to higher plasma density in high fields. The 6000G DRM enables 60.25μm deep trench etch at a rate of more than 1 μm/min, which is twice that of the current magnetron RIE. For the SiO₂ process, high field DRM was adopted according to the optimization of Vdc, ion flux and F radical concentration variations to field strength, to obtain the best etching performance. Finally, contact hole etch process with high selectivity over poly-Si(50), SiNₓ(20), and Al(40) was accomplished with the 120G DRM using fluorocarbon gas and CO chemistry.

The process technologies with DRM are concentrated in a next generation "UNITY" mainframe supported by an advanced control system.


We have introduced a new plasma etching system using a Dipole-ring Magnet (DRM) as a promising etch tool for quartet micro etch devices. DRM provides a parallel magnetic field up to 600 Gaus with excellent uniformity, and has achieved excellent performances such as high etch rate in Si deep trench and high selectivity of more than ten in SiNₓ etching over SiO₂Nₓ, as well as low charge-up damage (1).

For further improvement of the gate oxide reliability in SiO₂ etching, we have succeeded in compensation of the plasma non-uniformity due to F x B site electron drift, realized by use of an optimized magnetic field distribution. This enabled the use of higher rf power for higher etch rates, and a compact magnet design, still with no degradation of the gate oxide in a TDBB test. Finally, the DRM's low level of stray magnetic field eliminates interference between chambers, which makes it suitable for compact mainframe systems.


MS-TuP4 Profile Modeling of High Density Plasma Oxide Etching, J. S. Han, J. P. McVittie, J. Zheng, Stanford University, Center for Integrated Systems, Stanford, CA 94305.

Plasma dry etching is increasingly becoming a central part of IC manufacturing. As device dimensions continually decrease, the contact via sizes and lines begin to push the sub 0.35μm regime. In addition, aspect ratios approaching 5:1 for contacts and more than 20:1 for trenches are now commonplace. Such geometry can only be processed using dry etching techniques. To understand and predict etch profiles becomes essential to reduce costs in process optimization. In this paper, several profile issues that result from an industry accepted inductively couple high density plasma oxide etch system are investigated using oxide overhang test structures and vias. The key features observed from the test structures include ion enhanced polymer deposition, etch rate saturation effect, redeposition, direct polymer deposition (CVD polymer), and etch lag. In addition, via show trends of rewiring lag and microetching. A new model is presented and implemented in SPREDE, a topography simulator developed at Stanford design to use phenomenological models to predict profile evolution. The model separates the neutral flux into a deposition flux and a chemical flux, where the deposition species contribute to the growth of polymer and the chemical species contribute to the etching of the substrate, in this case, SiO₂. Deposition or etching takes place at a rate dependent on the ratio of the deposition flux to the ion (energy) flux or chemical flux to the ion (energy) flux. In addition, microetching is modeled by an ion induced chemical desorption component. Comparison with SEM results show that the new model can be used to accurately simulate profile evolution.


In this paper we describe real-time growth control of MBE grown GaAs/AlGaAs multiple quantum well, and MOCVD grown HgCdTe using a novel and low cost in situ multi-wavelength ellipsometer. This ellipsometer acquires in situ ellipsometric data in real-time at 44 wavelengths in the spectral range of 415-750 nm with maximum acquisition rate of 25 measurements/second (typical 1 ms measurement time/second).

A GaAs/AlGaAs multiple quantum well with varying well thicknesses was MBE grown on a GaAs substrate. In this experiment layer thicknesses of the multiple quantum well were controlled by the ellipsometer. The substrate was rotated at all times during the MBE growth. The in situ ellipsometer was also used in the composition control of MOCVD grown HgCdTe. Alloy compositions of 0.25 and 0.3 were stabilized by regulating the H₂ flow through a DMCD source. And, when an intentional temperature disturbance was introduced during the control experiment, the ellipsometer re-adjusted the H₂ flow to maintain the as-grown composition. Results from SIMS will also be described.

MS-TuP6 Electrical Monitoring of Surface Conditions in a Plasma Reactor, Mark A. Sobolewski and James K. Olthoff, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Measurements of the voltage and current of radio-frequency discharges can be used to detect plasma etching endpoints and monitor the conditioning of plasma reactor surfaces. However, the mechanisms that relate plasma electrical characteristics to surface properties are not well understood. We have therefore undertaken a detailed investigation of these mechanisms. In this work, voltage and current waveforms were measured at both electrodes of a parallel-plate reactor as gas composition and surface conditions were varied. To further characterize the electrical properties of the plasma, the rf voltage in the glow region of the plasma was measured, using a capacitive probe. Absolute concentrations of gas-phase species were measured simultaneously using a calibrated mass spectrometer.

Plasma electrical properties can depend indirectly on surface chemistry, through changes in the gas-phase concentration of species arising from the surfaces. We have also observed, for aluminum surfaces exposed to oxidizing plasmas, electrical changes that depend directly on surface conditions. At fixed gas-phase oxygen concentration, slow changes were observed in plasma impedance and dc self-bias, due to absorption of oxygen species by the aluminum. The electrical changes arise because the adsorbed oxygen increases secondary electron emission from the aluminum surface, producing greater current density in the plasma. The electrical changes can be reversed by exposing the surface to an argon plasma, which removes the adsorbed oxygen by sputtering.

We have developed a model of the electrical behavior of rf discharges
that can be used to interpret the data and to distinguish changes in surface conditions from competing effects. The model suggest that electrical sensors of surface conditions would succeed in a wide variety of plasmas. The model also allows values of other relevant plasma properties (such as the plasma potential and incident ion energies) to be obtained from the electrical data.


We have tailored field-emission tips with differing geometrical shapes using controlled electrochemical etching in combination with nanofabrication techniques including focused ion beam milling and reactive ion etching. Current investigations include studying the effects that a micromachined Schottky electrode, formed integrally with the tip, has on the tip's emission angle, extraction voltage, and energy distribution.

These emission properties are all relevant to micro-column based electron lithography which requires electron sources exhibiting: 1) low extraction voltages (~1 kV) to accommodate low-voltage microcolumn operation, 2) narrow energy distributions to reduce the effect of chromatic aberration on spot size, 3) narrow emission angles to reduce the effect of spherical aberration on spot size, 4) high current densities to allow faster scanning for a given electron dose, and 5) stable emission to minimize unintended dose variation.

Field-emission sources, best known for their narrow energy distributions and high current densities, have the potential to satisfy all these requirements simultaneously. Conventional sources of this type are, however, notoriously sensitive to contamination and sputter damage by ionized residual gases and usually operate under ultra-high vacuum conditions (~5 × 10⁻¹¹ Torr) for stability. They also have relatively high extraction voltages (few kV) and emission angles (~10°). These properties are affected by the geometry of the tip and the tip material which can be controlled.

Recent results from our study will be presented here.


Low energy electron beam lithography has been suggested for use in high resolution patterning because of low proximity effects and high resist-sensitivity at low energy. However, resists and electron-beam columns optimized for use in low energy lithography are not yet well developed. Micro-column lithography is one approach which addresses the difficulties of obtaining a finely focused, low energy electron beam. Using a miniature, micro-fabricated electron column (a few mm in length), scanning electron beams with diameters of tens of nm at 1 keV have been achieved. The response of resists to electrons of different energies must be considered for the development of low energy resists. For instance since electrons at low energy have very shallow penetration depths, the resists has to be very thin (tens of nm for 1 keV electrons). Some resists are capable of high resolution pattern transferring, but because of their low electron sensitivities at high energies, they are impractical to employ for conventional lithography. These resists are, however, attractive for use in low energy lithography because cross sections for 1 keV electrons are approximately two orders of magnitude higher than those for 30 keV electrons.

We are currently investigating several promising processes for use in low energy, high resolution pattern transferring. The resists are exposed using a low energy, field emission SEM, which has been modified to include a pattern generator. It has been used successfully to expose patterns with 1 keV electrons on PMMA and self-assemble monolayers. In addition, a Monte-Carlo program has also been developed for simulating the non-classical, low energy electron-matter interactions. We will discuss our most recent experimental results which include comparisons to the predictions of Monte-Carlo simulations.


MS-TuP9 The Design and Testing of a Multi-Task, Multi-Instrument Sample Transfer System, S. Thevuthasan and D. R. Baer, Pacific Northwest Laboratory, Richland, WA and J. N. Worthington, T. R. Howard, and J. R. Munn, Thermionics Northwest Instruments, Port Townsend, WA.

A prototype multi-instrument, multi-task UHV sample transfer system has been developed for integration with a wide range of synthesis and analysis instruments. This capability will be utilized in the Environmental Molecular Sciences Laboratory (EMSL), a new U.S. Department of Energy user facility under construction at Pacific Northwest Laboratory (PNL). The EMSL will have several state-of-the-art systems that can be used by scientists from around the world. A primary focus of EMSL is understanding the molecular level interactions that influence waste processing and contaminant transport in the environment. Because interfaces play a large role in these environmental issues, there is a significant element of interface science in EMSL. The specimen transfer capability allows a sample to be synthesized, processed, and characterized by several surface science techniques without exposing the sample to air. The temperature range of the specimen can be as high as 2000 K during heating and as low as 50 K during cooling. Components of the system are designed to allow pumped or ambient transfer of specimens from different locations. Several tests have been performed to verify the functional limitations and capabilities of the system. We will discuss the design and the capabilities of the transfer system along with test results.
Wednesday Morning, October 26, 1994

SURFACE SCIENCE
Room A205 – Session SS1-WeM

Surface Magnetism I
Moderator: H. Hopster, University of California, Irvine.

8:00 am SS1-WeM0 Spin-Polarized Electron Measurements: Foundations of Surface Magnetism Research, Robert J. Celotta and Daniel T. Pierce, Electron Physics Group, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Electron spectroscopies have long been used in investigations of atoms and solid surfaces. Although the number of electrons at a given energy or momentum resulting from a particular emission or scattering process is typically measured, new insights are obtained in many investigations if another parameter is considered, namely, the electron spin polarization. Such measurements have been facilitated in the last two decades by the development of improved electron polarization techniques, i.e., the GaAs polarized electron source and a variety of spin polarization analyzers. Surface magnetic properties such as the spontaneous magnetization, Curie temperature, temperature dependence of the magnetic order, anisotropy, spin-dependent electronic structure, magnetization curves, intermediate excitation, and magnetic microstructure have been determined with spin-dependent techniques.

Examples will be drawn from the work of the Electron Physics Group at NIST and from other laboratories. The outgrowth of this early work is evident in the current exciting investigations involving electron spin polarization.

INVITED


We have used a new spin-polarized low energy electron microscope (SPELEM) to measure the topographic and magnetic domain structure of thin layers of Co on W(110). In contrast to the first SPELEM instrument, the design of this new instrument is characterized by three main features: a) compactness of construction, b) multi-directional spin electron gun, and c) integration with a versatile sample preparation and exchange system. The new GaAs based and laser excited spin gun uses a combination of electrical and magnetic beam deflection elements to allow positioning of the electron beam polarization vector in any direction with respect to the sample surface.

Lateral resolution obtained to date with this microscope is in the order of 20 nm. These investigations in the magnetic contrast for different angles of the electron beam polarization, both in and out of the plane of Co/W(110) sample, will be discussed. SPELEM images have been measured both as a function of θ at constant ϕ and as a function of ϕ at constant θ. These images show that the magnetization of the domains is fully in-plane for 3-6ML Co, in contrast to the considerable out-of-plane magnetization previously measured for Co/Al(111) by secondary electron microscopy with polarization analysis (SEMPA).

Correlations between the magnetic and topographic structure of the Co/W(110) sample will also be shown.

2. K. Grzela and E. Bauer, to be published.
3. T. Duden and E. Bauer, to be published.


We have studied the spin polarization of the 2p3/2 main line and "6-eV" satellite photoemission features from Ni(110) by using Spin-Resolved X-ray Photoelectron Spectroscopy (SRXPS). The main line of the 2p3/2 spectrum displays a significant minority-spin polarization while the satellite displays a strong spin polarization and sizable exchange splitting of 0.5 eV. The observation of a finite polarization in the main line at a finite state valence configuration using core hole creation is not 3d6 for the main peak, in contrast with previous assignments, but is more likely 3d7. The SRXPS data directly reveal that the 3d electron count in the satellite is smaller than in the main line, and is most likely 3d5.

9:40 am SS1-WeM5 Electronic and Magnetic Properties of Pseudo-Domorphism Ferromagnetic Alloy Films on Cu(100), G. J. Mankey, S. Z. Wu and R. F. Willis, Department of Physics, The Pennsylvania State University, University Park, Pennsylvania 16802.

Ultrathin films of Fe, Co and Ni on Cu(100) exhibit a variety of magnetic phases which depend on film growth conditions, film thickness and temperature. We have measured the energy band dispersions and magnetic properties of these films. Fe films exhibit a perpendicular magnetization below 6 ML which switches to in-plane for thicker films. Co films exhibit an in-plane magnetization for both ultrathin and thick films. Ni films exhibit an in-plane magnetization below 7 monolayers which switches to perpendicular for thicker films. By simultaneously depositing Fe or Co and Ni on Cu(100) we can produce alloy films with a controlled stoichiometry. The electronic properties of the films are measured with angle resolved ultraviolet photoemission spectroscopy and the ferromagnetic properties are measured with the surface magneto-optic Kerr effect. We show that by varying the alloy stoichiometry the average spin moment of the ferromagnet can be tuned and demonstrate the effect of tuning the spin moment on the magnetic anisotropy behavior.


10:00 am SS1-WeM6 Ferromagnetic Phases at the Fe/Si(111) Interface, J. P. Woods, Y. L. He and D. Welapes, Department of Physics and Center for Materials Research and Analysis, University of Nebraska, 116 Brace Laboratory, Lincoln, NE 68588-0111.

Fe/Si layered structures have exhibited unique magnetic properties and the ferromagnetic Fe silicides at the Fe/Si interface are investigated. Fe is evaporated on the Si(111) surface at room temperature; the Fe concentration is measured with Auger and core-level electron spectroscopy and the surface order is monitored with low energy electron diffraction. The magnetic phases of atomically thin layers of Fe/Si(111) are identified with magneto-optic Kerr effect and photoemission experiments. Ferromagnetic ordering is first observed with Fe/Si Auger intensity ratio of 1.2 and an additional ferromagnetic phase (α Fe) is evident in hysteresis loops with additional Fe evapo-
The photoemission spectra are dominated by the Fe 3d electrons even at Si rich non-ferromagnetic phases, and the spin-resolved valence-band structure is measured. The diffusion of Fe into the Si substrate with various annealing steps and the diminishing ferromagnetic order is investigated.

Supported by ACS-PRF and NSF-EPSCoR.

10:20 am SS1-WeM7 Circular Dichroism in Core-Level Photoemission, H. Xiao\textsuperscript{ab}, A. P. Kaduwela\textsuperscript{c}, M. A. Van Hove\textsuperscript{d}, and C. S. Fadley\textsuperscript{ab}, \textsuperscript{a}Department of Physics, University of California, Davis, CA 95616, \textsuperscript{b}MSD, Lawrence Berkeley Laboratory, Berkeley, CA 94720. We have calculated the circular dichroism associated with core-level photoemission from both non-magnetic and magnetic systems using a multiple-scattering photoelectron diffraction theory. Our simulations for the non-magnetic cases of Cs 1s emission from CO on Pd(111) and Si 2p emission from Si(100) well predict the dichroism found in experimental results due to Schönfeng et al. \cite{1} and Daimon et al. \cite{2}, respectively. Magnetic circular dichroism has also been treated for the case of Fe 2p emission from Fe(110), including a generalization of the theory to allow for spin-orbit coupling in the initial state and spin-dependent scattering in the final state. Both total intensities and spin-resolved intensities resulting from circularly-polarized excitations will be discussed. A strong interplay between normal (non-magnetic) dichroism and magnetic dichroism is found. Photoelectron diffraction theory thus provides a general framework for modeling dichroism in core-level photoemission form both non-magnetic and magnetic systems.

\begin{thebibliography}{1}
\end{thebibliography}

10:40 am SS1-WeM8 Spin-Specific Photoelectron Diffraction Using Magnetic X-Ray Circular Dichroism, J. G. Tobin, and G. D. Waddill, Lawrence Livermore National Laboratory, X. Guo and S. Y. Tong, University of Wisconsin-Milwaukee. The first observation of spin-dependent photoelectron diffraction using circularly-polarized x-rays is reported for monolayer ferromagnetic Fe films on Cu(001). Circularly-polarized x-rays are used to produce spin-polarized photoelectrons from the Fe 2p spin-orbit split doublet, and intensity asymmetries in the 2p3/2 level of \( \approx 3\% \) are observed. The asymmetry is dependent on the relative orientation of the x-ray polarization vector and the magnetic moment, and is sensitive to the electron kinetic energy. Our results are compared to multiple scattering calculations which reproduce the experimental observations. A strong angular dependence is predicted by theory and is observed in the experiment. This spin-dependent technique promises the direct, element-specific determination not only of local atomic structure, but magnetic structure as well.

Work performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract number W-7405-Eng-48. Work at Wisconsin was supported by the US Department of Energy.

**SURFACE SCIENCE**

**Room A201 - Session SS2-WeM**

**Reactions on Metals: Diatomics**

**Moderator:** G. B. Fisher, General Motors Research & Development Center.

8:00 am SS2-WeM0 Structure Sensitive Selectivity of the NO+CO Reaction over Rh(110) and Rh(111), C. H. F. Peden, Pacific Northwest Laboratory\textsuperscript{*}, Richland, WA 99352, D. N. Belton and S. J. Schmiegel, General Motors Research and Development Center, Warren, MI 48090.

We have studied the effects of temperature, NO conversion, and NO:CO ratio on the activity and selectivity of the NO+CO reaction at high (1 Torr < P < 100 Torr) pressures over the Rh(110) and Rh(111) surfaces. Under the conditions used in this study, the NO+CO activity is between 1.3 and 6.3 times faster over Rh(110) than over Rh(111).

The (110) surface exhibits a lower apparent activation energy (\( E_a \)), 27.2 vs. 34.8 kcal/mol, than does the (111) surface. Although the turnover numbers for NO reaction can be quite similar on the two different surfaces, we find large differences between Rh(110) and Rh(111) with regard to their selectivities for the two competitive nitrogen-containing products, N\(_2\)O vs. N\(_2\). The more open Rh(110) surface tends to make significantly less N\(_2\)O than Rh(111) under virtually all conditions that we probed with these experiments. These results can be understood in terms of the relative surface coverages of adsorbed NO and N-atoms on the two surfaces. Notably, more facile NO dissociation on Rh(110) appears to lead to greater steady-state concentrations of adsorbed N-atoms than is present on the (111) surface. Higher N coverages on the (110) surface favor N-atom recombination (N\(_2\) formation) more than the NO+ N reaction (N\(_2\)O formation) on Rh(110) relative to Rh(111). Indeed, Rh(110) surfaces were found to be largely composed of adsorbed N and lesser quantities of NO in post-reaction XPS measurements. In contrast, Rh(111) surfaces only showed XPS features due to adsorbed NO.

*Pacific Northwest Laboratory is operated for the U.S. Dept. of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO 1830.

8:20 am SS2-WeM1 Dissociation Kinetics of NO on Rhodium(111), H. J. Borg, R. A. van Santen and J. W. Niemantsverdriet, Schuit Institute of Catalysis, TU Eindhoven, P.O. Box 513, 5600 MG Eindhoven, The Netherlands.

Temperature programmed static ion mass spectrometry (TPSIMS) and temperature programmed desorption (TPD) have been used to study the kinetics of adsorption, dissociation, and desorption of NO on Rh(111). At 100 K, NO adsorbs molecularly via mobile precursor state kinetics. SSSIMS suggests threefold adsorption at low coverage, with increasing amounts of bridged NO at higher coverages. Three characteristic coverage regimes appear with respect to NO dissociation. At coverages below 0.25 ML, NO dissociates completely at temperatures between 275 and 340 K, with an activation energy of \( 39 \pm 6 \) kJ/mol and a preexponential factor of \( 10^{6.1} \) s\(^{-1}\). Nitrogen atoms desorb as N\(_2\), with kinetics that is strongly influenced by the presence of coadsorbed oxygen. In the medium coverage range, 0.25 < \( \theta_{\text{O}_2} < 0.50 \) ML, part of the NO desorbs molecularly, with a desorption barrier of \( 116 \pm 10 \) kJ/mol and a preexponential of \( 10^{6.4(1.0)} \) s\(^{-1}\), suggestive of a mobile transition state. Dissociation of NO becomes progressively inhibited, the onset shifting from 275 K at 0.25 ML to 400 K, the NO desorption temperature, at a coverage of 0.50 ML. On these highly covered surfaces nitrogen atoms become destabilized, resulting in an additional low temperature N\(_2\) desorption state. For initial NO coverages higher than 0.50 ML, NO dissociation is completely self-inhibited, indicating that all sites required for dissociation are blocked. The desorption of the more weakly bound bridged NO does not generate the sites required for dissociation; these become only available after the desorption of triply coordinated NO.

8:40 am SS2-WeM2 Kinetics of the NO+CO+O Reaction over Rhodium, H. Pernama, D. N. Belton, S. J. Schmiegel, General Motors R&D, Warren, MI 48090-9055, and K. Y. Simon Ng, Wayne State University, Detroit, MI 48202.

The competition between NO and O\(_2\) to be the oxidant for CO is one of the primary determinants of the amount of NO emitted from the catalytic converter of a passenger car. Because Rh is the most active NO reduction catalyst, we have an ongoing program to understand the NO reduction over Rh single crystal catalysts with a combination of moderate pressure reaction kinetics and UHV measurements of elementary steps. Previously we focused on the NO+CO reaction over Rh(111) and Rh(110) and demonstrated that reaction can be understood in some detail when we have reliable measurements of the rates of elementary steps. In this talk we present new kinetic measurements for the NO+CO+O\(_2\) reaction over Rh(111) and show that the understanding we gained in the simpler NO+CO system extends quite readily to the more complex ternary mixture. Under our reaction conditions, addition of O\(_2\) to the NO+CO mixture has no effect on the NO reduction rate, the N\(_2\) and N\(_2\)O formation rates, nor the NO and CO pressure dependencies. In addition, we find that O\(_2\) is consumed with kinetics that are remarkably similar to those observed for the CO+O\(_2\) reaction. We understand these kinetics to mean that the dissociative adsorption of oxygen is proceeding on the surface vacant sites that are left-over by the NO+CO reaction. Surface NO and N\(_2\)O coverages are not affected by gas phase oxygen because the CO+O\(_2\) elementary step is fast so that surface O\(_2\) coverages remain quite low during the NO+CO+O\(_2\) reaction. Qualitatively, our
results suggest that for the NO + CO + O₂ system, both the NO + CO + O₂ reactions are occurring on the same surface with very little recognition that the other reaction is simultaneously occurring.

9:00 am  SS2-WeM2 A Model for Chaos in Oscillatory Surface Reactions: Pt(100)/NO + CO + O₂, G. Vazer, R. Imbihl and A. Mikhailov, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, D-14195 Berlin (Dahlem), Germany.

The NO reducing reactions with CO or H₂ on Pt(100) exhibit a number of similarities with respect to their dynamical behavior. Both reactions show sustained rate oscillations in the 10⁻⁴ mbar range which take place on a largely hex-reconstructed surface, and both oscillating systems display a periodic oscillatory cascade into deterministic chaos. Rate oscillations are found in the NO + CO reaction take place on a macroscopically uniform surface, i.e. the different local oscillators are completely synchronized via the gas phase. We propose a model for the occurrence of deterministic chaos in both reactions which is based upon the breakdown of synchronization as the system becomes chaotic.

The key feature of the model is the synchronization via a critical dependence of the 1x1 + hcp phase transition on PCO and PNO leading to nonlinear growth kinetics of 1x1 islands. The resulting 3-variable model exhibits complex dynamical behavior due to the time delay between generation and dissolution of 1x1 islands. Since the equations have been constructed on the basis of experimental data, we consider them to be the first realistic model for the occurrence of deterministic chaos in oscillatory surface reactions.

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9:20 am SS2-WeM4 Particle Cu on Ordered Al₂O₃ Reactions with Nitric Oxide and Carbon Monoxide, Ming-Cheng Wu and D. Wayne Goodman, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255.

The growth of small Cu particles, prepared by vacuum vapor deposition, on Al₂O₃ films on Mo(100) has been studied in the 80-800K temperature range with Auger electron spectroscopy (AES) and temperature programmed desorption (TPD). A model based on Auger measurements has been utilized to examine the number density and the average size of the Cu particles on Al₂O₃. Nitric oxide decomposition on the Cu particles has been further investigated using TPD and high resolution electron energy loss spectroscopy (HREELS). TPD and HREELS data clearly show that a fraction of NO molecules reacts with the Cu particles to produce nascent nitrogen and NO₂ in the 90-250K temperature range. The formation of N₂ at 77K is also evident and is due to the recombination of adsorbed N atoms from NO dissociation.

The effects of retained oxygen, adsorption temperature and particle size on the production of high-temperature N₂ have been examined. The reaction between nitric oxide and carbon monoxide, and CO₂ products arising from NO decomposition, a small quantity of CO₂ produced by the reaction between adsorbed CO and adsorbed oxygen.

9:40 am SS2-WeM5 The Effect of Oxidizing and Reducing Environments on the Regeneration of a Sulfur Deactivated Pd Foil Catalyst, Craig L. DilMaggio and Donald D. Beck, General Motors NAO R&D Center, Warren, MI 48090-9555.

A Pd foil has been treated in a reactor cell/UHV chamber with O₂, H₂, and SO₂ in an attempt to understand the mechanism by which sulfur poisons the Pd metal in a catalyst and test the effects of oxidizing and reducing environments on regenerating the catalytic activity of the Pd. Results indicate that the chemical state of the adsorbed surface sulfur to be very sensitive and independent of treatment scheme. Auger measurements of the surface strongly suggest that the sulfur penetrates the surface at temperatures in excess of 300°C and then redissolves out at temperatures above 500°C.

Although sulfur severely reduces the ability of the Pd catalyst to oxidize CO by blocking CO adsorption sites, the degree to which catalyst activity could be regained was dependent on the gas mix used during or after the SO₂ exposure. Auger measurements show that lean environments during SO₂ exposure maintain 50% to 75% of the CO adsorption activity while post exposure of a sulfur deactivated catalyst to O₂ removes greater than 90% of the sulfur present at relatively low temperatures. Conversely, rich environments (H₂) during SO₂ exposure or post exposure to H₂ were ineffective in removing more than 20% of the sulfur from the catalyst even though significantly higher temperature treatments were employed. This evidence supports the observation that a sulfur deactivated Pd foil under reducing conditions will not regain CO oxidation activity while oxidizing environments effectively remove sulfur and restore activity.

10:00 am SS2-WeM6 Structural Effects of Well-Defined Palladium Surfaces on CO + NO Oxidation and Reaction, Scott M. Vesecky, Xianwu Xu, Peijun Chen and D. Wayne Goodman, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255.

The coadsorption of CO and NO has been studied on well-defined Pd particles supported on silica and alumina and compared with coadsorption data on low index single crystals. At low pressures (<10⁻⁵ Torr), temperature programmed reaction spectroscopy (TPRS) was used to study the desorption temperatures of the reactants (CO and NO) and the products (CO₂, N₂ and N₂O). Under these conditions, N₂ production is strongly correlated to a high temperature (600 K) desorption feature. The concentration of this high temperature state and the rate of N₂O production increases with increasing Pd particle size (100-500 Å). At higher pressures (2-20 Torr), infrared reflection absorption spectroscopy (IRAS) was used to monitor the surface intermediate and gas phase product concentrations at thermal equilibrium. On the close-packed surfaces (Pd(111)) and large (>100 Å) Pd particles, a high temperature (600 K) NO state was again observed, which strongly correlates to the gas phase production of N₂O. On the lower coordination sites of Pd(100) and Pd(110) and smaller Pd particles (<100 Å), NO was found to be considerably more stable, with desorption occurring at 500 K under both high and low pressure conditions. The fact that N₂O production was practically negligible in comparison to N₂O production on these low coordination sites has important implications for the design of Pd-only exhaust gas catalysts. The strong correlation between the low and high pressure data validates the comparison of UHV measurements with higher, more "realistic" adsorption and reaction conditions in these experiments.

10:20 am SS2-WeM7 On the Kinetics of Methanation of CO on Nickel, J. Alstrup, Haldor Topsøe Research Laboratories, Nymøllevej 55, DK-2800 Lyngby, Denmark.

Number of macrokinetic models have in the past been suggested to account for experimental rates of CO methanation on nickel catalysts. However, it turns out that none of them can account for the published results obtained for nickel single crystal surfaces or foils. A set of microkinetic models with formation of carbon as an intermediate followed by stepwise hydrogenation of the carbon to methane is presented. Most of the parameters are determined from surface science experiments or from theoretical calculations. Agreement is obtained with the single crystal and foil results for a model with the hydrogenation of the CH⁺ surface intermediate as the rate controlling step. Analysis of this model shows that the surface is almost saturated by CO in the temperature range where the overall reaction rate is given by E₃, the reaction is approximately constant E₃ at temperatures below 400°C. E₃ is mainly determined by the chemisorption energy of CO in this temperature region. The decrease of E₃ at higher temperatures is mainly due to changes in the equilibrium constants of the hydrogen chemisorption and of the methyl formation step.

10:40 am SS2-WeM8 Experimental and Numerical Study of OH Radical Distribution and Temperature above a Platinum Foil in H₂/O₂ Mixtures, F. Behrendt, F. Gudmundson, B. Kasemo, A. Rosen, 1Department of Physics and 2Department of Applied Physics, Chalmers University of Technology and University of Göteborg, S-412 96 Göteborg, Sweden.

During the last years the formation of water in catalytic reactions of H₂ and O₂ on a platinum foil in a flow reactor at pressures from 10 to 200 mTorr have been studied. The formation of water proceeds through dissociative adsorption of molecular hydrogen and oxygen giving hydrogen and oxygen atoms on the surface, which then recombine to OH radicals. The vast majority of OH radicals are consumed in the formation of water, but if the catalytic foil is kept at high temperature a minor fraction of them leave the surface where they can be detected using laser-induced fluorescence (LIF). The studies have given detailed information about surface reaction steps.

In this work a flow of hydrogen and oxygen of various compositions at a total pressures of 0.2 Torr is directed towards the foil, which is resistively heated to 1000-1400 K. Under steady-state conditions two-dimensional LIF (2D-LIF) is used to determine the distribution of OH radicals outside the heated platinum foil. The fluorescence light from OH radicals excited by an excimer pump dye-laser system is imaged with a two-dimensional intensified CCD camera, and is used
to determine relative concentration profiles and the temperature outside the foil. A code to simulate laminar reactive stagnation point flows above catalytically active surfaces is applied to model the experiment. A detailed mechanism with elementary reactions is used to describe the gas phase reactions as well as a detailed mechanism for the surface reactions. The gas phase transport is described by a multi-component transport model. The experimentally found spatial distribution of OH radicals and temperatures as function of the distance from the platinum foil are compared with numerical results. The results are found to be well predicted by the detailed model calculations.

**NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY/MANUFACTURING SCIENCE AND TECHNOLOGY**

**Room A209 – Session NSMS-WeM**

**Industrial Applications of Scanned Probes**
Moderator: J. E. Griffith, AT&T Bell Laboratories.

8:00 am NSMS-WeM0 Industrial Applications of Friction Force Microscopy, J. Burger, A. Leijtala, M. Bingley and R. Christoph, Centre Suisse d’Electronique et de Microtechnique S.A. (CSEM), 2007 Neuchâtel, Switzerland.

Scanning probe microscopy has become a well established tool to enhance knowledge about nanometer scale properties of materials and has found applications in many fields of scientific interest. A new instrument developed at CSEM, the Atomic Scale Tribometer, is a very compact standalone force and friction microscope is being presented and its application to industrial problems, like surface characterization in general and questions of quality insurance in particular will be discussed. Due to the particular standalone design, our SFM/FFM is especially well suited to investigate samples under working conditions, i.e. realistic industrial environments as oils or other liquids (e.g. corrosive), or to investigate large samples as there is no need to scan the sample itself. A recently developed software module enables for the distinction of chemical heterogeneities on the sample surface by generating a friction histogram from the simultaneously acquired normal and lateral force data, thus providing further local analytical information on cleanliness and process yield. The characterization of surface roughness on bonding capillaries for microelectronics as well as nanotribological studies on SiC-Coatings and quantitative 3-dimensional measurements on prototype optical structures will be presented.

8:20 am NSMS-WeM1 Industrial Applications of Proximal Probes, H. Fuchs, Universität Münster, Physikalisches Institut, D-48149 Münster.

Proximal probe microscopes have displayed great versatility in analyzing materials from a wide range of fields and with varied properties. The scanning force microscope not depending on electrically conducting surfaces is currently of special interest of many industrial applications such as surface roughness measurements, local friction and stiffness investigations. Its derivatives such as magnetic force microscopy will be also developed as additional appropriate methods for industrial applications [1].

As compared to conventional surface analytical methods, AFM does not require any specific surface preparation techniques or vacuum conditions.

Examples for the industrial applications of proximal probes are given. The potential of novel techniques such as the scanning near field acoustical microscope (SNAM) and the scanning near field optical microscope (SNOM) for industrial applications are discussed.

9:20 am NSMS-WeM4 Scanning Tunneling Microscopy/Spectroscopy Doping Determination of Si and GaAs Bulk Materials and Device Structures, R. M. Silver, J. A. Dagata and W. Tseng, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Scanning tunneling microscopy/spectroscopy (STM/STS) of n- and p-doped bulk GaAs and Si was performed in air and UHP using recently developed passivation techniques to better understand the electronic nature of the passivated surfaces and air ambient STM [1]. The spectroscopic data, obtained over a range of doping densities for both Si and GaAs, is interpreted in a previously proposed model which includes thermoionic emission, space-charge tunneling, and vacuum tunneling [2]. Here, we extend this work to an investigation of the electronic properties of GaAs pn-junction heterostructures and Si device structures using STM imaging and position-dependent current-voltage spectroscopy. These results can be well understood by including electronic tip-induced effects on the bulk pn-junction depletion characteristics and surface states. This complete treatment of bandbending correctly predicts the apparent pn-junction locations observed in the STM imaging mode and the current-voltage characteristics across the junction.

9:40 am NSMS-WeM5 Ultra-High Density Data Storage Based on Proximal Probes, J. H. Mamin, B. Terris, S. Hoon, L. S. Fan and D. Rugar, IBM Research Division, Almaden Research Center, 650 Harry Road, San Jose, CA 95120.

Scanning probe microscopes have been used in a variety of ways to "write" on surfaces, on scales ranging from sub-micron down to the atomic. This technology thus forms an attractive approach to high density data storage, provided that some of the fundamental problems can be overcome. A number of formidable technical challenges must be met in order to produce a device with realistic data rate, error rate, and overall reliability. We will review a number of the modification techniques which have been demonstrated, and discuss their suitability for data storage. In particular, we will describe some techniques based on the atomic force microscope which appear to be among the most promising. We will also look at an alternative approach based on near-field optical recording with a high index solid immersion lens.

**INVITED**

**10:20 am NSMS-WeM7 Quantitative Dopant Profile Measurements on Si by Scanning Capacitance Microscopy, Y. Huang and C. C. Williams, Physics Department, University of Utah, Salt Lake City, UT 84112 USA, J. Slinski, IBM Microelectronics, Essex Jct., VT 05452 USA.**

The direct measurement of local dopant profiles on a nanometer scale will benefit sub-micron semiconductor device design and simulation. Presently, a nanometer scale capacitance voltage (C-V) method for the quantitative measurement of the dopant density distribution near a semiconductor surface is being established. An atomic force microscope is used to position a nanometer scale tip at a semiconductor surface, and local capacitance measurements are performed by a high-sensitivity capacitance sensor. The C-V measurements have been performed with tips of sub-50nm radius on test wafers in which the dopant...
density varies laterally by more than four orders of magnitude in a region of 300 nm. The experimental SCM data was inverted to provide quantitative dopant density profiles. The inverted dopant profiles were compared with profiles obtained by process simulation. First order agreement was found between the SCM profiles and the simulation profiles.


In order to meet the challenge of miniaturization of microelectronic devices on integrated circuits there is an increased demand for nanometer scale diagnostic tools for mapping out semiconductor doping profiles. We recently developed the scanning resistance microscope (SRM) as a nanometer scale technique for two dimensional delineation of P-N junctions [1]. The SRM utilizes a conducting probe to perform localized resistance measurements while scanning over a surface. Experimental results show that this technique can localize a P-N junction with a lateral spatial resolution of less than 35 nm. We will present images of surface polysilicon that demonstrate an artifact of the SRM technique where topographic features are coupled into resistance images. We will also be presenting results of SRM imaging of implant cross sections and silicon on insulator cross sections. In addition we will be presenting results from a new imaging technique that uses voltage modulation to maintain a constant current. This technique should overcome some of the limitations of the SRM and enhance the accuracy and dynamic range of the SRM technique.


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NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A207 - Session NS2-WeM

Atomic Manipulation
Moderator: H. Tokumoto, Joint Research Center for Atom Technology, Japan, and L. J. Whitman, Naval Research Laboratory.

8:00 am NS2-WeM0 Theory of Atom Extraction by the Scanning Tunneling Microscopy from the First-Principles Calculation, Kenji Hirose and Masaru Tsukada, Department of Physics, University of Tokyo, Tokyo 113, Japan.

We present a method of the first-principles calculation of the electronic states under strong fields and currents. This method is effective for the biocircuit systems with atomic structure. A microscopic distribution of the fields and currents is calculated self-consistently with the electron density in the surface region. Here the method is applied to the problem of atom extraction by the tip.

The picture shows the electron density for the surface bias of +8 V.

It is found, when a bridge structure of the electron density is formed between the tip and the surface, a strong extracting force is acted on the top surface atom. This explains the extreme specificity of the extraction site found in the experiments.

8:20 am NS2-WeM1 Investigation of the Mechanism for Removing Atoms from Si(001) Surfaces with the STM*, C. T. Salling and M. G. Legally, Univ. of Wisconsin-Madison, Madison, WI 53706.

We have used the scanning tunneling microscope (STM) to define regular crystalline structures as small as two atoms wide and one atom thick [1] by removing atoms from the Si(001) surface at small values of tip-sample separation (1-3 Å). Three types of features can result from an attempt to remove atoms [2]: a hillock, a hillock surrounded by a moat, or a pit. We have investigated the nature of the atom removal process by 1) measuring the probability of forming the three types of features as a function of voltage and tip-sample separation, 2) measuring the threshold voltage for forming a pit as a function of tip-sample separation, and 3) monitoring the piezo drive voltage and current during a removal attempt. At a tip-sample separation of 1.5 Å, we find that the most probable outcome is a hillock for low voltages, a hillock surrounded by a moat for higher voltages, and a pit for voltages at or above the threshold value. The threshold voltage is observed to increase with increasing tip-sample separation. The current during a removal attempt has been used to determine whether the atoms are transferred during or after the voltage pulse. We will use our results to evaluate possible mechanisms for atom removal, including chemically-assisted field evaporation [2] and simple thermal evaporation, and examine the hypothesis [2] that a bridge of atoms forms between the tip and sample as an intermediate state.

*Research supported by the NSF and AFOSR.

8:40 am NS2-WeM2 Atomic-scale Modification on Si(111) x 7 Surfaces, N. Lu, Q. J. Gu, Z. L. Ma, W. B. Zhao, X. Chu, Z. Q. Xue, S. Pang, Beijing Laboratory of Vacuum Physics, Academia Sinica, Beijing 100080, P. R. China.

As silicon is a very important material, so nanometer-scale structure modification on silicon surfaces is essential in developing new quantum devices. To do so, reliable modification and manipulation techniques must be found. We present a method to manipulate atom clusters and single atoms on silicon surfaces using the scanning tunneling microscope. With this method, we can remove a single atom at a predetermined site as well as deposit a single atom on a selected position. With this method, we can reproducibly scoop out nanometer-scale hollow lines (grooves), specially when it is along one of the three lattice elementary-vector-directions, the grooves appear a very regular structure, the linewidth can be easily controlled to be less than 1.5 nm. With this method we can routinely and alternatively create hollow lines (grooves) and protruding lines (ridges) on the Si(111) x 7 re-constructed surface. We also give a detailed discussion about the mechanical principle of the process.

9:00 am NS2-WeM3 Nanometer Scale Patterning of Silicon Surfaces with an Ultrahigh Vacuum Scanning Tunneling Microscope*, J. W. Leding, Department of Electrical and Computer Engineering and Beckman Institute, University of Illinois at Urbana Champaign.

Hydrogen passivation on silicon represents the simplest possible resist system for nanolithography experiments. We have achieved one nanometer resolution on the Si(100)-2×1:H monohydride surface by selectively desorbing hydrogen with an ultrahigh vacuum (UHV) scanning tunneling microscope (STM). The monohydride surface is prepared by exposing a heated sample (650K) to atomic hydrogen. This surface is structurally and chemically uniform and ideal for subsequent UHV-STM patterning. By operating the STM in field emission, the hydrogen passivation is selectively desorbed leaving behind atomically clean silicon. Depassivation occurs when the classical kinetic energy of the STM electrons exceeds the Si-H bond strength, and approximately 2×10^4 electrons are required to depassivate each silicon atom. The atomic hydrogen recombines to evolve as H_2, preventing repassivation of previously patterned areas of the surface. Depassivation of individual atomic dimer rows is possible by this technique. Results will be shown that demonstrate the variations in patterning as functions of the electron energy and dose. The recovery of clean silicon after STM patterning is suggestive of many interesting possibilities for selective surface chemistry. We have demonstrated selective oxidation.
of the patterned areas without degradation of the pattern resolution. Progress in applying these techniques to the nanofabrication schemes will also be presented.

INVITED

9:40 am  NSW-WeM5 Nanolithography with the STM on Si, N. Kramer, J. Jorissen, H. Birk and C. Schonenberger, Philips Research, Eindhoven, The Netherlands.

The STM has been used to locally oxidize hydrogen passivated silicon and hydrogenated amorphous silicon (a-Si:H). Oxide lines as narrow as 5 nm have been made on H-passivated Si. The oxide pattern acts as an etch mask. A proper choice of etch liquid enabled us to transfer 50 nm wide oxide lines 500 nm deep into the substrate; yielding trenches with a width below 50 nm and an aspect ratio > 10.

Oxide patterns can also be made in thin films of a-Si:H. a-Si:H clearly has the advantage that it can be deposited in very thin films (< 20 nm) on almost any surface, this makes a-Si:H a resist suitable for many applications.

In addition to the STM patterning, we studied the chemistry of electron induced oxidation. An UHV system was built that allowed us to expose a Si surface to electrons in an oxygen environment. Auger analysis revealed information about the oxide thickness as a function of electron energy and dose and the oxygen pressure.

The direct patterning of oxide on Si is not only interesting for the fabrication of etch masks, many other applications are possible. The oxide patterns can form a mask for selective growth of metals with CVD or for the selective chemisorption of molecules.

10:00 am  NSW-WeM6 Local Deposition of Si on Si(111)(7\times7) with the STM, H. Rauscher, U. Memmert, R. J. Behm, Abteilung für Oberflächenchemie und Katalyse, Universität Ulm, D-89069 Ulm, Germany.

In search for a method to produce small artificial surface structures, the STM is used for direct local deposition of Si from a SiH₄ precursor on Si(111)(7\times7). For material deposition, the tip is brought into the field emission regime and the emitted electron beam is utilized for direct proximal probe lithography at room temperature. The SiH₄ precursor gas is activated by electron bombardment from the STM tip with energies from 40 to 100 eV and emission currents of up to 100 nA. This produces local accumulation of the dissociation products on the surface just below the tip. Direct writing of nanostructures with lateral dimensions of down to 40 nm is accomplished. Structures with a height from ~3 Å (1 ML) up to ~50 Å have been produced. Deposition and imaging of the cracking products is carried out with the same tip. Possible deposition/activation mechanisms and the chemical nature of the deposited species will be discussed.

10:20 am  NSW-WeM7 Nanofabrication with Laser Focused Atomic Deposition, J. J. McClelland, R. E. Scholten, R. Gupta, and R. J. Celotta, Electron Physics Group, National Institute of Standards and Technology, Gaithersburg, MD 20899 USA.

When an atomic beam passes through a laser beam, the optical field can act as a lens, focusing the atomic beam to a spot which can reach the nanometer scale. Such a laser-atomic beam can be used to focus atoms as they deposit onto a substrate, creating nanostructures. When the laser field is generated in an interference pattern, such as a standing wave, a large array of micro-lenses is created, each with dimension of the order of an optical wavelength. This arrangement allows massively parallel fabrication of nanostructures with potential dimensions of 10 nm or less. We have demonstrated this technique by focusing chromium atoms as they deposit onto a silicon substrate. A one-dimensional standing wave was used, and the resulting pattern is a series of lines with width 65 nm, spaced at 212.78 nm, covering an area of approximately 0.5 mm² (see figure). Future applications and extensions of this technique include two dimensional patterns, use of the chromium as an etch mask, and generalization to other atomic species and arbitrary patterns.

INVITED

Work supported in part by NSF grant PHY-9312572.

APPLIED SURFACE SCIENCE
Room A101 – Session AS-WeM

Quantitative Analysis, Emphasizing Angle-Resolved XPS
Moderator: J. T. Grant, University of Dayton.

8:00 am  AS-WeM0 Depth-Profiling by Angle-Resolved XPS and AES; Elastic Scattering Corrections, and Intrinsic Limits on Depth-Resolution, R. J. C. Campion, National Physical Laboratory, Teddington, UK.

Angle-resolved data has often been used to obtain information on variations of concentration with depth, down to about 5 nm below the specimen surface. For a decade this has been a successful method of measuring the thicknesses of overlayers, but a number of algorithms for the reconstruction of complete depth profiles have also been proposed. The usefulness of these methods is limited by the low information content of angle-resolved data; this is quantified, and used to show that the fractional depth-resolution in ARXPS or ARES is limited to:

$$\frac{\Delta z}{z} \approx 0.8 \exp\left(\frac{\epsilon}{8.9(n-1)^{1/2}}\right)$$

where $\epsilon$ is the %-precision of the peak intensities measured at n emission angles. It is shown how this limit can be improved by a factor of about 2 using a priori information which is justified for some specimens.

Elastic scattering leads to deviations from commonly used quantification equations. Results of efficient Monte-Carlo calculations are used to define precisely the angular range for which these equations are reliable.

8:20 am  AS-WeM1 Assessment of Overlayer Thickness Determination Model by Controlled Monolayers, Bruce C. Beard*, and Robert A. Briczkoard*, A) Knox Novel, 1 Livingstone Ave., Dobbs Ferry, NY, 10522, b) Naval Surface Warfare Center, 10901 New Hampshire Ave., Silver Spring, MD 20903.

Thickness determination of carbonaceous overlayers on solid surfaces is of great importance for the quantitative analysis of x-ray photoelectron spectroscopy data. Several approaches can be taken for the determination of such overlayer thicknesses. In this discussion the C(1s)/C(KW) peak area ratio method will be evaluated. This approach depends on the difference in energy dependent inelastic mean free path of the two electron emission transitions to act as a measure of the overlayer thickness. A theoretical formalism was previously presented employing a ratio of the classic overlayer intensity equation:

$$I_o = I_N \cdot T(E_o) \cdot E_o \cdot I_e [1 - \exp(-d/sin \theta \cdot \lambda(E_o))]$$

Earlier investigators have found this formalism to poorly represent

AFM image of laser focused Cr lines

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experimental data. In this study self assembled overlayers of alkane thiols have been employed to more closely evaluate the potential sources for error in the theoretical expression. Thickness of the overlayers was determined by angle dependent XPS and confirmed with published ellipsometry values. These results also do not fall on the theoretically predicted curve. Evaluation of the theoretical formalism will be discussed in light of these results to provide an indication of the source of error and means for correction.

8:40 am  AS-WeM2 Inelastic Mean Free Pathlengths of X-Ray Photoelectrons for Quantitative Investigation of Technological Ultrathin Layers, Werner H. Gries, Research and Technology Center, Deutsche Bundespost Telekom, P.O. Box 10 0603, 64276 Darmstadt, Germany.

Angle-Resolved Self-Ratio X-ray Photoelectron Spectroscopy (ARXPS) is being developed for the quantitative investigation of (disordered) technological ultrathin layers (this author, at previous AVS symposia and elsewhere). The thicknesses and stoichiometries of a succession of surface layers within a total depth of 10 nm can be derived from angle-dependent intensity measurements, provided (1) the layers are sufficiently dissimilar for each to give rise to at least one unique spectral line and (2) the inelastic mean free pathlengths (IMFP) of the characteristic x-ray photoelectrons are known for each material involved.

The rather work-intensive procedure of evaluation has now been simplified to the extent that routine application to technological surfaces is possible. The major obstacle to quantitative interpretation, i.e., the lack of IMFP data for the great variety of technological layers, has been overcome by the introduction of a new universal predictive formula which allows IMFP to be predicted for all elements, atomic mixtures and compounds of varying stoichiometry found on technological surfaces. The formula has been derived on the basis of a recently published selection of IMFP data (Tanuma, Powell and Penn) as well as a large collection of stopping power data of charged particles from various sources.

9:00 am AS-WeM3 Corrections to the Angle-resolved X-ray Photoelectron Spectroscopy with Consideration of the Solid Acceptance Angle, Wong Kaa Wai and R. W. M. Kwok, Department of Chemistry, The Chinese University of Hong Kong.

Angle-resolved x-ray photoelectron spectroscopy (ARXPS) is a non-destructive depth-profiling technique in which “lines” or “columns” of photoelectrons are assumed to be ejected from the sample. However, it is widely known that “corners” of photoelectrons are actually collected and the corner is characterised by the so-called solid acceptance angle. As most scientists neglect this factor even in quantitative XPS analysis, the author found that there was an uncertainty of over 20% for the deduced thickness of SiO2 overlayer in an ARXPS analysis on a Si02/Si sample at three polar angles: 0°, 15° and 30°, and three solid acceptance angles (controlled by iris). Therefore the author derives a set of basic equations that describe the signal intensity detected from these “corners” in terms of acceptance angle, polar angle and the thickness of layers in multilayer structure. The aim is to improve the accuracy of the current ARXPS technique.


We have studied theoretically the variation of effective sampling depths with direction for core photoelectron emission from single crystals, including the effects of isotropic inelastic scattering and single and multiple elastic scattering with real and complex phase shifts. Our calculations were carried out for emission in the 1 keV energy range on both simple chains of atoms embedded in an isotropic inelastic medium, and on larger atomic clusters that should more realistically simulate extended inelastic scattering from a semi-infinite single-crystal substrate or epitaxial overlayer. The mean emitter depth is found to vary by as much as a 30% with direction, being lowest just adjacent to low-index chains of atoms because of destructive interferences in photoelectron diffraction, and highest along low-index directions due to forward scattering, in spite of well-known reductions in intensity along these directions due to multiple-scattering damping effects. These variations of effective sampling depths due to photoelectron diffraction effects (as well as analogous Auger diffraction effects) thus should be taken into account in the quantity analysis of surfaces and surface concentration profiles using photoelectrons and Auger electrons.

9:40 am AS-WeM5 A Novel Algorithm for Quantitative Elemental Depth Profiling by Angle-Resolved XPS, John M. Williams and Thomas P. Beebe, Jr., Dept. of Chemistry, University of Utah, Salt Lake City, Utah, 84112.

We have developed an algorithm for analysis of angle-resolved x-ray photoelectron spectra (ARXPS) that allows quantitative depth profiling of the surface elemental composition with atomic resolution. This method has non-destructively yielded valuable new information about adsorbate bonding and conformation for several of the chemical systems listed below, permitting adsorbate modeling at the atomic level. The computation implements a profile-smoothing search, gives error limits on the profiles, and requires only minutes on a 33-MHz processor. The method is capable of computing at least six simultaneous elemental profiles, and performs well even under unfavorable conditions such as patchy overlayers, complex organic adsorbates (such as DNA), and trace elemental quantities (<10%). The depth profiles obtained for Cu on Ru(0001), and for sulfur-modified DNA, C6H3S3H, and cysteamine adsorbed onto Au(111) show close correspondence to scale molecular models and to expected elemental percentages for the surface structure. The algorithm also has verified depth profiles from other researchers on other chemical systems.

10:00 am AS-WeM6 A Comparison of XPS Quantitation using Electrostatic and Magnetic Lens-Based Analyses, D. J. Sarman and A. R. Walker, Kratos Analytical Inc., 353 E. Crescent Ave., Ramsey, NJ 07446, and T. A. Zupp, J. Lucas, L. Paleudis, B. J. Tielens, and J. E. Fulghum, Chemistry Department, Kent State University, Kent, OH 44242.

Magnetic immersion lenses have not, until recently, been incorporated into x-ray photoelectron spectrometers. These lenses do, however, overcome the limitations of electrostatic lens-based systems for small area analysis and imaging. The spherical aberrations that provide a practical limit of low magnifications and collection angles in electrostatic lenses are minimized by magnetic immersion lenses, which provide large magnifications and collection angles. This results in a dramatic improvement in sensitivity from small analysis areas. An important aspect of X-ray Photoelectron Spectroscopy, XPS, is the ability to obtain quantitative information. Previous work evaluating spectrometer transmission functions in order to improve quantification has focused on spectrometers incorporating only electrostatic lenses. This paper will compare XPS data acquired using both electrostatic and magnetic lenses on the same instrument. The results demonstrate that operation of the spectrometer with either the electrostatic or magnetic lens results in a similar transmission function, and that qualitative results are easily obtainable from small analysis areas using a magnetic immersion lens. The discussion will also illustrate quantitative imaging XPS.

10:20 am AS-WeM7 Quantification in Molecular SIMS, B. Hagenhoff, K. Meyer, M. Deimel, D. Rading, W. Sichermann, and A. Benninghooven, Physikalisches Institut der Universität, Wilhelm-Liepmann-Str. 16, D-48149 Münster, Germany.

In the last years TOP-SIMS has been proven to be a suitable tool for the qualitative characterization of molecular species with high surface sensitivity. However, quantification in molecular SIMS is difficult because of the dependence of secondary ion yields (transformation probabilities) on the chemical composition of the surface sample. This matrix effect is determined by the nature of the surface species considered and by its chemical environment (matrix, substrate). Nevertheless, quantification can be realized by three different procedures. The application of 1. additional quantitative surface analytical techniques (e.g., XPS, Laser-SNMS), 2. external standards, and 3. internal standards.

We applied these calibration techniques to a variety of organic molecules (e.g. cyclosporin and steroids), several functional groups on polymers (e.g. OH), self assembling layers (e.g. binary mixtures of different thiols on Au), etc.

Our investigation reveal that the application of internal standards— a well established technique in general mass spectrometry—is most valuable for molecular SIMS. Not only isotope labelled molecules but also chemically similar molecules could be applied successfully as internal standards.

Examples for molecular quantification, in particular for organic molecules, will be presented in detail.

10:40 am AS-WeM8 Simultaneous Dual-Element Analyses of Platinum-Group Elements in Natural Occurring Matrices using Resonance Ionization of Sputtered Atoms, W. F. Calaway, R. C. Wiens.
The combination of secondary neutral mass spectrometry (SNMS) and resonance ionization spectroscopy (RIS) has been shown to be a powerful tool for the detection of low levels of elemental impurities in solids. Drawbacks of the technique have been the laser-repetition-rate-limited low duty cycle of the analysis and the fact that RIS schemes are limited to determinations of a single element. These problems have been addressed as part of an on-going program to explore the usefulness of RIS/SNMS instruments for the analysis of naturally occurring samples. Efficient two-color/two-photon resonance ionization schemes were identified for five platinum-group elements (Mo, Ru, Os, Ir, and Re). Careful selection of the ionization schemes allowed Mo or Ru to be measured simultaneously with Re, Os, or Ir using two tunable dye lasers and a 1 kW excimer laser. Resonance frequency could be switched easily under computer control, so that all five elements were analyzed while removing less material than the depth resolution of ion sputtering methods (~1 nm). Quantitative measurements of these elements in metal grains of five meteorites at sub-ppm levels were determined. Detection limits were calculated from the sensitivity of each element and the background signal. From the analyses, estimates of the accuracy and precision our RIS/SNMS instrument were determined.


**PLASMA SCIENCE**

**Room A109 – Session PS-WeM**

**Plasma Diagnostics**

**Moderator:** S. C. McNevin, AT&T Bell Laboratories.

**8:00 am PS-WeM0** Laser-Induced Thermal Desorption as an in situ Surface Probe During Plasma Processing of Si, I. P. Herman, C. C. Cheng, K. V. Guinn, and V. M. Donnelly, AT&T Bell Laboratories, Murray Hill, NJ 07974.

Laser-induced thermal desorption (LITD) is used to detect adsorbates in real time during plasma processing. The steady-state surface layer formed during etching of Si(100) in a helical resonator is examined with Cl₂, HBr, Cl₂/O₂, and Cl₂/HBr gases. During Si etching by Cl₂-pulsed XeCl excimer laser irradiation of the sample produces transient heating and thermal desorption of SiCl₄. The desorbing products can be detected by either laser-induced fluorescence (LIF) excited by the same pulse or by plasma induced emission resulting from electron impact excitation. LIF analysis provides time-resolved measurements on a scale that is less than that required to etch one monolayer, and has been used to show that the surface layer formed during plasma etching of Si by Cl₂ remains essentially unchanged after processing, so post-processing XPS analysis probes a layer that is representative of the real-time processing conditions. Supportive XPS measurements are also presented. Using this LITD technique, the steady-state surface layer during chlorination is observed to contain twice as much chlorine and to be more weakly bound when the plasma is on vis-a-vis when the plasma is turned off. Near typical operating conditions, the Cl coverage depends weakly on discharge power, substrate bias voltage, and pressure, which suggests that ion flux, and not neutral etchant flux (i.e., Cl and Cl₂) apparently still limits the etch rate, even in a high-density, low pressure plasma. Competitive chlorination/bromination of the surface layer during etching by Cl₂/HBr mixtures is also probed, by comparing LIF by desorbed SiCl₄ and SiBr₂.

**8:40 am PS-WeM2** Measurement of the Electron Energy Distribution Function with a Differentially Pumped Spherical Sector Electrostatic Energy Analyzer, Ken D. Schatz, David N. Ruske, University of Illinois, Urbana, IL 61801.

The fundamental information about a plasma is contained within its electron energy distribution function (EEDF). Typically the EEDF is derived from Langmuir probe measurements by taking the first or second derivative of the probe electron current versus voltage data. Several difficulties are inherent with this scheme, including smoothing and fitting of data prior to differentiating, subtraction of ion current, and accounting for a drifting EEDF. A diagnostic which avoids these difficulties is the spherical sector energy analyzer. This type of energy analyzer is energy discriminating rather than energy integrating as is the Langmuir probe.

The diagnostic apparatus samples the plasma through a 100 micron diameter pinhole into a differentially pumped diagnostic chamber. The energy analyzer, located within the low pressure, ~10E-5 Torr, diagnostic chamber, records electron collection rates with an energy resolution of ~1 eV around the detection energy. Also the potential of the 6 mm diameter pinhole disk is adjustable, and disk voltage and current are recorded. This feature allows the pinhole to be biased at plasma potential and so remove the effects of the sheath as much as possible from in front of the pinhole.

Results are presented for the EEDF of an electron-beam sustained discharge in 100 to 400 mTorr of Helium. The discharge, powered by the combination of an electron beam and DC electric field, is measured to produce plasmas with electron temperatures between 0.2 eV and 5 eV and interesting, non-maxwellian, beam effects are observed. Also, deviation of the EEDF from a simple inverse exponential at energies corresponding to excitation and ionization thresholds is observed. A comparison is made with Langmuir probe data for the same discharge conditions.

**9:00 am PS-WeM3** Applications of Real Time Ellipsometry for Characterizing Etch Profiles in Sub-Micron Features, Nadine Bayou, A. Greenez, J. T. C. Lee, and D. E. Emberson, AT&T Bell Laboratories, Murray Hill, NJ 07974.

Microelectronics processing of ULSI devices, which incorporate thin films and sub-micron design rules, requires sensitive diagnostic techniques to achieve tightly controlled processes. Wafer film thickness and etch profiles are commonly characterized by post-process or destructive techniques such as cross-sectional scanning electron microscopy. In situ spectroscopic ellipsometry is a preferable diagnostic because of its sensitivity and compatibility with plasma processing environments, but until recently was thought to require a sacrificial section of unpatterned wafer, which could provide film thickness, but not etch profile information. We show, that with careful analysis, ellipsometry can be used to distinguish between undercut, tapered and vertical sidewall profiles, and thus provide real time etch pressure, axial ratio information, multilayer stack etching and film thicknesses without the use of the sacrificial pad. To assist in distinguishing etch profiles, in-situ UV-visible spectroscopy is collected in addition to the real time ellipsometry. We discuss the sensitivity of the technique to detect changes in linewidth and wall angle and present different models to understand how the ellipsimetric traces relate to the etch profiles and line features.
The time-averaged spatial concentration distribution of the argon (1s) metastable state in pure argon and argon/CF_4/O_2 rf plasmas, generated in a parallel-plate, asymmetric discharge, have been studied using planar laser-induced fluorescence imaging. This metastable state was examined with a gas reactor pressures ranging from 100-1000 mTorr and applied rf voltages from 75-300 V. The measurements were obtained using pulsed, laser-sheet excitation of the 1s-3p transition at 394.9 nm and right angle detection of the fluorescence (at 418 and 433 nm) with an intensified, cooled CCD camera. The fluorescence signals were corrected for the excited state quenching, based on the observed fluorescence lifetime at each condition. The imaged region covered one-half of the discharge, extending from the centerline to ~1 cm beyond the edge of the electrodes.

In general, our measurements indicate that the metastable density increases with pressure and, to a lesser extent, with applied voltage. At a given pressure, significant radial variations in the metastable concentration are observed, with the peak occurring near the outer edge of the plasma. While radial variation in the metastable density is observed at all conditions examined, it is most pronounced at the lower applied voltages where the variations are as high as 50%. Significant axial variations in the metastable density are observed as well. For example, as the pressure is increased from 100-1000 mTorr, the distribution changes from a parabolic type profile, with a peak at the discharge center, to one in which the metastables peak sharply near the glow/sheath interface of the powered electrode. By providing two-dimensional spatial distributions and the relative metastable concentrations of the argon (1s) metastable state over a range of discharge conditions, these results provide insight into the uniformity of these plasmas and valuable data for the validation of 2-D plasma processing models.

9:40 am PS-WeM5: Diagnostics and Control of Radicals in an Inductively-Coupled Etching Reactor, H. Sugai, K. Nakamura, Y. Hikosaka, N. Nakamura, A. Nagoya University, Nagoya, 464-01, Japan; b) Process Development Division, Fujiitsu Ltd., Kawasaki 211, Japan.

Advanced high-density etching reactors such as ECR, helicon and ICP (inductively coupled plasma) have common problems of irreproducibility and a low etch selectivity of SiO_2 to Si. Lack of information on radical behaviors have hindered from understanding and controlling the high-density plasma etching. In this paper, we report comprehensive measurements of ion and neutral radicals in a fluorocarbon ICP, together with an innovative radical control based on a reactor temperature and/or pulse modulation. Neutral radicals Cm (m = 1-4) were detected by appearance mass spectrometry and F atom by an actinometry technique. The measured densities and surface loss probabilities are presented in comparison with a low-density high-pressure CCP (capacitively coupled plasma). The most abundant species is CF_2 in the ICP and CF_3 in the CCP. The density of F atom is an order of magnitude higher in the ICP than in the CCP while the densities of CF_m radicals are lower in the ICP than in the CCP. The high etching rates and low selectivity of SiO_2 to Si observed in the CCP are tentatively attributed to numbers of F atoms with less CF_m radicals. In order to control the radical composition of ICP, the reactor vessel was heated up to 100-200°C. The measurement revealed a drastic increase in the CF_m densities with the F atom density almost unchanged. As a result, a high etch selectivity of SiO_2 to Si was obtained in the hot CCP reactor. Another possibility of radical control by a pulse modulation will be presented.


The energy distribution of ions exiting a plasma through an rf modulated sheath is important in many surface processing applications, and also as a plasma diagnostic. It is well known, that the rf excitation frequency lies between the ion and electron plasma frequencies, the collisionless energy distribution is made up of two peaks (the so-called ‘bimodal’ distribution) at energies related to the amplitude of the rf modulation and the ion transit time across the plasma. We have measured almost collisionless, mass-resolved ion energy distributions in a 13.56 MHz rf plasma at very low pressures (5 to 20 mTorr) and compared the results to numerical simulations of ion loss in both model and self-consistently obtained electric fields. Contrary to expectation, the distributions of both minority species (such as He in a mixture of Ar + 8% He) clearly show the presence of a third peak. The simulations are used to study the genesis of this third peak and its dependence on the plasma and sheath parameters. The results support the conclusion that the third peak is a direct indication of the Bohm velocity of the ions entering the sheath. The measurement of its position can thus be a valuable diagnostic of the plasma sheath and presheath. Results from both electric and electrostatic plasmas (obtained by mixing a few percent CF_4 with Ar) are discussed and a previously unexplained feature of collisionless ion energy spectra is clarified.


Optical emission spectroscopy (OES) has been shown to be a valuable tool for monitoring the etching process of III-V materials. This non-invasive in-situ diagnostic can be related to the etch rate, surface morphology and etch front. A Cl_2/Ar plasma generated by an electron cyclotron resonance (ECR) source is used for the etching of InP and GaAs since it can provide low surface damage. Additionally, there is no polymer deposition when Cl_2/Ar is used compared to Cl_2/H_2 and the process is more controllable and reproducible. A 110 μm deep, 30 μm wide via holes in InP have been etched with Cl_2/Ar discharges. The etch rate is ~3 μm/min and a vertical profile with smooth morphology is obtained. Etching should be stopped at a 40 nm thick Ti layer to prevent pitting of the underlying Au which can cause device failure and contamination. Therefore, end point detection using OES is essential. It is found that the optical emission signals for In at ~410.2 and 451.1 nm can be used to monitor the end point. For GaAs etching, the optical emission signal for Ga at ~417.1 nm shows close correlation with etch rate for varying microwave power, rf power, Cl percentage in Ar, and pressure. For example, the Ga signal increases 9× for 5% Cl_2 addition to Ar which results in 8× increase in etch rate. The response time for the increase in Ga signal is found to be less than 5 sec. Monitoring the Ga signal as a function of time, the etching of surface oxide can be detected and the removal rate is found to increase with ion energy. The sensitivity of OES will be investigated for controlling via hole etching in InP and emitter etching for heterojunction bipolar transistors and the results will be compared to mass spectrometry.

SURFACE SCIENCE/ELECTRONIC MATERIALS
Room A102 – Session SSEM-WeM
Semiconductor Surface Reactions I
Moderator: K. Raghavachari, AT&T Bell Laboratories.
8:00 am SSEM-WeM0: Structure Sensitivity of Trimethylgallium Chemisorption on GaAs Surfaces, J. Randall Creighton, Org., 1126, MS 0601, Sandia National Laboratories, Albuquerque, NM 87185-0601.

Compound semiconductors deposition by metal organic chemical vapor deposition (MOCVD) and atomic layer epitaxy (ALE) typically exhibits a marked structural sensitivity, i.e., a large variation in growth rate for different surface crystallographic orientations. We have investigated the chemisorption and reactivity of trimethylgallium (TMGa) on non-polar (i.e., [110]) and polar (i.e., [100] and [111]) surfaces of GaAs and found significant differences in behavior. Surface infrared spectroscopy and static-SIMS were used to examine the surface intermediates formed during TMGa dissociation, while TPD was used to monitor the overall reaction pathway. Results indicate that the dominant factor in determining reactivity is the presence or absence of atomic vacancies at the surface. The polar [111] and [100] surfaces generally reconstruct into structures with intrinsic vacancies or ad-atoms, whereas the [110] surface is stoichiometric. During TPD a significant fraction (10-30%) of a monolayer of TMGa adsorbs irreversibly on the gallium-rich ([100]) and [111]A surfaces, evolving methyl radicals at 705 and 730 K, respectively. In contrast, there is a complete lack of methyl radical desorption from the [110] surface. Although TMGa chemisorbs strongly on the [110] surface, the process is reversible and virtually all of the TMGa desorbs at 400 K during
TPD. Apparently the gallium atom vacancies intrinsic to the (100) and (111A) reconstructions offer a convenient site for the gallium atom to occupy during TMGa dissociation, while the stoichiometric (110) surface has no atom vacancies and cannot accommodate the extra gallium atoms into a stable surface structure.

8:20 am SSEM-WeM1 Molecular Beam Scattering as a Probe of Thin Film Deposition Processes, J. R. Engstrom, School of Chemical Engineering, Cornell University, Ithaca, NY 14853.

Supersonic molecular beam scattering techniques have been employed to examine the prerequisite reaction to the deposition and growth of thin films: dissociative chemisorption. Concerning the reactions of SiH₄ and SiH₂ on clean Si surfaces we have found that incident translational energy (E₀) and incident angle (θ₀) are the dominant variables controlling the probability of dissociative chemisorption. E₀, SiH₂ and SiH₄ have been examined on Si(100), Si(111) and Si(110) surfaces. We find that for incident translational energies examined adsorbed P acts to inhibit chemisorption of both silanes in a sub-linear fashion, i.e., Sₐ = S₀(1 - θ₀)ⁿ, where n < 1, and θ₀ is the fractional coverage of P(a). Adsorbed P(a) is also observed to influence the dissociation pathway for SiH₂ on Si(100), where at finite values of θ₀, a SiH₂(a) production channel is emerging.

INVITED

9:00 am SSEM-WeM3 GaF₃, Thin Films Grown on GaAs via Exposure to XeF₂, W. C. Simpson, P. R. Veenkamp and J. A. Yarmoff, Department of Physics, University of California, Riverside, CA 92521, and Materials Sciences Division, Lawrence Berkeley Laboratory, Berkeley, CA 94720.

A study of the interaction of atomic F (via XeF₂) with GaAs(100) and (110) has been carried out, employing soft x-ray photoelectron spectroscopy (XPS), temperature-programmed desorption (TPD), and electron energy loss spectroscopy (EELS). It is found that the fluorination of GaAs results in the formation of a thin film of GaF₃, which acts as a 10 eV band gap insulator. This is a simple chemical process for growing an insulator on GaAs, similar to how SiO₂ is grown on Si via exposure to O₂ and thus has potential technological importance, motivating further study of GaF₃ films grown in this manner.

From the results of this work, a model of the film growth mechanism is proposed and the nature of the film-substrate interface is discussed. In addition, the electronic structure of GaF₃ films is investigated. It is found that, once the films reach a thickness of ~10 Å, they acquire the electronic characteristics of bulk GaF₃. From XPS and EELS measurements, the band-gap of the films and the band-line-up of the films with the substrate are determined. From the PSD data, absorption spectra of the GaF₃ films are obtained, which provide information on the unoccupied density of states.

9:20 am SSEM-WeM4 Initial Stages of Phosphorous Desorption from InP(110) Surfaces, Ph. Ebert*, M. Heinrich*, M. Simon*, K. Urban*, M. G. Lagally*, *University of Wisconsin, Madison, WI 53706; *Institut für Festkörperforschung, Forschungszentrum Jülich GmbH, D-52425 Jülich, Germany.

The initial stages of phosphorus desorption from InP(110) surfaces were investigated quantitatively on the atomic scale as a function of the duration of thermal treatment, in a temperature range between room temperature and 200°C. The measurements were performed using a scanning tunneling microscope to investigate in particular the phosphorus monovacancies created during the thermal treatments. The formation rate of the P vacancies increases up to 160°C. Above 160°C the formation rate decreases. This non-Arrhenius behavior is explained by the interaction of two thermal processes, a vacancy-adatom pair formation, creating vacancies, and a P segregation from the bulk toward the surface, annihilating vacancies. The evaporation of P itself occurs through diffusion of P adatoms along the surface and formation of P₂ molecules, which immediately desorb. A saturation of the vacancy density is observed and explained by a charge-induced repulsion between vacancies.

*P. E. thanks the Humboldt Foundation for a Lynen Research Fellowship. Permanent address: Institut für Festkörperforschung, Forschungszentrum Jülich GmbH, D-52425 Jülich, Germany.

9:40 am SSEM-WeM5 Interaction of 50 eV Electrons with D₂O on GaAs(100): Electron and Thermal Induced Oxidation, Derrell W. Sloan, Yang-Ming Sun, and J. M. White, Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712.

The interaction of 50 eV electrons with D₂O on GaAs(100) has been investigated using temperature programmed desorption (TPD), x-ray photoelectron spectroscopy (XPS), and high resolution electron energy loss spectroscopy (HREELS). The interest in this study was to determine if an oxide layer could be produced on a semiconductor surface by a nonthermally activated pathway. D₂O adsorbs molecularly on GaAs(100) at ~100 K as indicated by XP spectra that show an O(1s) binding energy of 534 eV. Substantial hydrogen bonding at submonolayer coverages is seen in both TPD and HREELS results. Electron irradiation induces both desorption and dissociation of D₂O forming OD, O, and D. XPS spectra taken after electron irradiation show that the O(1s) binding energy shifts to 532 eV and broadens. HREELS spectra show that hydrogen bonding as well as D₂O binding decreases after electron irradiation. These results are consistent with molecular dissociation and oxidation leaving OD, O, and D. Flashing to 600 K following electron irradiation removes OD and O leaving an oxide surface as indicated by an O(1s) binding energy of 530 eV and the loss of all D₂O and OD features in the HREELS spectrum. The results indicate that electron irradiation can produce an oxide but not a clean oxide layer. D and OD that remain on the surface after electron irradiation must be removed thermally.

10:00 am SSEM-WeM6 Characterization of Silicon Surfaces and Interfaces by Vibrational Spectroscopy, Y. J. Chabal, M. A. Hines and D. Fei, AT&T Bell Laboratories, Murray Hill, NJ 07974.

The understanding of chemical reactions at silicon surfaces and interfaces required spectroscopic characterization of the chemical species (reactants, product species, and intermediate species). We consider here the various techniques available (Infrared Absorption, Raman and Sum-frequency spectroscopies as well as Electron Energy Loss Spectroscopy) for the study of etching and bonding of silicon wafers.

Etching plays a key role in wet chemical processing, including chemomechanical polishing. The use of aqueous fluoride etch leads to a termination of silicon surfaces often involving hydrogen with contamination species such as OH, CH₃, F. The combination of electron and photon spectroscopies have made it possible to characterize both flat and stepped surfaces. The data have been used to suggest specific chemical reactions responsible for the preferential etching observed in buffered HF solutions.

Silicon bonding occurs upon mechanical contact of oxidized (hydrophobic) wafers. It is an important technique to produce Silicon-on-insulator substrates, to substitute for epitaxial growth and to manufacture micro-mechanical systems. We have used multiple internal reflection (MIR) infrared absorption spectroscopy to characterize the concentrations of water, hydrogen, hydroxyl and hydrocarbon species at the interface of both hydrophobic and hydrophilic Si(100) wafers as a function of bonding temperatures. This sandwich geometry provides a large (≈ 10⁴) enhancement (of the component of vibrations perpendicular to the interface plane) over the standard MIR geometry, traditionally used to study semiconductor external surfaces. The magnitude and polarization dependence of this enhancement factor make it possible to perform accurate measurements in air without interference from impurities adsorbed on the outer surfaces, and to detect as few as 1% monolayers of various species in these chemically complex interfaces. The data are used to critically evaluate the various proposed bonding mechanisms.

INVITED
Polarized infrared spectroscopy reveals these arsine species bond to the second-layer gallium atoms exposed at As-dimer vacancies. Infrared peaks for arsine adsorption on (1 × 6) reconstruction appear at 2135 and 2100 cm⁻¹. Increasing the arsine adsorption temperature causes additional peaks to appear at 2045 and 2020 cm⁻¹. This indicates that the arsine adsorbs on gallium dimer sites and decomposes to form arsenic dimers. These results are used to present a mechanism for arsine adsorption and decomposition on GaAs(001) surface.

THIN FILM
Room A105 - Session TF-WeM

Optical, Piezoelectric and Ferroelectric Films
Moderator: D. K. Chatterjee, Eastman Kodak Company,

8:00 am TF-WeM0 Thin Film Optical Coatings, H. A. Macleod, Thin Film Center Inc., 2745 E Via Rotonda, Tucson, AZ 85716-5227.

Optical materials are quite limited in their range of intrinsic optical properties. Thin-film optical coatings open up an almost limitless range of possible performance by combining optical materials into regular layered structures. The resulting behavior may be far from that of the component materials and this includes properties such as mechanical, thermal, electronic and acoustic. The distinguishing feature of an optical coating is that its primary purpose is optical but all the other properties may be, and often are, involved. The optical properties are normally achieved by controlled interference and this implies a high degree of uniformity and precision of layer thickness. The structures may range from a few to several thousand layers. Their optical purpose may be to reflect strongly, to select or reject spectral regions, to control polarization, to suppress reflection, to combine or split beams. The list is almost limitless. Optical considerations have traditionally been dominant and optimum, with mechanical, thermal and other considerations of secondary importance. As requirements change this situation is also changing and much effort is now directed at improved understanding of these other factors and especially at the influence of the various processes that may be used for optical film deposition.

INVITED

8:40 am TF-WeM2 Composition and Morphology of Al Multilayer Thin Film Reflective Coatings, J. Kim*, J. J. Wietman**, D. Torr*, and M. Zukic.** *Department of Physics, **Department of Chemistry and Department of Chemical & Materials Engineering, University of Alabama in Huntsville, Huntsville, AL 35899.

Successful design of Al multilayer optical devices depends critically on accurate knowledge of optical constants for the device. Obtaining detailed information about thickness, compositions, and morphologies of layers in such devices is therefore vital. Multilayer Al thin films with interleaving and protective MgF₂ dielectric layers for use as far and extreme UV reflectors have been characterized using x-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) with this goal in mind. The devices were prepared in a conventional high vacuum system, and sputter depth profiles were obtained using XPS in a separate UHV system. Thin, oxidized regions were found at the top- and bottom-most interfaces of the Al layers. These oxide regions may be thicker than previously reported from optical measurements. Interdiffusion of elements was apparent at boundaries between the Al and MgF₂ layers. The AFM images showed column-like growth of the Al and MgF₂ layers, and roughnesses were determined on surfaces at the microscopic scale. Since oxidation of Al is probably the main cause of discrepancies between design and actual performance, a relationship was derived to predict the extent of oxidation in Al layers from parameters measured in situ during film deposition. A model for determining optical parameters of multilayer devices is presented that incorporates Al oxide layers as a first step toward improving agreement with design values.

9:00 am TF-WeM3 Production and Characterisation of Multilayer KCl:LIF Thin Films on Glass, F. Somma, A. Ercoli, Dipartimento di Fisica, III Università di Roma, 00146 Roma, Italy, S. Santucci, L. Lozzi, M. Passacantando, P. Piccotti, Dipartimento di Fisica, Università dell’Aquila, 67010 Coppito (AQ), Italy.

Alkali halide films with colour centres have important applications in information storage, lithography, radiation dosimetry and solid state lasers. KCl:LIF multilayers have been grown by thermal evaporation on glass substrates at p < 10⁻⁶ mbar under continuous monitoring of the substrate temperature (T = 300°C) and deposition rate (V = 0.4–0.6 nm/s). The centre colours have been produced by low energy (3 keV) electrons. The complete characterisation of the samples has been performed using depth profile XPS, for the profile composition, X-ray diffraction and SEM, for the structural analysis and optical absorption, for the optical properties. When the substrate temperature is low (30°C) the LiF layer deposited onto KCl (h00) film grows as (111) and a low diffusion between KCI and LiF layers are observed. Instead when the substrate temperature is high (250–300°C) a (100) growth of LiF onto KCl (h00) and a strong diffraction are shown.


The thermal conductivity of dielectric optical coatings is thought to be a major factor in determining the laser damage threshold. Micron thick films of TiO₂, SiO₂, Al₂O₃, and stabilized zirconia are deposited on single crystal Si and MgO substrates using radio-frequency and ion-beam sputtering. We measure the thermal conductivity of the films in the temperature range 80 to 400 K using a new extension of the 3o method. The accuracy of this technique is demonstrated by the fact that the thermal conductivity of a-SiO₂ grown thermally on Si(001) is identical to bulk a-SiO₂. The temperature dependence of the data for sputtered oxide films is comparable to bulk a-SiO₂ and the magnitude of the conductivity at high temperatures is in good agreement with the predicted minimum thermal conductivity for these materials.

9:40 am TF-WeM5 Review of Recent Developments in Ferroelectric, Piezoelectric and Electrooptic Thin Films, A. I. Kingon, H. N. Al-Shareaf, A. Chow, D. J. Lichtenwalner, and O. Auciello, Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695-7919.

We review recent developments in ferroelectric, piezoelectric and electrooptic films, with special emphasis on our research at NCSU. Ferroelectric films are of current interest for non-volatile memories (NVMs) and DRAMs. Lead zirconate titinate (PZT) solid solutions are a leading choice for NVMs. Successful utilization requires careful control of stoichiometry and microstructure, via the chosen process method. We show the importance of the early stages of film growth on the resultant microstructure. The control of key properties such as polarization fatigue and film leakage is discussed.

Electrooptic films have potential application for optical switches and frequency doubling (for blue light sources). In this case, high quality epitaxial films are required. Improvement of optical losses is the major technical obstacle to exploitation. The situation is discussed with reference to niobate films (KNbO₃ and LiNbO₃).

The final portion of the presentation deals with the status of piezoelectric films. Applications include electromechanical (MEMS) and surface acoustic wave (SAW) devices. The review covers simple oxides such as ZnO as well as perovskite solid solutions.

INVITED

10:20 am TF-WeM7 Smart Thin Film TiN/Piezoelectric Heterostructures, A. Peter Jardin and Peter G. Mercado, Dept. of Materials Science and Engineering, SUNY at Stony Brook, Stony Brook, NY 11794-2275.

The development of smart materials relies on developing heterostructures with both sensing and actuation capabilities which are coupled by an intrinsic control mechanism. It has been previously demonstrated that heterostructures of Shape Memory Effect TiN and piezoelectric (53/47) Pb(Zr,Ti)O₃ (PZT) coupled by a TiO₂ interlayer are capable of being coupled as "smart". As TiN wafers with a titania surface, the use of piezoelectric titanates was a natural system for exploiting these heterostructures. In this paper, we outline the development processing of TiN using UHV deposition techniques and the development of the ceramic layers using sol-gel techniques.

The current post-deposition state of the heterostructures was initially amorphous. Of particular concern to smart materials development is that the post-deposition processing to crystallize the correct phase of one component of the smart heterostructure can adversely affect the microstructure and active properties of the second phase of the material. We have found that there is a narrow range of processing temperatures between 550°C and 600°C where the PZT thin film is in its perovskite phase, the TiN is in its superelastic austenite state and the TiO₂ interface between the TiN and PZT is rutile. Detailed characterization of the physical properties was performed using SEM, XRD and optical microscopy and the piezoelectric properties of these het-
erostructures was performed using an RT-11 Ferroelectric Test station. These results will be presented and implications for possible devices discussed.


Surface and/or microstructures of thin films of the perovskites, which will affect their physical and chemical properties, have been not fully understood yet, although the extensive works have been done on a deposition of the perovskites including sputtering, reactive evaporation, laser ablation, and chemical vapor deposition. This study describes the surface and the microstructures of perovskites lead titanate (PT) thin films of PbTiO$_3$ families grown on (100) MgO and (001) sapphire. Their surface and microstructures have been studied by a scanning electron microscopy (SEM) and a transmission electron microscopy (TEM). RF magnetron sputtering was used for the deposition of the perovskites thin films. It was found that the (001) lead titanate thin films were epitaxially grown on the (100) MgO single crystal at stoichiometric composition. The composition of the resultant thin films and the thin film thickness have strongly influenced their surface and microstructures.

This research was supported in part by the NEDO Environmental Catalyst Research Programme.

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**ELECTRONIC MATERIALS**

Room A108 - Session EM-WeM

**Heterostructures for Optoelectronics**

Moderator: C. W. Wilsen, Colorado State University.

8:00 am EM-WeM9 MBE Growth of II-VI Blue/Green Laser Diodes, D. C. Grillo, M. D. Ringlo, J. Han, R. L. Guenther, G. C. Hua, Purdue University, West Lafayette, IN 47907, A. Salokatve, H. Jeon, A. V. Nurmiko, Brown University, Providence, RI 02912.

The high-injection lasing conditions mandated for semiconductor laser diodes present stringent challenges to both material and device scientists. Issues such as defects, dopings, contacts, and processing have to be properly addressed in order to achieve prolonged operation at room temperature under continuous wave (CW) excitation. In this paper we describe the MBE growth and characterization of widegap II-VI (Zn,Mg)$_2$Se/ Zn$_2$(S,Se)$_2$/Zn$_2$(Cd,Se)$_2$ separate-confinement heterostructure (SCH) quantum well lasers. By employing an elevated temperature during growth of the active regions (confinement region and quantum wells), we were able to reduce, as evident from photoluminescence, device degradation, and TEM characterizations, the density of non-radiative point defects. Devices operated at room temperature have CW lifetime exceeding 30 seconds with threshold current densities at low as 250 A/cm$^2$ and lasing voltage below 6 volts. To eliminate the residual strain built-in to the (Zn,Mg)$_2$(S,Se) cladding layers due to compositional drifting, as manifested by the presence of multiple diffraction peaks in the x-ray rocking curves, the substrate temperature was carefully monitored by an infrared pyrometer taking into account the thin-film interference effect arising from the II-VI/III-V heteroepitaxy; rocking curves with single diffraction peak of 24 arcsec were obtained from a 2 µm quaternary epiayer.

This work was supported by ARPA/ONR University Research Initiative grant number 286-25043, AFOSR grant number F49620-92-J-0440, NSF/MRGR grant number 9221390-DMR, and NSF grant number 9202957-DMR.

8:20 am EM-WeM1 MBE Growth of ZnMgSSe and Its Application to Blue and Green Laser Diodes, M. Ikeda and A. Ishibashi, Sony Corporation Research Center, 174 Fujitsu-cho, Hodogaya-ku, Yokohama 240, Japan.

The ZnMgSSe alloy, which was originally developed by Sony researchers in 1989, is an attractive material for cladding layers of wide band gap II-VI laser diodes because it offers a wide range of band gap energy from 2.7 to ~ 4 eV at room temperature maintaining the

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**Inorganic Materials**

9:00 am EM-WeM3 Structural and Compositional Characterization of HgSe Contacts to p-ZnSe, J. Fijol, P. H. Holloway, J. Treuler, J. King, and K. Jones, Department of Materials Science and Engineering, University of Florida, P.O. Box 116400, Gainesville, FL 32611.

HgSe contacts to MBE grown p-ZnSe (nitrogen doped to 3 x 10$^{17}$ cm$^{-3}$) have been formed by the reaction of an amorphous Se capping layer with Hg vapor, yielding pseudo-ohmic behavior. This process was performed using atmospheric processing which involves heating the Se capped samples to 150°C in Hg vapor. Current densities of 2.5 A/cm$^2$ at 10 volts were observed for these contacts and temperature dependent I-V measurements have been used to identify thermionic field emission as the mechanism of current transport across the HgSe/ ZnSe interface. The properties of the amorphous Se capping layer, such as glass transition temperature ($T_g$) and peak re-crystallization temperature ($T_r$) have been identified as critical factors in the formation of HgSe. To improve contact performance, the reaction of Hg with Se and the structural and compositional properties of the resulting HgSe contacts have been studied. Auger electron spectroscopy (AES) and x-ray diffraction (XRD) have been used to determine contact composition and structure at stages throughout the reaction. TEM analysis indicates that under specific processing conditions, regions of HgSe are formed which are epitaxial to the p-ZnSe. The implications of these reactions to the contact properties will be discussed.

9:20 am EM-WeM4 Deep Level Interface States Near ZnSe/GaAs(100) Heterointerfaces, A. D. Rabinen and L. J. Brillson, Xerox Webster Research Center, Webster, NY 14580, L. Vassalli, L. Saue, and A. Franciscato, Dept. of Chemical Engineering and Materials Science, Univ. of Minn., Minneapolis, MN 55455.

We have used low energy cathodoluminescence spectroscopy (CLS) and laser photoluminescence spectroscopy (PL) to characterize the energies, and depth distribution of deep levels near the buried, MBE-grown ZnSe/GaAs(100) heterointerface. Previous studies have described a variation in heterointerface band offset with ZnSe/Se beam pressure ratio (BPR) during epilayer growth. Using a combination of electron excitation energies and laser wavelengths to obtain CLS/PL "depth profiles" through the interface regions of 50, 1500, and 5000 Å ZnSe/GaAs structures, we find that deep electronic states are formed near the heterojunction and within the epilayer whose relative emission intensities vary dramatically with BPR. For Zn-Se-rich interfaces, features at 0.9 and 1.0 eV (1.3 and 1.9 eV) photon energies are dominant. In general, intensities of the 0.9 and 1.0 eV features decrease away from the free epilayer surface, whereas the 1.3 and 1.9 eV features appear most intense for excitation depths corresponding to the buried interface regions. The CLS/PL spectra reveal characteristic features of intermixing at elevated temperatures consistent with known atomic diffusion behavior. These spectral features demonstrate substantial differences in the localized interface states formed under variant epi-
layer growth conditions. In addition, they provide effective in-situ monitors of heterointerface quality and stability.


Since the first report of II-VI blue-green laser diodes in 1991 rapid progress has been made in advancing both the II-VI materials and devices. Although continuous operation of laser diodes at room temperature has been demonstrated, the lifetimes of the best devices are limited to a few minutes. TEM, cathodoluminescence and electroluminescence studies have shown that laser degradation proceeds by the evolution of dark defects that are formed in the vicinity of pre-existing defects such as V-shaped stacking faults. The stacking faults responsible for the degradation appear to be formed during the nucleation of the II-VI layers on the GaAs substrate. Possible causes of the stacking faults and efforts to eliminate them will be discussed.

INVITED


The control of the properties of heteroepitaxial films of compound semiconductors on silicon is impeded by surface structure/chemistry and strain effects that result in the generation of defects. In this paper, the low temperature growth of nearly lattice-matched heterostructures of compound semiconductors on silicon is reviewed, separating the study of surface structure and chemistry effects from the problem of strain-induced defect formation. Using GaP on silicon as an example, the growth of heteroepitaxial structures under the conditions of interrupted cycle chemical beam epitaxy is discussed. Excellent selectivity of GaP heteroepitaxy on Si as compared to SiO2 and SiC covered surface areas is observed. Although selectivity is a desirable aspect in the context of device processing, the impedance of epitaxial overgrowth by residual oxygen and carbon on the silicon surface points to the fundamental importance of surface cleaning and the maintenance of a clean silicon surface during the initial stages of nucleation and GaP growth. The kinetics of heteroepitaxial GaP growth is evaluated on the basis of RBS, AFM, HREM, RHEED and mass spectrometric studies that focus on the evolution of chemical and structural changes during the heating of the initially hydrogen terminated silicon surface in the presence of a phosphorus precursor beam and the subsequent nucleation and sealing stage that establishes a contiguous GaP epitaxial layer on the silicon surface. Also, the heteroepitaxial growth of related nearly lattice-matched II-IV-V2 compounds and alloys is considered, using the ZnSiSe2-P2 system as an example.

INVITED

MANUFACTURING SCIENCE AND TECHNOLOGY
Room A110 – Session MS-WeM

Advanced Manufacturing Equipment-B
Moderator: C. B. Whitman, CVC Products, Inc.

8:00 am MS-WeM0 Long-throw Low-pressure Sputtering Technology for ULSI Devices, Yashuyuki Kadokura, Yuzou Kashimoto, Tetsujii Kiyota, Nobuhiro Motegi, I. Nakayama, ULVAC Japan Ltd., Shizuoka, Japan.

The process of metallizing contact/via holes plays a very important role in semiconductor device fabrication. As LS becomes increasingly high integrated, the size of the contact/via hole is becoming smaller, producing higher aspect ratios. As a result, sufficient bottom coverage cannot be obtained by the conventional sputtering techniques. To solve this problem, a collimated sputtering technique has been investigated recently. Though it has been shown that the bottom coverage is highly improved by using a collimator, the technique has still disadvantage for production, such as generation of particles, degrading from the collimator, and clogging of the collimator itself. We have developed a new technology, called long-throw low-pressure sputtering technology, for sputtering high-aspect-ratio ULSI devices without employing collimators in the system. The basic feature of our design is to make the distance between target and substrate much longer than that of the conventional sputtering method so that sputtered atoms are incident perpendicular to the wafer, while lowering the discharge pressure by an order of magnitude in deposition (10-2 Pa range) so that sputtered atoms are not scattered by gas molecules between the target and the substrate. Our results indicate that this technique has very good potential for applications in high-aspect-ratio ULSI sputtering processes. In this paper, we will describe the basic principle of our design, the unique properties of metal thin films, such as TiN/Ti and Al-alloy formed by our method, and the variation of the method as an advanced manufacturing technology for next generation ULSI devices.


Faster circuit performance requires tight control of gate linewidth and profile to avoid short-channel reliability failures or speed degradation; to provide this control, the gate etch process must be insensitive to variations in loading. Problematic loading densities include very local (<1μm), intermediate (1-20nm) and global (100nm) interactions. Our efforts to quantify and reduce these effects are discussed in this paper.

For this study, we used an integrated three-step gate-etch process in a single wafer RIE tool which included a F-based breakthrough etch, followed by a two-step HBr/Cl2/O2 polysilicon etch. Cross-sectional and plan-view SEM and electrical linewidth measurements were used to characterize the etch results.

The optimized process showed similar profiles and negligible offset between nested and isolated lines. For very local effects, the offset between isolated and dense features was increased by the passivation produced in the breakthrough step, the degree of sputtering in the first polysilicon etch step, and the oxygen flow ratio in the final gate-oxide selective step. The local loading effects were further perturbed by the intermediate- and global-scale loadings. The length scale of the intermediate loading effects for the gate etch process was determined by using a variable loading structure positioned next to an array of measurement structures. For the most severe loading changes, significant variations in the isolated features extended over 20nm, while nested lines were almost unaffected. Higher global resist loading was observed to decrease etch bias and increase the isolated-dense offset.

8:40 am MS-WeM2 Interlayer Dielectrics with Low Dielectric Constant for Multilevel Interconnection, R. Aoki, N. Hayasaka*, Y. Nishiyama, H. Miyajima*, Y. Nakasaka* and H. Okano*, Integrated Circuit Advanced Process Eng. Department, Toshiba Corp., ULSI Research Center, Toshiba Corp.

The high quality insulator with low dielectric constant for interlayer dielectrics is the key technology for future high speed ULSI devices. We realized F doped SiO2 with low dielectric constant (less than 3.5) and high water absorption resistance. F was incorporated into SiO2 by adding fluoride contained gases into TEOS/O2 dual frequency excited plasma.

We studied the CVD mechanism by investigating the plasma chemistry and physics, and we have clarified the role of F on film formation properties (film quality and step coverage). And also the mechanism of reducing dielectric constant due to F addition into SiO2, which was clarified by using molecular orbital calculation, will be presented.

9:00 am MS-WeM3 Manufacturing Issues of Electrostatic Chucks, D. R. Wright, L. Chen, P. Federlin, K. Forbes, SEMATECH, Austin, TX 78741.

In the past few years, Electrostatic Chucks (ESCs) have become much more widespread in semiconductor manufacturing equipment. In addition to the elimination of moving parts, ESCs promise the promise of decreasing the wafer edge exclusion, that is, to allow more good chips to be made on each wafer. A number of technical, material, and business challenges remain in making ESCS workable and reliable in all tools across the semiconductor factor, or fab.

We will discuss issues of clamping force, clamping and declamping time, and wafer temperature control, describing how they affect design and choice of materials. The effect of these choices on adapting ESCS
to various tools over various temperature ranges will also be discussed. The role of models and test results in accelerating development will be addressed.

Finally, we will list some of the business challenges to implementing ESCs. Despite technical successes, many high-volume fab lines are always reluctant to risk installing new technology, despite promises of improvement. SEMATECH has addressed these issues with Work- ing Groups, which help address standard specifications, study early testing results, and share manufacturing performance data.

INVITED

9:40 am MS-We55 Equipment Engineering Methods for Improvements in Particle and Uniformity Performance during Plasma Processing, G. S. Selwyn, M. Dalvie, C. R. Guarneri and M. Surendra, IBM Research Division, Yorktown Heights, NY 10598.

Particle contamination and process uniformity are key yield detractors in plasma processing. Often, these issues are viewed as separate problems with separate corrective actions. However, recent results demonstrate that uniformity and contamination problems can be interlinked by the formation of locally-disturbed plasma regions called “traps”. Traps are induced by changes in the electrode including the electrode topography and the emission of secondary electrons. Traps are characterized by a localized increase in the plasma potential. Wafers, clamp rings, focus rings and wafer patterning induce trap formation. In addition to these causes, we demonstrate for the first time that changes in materials under the electrode surface also induce traps, by altering the coupling of rf power into the sheath. Traps influence contamination problems by helping to confine particles. Similarly, traps can influence process uniformity by altering the flux and trajectory of ions to the wafer. The latter effect has strong influence on the wafer edge exclusion, the highly nonuniform region close to edge of the wafer. Typically, product yield is lowest in this region.

We correct this problem through the use of specially-designed buried layers of metal and dielectric to compensate edge effects and topology changes. The results are verified by optical emission studies, uniformity mapping and improvement in particle contamination performance.

INVITED

10:20 am MS-We57 Plasma Process Uniformity in a High Density System: Experiment and Modeling, C. R. Guarneri, M. Surendra, G. S. Selwyn, and M. Dalvie, IBM T. J. Watson Research Center, Box 218, Yorktown Heights, NY 10598.

In plasma processing of large area substrates, nonuniformities in etching or deposition result in lower yields or a wide distribution of device characteristics. Process uniformity is affected by variations in ion flux, energy, and direction.

Experiments have been carried out in a high density, rf inductively coupled system with a rf capacitively coupled substrate. Results indicate that by spatially varying the rf coupling to the substrate alone, we are able to vary the etch rate uniformly of SiO2 on blanket wafers from a profile that is higher in the center by ~20% to one which is lower in the center by ~10%. The rf coupling impedance is varied by both controlling the shape of the metal electrode and the use of di-electrics. The effect of spatially varying substrate rf coupling is dependent on substrate resistivity and applied frequency. Situations where substrate rf coupling is unintentionally nonuniform, e.g., wafer bowing due to backside cooling gas pressure, are also examined.

Process rate uniformity has been modeled with two dimensional analytic models of the plasma source and rf sheaths, coupled to an equivalent circuit element discretization of the electrode assembly, substrate, plasma source and sheath. Results from the model are in reasonable agreement with experimental measurements, and serve as a useful design tool for electrode assembly design.

10:40 am MS-We58 The Complex Impedance of a Dusty Processing Plasma, Weston C. Roth and Robert N. Carillo, Dept. of Electrical and Computer Engineering, University of Arizona, Tucson AZ 85721.

It is now understood that all plasmas used for processing silicon wafers contain large numbers of particles (dusty plasma) which have the potential to contaminate the wafer being processed. It is the purpose of this paper to examine the complex plasma impedance. Specifically, we measured the complex impedance (magnitude and angle) at the fundamental frequency of 13.56 MHz and also at the first, second, and third harmonics as a function of time at the RF input to a modified Tegal MCR-1 etch chamber. The plasmas were derived from argon and also SF6. For SF6, using a Si wafer on an aluminum electrode, we observed rapid variations of all angles at early times, probably indicating initial particle nucleation and growth within the plasma volume and transport to particle traps at the plasma-sheath interface. Subsequent to this period, the angles changed slowly. For argon, with a silicon wafer on a graphite electrode, there was no initial rapid variation, but only a slow change with time, probably suggesting that particle formation is a surface phenomenon. It may be possible to correlate the slow changes in the impedance angles at long times with particle deposition on the wafer, thus eliminating the need for the common and expensive use of witness wafers.

BIOLOGICAL MATERIALS NANO 3/ NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room A106 – Session BINS-WeM

Artificial Cellular Assemblies

Moderator: D. A. Stenger, Naval Research Laboratory.

8:00 am BINS-WeM0 Electrical Characterization of Artificial Neuronal Networks, J. J. Hickman, K. E. Foster, R. C. Opreson, D. A. Stenger*, A. E. Shaffner, and J. L. Barker*, Science Applications International Corporation, McLean, VA 22102; Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, DC 20375; Laboratory for Neurophysiology, BNP, DIR, NINDA, National Institutes of Health, Bethesda, MD 20892.

We are creating in vitro circuits of mammalian neurons by controlling their adhesion and neurite outgrowth on artificial surfaces. We are using self-assembled monolayers (SAMs) to control the intrinsic and geometric properties of the culture growth surfaces. The ability to control the surface composition as well as other variables, such as growth media and cell preparation, all play important roles in neuron pattern viability. We have recorded the electro-physiological signals produced by neurons on the artificial surfaces in response to stimuli. The surfaces have been characterized by XPS and contact angle measurements and we are relating the intrinsic properties of the surfaces to the cellular development. We are using what we learn for a more fundamental understanding of neuronal circuit development as well as to develop new algorithms for training neural networks.

8:20 am BINS-WeM1 Neuronal Cells Cultured on Modified Microelectronic Device Surfaces, A. Offenhauser*, J. Ruhe*, W. Knowlton*, 1Frontier Research Program, RIKEN, 351-01 Wako-shi, Japan and 2Max-Planck-Institute of Polymer Research, 55128 Mainz, Germany.

The recording of the electrical activity of a large number of neurons in tissue culture over a period of weeks or even months should be very helpful in the understanding of the development and function of biological neuronal networks. Ideally, a method for the detection of the changes in intercellular voltage in such a system should have both high spatial and temporal resolution. One approach could be to record with a fixed electrode array built into the floor of the tissue culture chamber.

We have chosen to detect the electrical signal of the neuron by direct coupling with a field effect transistor. Such a coupling is the first step towards multi-site recording in neuronal nets and the development of neuronal network on a microelectronic device surface it is necessary, to control adhesion and outgrowth of neurons on a microscopic scale. To achieve biocompatibility the chemical composition of the surfaces of such devices have to be modified.

Our approach to control the chemical architecture at the interface is to attach ultra thin polymer films to the device surface by using a novel “grafting from” procedure. It could be shown, that the chemical composition of the interface could be tuned in such a way that Purkinje neurons show good adhesion to such a surface and survive and grow on it for weeks.

8:40 am BINS-WeM2 Using Both Topographic Control and Microspheres Protein Substrates in Controlling Neuron Extension and Connection in Culture, Professor A. S. G. Curtis, Department of Cell Biology, University of Glasgow G12 8QO, Dr. Stephen Britland and Professor C. D. W. Wilkinson, Department of Electronics and Electrical Engineering, University of Glasgow G12 8LT.

We describe techniques of combining topographical guidance and patterning of protein molecules on the surface and other adhesive, non-adhesive and activating and inactivating molecules on surfaces,
to control and to aid the formation of functional neuronal networks in vitro. These techniques have been used with rat CNS neurons in culture to obtain good guidance and electrical activity in culture. The activity of the cells is neither spontaneous or can be stimulated with extracellular electrodes. The same electrodes also fabricated in the substrate can be used for long-term multisite recording. Problems associated with patterning proteins over electrodes will be reported.

9:00 am BINS-WeM3 Photochemically-driven Surface Modifications and Cellular and Biomolecular Assemblies with Micron-order Precision. T. Matsuda, National Cardiovascular Research Institute, 5-3-1 Fujishirodai, Suita, Osaka 565, Japan.

If we can manipulate precisely surface microprocessing, three dimensional (3-D) functionalized surfaces can be created to provide advanced artificial organs, microbiosensors and micromachines in which surface properties play a decisive role in functioning. To this end, precise control of the surface chemical composition at X-Y-plane and microstructure at Z-axis is essentially needed. Since photochemical process proceeds only at irradiated portions, such a surface modification can be dimensionally controlled with micron-order precision in principle.

In this talk, our recent several years-efforts focusing on the development of surface photochemical modification methods will be summarized. At first, XY-manipulation was attained by photoreactivities of p-azidophenyl group, diithiocarbamate group and azide group. After that, we can form a covalent bond with a neighboring chemical group upon ultraviolet (UV) irradiation. Photoreactive group-derivative synthetic and biological polymers were prepared and cast on a substrate. Subsequently UV light was irradiated. Chemical fixation was noticed. When UV was irradiated through a photo mask, a patterned surface was created, which was clearly observed under atomic force microscopy. Photoreactive proteins such as albumin and gelatin were also chemically fixed at UV-irradiated portions. Z-axis manipulation was attained by an excimer laser ablation technique combined with surface photochemical process, resulting in the formation of microtextured surface.

When 2-D surfaces with cell-adhesion promoting regions such as hydrophobic gelatin-fixed parts and cell-adhesion-free regions such as nonionic, hydrophilic or albuminated parts were created, cells adhered, spread, migrated and proliferated only on cell-adhesion promoting regions, resulting in the formation of a 2-D tissue. 2-D and 3-D neural circuits were also demonstrated.

9:40 am BINS-WeM5 Cell Patterns and Whole-cell Biosensors Using Photonic Tweezers Technology. J. Renken and S. Seeger, Universitat Heidelberg, Physikalisches-Chemisches Institut, Im Neuenheimer Feld 253, 69120 Heidelberg, Germany.

Photonic tweezers technology exploits the light pressure in order to trap and transport microscopic particles such as mammalian cells. In our photonic tweezers system, a Nd:YAG laser (1064 nm) is directed into an inverted microscope, which is equipped with a computer-controlled XY-stage and an image processor. This setup was used for the directed transport of cells from chambers showing weak cellular adhesivity to chambers coated with Cell-Tak, a strong cell adhesive allowing the rapid immobilization of cells within cell diameter accuracy. Patterns of different cell lines were fabricated on a single chip. Thus the direct observation of the interaction between different cell classes is possible. By staining the cells with different ion-sensitive fluorescent dyes, concentrations of various ions can be monitored simultaneously and related to the metabolic responses of various cells to drugs. This way, a new tool for in vitro-toxicology is established.

10:00 am BINS-WeM6 Modification and Refunctionalization of Fluoropolymers for Controlling and Directing Neurons at Interfaces, T. G. Fargo et al., State University of New York at Buffalo, Buffalo, NY 14214.

Recent modifications to fluoropolymer substrata have been shown which allows their surfaces to be refunctionalized either homogeneously or in patterns with a variety of materials including conducting metals, proteins or bioactive molecules. Covalent attachment of peptides with bioactive receptor sites for neurons is one of the most recent results. Through lithographic techniques, fluoropolymer surfaces can be patterned with peptides which can act to influence or direct specific attachment of neurons via their active receptors. In this discussion results will be shown illustrating the specificity of NG 108-15 neuroblastoma and PC12 cells to well characterized FEP (fluorinated ethylene propylene) surfaces refunctionalized with laminin derived oligopeptides. Receptor mediated cell attachment is determined using competitive binding assays. Detailed analysis of the refunctionalized FEP surfaces is obtained using ESCA, ToF-SIMS, ATR-FITR, and fluorescent spectroscopy. FEP films having covalently immobilized fluoromeric IKVAV sequences are used to demonstrate a high percentage of receptor mediated cell attachment sites on the IKVAV sequence.

Results are shown where a 6 fold decrease in PC12 cell attachment is observed as determined through a competitive binding assay medium containing the IKVAV oligopeptide.


We report on the initial findings of a program to investigate the directed growth of fungal hyphae on custom-designed integrated test circuits. The motivation behind such a combination is to allow the study of hyphal growth/steering mechanisms at the microscale and to determine the electrical characteristics of the resulting bio-hybrid integrated system. The primary biological element in this work is Urocytes appendiculatus, an organism which has been shown to exhibit profound and unique topographical signal recognition. The integrated circuit substrate is fabricated using standard semiconductor processing techniques. Guiding or "vectoring" elements, a series of 1 micron wide and 0.1 micron high ridges, are etched into the final layer of metallization (aluminum). A self-assembling hydrophobic monolayer is then applied to the circuit to create a suitable bio-compatible layer for cell attachment and growth. The Urocytes spore is placed on a contact pad in the center of the circuit, where it takes in water to allow it to begin the germination process. Germination results in the formation of a germ tube from the spore which grows out to touch the surface. This narrow, cytoplasm-filled structure is then guided by the vectoring elements toward another contact. Since all the structures are part of the metallization pattern, they may be biased to control ion movement in the tube. The integrated test structures have been used to determine the influence of topography and surface coatings on hyphal growth. The vectoring structures were shown to be capable of steering the hyphae to intended connection/termination points through a wide range of angles. In addition, self-assembled monolayers of n-octylchlorosilane on the circuit materials resulted in superior germination characteristics compared to untreated surfaces.

10:40 am BINS-WeM8 Patternning of Microtubules Observed with Atomic Force Microscopy. D. C. Turner, C. Chang, S. L. Brandow, and D. B. Murphy, Jr., Center for Biomolecular Science and Engineering, Code 6900, Naval Research Laboratory, Washington, D.C. 20375-5348, and 1Department of Cell Biology, Johns Hopkins University Medical School, Baltimore, MD 21205.

Microtubules are cylindrical microtubular structures of diameter 25 nm that self-assemble from monomeric tubulin protein in the presence of the nucleotide GTP. In the cell microtubules act structurally, as part of the cytoskeleton, and as substrates for active transport of organelles. This transport is mediated by molecular motor proteins, such as kinesin and dynein, which "walk" along microtubules in the presence of ATP, carrying cargo along with them. We have been studying the adhesion characteristics of microtubules on silica modified surfaces with the goal of producing biologically active, patterned microtubules for the examination of two-dimensionally constrained molecular motor traffic in vitro. Silanes which promote adhesion and surfaces which reduce adhesion will be discussed, as well as a silica which causes depolymerization of the microtubules. Characterization of the silica films was carried out using AFM (atomic force microscopy) and wettability. Biological activity of microtubules immobilized on the silica surfaces was assayed by observing ATP induced binding and movement of kinesin coated beads on the microtubules. Silanes which promoted adhesion were patterned using the method of Calvert and coworkers (Calvert, J. (1993) J. Vac. Soc. B 11, 2155) to produce substrates for selective adhesion of microtubules. Patterns of isotropic and aligned microtubules which were imaged with AFM will be discussed.

Supported by the Office of Naval Research.
Surface Magnetism II
Moderator: B. P. Tonner, University of Wisconsin, Milwaukee.

2:00 pm SSI-WeA1 Experimental Studies of Surface and Interface Magnetism, H. Hopster, University of California, Irvine, CA 92717.

The magnetic coupling between ferromagnetic films through spacer layers is of great current interest due to the associated giant magnetoresistance (GMR) effects. The exchange coupling at various transition metal interfaces between ferromagnetic and non-ferromagnetic materials has been studied by spin polarized electron spectroscopies (secondary electrons, core level photoemission, Auger electrons, and energy loss spectroscopy) by several groups [1-6]. Although surface magnetic moments are difficult to extract directly from the data, it is evidence that the surface moment at Fe(100) is enhanced with respect to the bulk value. Monolayers of Cr, Mn, and V have been shown to couple antiferromagnetically to Fe(100). While Cr and Mn(100) layers order in a layer-by-layer antiferromagnetic structure on Fe(100) no intrinsic magnetic structure has been found in thicker V layers on Fe(100). A largely enhanced surface moment is found on the Cr(100) overlayers.

INVITED


2:40 pm SSI-WeA3 Strain Accommodation and Atomic Order in the Nonpseudomorphic Growth of Ultrathin Fe Films on Cu(001), D. E. Fowler and J. V. Barth, IBM Research Division, 650 Harry Rd., San Jose, CA 95120.

The detailed role of atomic order and of strain in a magnetic film and its interfaces in determining the magnetic anisotropy of the film is not well understood. To probe connections between these properties, we have made a structural study of Fe on Cu(001). Fe films from 0.5 ML to 6 ML were grown at 310 K and at 100 K, then annealed to 300 K. Medium Energy Ion Scattering (MEIS) measurements clearly show that there are deviations from pseudomorphic growth for even low Fe coverages. Structural changes continue to occur as the Fe coverage increases suggesting that the Fe film is attempting to take up a non-Cu fcc-like lattice introducing strain at the interface. By 6 ML (310 K growth) the Fe almost completely achieves this new lattice and, also, shows a surface reconstruction. Our MEIS data are consistent with recently proposed nonpseudomorphic model (1). For 100 K growth and to a lesser extent for 310 K, the interfacial strain is apparently taken up by disruption of the near-interface Cu layers as shown by increased ion scattering from Cu. On increasing from 3 to 4 ML of Fe there is a particularly large increase in Cu scattering from the 2nd and 3rd layers below the interface. At 6 ML (100 K growth) of Fe nearly 5 full Cu layers are exposed to the ion beam. We compare these structural changes with the changes in the perpendicular, magnetic anisotropy constants of these Fe films as determined by an in-situ Magneto-Optic Kerr Effect (MOKE).


3:00 pm SSI-WeA4 Magnetic Structure of Ultra-Thin Mn Films on Fe(100)/Pd(100)*, G. Stierle and C. Rau, Department of Physics and Rice Quantum Institute, Rice University, Houston, TX 77251-1892.

The electronic and magnetic structure of Mn films epitaxially grown on Fe(100)/Pd(100) is studied using spin-polarized electron emission spectroscopy (SPEES). In SPEES surface probed electrons with an energy of typically 150 keV are used to induce the emission of Auger and secondary electrons. Spin analysis of the emitted electrons allows us to determine the spin polarization in the topmost Mn layer. We report on the thickness dependence of the Mn surface magnetization within a thickness range of 1 to 20 monolayers. The relevance of our results towards the exchange coupling mechanism found between ferromagnetic layers separated by a spacer material is discussed.

*Supported by the National Science Foundation, the Welch Foundation and the Texas Higher Education Coordinating Board.

3:20 pm SSI-WeA5 Mössbauer Spectroscopy Study of Tetragonally-Distorted fcc Fe(100), J. W. Freeland, D. F. Storm, I. L. Grigorov, D. J. Keavney, and J. C. Walker, Johns Hopkins University, Baltimore, MD 21218.

Ultrathin films of fcc Fe(100) have been grown by MBE on Cu(100) and Cu1-xAr(x) substrates, and their magnetic properties studied by Mössbauer spectroscopy and SQUID magnetometry in an effort to determine the effect of substrate lattice parameter on the magnetic moment of fcc Fe. The alloy substrates were grown in-situ by co-deposition, and their composition was varied between pure Cu and x = 0.57 Au. This composition range corresponds to a lattice parameter range of 3.61 Å to 3.702 Å. RHEED and Mössbauer structural information suggested that the Fe is pseudomorphic with the substrate, with a tetragonal contraction to compensate for the in-plane expansion.

Mössbauer spectroscopy detected two different magnetic phases coexisting in the film: an antiferromagnetic (AF) phase with low spin, and a higher spin ferromagnetic (FM) phase. Interestingly, the moments of these phases appear to be independent of the substrate lattice parameter. Rather, the relative amounts of the film that exist in each phase changes dramatically with the alloy substrate composition. Over the lattice parameter range studied, the FM phase increases in intensity from about 20% to more than 60% of the film, at the expense of the AF phase. Therefore, it appears that there are two metastable states, and increasing the lattice parameter drives the Fe structure closer to one of these states. SQUID magnetometry observed this as an increase in average moment per Fe atom, in agreement with other total moment studies done on fcc Fe(100).

3:40 pm SSI-WeA6 Magnetic Properties of Fe and Mn Overlayers on Ir(111) by Soft X-ray Circular Dichroism, W. L. O'Brien and B. P. Tonner, Synchrotron Radiation Center, Univ. of Wisconsin-Madison, Stoughton 53589.

A comparison has been made between the structure and magnetic properties of Mn and Fe ultrathin films grown on an fcc(111) substrate. Initially, Mn/Ir(111) gives split (1 x 1) LEED spots, which evolve into a simple (1 x 1) pattern between 2 and 4 monolayer thicknesses. Above 4 ML a new structure with a (3 x 3) LEED pattern forms. In Fe/Ir(111) case, however, the films grow in the more common Kurdjumov-Sachs bcc(110)-fcc(111) structure. A combination of X-ray absorption, photoemission, and X-ray magnetic circular dichroism (XMCD) spectroscopies was used to correlate the changes in film structure with local magnetic moment and magnetic anisotropy (MF) order. Only the Fe films show FM ordering at room temperature (as detected by XMCD). The XMCD data does not support a model of magnetic "dead layers" at the Ir interface, as proposed to explain earlier Fe/Ir superlattice experiments. The Mn/Ir(111) films show an enhancement of the Mn local atomic magnetic moment for atoms at the surface, but this is not associated with the structural changes that occur at different film thicknesses. A small change in the Mn 3s core-level photoemission exchange splitting is found to accompany the increased local moment. These results will be compared to other studies of Mn magnetic films, including c(2 x 2) MnCu and c(2 x 2) MnNi alloys.

4:00 pm SSI-WeA7 Impact of Magnetism on the Stability of Ultrathin Films: The Magnetically Driven Two Dimensional Surface Alloys, S. Blügel, IFI, Forschungszentrum Jülich, D-52425 Jülich, FRG.

The magnetism of 3d, 4d, and 5d transition metal monolayers on various noble metal (100) substrates is shortly reviewed. The existence of two magnetic phases (p(1 x 1) ferromagnetic and c(2 x 2) antiferromagnetic) will be discussed. The main emphasis of this talk will be on the importance of magnetism for the structure and stability of ultrathin magnetic films. Exemplary for 3d transition metal monolayers on noble metal substrates detailed calculations for 3d monolayers on Cu(100) have been performed, which show a clear trend: i) Magnetism acts against interdiffusion. At low coverage, where the
magnetism of Cr and Mn is very large, interdiffusion is suppressed. ii) Magnetism reduces clustering and promotes alloy formation. For Mn we find a strong tendency for alloy formation. From i) + ii) we expect for the case of the Cu substrate a thermodynamically stable, two dimensional surface alloy: Cu(100)c(2×2)Mn. Although in detail, the results depend on the Cu substrate, the trends presented above have an universal character. Therefore, Cu(100)c(2×2)Mn is one example of a much wider class of magnetic surface alloys. Ab-initio calculations have been performed using the FLAPW-method (Full Potential Linearized Augmented Plane-Wave) and are based on the density functional theory in the local spin density approximation.

**SURFACE SCIENCE**

**Room A201 - Session SS2–WeA**

**Solid Liquid Interfaces**

**Moderator:** E. M. Stuve, University of Washington.

**SS1-WeA9 Enhanced Exchange Splitting of Gd at the Monolayer Limit, Dongqi Li**, Materials Science Division, Argonne National Lab, Argonne, IL 60439, Jiana Zhang, P. A. Dowben, Dept. of Physics, Univ. of Nebraska, Lincoln, NE 68588, M. Onellion, Dept. of Physics, Univ. of Wisconsin, Madison, WI 53706.

Ultrathin Gd films on W(110) have been studied with constant-initial-state spectroscopy (CIS) for photoemission utilizing linearly polarized light of synchrotron radiation. The photoemission cross-section of a 5d surface state near E_F shows strong photon energy dependence, i.e., sharp peaks in CIS spectra near the Gd 5p_{3/2} ad- sorption edge. These peaks originate from resonant photoemission processes involving the occupied 5p and unoccupied 5d levels. In particular, mainly the 5d surface states/resonances were utilized in this resonant process. The symmetry of the occupied 5d states were determined experimentally by changing the polarization of light, where p-polarized light excites only to the unoccupied 5d^2 or 5d^3 states and s-polarized light to the 5d_5/2 and 5d_3/2 states. The lower bound of the exchange splitting of the Gd 5d_5/2 surface state can therefore be abstracted. This minimum exchange splitting increases when the thickness of Gd film decreases. The effect is particularly apparent at the monolayer limit, where the minimum exchange splitting increases by about 1 eV compared to that of bulk. Our results are qualitatively consistent with the theoretical predictions that the systems with lower coordination numbers have larger magnetic moments, and therefore larger exchange splitting.

*Work at Argonne was supported by the US DOE BES-Materials Sciences under contract #W-31-109-ENG-38.

2:00 pm **SS2-WeA1 Surface Reconstruction at the Metal-Electrolyte Interface, D. M. Kolb,** Dept. of Electrochemistry, University of Ulm, D-89069 Ulm.

The low-index faces of gold and platinum are well-known to reconstruct when annealed under ultra-high vacuum (UHV) conditions. We have shown in the past that reconstructed gold surfaces are also stable in an electrochemical environment, if certain precautions are taken [1, 2]. At positive potentials specific adsorption of anions usually removes the reconstruction, while by applying negative surface charges the reconstruction is readily restored. The transitions between reconstructed and unreconstructed phase have been studied for Au(100) by in-situ STM which has led to an atomistic view on the dynamics of the potential-induced reconstruction and its anion-induced lifting. It will be shown that the transitions always start at surface defects such as monoatomic steps and therefore, the overall kinetics of these processes depends markedly on the step density. The corresponding current transients point to a nucleation-and-growth process which, however, cannot satisfactorily be described by the standard models of instantaneous or progressive nucleation. For many adsorption processes on gold the potential-induced reconstruction has to be taken into account, because depending on the potential region the reaction may take place on a surface structure vastly different from the one expected for the unreconstructed case. This is demonstrated for pyridine on Au(100) where the molecule adsorbs flat on the reconstructed surface, but assumes a vertical position on Au(100)-(1×1). A reorientation of the molecule occurs concomitantly with the lifting of the reconstruction.


5:00 pm **SS1-WeA10 The Connection Between Morphology and Magnetic Characteristics for Gd Thin Films Grown on W(110),** 


Post-deposition annealing of Gd thin films grown epitaxially on W(110) substrates has been observed in prior studies to drastically alter the magnetic properties of these films. We have used Scanning Tunneling Microscopy to examine the morphology of such films for two different post-deposition annealing temperatures (530 and 710 K). For coverages between 7 and 20 ML, annealing to 530 K is observed to produce smooth, monotonically stepped films. However, annealing to 710 K causes the films to break up into large 3-D islands resting on a base monolayer. The base monolayer is found for the first time to form a 7 × 14 superstructure arising due to the lattice mismatch between the W(110) surface and the hexagonal Gd monolayer. The dimensions of the Gd islands produced by 710 K annealing are used to calculate the in-plane demagnetization factor and Curie temperature for 11 ML thick films. These results are compared to previous magnetic ac-susceptibility (χac) measurements. Our calculations accurately predict the 97% decrease observed in the χac signal between the 530 and 710 K annealed films as well as the increase in the Curie temperature from 247 to 284 K, respectively.


The interactions of sulfate anions in sulfuric acid solutions with the Au(111) and Ag/Au(111) surfaces were studied using Auger Electron Spectroscopy (AES), Low Energy Electron Diffraction (LEED), Atomic Force Microscopy (AFM), and Core Electron Energy Loss Spectroscopy (CEELS) and electrochemistry. At electrode potentials more positive than those within the silver deposition range sulfate adsorbate forms an ordered Au(111)/(√3×√3)R30° adlattice, and gives rise to a corresponding LEED pattern. Following extensive rinsing, silver forms two well-ordered structures: Au(111)p(3×3) and Au(111)p(5×5). However, AFM images reveal a clear p(3×3)-Ag structure, which condenses to a close packed p(1×1)-Ag overlayer at more negative potentials. Distinctive S(LMM) silver electron transitions and the S(L2,3) electron energy loss of surface sulfate show a characteristic S2+ sulfur valency, giving evidence that the sulfur oxidation state is not altered in the UHV environment. The downshift in the electron loss energy at the S(L2,3) level is indicative of increased electron density on sulfur with increasing sulfate-surface bonding. This increased sulfate-surface interaction arises, most probably, from back-donation into empty Rydberg states in the outer-potential wall. These data suggest that metal conduction electrons participate in the anion chemisorption. We conclude that the techniques used are complementary and together provide considerable insight into the Ag upd process.

With the advent of synchrotron radiation, surface x-ray scattering has emerged as an important new probe of the structure and phase behavior of electrode surfaces. The low-index faces of Au, Pt, and Ag have been investigated in a variety of electrolytes. These studies reveal that well-ordered monolayers of electrodepositied anions (I-, Br-, and Cl-) and metals (Cu, Pb, Bi, and Tl) can be formed at the electrode surface. The structure of these monolayers, and of the underlying substrate, can be studied with great precision using surface x-ray scattering. These studies show that commensurate, uniaxial-incommensurate, and biaxial-incommensurate adlayer structures may form and that transitions between these structures can be induced via the applied potential. For adsorbed anions, all three structure types have been observed and the incommensurate structures always electrocompress in the incommensurate phases. Adsorbed phases, composed of anions and cations, may exist over a range of potential. An overview of x-ray scattering from electrode surfaces will be presented, and a comparison with vacuum studies will be provided.

This work was supported by the Department of Energy, Materials Sciences Division under Contract No. DE-AC02-74CH00016.

4:20 pm SS2-WeA8 Model Electrochemical Interfaces in Ultrahigh Vacuum: Ionic and Surface Solution Probed by Infrared Spectroscopy, M. J. Weaver, N. Kishakevich, and V. Illeges, Department of Chemistry, Purdue University, West Lafayette, Indiana 47907, USA.

The last few years have witnessed notable advances in our ability to acquire atomic- and molecular-level structural information for in-situ metal-solution interfaces. The emergence of such spectroscopic and microscopic methods, applicable at metal-UHV and metal-solution interfaces, is also enhancing the significance of fundamental electrochemical modeling studies utilizing the former interfacial environment. This talk will focus on the use of one such method, infrared reflection-absorption spectroscopy, along with work-function measurements (i.e. surface potential) to yield information regarding ionic and surface solution for model electrochemical interfaces ("synthetic double layers") in uhv. Specifically considered will be aqueous and non-aqueous solvation on Pt(111) surfaces in the presence of potassium ions and carbon monoxide. Although this type of information is still largely unobtainable for in-situ systems, the use of a common technique enables the intercomparison, and possibly enrichment, of the information obtained in both metal-liquid and metal-vacuum environments.

4:40 pm SS2-WeA9 Thermal and Photon-stimulated Interactions of Oxygen and Water Coadsorbates with Potassium on Graphite, D. Chakarov, L. Osterlund, B. Kasemo, Department of Applied Physics, Chalmers University of Technology and University of Gothenburg, 412 96 Göteborg, Sweden.

Interactions of O2 and H2O with clean and potassium covered graphite constitute interesting model systems both for purely scientific and technological relevance. We have investigated these adsorbate and coadsorbate systems on graphite (0001) using Temperature Programmed Desorption (TPD), Photo Induced Desorption (PID), and High Resolution Electron Energy Loss spectroscopy (HREELS) at temperatures 85-1000 K. The clean graphite surface is non-reactive toward molecular oxygen or water: at 85 K O2 sticking is below the detection limit, while H2O condenses to form hydrogen-bonded ice aggregates that sublimate at ~150 K. In contrast, O2 readily adsorbs on the K-covered surface to form K-O complexes with stoichiometry depending on the concentrations of the coadsorbates and on temperature. Water coadsorption with K, at low θK, results in substantial structural changes of the H2O layer without dissociation. Above a critical coverage of K, H2O dissociates and reacts with K, to form KOH and KH. During subsequent annealing a number of surface reactions and transformations occur, including formation of potassium oxides and CO2. Photon irradiation causes desorption from the pure K-adlayer but does not desorb pure H2O. It induces a variety of transformations in coadsorbed H2O + K layers including formation of CO2.

5:00 pm SS2-WeA10 Adsorption, Desorption, and Phase Transformation Kinetics of Multilayer D2O and H2O on Au(111) and Ru(0001), C. Huang, E. K. L. Wong, R. Scott Smith, and Bruce D. Kay, Molecular Science Research Center, Pacific Northwest Laboratory, Richland, WA 99352.

Molecular beam scattering and programmed desorption (both TPD and isothermal) are used to study the adsorption, desorption, and phase transition kinetics of H2O and D2O on multilayer ice surfaces. Two substrates, Au(111) and Ru(0001), are used as templates for the multilayer (5-200) water film growth. Water does not wet Au(111) but is known to form an ice-like bilayer structure on Ru(0001). Below 130K the adsorption probability is unity for both surfaces. Above 130K the desorption rate becomes comparable to the incident beam flux and, as such, the net condensation rate decreases rapidly with increasing temperature. On both substrates water films grown below 130K are initially amorphous in nature but undergo an irreversible phase transformation to a crystalline phase at a rate that is both strongly temperature and thickness dependent. Accompanying this phase transformation the multilayer shows complete isotopic scrambling over a thickness range exceeding 60 layers. The desorption kinetics from both phases are markedly non-zero order and substrate dependent. The desorption kinetics can be quantitatively described by a unidirectional scale law. The non-zero order kinetics and the attendant universality class are attributed to the morphology of the multilayer ice films. The experimental results, a quantitative kinetic model, and their implications concerning the structure of thin multilayer films will be discussed.

* Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO 1830.

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NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY/BIMATERIAL INTERFACES 
Room A209 – Session NSBII-WeA

Biology at the Nanoscale: I

Moderator: L. Bottomley, Georgia Institute of Technology.

2:00 pm NSBI-WeA1 Non-contact Scanning Force Microscopy of F-actin, David Braunein, and James A. Spudich, Dept. of Biochemistry, Stanford University, Stanford CA 94305.

Filamentous actin or F-actin, a ubiquitous cytoskeletal protein, consists of 5 nm monomers assembled into a helical structure of 36nm pitch, and 7 nm in diameter. F-actin is one of the best characterized cytoskeletal proteins, and serves as a good sample for testing the limits of resolution of non-contact scanning force microscopy (NCSFM). Unlike contact SFM, NCSFM probes the surface topography of a sample without direct physical contact of the sample with the cantilever tip, hence avoids the type of tip inflicted sample damage that one encounters with contact SFM. Rhodamine-Phalloidin-stabilized F-actin was deposited on sapphire substrates, which provide a flat, reusable surface, that upon submersion to an aqueous environment at pH 7 exhibits a positive net charge that complements the negative surface charge of F-actin. The F-actin is sufficiently immobilized on the surface to withstand subsequent rinsing with distilled H2O and drying with dry N2 gas. NCSFM images of F-actin clearly show filamentous structures, an ~40nm periodicity, an apparent thickness of ~20 nm, and ~3nm high, the reduced height probably the result of sample shrinkage. Although tip artifacts are apparent, such as the larger apparent diameter of the filaments, they are much less pronounced than that seen in contact images of the same samples. Contact SFM images, although showing the filamentous structure of the F-actin, display less of the periodic structure of the filaments over shorter stretches of the filaments, probably as a result the changes in orientation of the tip relative to the filament on the surface. The use of NCSFM in combination with the sapphire substrates should make it possible to image the ultrastructure of other cytoskeletal proteins.

This work is supported by NIH fellowship PHS AR08202-02.


We are studying how the extracellular matrix is built up from individual molecules of collagen and associated polymers. We used a TappingMode™ AFM (NanoScope III) to image Type I collagen monomers, fibrils and complexes of collagen with the proteoglycan decorin. Collagen monomers (obtained from pepinized bovine skin) appeared as flexible rods of variable length. Knob-like structures may be non-helical regions produced as a result of enzymatic digestion. We also found some oligomers in these preparations. Reconstituted collagen fibrils showed well-defined D-bandig, with a period of 68 nm, as well as sub-bands at intervals as small as 20 nm. Asymmetry in these sub-bands was sufficiently distinct to indicate the direction of the carboxyl termini within the polymerized fibrils. The sub-band height contrast was increased by using TappingMode in a dry He atmosphere, suggesting that dehydration is a factor in visualizing the sub-bands. On fibrils reconstituted from a collagen-decorin mixture, decorin was visualized as an orderly pattern of bumps in the gap region. This is the first report of stable imaging of collagen monomers by AFM at room temperature. It is also the first report of direct visualization of collagen-bound decorin without using antibodies or other labels.

We thank Kathryn Vogel for providing the decorin and Helen Hansma and Megdalena Bezanilla for AFM work in dry He.

2:40 pm NSBI-WeA3 Imaging Biological Systems with Near-Field Scanning Optical Microscopy, Jeesong Hwang*, Eric Betzig*, and Michael Elzish*, The Johns Hopkins University, Baltimore, MD 21209, *AT&T Bell Laboratories, Murray Hill, NJ 07974.

The Near-Field Scanning Optical Microscope (NSOM) exploits the near-field optical interaction between a sharp probe and a sample of interest to image surfaces at a resolution (<50 nm) inaccessible by traditional far-field microscopy. In addition to the resolution enhancement, many of the advantages in the conventional optical microscopy such as noninvasiveness, contrast mechanisms and reliability are retained as well in the NSOM technique. Here, the application of the NSOM will be discussed in imaging a variety of biological systems, including phospholipid domains and HLA-I proteins in the membranes of human skin fibroblasts and dynamic features of the model membrane systems during phase transition.

INVITED


Scanning tunneling microscopy (STM) and atomic force microscopy (AFM) have been used to study the tertiary structures of protein molecules such as Chaperonin (Gro-EL) and Immunoglobulin (IgG). We have obtained nanometer-scale STM images of individual, randomly-spaced molecules deposited on thin films of sputter-deposited Au (111) on mica in ultrahigh vacuum. The many well-defined defects such as steps and kinks among the atomically flat (111) terraces act as physical traps for anchoring the molecules for imaging. The two-layered, ring-like structure of Chaperonin agree well with that deduced from transmission electron microscopy. The seven domains in the ring are not distinct. In addition, a structure attributable to a complex of Gro-EL and Gro-ES was also observed. The IgG molecule was observed to be lying flat on the surface. The well-known Y-shaped structure derived from x-ray crystallography of the IgG was readily observed. Although the dimensions of both molecules in the plane of the gold surface agree well with the known dimensions of the molecules, the vertical dimension of the Chaperonin molecules is severely compressed from over 10 nm to ~2 nm, whereas the vertical height of the IgG molecules was compressed much less severely from ~3 nm to ~2 nm. Possible mechanisms for imaging these molecules with the STM will be discussed. The STM results will also be compared to those obtained with the atomic force microscope (AFM).


Manipulation and modification at atomic and nanometerscales of some semiconductor and metallic surfaces has been demonstrated by using one or a combination of the interactions present in Scanning Tunneling Microscopy. This has stimulated a variety of methods for fabrication of nanometer-scale structures and devices. The poor electronic conductivity of biological molecules has prevented the extension of these methods to them. Here, it is described how a low current Scanning Tunneling Microscope (STM) operated in the field emission regime allows to do, reproducible imaging and selective modification of biological membranes. A method is presented (i) to visualize at high resolution hydrated purple membrane sheets, (ii) to produce nanometer-scale marks on them and (iii) to image the altered membranes. These experiments pose the problem of electron transport through 5-10 nm thick insulating materials. We propose a model where the contrast mechanism is controlled by two factors, the electric field at the interface and the transmission through empty states in the membrane.

4:00 pm NSBI-WeA7 SPm Studies of Supramolecular Architecture at Biological Interfaces, M. Hara and W. Knoll, Frontier Research Program, RIKEN, Wako, Saitama 351-01, Japan.

Supramolecular architecture, which is composed of base substrate, single-crystal metal layer, modified layer and biological macromolecules, has been studied by scanning probe microscopy (SPM) to design...
and build up novel artificial layered structures with well-controlled inorganic-organic interfaces. In order to fabricate such heterogeneous interfaces, we have combined self-assembly (SA) and LBK wet processes with dry MBE technique in one system.

Single-crystal metal layer, Au(111) was prepared by metal MBE with in situ RHEED monitoring under UHV condition. The substrate was transferred into an aqueous solution of SA materials such as thiol derivatives, and macro molecules were introduced onto the modified Au(111) surface by SA and/or LBK.

STM/AFM imaging has been carried out in air and solution at room temperature. The images showed homogeneous domains and dislocations of protein monolayers immobilized and superimposed in the well-defined array of the modified Au(111) surfaces with high reproducibility.

This is the first step toward realizing novel material structures with well-controlled biological interfaces, which will provide an outstanding capability to identify specific sites and reveal nanoscopic aspects of supramolecular structures at individual molecular level.

4:20 pm NSBI-WeA8 Control of the Adsorption of the Photobiological Purple Membrane to Surfaces Using Self-Assembled Monolayers, R. A. Britzola, NSWC-White Oak, Code R34, Silver Spring, MD 20903.

Bacteriohodopsin is a photobiological protein found in the purple membrane of the Halobacterium Halobium. Bacteriohodopsin is of interest for applications which utilize its photovoltaic properties, for example in devices for next-generation computational architectures. Potential benefits over semiconductor-based implementations include reduced device size, lower power consumption. In order to realize the goal of microfabricating devices based on purple membrane, a method is needed which allows the site-selected attachment of the purple membrane to a surface with a controlled orientation. Self-assembled monolayers (SAMs) are being investigated as a means to accomplish this. By patterning SAM terminal groups which inhibit and promote purple membrane adsorption, attachment sites for the purple membrane can be defined on a substrate. This talk will present results on the affinity of various SAM terminal groups for the purple membrane. Silane-based SAMs on glass were characterized by angle-resolved x-ray photoelectron spectroscopy (XPS) and contact-angle measurements. Purple membrane films on these SAMs were characterized by XPS and fluorescence microscopy. SAM terminal groups which promote and inhibit purple membrane adsorption have been identified. By using this information together with one of the SAM patterning approaches described in the literature, PM adsorption sites may be formed on a substrate. Other approaches which may allow the attachment of different genetic variants or other functional biomolecules to adjacent sites on a surface will also be discussed.

4:40 pm NSBI-WeA9 Force Modulation Imaging of Protein Membranes, Hirofumi Yamada, Yoshiki Hiritari and Jun Miyake*, Joint Research Center for Atom Technology, 1-1-4 Higashi, Tsukuba 305, Japan, *National Institute for Bioscience and Human-technology, 1-1-4 Higashi, Tsukuba 305, Japan.

Force modulation (FM) technique in atomic force microscopy (AFM) provides new information about the local, mechanical properties of samples using the modulation of the applied load through vertical vibration of the sample at a frequency exceeding the feedback bandwidth of the AFM. This technique allows to detect the difference in composition and/or conformation of organic molecular films. Using the FM technique, we have investigated Langmuir-Blodgett films of the photosynthetic protein membranes, chromatophores, obtained from photosynthetic bacteria Rhodopseudomonas viridis deposited on glass substrates. Chromatophores with various sizes from 50 nm to 1 µm are composed of lipids and protein membrane proteins, the photosynthesis units (PRUs). The modulation frequency used here was 30-40 kHz and the amplitude was about 0.5 nm. The FM images show a remarkable contrast between the glass substrate and the membrane. The hexagonally packed structure of PRUs with the average spacing of 10 nm can be clearly observed in the FM images even when the corresponding structure cannot be found in the normal topography images taken simultaneously. In addition, the FM images reveal that some membranes do not contain PRUs while the topography data show no difference between these membranes and the others.

5:00 pm NSBI-WeA10 LB Films of Disintegrated Purple Membranes: Photo-Electrical Properties and STM Investigation, E. A. Fedorov, V. V. Prokhorov, V. V. Panov, A. A. Kononenko, E. P. Lukashov, D. S. Chernasvitski.

The purple membranes (PM) fragments obtained by ultrasonic disintegration were used as a material for preparing monolayer LB films. The procedures of sample preparation, photo-electrical and STM methods of their study are described. As shown, the LB technique for a buildup of thin PM fragment films gives preparations in which the native optical and photo-induced electrical properties of bacteriohodopsin (bR) are completely retained. The method of STM (under normal conditions in the absence of any conducting coatings and/or replicis) makes it possible to obtain well contrast images of membrane surface which have a regular quasicrystalline structure of BR trimers with parameters in the range of (6.19-6.31) nm. The optimum conditions to obtain contrast images are found to be namely modulation regime, initial bias voltages about (150-300) mV, negative potential at probing tip and rather deep modulation Umod = 100 mV at the frequency of 2.4 kHz. The problems of anomalous high conductivity, superresolution, nature of contrast in STM experiments with biological objects are discussed. The analysis is accomplished of the possibility of electron conductivity and conductivity closely related to protein hydration state, where charge transfer occurs through network of collective surface hydrogen bonds coordinated by polar hydration centers.

NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A207 – Session NS2—weA

Optical Properties of Silicon Nanostructures
Moderator: O. J. Glensbek, Naval Research Laboratory.

2:00 pm NS2-WeA1 Light from Silicon, F. Koch, Physics Department, Technical University Munich, 85747 Garching, Germany.

In searching for ways to employ Si light emitting sources as efficient light-emitting sources for optoelectronic applications, a great deal of progress has been made recently in the understanding of the fundamental processes involved. We review the various emissions between 0.8 and 2.7 eV that can be observed for bulk, crystalline Si (band gap 1.1 eV) by appropriate electrical and optical excitation. Particular interest attaches to the luminescence of Si which is finely divided as nanometer-sized crystalline grains in porous Si or colloidal suspensions. This is quite efficient and persists at room temperature. We show how the physics of quantum confinement and the chemistry of realistic surfaces combine to account for the observed luminescence.

INVITED


We report on efficient visible luminescence from nanocrystalline silicon thin films deposited by plasma enhanced chemical vapor deposition using hydrogen diluted silane source gas mixtures and 110 MHz high frequency plasmas. This technique produces thin films consisting of silicon grains < 100 nm in size in an amorphous silicon matrix. The grain size was controlled by varying the hydrogen content in the substrate temperature. Films have been deposited on glass, ITO coated glass, Cr coated glass, metal foils and silicon wafer substrates. Stain etching with HF:HNO3:H2O solutions yielded films with orange-red photoluminescence at room temperature with efficiencies close to anodically etched porous silicon films on wafer substrates. Temperature dependent photoluminescence measurements were performed on as-deposited and etched films. The luminescence from as-deposited nanocrystalline films was virtually identical to the luminescence from hydrogenated amorphous silicon films deposited from pure silane plasmas with a single PL peak at 1.5 eV. After stain etching, a visible luminescence peak at 1.7-1.8 eV developed while the amorphous silicon peak intensity decreased considerably. Variations in the luminescence with deposition conditions, including doping with boron, phosphorus, or carbon, will be presented. The implications of these results on the explanation of the origin of visible luminescence in nanocrystalline silicon thin films produced by various methods will be discussed.

3:00 pm NS2-WeA4 The Mechanism of Room Temperature Red Light Emission in Porous Silicon and Fabricated Silicon Nanostructures, S. M. Prokes, W. E. Carlos and O. J. Glensbek, Naval Research Laboratory, Washington, D.C. 20375.

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Raman spectroscopy, photoluminescence (PL), and electron spin resonance (ESR) experiments have been performed on as made and atmosphere-oxidized porous silicon. Results indicate the presence of oxygen shallow donors of binding energy of about 0.1 eV, which show a distinct correlation with the intensity of the red room temperature PL observed. It is suggested that the shallow donors become paramagnetic by photo-induced capture of carriers created by light absorption into the silicon crystallites. Annealing studies have shown a correlation between the PL intensity and the paramagnetic shallow donor signal strength. In view of these results, a model is suggested linking the presence of the shallow donors to the presence of non-bridging oxygen hole centers, which are thought to be responsible for the intense red PL reported in porous silicon. In addition, lithographically fabricated silicon nanostructures have also been formed, and they have been examined using micro-photoluminescence and micro Raman spectroscopy. Their light emission and structural properties will be discussed in view of the above suggested model.

3:20 pm **NS2-WeA5 Properties of Ultrathin Films of Porous Silicon**, J. von Behren, Y. Kostoulas, L. Tsybeskov, Ju. V. Vanyushov, and P. M. Fauchet, Department of Electrical Engineering, University of Rochester, NY 14627.

We have produced films of light-emitting porous silicon (LEPSI) as thin as ~1 μm, lifted them off the silicon wafer by an evaporating step, and deposited them onto sapphire windows where they remain attached by Vanderwaal's forces or electrostatic forces. Although others have previously obtained free-standing LEPSI films, our films are one order of magnitude thinner, luminescence strongly and have excellent mechanical properties because of the sapphire substrate. The important steps in this procedure will be discussed in some detail and the structural, chemical and optical properties of these films as measured using a variety of probes will be reported. These films are semi-transparent in the visible and thus make several new optical measurements possible. We will report the results of photoinduced absorption measurements performed with 100 femtosecond time resolution which reveal the first step in the carrier dynamics, namely the trapping of carriers in surface states.

3:40 pm **NS2-WeA6 Luminescent Porous Silicon Layers Fabricated by Anodisation and Supercritical Drying**, L. T. Canham and T. I. Cox, Defence Research Agency, St. Andrew's Road, Great Malvern, Worcestershire, WR14 3PS, United Kingdom.

There is currently enormous interest in the optoelectronic properties of luminescent porous silicon. We have recently demonstrated that ultrathin porous layers ('aerocryals') can be realized by removing the electrolyte from the pores with supercritical CO₂ (1). We show here that a wide range of porous silicon structures, not previously attainable, may be fabricated with good structural integrity using supercritical drying. In many high porosity layers, much of the inhomogeneity reported to date is shown to arise from the capillary force induced collapse of the silicon network during normal ambient drying. Supercritical drying enables us to reveal clearly the large porosity gradients that can result from the electrochemical etching process itself. Oxidation of complete layers gives rise to films of high porosity silica which have subsequently been impregnated with laser dyes to give very bright photoluminescent films. Cross-sectional PL spectroscopy and EDX of the structures described above is presented revealing the depth dependent luminescence properties of such layers. The importance of porosity gradients in elucidating optoelectronic properties will also be discussed.

4:20 pm **NS2-WeA8 Properties of Partially Oxidized Porous Silicon**, L. Tsybeskov and P. M. Fauchet, Department of Electrical Engineering, University of Rochester, Rochester, NY 14627, USA.

We report the preparation and characterization of partially oxidized porous silicon layers obtained for example by anodization of p-type silicon wafers in an HF solution with light assistance. In these samples, FTIR measurements detect a significant amount of O₃-Si-H bonds and the photoluminescence (PL) is stable, peaks between 600 and 570 nm, and has a temperature dependence that can be fitted by an activation energy of only 10 meV. Light-emitting diodes have been made in this type of porous silicon and their very promising electrical and optoelectronic properties have been fully characterized. We find that the electroluminescence (EL) occurs at 760 nm (and not at 600 nm) and is stable (no degradation after 100 hours of operation). In addition, the EL intensity increases linearly with current up to ~ 1 A/cm². The origin of the PL and EL and the prospects for commercial devices will be discussed.


We present the results of an experiment of various means to enhance or suppress the formation of porous silicon. Our results demonstrate that porous silicon LEDs and silicon electronic devices can coexist in close proximity (within 1 μm or less) on the same chip. The first method uses a trilayer photolithographic process, together with silicon nitride, to produce light-emitting porous silicon (LEPSI) lines as narrow as ~2 μm adjacent to fully protected silicon regions. The second method consists of amorphizing regions of the wafer prior to anodization with high energy/high dose ions (ion implantation) such as Si, B or P, followed by anodization and annealing. In this method, LEPSI is produced on the unimplanted regions only. The crystallinity
and electrical properties of the implanted region have been fully character-
ized after annealing. Using focused ion-beam implantation, 50 nm patterns have been obtained. The third method consists of low energy/low dose bombardment (ion milling/reactive ion etching) with argon ions prior to anodization. Under appropriate conditions, we have observed a strong enhancement of the formation rate of LEPSi where bombardment took place, possibly due to the generation of a large number of defects on the wafer surface.

*Work supported in part by grants from the New York State Energy Research & Development Authority and Rochester Gas & Electric.

APPLIED SURFACE SCIENCE
Room A101 – Session AS-WeA

Depth Profiling

2:00 pm AS-WeA1 Fundamental Limits to Sputter Depth Profiling: Atomic Force Microscopy of Ion Beam-Induced Topography, Eun-Hee Cirlin, Hughes Research Laboratories, Malibu, CA 90265.

Sputter-induced surface topography, atomic mixing, and the statistical nature of the sputtering process contribute to degradation of the depth resolution and changes in secondary ion yields of sputter depth profiles. Hence accurate sputter depth profiling of atomically abrupt interfaces of modern layered structures poses stringent requirements for the understanding of the transport of ions in matter, and the physics and chemistry of ion-target interaction at the surface and interface. In this talk, first, recent advances in understanding of the mechanism of the ion bombardment–induced roughening and smoothing of surfaces caused by various factors—sputtering, redeposition, implantation of primary ion, ion-target interaction, surface diffusion, and segregation—will be reviewed. Sputter-induced roughening has long been attributed primarily to variations in secondary ion yield with angle of incidence and surface temperature for a given primary ion species. To illustrate, an exciting video involving AFM investigation of ripple nucleation, formation, evolution on a GaAs surface during 3 keV O2− bombardment at θ = 45° will be shown. In addition, recent insights gained on the fundamental limits to sputter depth profiling from the studies of SIMS and AFM of delta-doped layers and superlattices will be discussed. Next, using AFM measurements together with SIMS depth profiles of Si delta-doped GaAs, with and without sample rotation, various contributing components of the depth resolution including sputter-induced roughness, atomic mixing, statistical sputtering, and inhomogeneity of the ion beam current density will be quantified. Also, the Gaussian resolution functions of 2σ = 0.2λ before and after ripple formation will be examined. Finally, recent international SIMS round robin studies of the depth resolution of Si delta-doped GaAs from 25 laboratories will be discussed.

3:00 pm AS-WeA4 Resolution in Sputter Depth Profiling Assessed by AlAs/GaAs Superlattices, K. Kajiwara* and R. Shimizu*, Sony Corp. Research Center, Yokohama, 240, Osaka University, Suita, 565 Japan.

AlAs/GaAs superlattices are proposed as a potential reference material for sputter depth profiling since, at present, AlxGa1−xAs/GaAs superlattices are grown in a defect-free single crystalline state with atomically flat and abrupt interfaces by metalorganic chemical vapor deposition. It is possible to determine accurately the depth resolution of AES, XPS and SIMS depth profiles using AlAs/GaAs superlattices. A preliminary round robin test was performed by the ISO/TC201 Depth Profiling WG in Japan.

As a result, it is clear that in all the AES and SIMS data, the depth resolution is constant having no relation to the sputtered depth. On the contrary, in the XPS data, which have a larger probing area, a degradation of depth resolution occurs with increased sputtered depth. It is estimated that the depth resolution is correlated with the probing area owing to the crater edge effect. In addition, in the SIMS data, the resolution of the leading and trailing edges depends strongly on primary reactive species (O2−, Cs+). This is in contrast to AES and XPS data using inert ions (e.g. Ar+). It was confirmed that AlAs/GaAs superlattices as a reference material are very useful for accurate evaluation of the resolution in sputter depth profiling.


The effects of misorientation on the ion yield during SIMS sputter depth profiling have been studied for the GaAs(100) surfaces miscut by 0°, 2°, 5° and 7°. The MOCVD grown samples with the structure of AlGaP/GaAs/GaAs (Si doped substrate) were sputtered with 5 keV O2− or Cs+ primary ion beam at an incident angle of 60° from the surface normal. The relative secondary ion yields of Al and In were measured. In case of Cs+ primary ion beam, there was no detectable change in the secondary ion yield with the surface misorientations. On the contrary, the analysis with O2− primary ion beam showed a correlation between the change of secondary ion yield and the degree of misorientation of the surface. The relative secondary ion yield was increased with increasing the degree of misorientations in order to investigate this dependence from the topographical point of view, the crater bottoms after sputter erosion were scanned by AFM. The AFM results revealed a close resemblance to the observed correlation between the change of ion yield and the degree of misorientation. The relationship among the degree of misorientation, the secondary ion yield and the topographical change will be discussed.


The high sensitivity of SIMS makes it a desirable technique to quantitatively study the compositional gradients produced in glass surfaces due to manufacturing and secondary processing conditions. Earlier studies of commercial soda-lime silicate glass, done in a Cameca IMS-3F using negative primary beam bombardment, showed unexpected gradients in the sodium, silicon and calcium concentrations within 100 nm of a fracture surface of this glass. A series of similar sodium-silicate and soda-lime glasses has been prepared and analyzed to study these unexpected gradients. Both the magnitude and depth of the gradients observed in the silicon profile show a strong dependence on the presence of calcium in the glass. The same depth profiles in the glasses show an apparent surface enrichment, however the in-depth behavior of the sodium profile varies with the glass composition. This result, coupled with the difficulty in reproducing the measured Na/Si signal ratios suggest that sodium may be mobile during the analysis, despite stable surface charge compensation. The calcium profiles in all the glasses show an apparent surface depletion, and the approach to steady state signal levels is not effected much by the glass composition. These gradients are not expected on fracture surfaces, there-
fore their presence on manufactured or processed surfaces does not necessarily indicate true compositional changes. Methods to correct for these gradients will be discussed.

4:00 pm AS-WeA7 The Effect of Tilt Angle on As Impacts in Si, J. M. Anthony, and J. A. Keenan, Texas Instruments, Dallas, TX 75265.

Arsenic implantation is often used to dope the walls of trench capacitors in high density DRAM structures. In order to investigate As incorporation during angled implantation, Si wafers have been implanted with As ions (100 keV, 1.25 x 10^15 at/cm²) at a variety of tilt angles from 0–80 degrees. BF₂ ions (80 keV, 5 x 10^15 at/cm²) were also implanted at 0 degrees in each wafer to establish a consistent depth marker during SIMS analysis. Range and peak concentration data were extracted from the SIMS data and are compared to predictions from simulation programs. Dose measurements for the steeply angled implants are difficult with SIMS due to surface ion yield transients, so absolute dose measurements have been made using RBS and neutron activation analysis. At high tilt angles (similar to the geometries used for trench implants) backscattering of the As ions greatly reduces the implanted fraction, and this is also compared with computer simulations.


Ion beam sputtering has been widely used for sputtering depth profiling with XPS and AES. However the problem of the surface compositional change due to ion bombardment has remained to be understood and solved. In this work, the altered surface layer of amorphous Ta₂O₅ thin films due to Ar⁺ ion bombardment was depth profiled nondestructively and quantitatively, for the first time by Medium Energy Ion Scattering Spectroscopy as a function of the ion incidence angle, the ion energy, and the ion dose with the depth resolution of better than 1 nm. The MEIS spectrum showed that the oxygen depleted depth is 30Å under normal incident 3 keV Ar⁺ ion bombardment and it decreases continuously with the incidence angle to 1Å under the 80° grazing incidence angle. The increased Ta concentration at the surface due to preferential sputtering of oxygen atoms was about 47% under normal incident 3 keV Ar⁺ ion bombardment and about 20% under 80° grazing incidence. The altered zone was saturated in the ion dose of 3 x 10¹⁸ ions/cm². With the increase of the ion energy from 3 keV to 5 keV, the depth of oxygen depleted zone increased slightly from 30Å to 40Å. The above experimental results are compared with XPS results and the Monte Carlo Simulations.

4:40 pm AS-WeA9 Nondestructive EPMA Depth Profiling of Buried Oxide Layers in Silicon, A. P. Alexeyev, Research Centre for Surface and Vacuum Investigation, 2 Andrejevskaya nab. Moscow 117334 Russia.

The application of electron probe X-ray microanalysis (in wavelength dispersive mode) to obtain the depth profiles of buried oxide layers in silicon is discussed. If the electron beam of energy Ep falls onto the surface of the analyzed specimen, the characteristic X-ray intensity of oxygen atoms will be proportional to the convolution of two functions: the depth profile W(Z) of oxygen atoms and the depth distribution of X-ray production for the characteristic X-rays of oxygen atoms Φ(Z,Ep). One needs to take into account the absorption of X-rays in the analyzed specimen. One can obtain m coupled equations with respect to W(Z) by measuring the oxygen intensities at m values of Ep. In this case one can find the input parameters (m more than this unknown profile which will provide the coincidence between the calculated and measured X-ray intensities. 4–5 parameter fitting of W(Z) curve was developed to characterize the buried near surface oxide layers. Most of the "polysilicon/ SiO₂/bulk Si" sandwich structures having the polysilicon and SiO₂ layers of different thickness was prepared. The measurements of O-Ka intensities on these structures made possible to find the Φ(Z,Ep) curves that were used for W(Z) determination

5:00 pm AS-WeA10 Real Time Monitoring of Depth Profiling, L. A. Vasylev, A. V. Naumkin, V. I. Rakovsky, Research Center for Surface and Vacuum Investigations, 6, bld. 1, Kazarnennaya per., 109028 Moscow, Russia.

Depth profiling (DP) is a widespread procedure in XPS, AES and SIMS. Ion sputtering is very convenient for DP. Usually DP experiment takes a lot of time and destroys a sample. If the region of interest has been passed one has to get another sample and to repeat an experiment. Ion sputtering can modify the sample composition and affect DP results (broad the interfaces, alter the composition of the inhomogeneities and the interfaces, etc.) So it is a good idea to change the sputtering conditions when the region of interest is at the depth of 100–200 Å. Under the surface sputtered. In our investigation we studied the ion induced X-ray excited Auger electron peak (IXSAMPS) sensitivity to monitor the interfaces and inhomogeneities appearance. The results show that with samples consisting of the insulating film on the conducting substrate (SiO₂ at Si-like) it is possible to detect the interface at the depth of 100–300 Å under the surface sputtered.

PLASMA SCIENCE

Room A109 – Session PS—WeA

PLASMA-INDUCED CHARGING AND CONTAMINATION EFFECTS

Moderator: M. G. Blain, Sandia National Laboratories.

2:00 pm PS-WeA1 Effect of Plasma Overetch of Polysilicon on Gate Oxide Damage, J. P. McVittie and C. T. Gabriell*, Stanford University, Stanford, CA 94305-4070, *VLSI Technology, San Jose, CA 95131.

Modeling and previous experiments have indicated that charging damage during polysilicon etching occurs just before endpoint when the last of the exposed polysilicon can collect excess plasma currents which cause damage by flowing through the gate oxides. During overetch, this damage mechanism is suppressed by the poor collection efficiency of the exposed polysilicon edges, and as such damage from this mechanism is expected to remain constant during this period. Although there is an additional edge damage mode where damage can increase during overetch, we here report results showing a decrease in damage with overetch time. The experiment consisted of two lots of fully processed CMOS wafers where the polysilicon overetch percentage was varied from 0 to 90%. The etch tool was a triode system which showed antenna dependent charging damage. The test structures were large area capacitors with a 0.5 nm thick oxide whose breakdown statistics were measured after the full process including anneals. Both the n-channel and p-channel devices showed damage which decreased with overetch time. The damage was significantly worse for the n-channel devices—the mean n-channel gate oxide breakdown voltage was 3 V at endpoint and increased to 11.5 V at overetch. The effect was repeatable in that both lots showed the same results.

The results indicate an apparent annealing during overetch. In the past, rf annealing has been reported for e-beam damage with a proposed mechanism involving rf heating and carrier motion. We consider this unlikely and suspect that low plasma currents collected by the gate edge could be playing a role in this annealing; however, etching of the exposed gate oxide during overetch may also be important. The n and p channel differences can be explained by a voltage drop across the n-well which reduces the tunnel current through the p-channel gate oxides.

2:20 pm PS-WeA2 Plasma-Induced Gate Oxide Charging Issues for sub-0.5µm CMOS Technologies, A. K. Stamper, J. B. Lasky, and J. W. Adkisson, IBM Microelectronics, Essex Junction, Vermont 05452.

Gate oxide charging issues which occurred during the development of IBM's 200mm wafer sub-0.5µm 16-Mb DRAM and logic processes are discussed. We show that gate oxide degradation is caused by a constant electron current, but it can be modulated by systematically changing the plasma properties. Gate oxide charging can occur during reactive ion etching (RIE) and plasma enhanced chemical vapor deposition (PECVD) processes. Using poly-conductor (PC) antenna structures and fully integrated PC test sites, we investigated three RIE processes: metal etch (BCL/CW/N₂), oxide etch (CHF₃/C₂F₆/Ar), and polycrystalline etch (HCl/O₂/H₂); Ar sputter processes; and TEOS and SiH₄ based PECVD processes. Charging increased when the PC was
exposed during the etch and decreased when the PC was coated with photoresist. Oxide RIE and Ar sputter were the most charge-sensitive etch processes; TEOS oxide was the most charge sensitive PECVD process. I-V measurements on PC antenna structures showed that the degradation can be separated into two components: gate oxide trap creation and catastrophic gate oxide defects. The gate oxide traps result in a transient current during I-V testing and can be passivated (or activated) by a 400°C H2/N2 anneal. Significant charging can occur during TEGIS. In addition, RIE and Ar sputtering will cause TEOS partial pressure decreased, as phosphorus doping decreased, as RF power decreased, and as overall pressure increased. By measuring the optical emission, rf current, and rf voltage during the film depositions, we concluded that charging decreased as rf voltage and optical emission of the plasma decreased, indicating that gate oxide charging is a strong function of electron energy and density in the plasma.

2:40 pm PS-WeA3 Gate Oxide Damage due to Local Charging Caused by Electron Shading, Koichi Hashimoto, Fujitsu Limited, C832, 1015, Kamikodanaka Nakahara-ku, Kawasaki 211, Japan.
Degraded gate oxide breakdown voltages have been observed after exposing antennas (10,000:1 area ratio) to an electron cyclotron resonance (ECR) plasma when the antennas were covered with resist patterns having high-aspect-ratio openings. Similar antenna structures appear near the endpoint of ordinary metal etching transiently, because of microloading effect, and also appear in via hole etching inherently. In contrast to the well-studied damage caused by charge-up voltage difference across the wafer, the damage reported here was observed even when the test wafer was cut into small (5 mm square) chips where the wafer-scale voltage difference, if existed, could not affect any longer. In addition, the aspect ratio of the resist patterns raised the gate oxide failure frequency. These results indicate that the structure itself induced a local differential charging; and they led the author to "electron shading" model: the resist patterns above antenna metal shaded it from oblique-incidence electrons, resulting in an excessive positive current from ions impinging normally. This effect was observed in various such as parallel-plate and ECR plasmas, for metal etching and via hole etching. Moreover, the damaging current appeared to increase by a factor of more than ten with a decrease in gate oxide thickness from 8 nm to 6 nm, and hence serious damage could be expected for future ULSI. This dependence on gate oxide thickness can be explained with a somewhat complicated model taking into account the electric field building up in and around the resist patterns.
INVITED

Refractory metal thin films for non-all oxidized ohmic and Schottky contacts are finding wide application in the fabrication of high-speed GaAs FETs and IETS. Although reactive ion etch (RIE) and electron cyclotron resonance (ECR) etches have been reported to yield submicron dimensions and anisotropic profiles, plasma-induced-damage to the GaAs active layers has not been reported.
We have studied the effect of plasma-induced-damage on the majority carrier transport properties of GaAs by monitoring changes in sheet resistance of thin film conducting layers under etch conditions for refractory metal contacts. Sheet resistance (R_s) determined from transmission line measurements are used to evaluate the depth of plasma-induced-damage for ECR and RIE etch conditions by varying the thickness of active epitaxial layers. At high dc-bias ECR conditions (300 V), damage extends throughout the conductor layer yielding infinite R_s, for 150 cm to 3E12 cm^-2 at thicknesses of 100 Å and 1800 Å, respectively. Under RIE conditions at 400 V dc-bias, only the 1E18 cm^-2 material was completely depleted. This data suggests the bulk of the damage occurs within 1000 Å of the surface, however increasing the dc-bias can significantly extend this depth. For low dc-bias conditions in the ECR and RIE plasmas (70 and 190 V, respectively), the lower R_s layer (by approximately a factor of 2) indicative of less plasma-induced-damage. This observation may be attributed to significantly higher ion densities in the ECR which will be investigated as a function of microwave power.

4:00 pm PS-WeA7 The Role of Electrode Characteristics in Particulate Trapping in RF Discharges, J. E. Daugherty and D. B. Graves, University of California at Berkeley, Chemical Engineering, 201 Gilman Hall #1462, Berkeley, CA 94720-1462.
The control of plasma-induced particulate contamination is a matter of increasing concern in the microelectronics industry. It has been observed that particles are often "trapped" in dome and ring shaped clouds. Since these clouds are usually located in close proximity to the wafer, they pose a considerable contamination threat. It has been suggested that topographical features on the electrode surface (e.g., the wafer) may cause weak electric field nonuniformities that result in cloud formation. However, electrode topography could also cause thermal nonuniformities as well as disruptions in the gas flow and ion motion in the vicinity of the topography. Consequently, it is not certain that the electrostatic force is the dominant force trapping the particles. We present the results of a series of experiments with a specially designed aluminum electrode. This electrode has an electrically and thermally isolated aluminum ring imbedded in its surface. The temperature, electrical bias, and local geometry of the ring may be controlled independently from the rest of the electrode. In addition, the aluminum ring may be replaced by an alumina (electrically insulating) ring. In this way we examine the relative importance of thermal, electrical, topographical, and materials properties in forming particle traps. The particle traps are analyzed in terms of the fundamental forces that act on the particles.

Acknowledgments: This work was funded by Swiss Federal Research Grant EF-REN(93)035 and BBW93.0136 for BRITE-EURAM project BE-7328.

4:20 pm PS-WeA8 Strategies for Controlling Dust Particle Transport in Inductively Coupled Plasmas, Helen H. Hwang, Michael Grapperhaus and Mark J. Kushner, University of Illinois, Dept. Electrical and Computer Engineering, Urbana, IL 61801.
Dust particle transport and contamination of wafers in plasma etching and deposition tools depend on the relative importance of fluid drag, ion drag, electrostatic, thermophoretic and gravitational forces on the particles. Previous work has shown that electrode topography (i.e., grooves) in conventional RIE tools can influence dust particle transport by perturbing the sheath edge at which particles accumulate. Similar strategies are less successful in high plasma density etching tools since the sheath voltages are lower and ion fluxes (producing ion drag forces) are larger. We have developed set of computer models to investigate the transport of dust particles in an inductively coupled plasma (ICP) reactors. A 2-dimensional plasma equipment model is used to produce ion fluxes, electric fields and advective gas flow fields. A dust particle tracking model uses these quantities to formulate the forces on the dust particles and resolve their trajectories. Results from these models will be discussed with regard to the trapping of dust particles and contamination of wafers.

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The dominance of ion drag forces results in most particles produced in the volume being swept out of the plasma to surfaces. However, particles do accumulate in the periphery of the reactor where ion fluxes are low. These particles may then be redistributed when the power is lowered, and ion drag forces decrease.

1Work supported by NSF, SRC, Sandia/Sematech and Univ. of Wisconsin ERC for Plasma Aided Manufacturing.

4:40 pm SASE-WeA9 The Dynamics of Particulates in the Afterglow of an RF Excited Plasma, C. K. Yoon, J. H. Kim, K. W. Whang, Department of Electrical Engineering, Seoul National University, Seoul, Korea.

The formation of the particulate cloud was examined by the laser light scattering method with the DLC film deposited wafer in an RF excited Ar plasma. The particulates suspended around the plasma sheath boundary above the substrate and continued to grow during the discharge. The absolute value of the negative self-bias voltage and the plasma potential decreased when the particulates agglomerated around the plasma sheath boundary. We used the laser sheet and the medium speed camera to obtain the time-resolved images of the moving particulates and observed the particulates showing directional motions in the afterglow. When the RF power was turned off, the direction and the characteristic time of the particulate density decay were highly dependent on the experimental conditions such as the electrode temperature, the gas flow rate and the pressure. In some experimental conditions, most of the particulates were observed to drift onto the wafer surface in a few tens of milliseconds. The afterglow particulate behavior was highly influenced by the DC bias voltage which was applied to the electrode and it indicates that the particulates have residual negative charges. The dependence of the afterglow particulate behavior on the experimental conditions suggests some modified plasma turn-off process which can reduce the particulate contamination of the substrate surface.


Particle contamination in plasma tools used for the manufacture of VLSI semiconductor devices on silicon wafers is a major cause of yield loss. Understanding the dynamics of particle movement in the post-plasma regime is important to explain the process of their transport to the wafer. The movement of particle contamination in a Gaseous Electronics Conference Standard Cell in the post-plasma regime was investigated using a novel technique. Particle clouds were observed using laser light scattering together with an image intensifier and a monochromator. This technique allows particle clouds of low density to be seen which cannot otherwise be detected. Video analysis of the particles showed movement of the cloud front in the first few milliseconds after the plasma is turned off. The particle cloud is destroyed in the first 10 to 20 milliseconds of the post-plasma. The role of the thermophoretic force on particles during the post plasma is investigated. Values for this force are obtained from the video analysis of particle movement for various electrode temperatures and are compared with theoretical values.

SURFACE SCIENCE/ELECTRONIC MATERIALS
Room A102 – Session SASE-WeA

Semiconductor Reactions II
Moderator: T. W. Engel, University of Washington.

2:00 pm SASE-WeA1 Thermal Stability of Hydroxyl Species on the SiO2 Surface, Ofer Sneh and Steven M. George, Department of Chemistry and Biochemistry, University of Colorado, Boulder, CO 80309.

Many semiconductor device processing steps occur on the SiO2 surface. The main functional group on the SiO2 surface is the hydroxyl (SiOH) species. The chemical properties of the SiO2 surface depend on the hydroxyl coverage and the stability of the SiOH species versus temperature. In this study, the thermal stability of SiOH species was investigated on SiO2 films deposited by chemical vapor deposition on Si(100) substrates. The hydroxyl coverage was determined using two monolayer-sensitive methods in ultrahigh vacuum. In one set of experiments, methanol (CH3OH) was used to "titrate" the hydroxyl species because CH2OH forms hydrogen bonds to SiOH species. The methanol coverage hydrogen-bonded to SiOH species was measured by CH2OH temperature programmed desorption (TPD) using an in situ mass spectrometer. Additional experiments measured the hydroxyl coverage by desorbing the SiOH species as H2O (SiOH + SiOH → H2O + Si-O-Si) using laser induced thermal desorption (LITD) techniques. Both the CH2OH TPD and H2O LITD experiments monitored the progression and nearly linearly decrease of the hydroxyl coverage versus thermal annealing from 300–900 K. The declining hydroxyl coverage versus annealing temperature is attributed to the thermolysis of the Si-O-Si surface. i.e., SiOH + SiOH → H2O + Si-O-Si. The results on these well-defined SiO2 surfaces are consistent with earlier infrared and nuclear magnetic resonance measurements of hydroxyl species versus thermal annealing on high surface area silica powders. Implications of these measurements for semiconductor device processing of SiO2 surfaces will be discussed.

2:20 pm SASE-WeA2 Correlation of Surface Core Levels and Structural Building Blocks Through High Resolution Core Level Spectroscopy: Si(111)-7×7, J. J. Pagel, W. Theis, K. Horn, Fritz-Haber-Institut der MPG, D-14195 Berlin, Germany, Ch. Jung, C. Hellweg, and H. Petersen, BESSY GmbH, D-14195 Berlin, Germany.

The Si(111)-7×7 reconstruction is among the most frequently studied semiconductor surface, and in view of its complexity it is particularly important to understand the relationship between the geometric and electronic structure of its building blocks. Surprisingly, very little is known about the exact nature of the SCL shifts is still a subject of controversy. Here we demonstrate by high resolution core level photoemission, using charge of one of the new generation of plane grating synchrontron radiation monochromators, that apart from the surface line that was previously found, additional core level shifts exist. In agreement with a previous study, it is found that, due to diffraction effects, the intensities of the surface components exhibit maxima at fixed photon energies, and its use in assignment on the basis of electron mean free paths and relative numbers of contributing atoms may be subject to error. We arrive at a consistent assignment of the SCL peaks to the different structural building blocks of the (7×7) structure by comparing our low temperature data with spectra recorded above the temperature of the (7×7) → (1×1) phase transition at 1100 K, and through an analysis of SCL intensities over a range of photon energies. These data yield new insight into the influence of different bonding environments on photoemission core level line shifts.


Real-time studies of abstraction of surface H and D by atomic H and D (D2 and H2) from polytetrafluoroethylene and diamond, and single crystal Si and Ge surfaces are presented. Time-of-flight detection of low energy H* and D* ions enables real time kinetic measurements of the abstraction yields H D and D D. The abstraction rates are linear in D2 and H2 flux, 1st order in D2 and H2, and independent of the initial D2 and H2. The abstraction energy is due to enhanced abstraction rates from v = 1 vibrational states of the adsorbed H2 and D2Si and Ge surfaces. Using these results, we find that the abstraction barriers are 4.5–5 Kcal/mole on Si and Ge are approx 16 Kcal/mole on diamond. The origin of the abstraction barrier on Group IV surfaces appears to depend on the energy transfer between the clean and H terminated surfaces. All of our results are consistent with a generalized Eley-Rideal abstraction mechanism.

3:00 pm  SSEM-WeA4  Local Adsorbate Structures of F$_2$ Chemisorbed onto the Si(111)-7×7 Surface: A Molecular Beam/STM Study, I. A. Jensen, C. Yan, A. C. Kimmell, U.C. San Diego Chemistry, La Jolla, CA 92039.

The chemisorption of a mono-energetic molecular beam of F$_2$ onto a room temperature Si(111)-7×7 surface is examined using scanning tunneling microscopy and the resulting adsorbate structures are studied as a function of translational energy/chemisorption mechanism. For F$_2$ chemisorption on Si(111)-7×7, there is no intrinsic physisorption state and therefore no island formation at low incident translational energy, E$_{trans}$. Instead at low E$_{trans}$ (0.03eV), we observe that the dominant adsorption sites are single reacted adatoms (Si-F), while at higher E$_{trans}$ (0.27eV) dimers/pairs of reacted adatoms are commonly observed. From previous work by the Ceyer and Carter groups, it is known that at low E$_{trans}$, F$_2$ can adsorb via abstraction (F$_2$ collides, one F atom chemisorbs, the other is ejected into the gas phase), while at higher E$_{trans}$, dissociative chemisorption becomes the predominant adsorption mechanism. Our STM experiments, in conjunction with a simple Monte Carlo model, show that at low E$_{trans}$, the abstraction mechanism accounts for nearly all chemisorption because nearly all adsorption sites are single reacted adatoms. At higher E$_{trans}$, the dissociative chemisorption mechanism becomes important and the predominance for single site adsorption is greatly reduced while the occurrence of dimers/pairs of reacted adatoms is increased. Comparisons are made to the high E$_{trans}$ chemisorption of Cl$_2$ onto the Si(111)-7×7 surface which produces almost exclusively reacted dimers/pairs of adjacent adatoms.

3:20 pm  SSEM-WeA5  First-Principles Study of Structures and Reactions on Si/SiO$_2$ Surfaces, Krishnan Raghavachari, AT&T Bell Laboratories, Murray Hill, NJ 07974.

In this work, we have used first-principles quantum chemical techniques to understand the nature of the structural species and microscopic reaction mechanisms on Si/SiO$_2$ surfaces. We use the “cluster approach” where the surface is represented by clusters of atoms designed to model the important aspects of the local interactions. Examples will be given on detailed structural assignments based on correlations between the local structures and the experimentally observed vibrational frequencies of hydrogen terminated silicon surfaces. Additional studies investigating microscopic reaction mechanisms for processes such as etching reactions of HF and H$_2$O on Si/SiO$_2$ surfaces are also reported. Kinetic considerations are found to be extremely important and an understanding of the relevant energy barriers is critical to provide a proper description of such surface reactions.

INVITED

4:00 pm  SSEM-WeA7  Translational-Energy-Induced Etching of n$^+$ Si(100) by Hyperthermal Cl$_2$ Molecular Beams, Y. Teraoka, I. Nishiyama, Microelectronics Research Laboratories, NEC Corporation, 34 Miyukigaoka, Tsukuba, Ibaraki 305, Japan.

The reactivity of Cl$_2$ molecules with n$^+$ Si(100) in hyperthermal translational energy range was investigated using molecular beam etching technique with a high temperature nozzle. The translational energy threshold had been already found to be 2.1 eV and anisotropic etching profiles had been also achieved owing to the stimulated etching [1]. In order to understand the role of translational energy, the etch rates were measured as a function of substrate temperature as well as translational energy. The two kinds of thermal desorption processes were found by the resulting Arrhenius plots of etch rates. The activation energies, correspondent to each desorption process, are 2.7 eV and 1.2 eV, respectively. The former is the same as those of a conventional gas etching and the latter governs the translational-energy-induced etching. The small activation energy implies that a new type of cloration to Si structure is formed on the surface. In addition, the small activation energy was not changed by the translational energy up to 3.0 eV. Even though the impinging Cl$_2$ molecules have translational energies over the small activation energy, the etching is driven by the thermal desorption of the chlorinated structure. The translational energy over 2.1 eV may be necessary for overcoming an energy barrier to produce such new chlorinated structure.


Surface diffusion of adsorbates has been notoriously difficult to measure on semiconductors, and the effects of techniques intended to speed diffusion have been even more difficult to quantify. However, an understanding and quantification of such effects is needed to develop improved processes for chemical vapor deposition and molecular beam epitaxy. We have been employing the scanning hot electron microscopy (SHM) for this purpose. In SHM, the decay of a step concentration profile is measured directly. Recent experiments on Si have confirmed the results obtained previously in our laboratory for several adsorbates on Ge. That is, under processing temperatures surface diffusion occurs by a vacancy mechanism with an extraordinarily high activation energy and prefactor. For example, for SB on Si(111) the activation energy is 54 kcal/mol and the prefactor is $1.5 \times 10^{13}$ cm$^2$/s. Molecular dynamics simulations give insight into this mechanism. The diffusion rate may be enhanced substantially by bombardment with low-energy (50 eV) argon ions. Enhancements of pure diffusive motion by factors of 100 have been quantified. The enhancement appears to be a momentum transfer effect, because electron bombardment even at high energies provides no measurable enhancement. When the ion bombardment is near glancing evidence, there is evidence that a net convective motion of adsorbate is induced as well. That is, adsorbate atoms are actually "blown" across the surface. These results are important for processing applications because the ion energies are so low that little surface damage results.

4:40 pm  SSEM-WeA9  Thermal and Photochemical Reactions of HN$_3$ on Ge(100): A Lower Temperature Route to Germanium Nitride, Craig Tindall, John C. Hemminger, Department of Chemistry and the Institute for Surface and Interface Science, University of California Irvine, Irvine, CA 92717.

Because of the reactivity of its thermally grown native oxide, an optimal process for passivating the germanium surface has yet to be found. Ge$_2$N$_2$ and GeO$_2$, with compounds found to be promising candidates. However, the most commonly used nitrogen precursor, NH$_3$ requires a relatively high temperature in order to thermally grow Ge$_2$N$_2$. For this reason, alternative precursors for growing Ge$_2$N$_2$ are desirable. We describe here the first use of HN$_3$ as a nitrogen precursor for forming Ge$_2$N$_2$. We have studied the chemistry of HN$_3$ with Ge(100) using HRREELS, TDS, LEED, and AES. HN$_3$ adsorbs molecularly below 25°C. It begins to decompose around 100°C, desorbing N$_2$ and leaving N-H groups on the surface. At 300°C the N-H is dehydrogenated and germanium nitride is formed. We have found that HN$_3$ facilitates the formation of Ge$_2$N$_2$ at a substantially lower temperature (350°C) than that required previously when using NH$_3$ (700°C). In addition, HN$_3$ adsorbed on Ge(100) is photosensitive. Visible light decomposes HN$_3$ on the germanium surface, again desorbing N$_2$ and leaving N-H groups on the surface.


Using STM we find that quench cooling a Ni/Si(111) surface results in a 1×1 lattice gas of “ring-clusters” (RCs) plus small 2×2 patches of Si adatoms on T$_2$ sites, similar to the Co/Si case. [1] LEEM observations show that the 1×1 structure forms preferentially on wide terraces, decorating steps with a denuded zone of clean 7×7 whose width decreases with cumulative Ni dose and quench rate. As coverage is increased above 0.16ML a 1/19 structure forms followed by islands of Ni$_{52}$. At 700°C the 1×1 structure dissolves without change of domain shape, leaving clean 7×7. This shows that the RCs do not diffuse across the surface. For warm deposition (reactive epitaxy), islands of Ni$_{52}$ form in a matrix of 7×7 with no RCs. The island size distribution is narrowly peaked at 100nm, suggesting coherent, strained islands, similar to the Ge/Si(100) system. These dissolve at 700°C, leaving clean 7×7. Implications for silicide processing are described.

THIN FILM
Room A105 - Session TF-WeA

Deposition and Characterization Techniques of Nanostructures in Thin Films
Moderator: R. Sargent, OCLI.

2:00 pm TF-WeA1 Deposition and Characterization Techniques for Nanostructures in Thin Films, J. C. Bietello and S. M. Yalisove, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109.

Traditional methods of characterizing the magnitude of the internal stresses in thin film nanostructures involves measuring the physical curvature of the film-substrate couple. These common procedures, which include laser scanning and optical interferometry; measure only the average stress throughout the film thickness and are sensitive to surface conditions. Two new X-ray diffraction techniques will be discussed in the present paper. The first, double crystal diffractometry (DCDT) allows an assessment of the strains of the crystal lattice planes and is hence inherently less sensitive to the state of the sample termination surface. Rough surfaces, as well as studies of buried layers, are possible by this method. It is feasible to obtain a complete map of the strain tensor field over the entire area of a large wafer using DCDT. The second characterization technique, which will be described, uses grazing angle incidence X-ray scattering (GIXS) measurements of the strain distribution through out the depth of a thin film, i.e. depth strain profiling. On a microscopic level, the details of the strain distribution as a function of depth through the thickness of the film can have important consequences in governing film quality and the ultimate morphology of the microstructure. The information obtained from GIXS observations allows one to determine the depth dependence of the principal strains for polycrystalline thin films of virtually any composition. In practice, for very thin films, the GIXS technique requires using a synchrotron to have sufficient intensity in the scattered X-rays to make measurements practical. The application of these techniques will be reported for a variety of refractory metal films, including Mo, W, and Ta on Si substrates. The implications with respect to controlling the internal stress in thin films and their concomitant microstructure will be discussed.

INVITED

Research supported by the USARO and ARPA under contract DAAH03-91-0235 and at SSRl, funded by the USDOS, Office of Basic Sciences.

2:40 pm TF-WeA3 Crystallographic Relations in Zirconia-Alumina Multilayer Nanolaminates, M. Gajdarziska-Josifovska and C. R. Aita, Laboratory for Surface Studies, U. Wisconsin-Milwaukee, P.O. Box 784, Milwaukee, WI 53201.

Nanoscale multilayer films of polycrystalline zirconia and amorphous alumina were characterized by high resolution electron microscopy. Previous results (1) show that the layer spacing can be scaled to insure the growth of metastable tetragonal (t) zirconia, the desired phase for good film mechanical behavior. Here, we report the crystallographic relations between tetragonal zirconia and adjacent regions that have transformed to monoclinic zirconia (m), the STP phase. The growth orientation of the parent phase is t(111) parallel to the growth interface, t(111) transforms to m(001), resulting in a displacement of 9° vicinal to the growth interface. m(200)/t(200) planes are invariant under transformation. Twinning in the monoclinic phase is the dominant mode by which strain is accommodated. Monoclinic unit cell distortions and the implications for transformation toughening behavior of these nanolaminates are discussed.


Research supported under ARO Grant No. DAAH0493G0238 and NSF Grant No. DMR9113680.


Nanostructure fabrication often requires the conformal deposition of thin films in trenches, via, and other high-aspect-ratio structures. Characterizing the surface coverage of thin films deposited in such structures usually requires cross-sectional SEM, which can be difficult. To study more easily the conformality of nanometer-scale thin films deposited in high-aspect-ratio structures, we have developed a process for producing a planar array of silicon micro-capillary structures on a silicon wafer. Capillaries with cross sections as small as 1 μm x 2 μm and lengths as long as 400 μm have been fabricated using conventional microfabrication processes. After fabrication, silicon oxide or silicon nitride films are deposited on the inner and outer surfaces of the capillaries by LPCVD. After deposition, the capillary "roof" is removed via selective etching and/or mechanical techniques. This exposes the oxide or nitride deposited on the silicon capillary "floor". The film thickness as a function of distance down the capillary can be accurately measured on the capillary floor by interferometry. We will present surface coverage measurements for several LPCVD dielectric thin film processes in structures with aspect ratios from 10:1 to 200:1 or greater.

This technique of optically measuring thin films deposited inside micro-capillaries is useful for studying the surface coverage, kinetics and transport characteristics of deposition processes in ultra-high-aspect-ratio structures.

INVITED

2 μm x 2 μm x 400 μm
Silicon Micro-Capillary

3:20 pm TF-WeA5 Fabrication of Thin Films with Highly Porous Microstructures, K. Robbie, S. K. Dew, T. Smy* and M. J. Brett, Univ. of Alberta, Edmonton, Canada and *Carleton University, Ottawa, Canada.

Metal and insulator thin films of densities less than 50% of bulk and with very high surface area to volume ratios have been deposited by an evaporation technique. Multiple evaporation sources were used to provide a symmetrical but very oblique (>80°) flux incident on the substrate. Extreme self-shadowing produced vertical columnar microstructures consisting of isolated and evenly spaced columns. Significant columnar anisotropy may be produced in the plane of the substrate by variations of the deposition geometry. For metal films, this anisotropy gives rise to a directional conductivity variation exceeding 20% between two axes parallel to the substrate surface. The growth of these structures has been simulated by a model incorporating ballistic deposition and minimization of chemical potential in order to better understand and optimize their formation. Applications of these films as catalytic surfaces for gas sensors, thermal insulators, and polarizers will be discussed.

Highly Porous MgF₂ Film

UVH sublimation has been used to deposit C$_60$ fullerite on in-situ cleaved lamellar substrate MoS$_2$(0001). The van der Waals epitaxy is evidenced by the observation of the hexagonal LEED pattern characteristic of the close-packed C$_60$ structure with an intermolecular spacing of $\sim 10.9$ Å. At low coverage, the simultaneous observation of LEED spots corresponding to the substrate and the C$_60$ overlayers allows to specify the geometry of the epitaxy. The intensity curve of C$_60$ photoemission line as a function of the C$_60$ dose indicates a layer by layer (Franck-van der Merwe) growth mode. A detailed analysis of the photoemission lines of the substrate and the C$_60$ core levels recorded at monolayer and multilayer coverage indicates a small electron transfer ($\leq 0.02$ electron per C$_60$ molecule) from the substrate to the first C$_60$ layer. A C$_60$ line shift of $\pm 100$ meV to higher binding energy, for emission from a multilayer film with respect to a monolayer, is observed and interpreted as reflecting a solid state effect. The valence band probed by UPS (hv = 21.21 eV) at various coverages shows only a weak hybridization between energy bands of the substrate and those of C$_60$, notwithstanding a work function decrease of $\sim 1$ eV upon C$_60$ deposition.

4:00 pm **TF-WeA7** Compositional and Microstructural Characterization of Fe-N Thin Films for Recording Sensor Applications, Y. K. Kim and P. B. Narayan, Rocky Mountain Magnetics, Inc., 2270 South 88th St., Louisville, CO 80303-3188.

Polycrystalline Fe-N thin films with typical thicknesses less than 100 nm have been characterized. Fe-N films are promising for recording sensor applications due to good magnetic properties and materials reliability. Films were fabricated reactively on Al-oxide precoated Si wafers by a dc magnetron sputtering system under Ar and N$_2$ environment. Magnetic properties measurements indicate that the control of N is critical to achieve desired magnetic softness. The purpose of this study is to understand processing, properties, and microstructure relationship as a function of N content in the film. Characterization of compositional and microstructural evolution will, therefore, provide valuable insights to uncover the magnetic behavior of Fe-N films. Figure shown on the right represents the XPS composition-depth profile of an Fe-N film prepared at R = 0.35 where soft magnetic properties were achieved. R is defined as a ratio between N$_2$ and Ar flow rate. TEM studies showed that when R = 0, the film has an average grain size of 30 nm with many grain clusters. As R increased, the grain size became as small as 10 nm with nanocrystalline selected area diffraction pattern. Annealing at 150°C for two hours did not produce any noticeable change in the microstructure.

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**ELECTRONIC MATERIALS**

Room A108 – Session EM-WeA

**Wide-bandgap Nitrides**

**Moderator:** P. D. Holloway, University of Florida, Gainesville.

2:00 pm **EM-WeA1** Low Carrier Concentration GaN Grown by MOCVD at Low Temperature (720°C), J. C. Chen, Bing Yong, F. Semonds*, and N. Bambha*, Dept. of Electrical Engineering, Univ. of Maryland Baltimore County, Baltimore, MD 21228; *IR Optics Technology OFC, Army Research Lab., Ft. Belvoir, VA 22060.

Gallium nitride (GaN) based materials are wide-band-gap III-V compound semiconductors ideally suited for devices in the visible-to-ultraviolet region of the spectrum. Successful growths of GaN have been reported by several groups. However, the growth of device-quality GaN films is still a difficult task. Problems such as high growth temperatures, difficulty in achieving low n-type background concentration and difficulty in achieving p-type material make it difficult to fabricate GaN-based devices.

In this study, we have carried out a series of experiments in the growth of GaN/AlGaN by MOCVD. The typical growth was conducted at atmospheric pressure. Sapphire with (0001) orientation, one inch diameter, was used as a substrate. TMGAs, TMAI, NH$_3$ and H$_2$ were used as Ga, Al, N and carrier, respectively. The GaN films were characterized by C-V, PL and double crystal x-ray diffraction methods. The preliminary results indicates that we are able to get the crystalline GaN (confirmed by double crystal x-ray measurement). The FWHM of GaN (002) peak is around 220 arc seconds which is comparable to the result grown at high temperatures ($> 1000°C$). The background n-type concentration as low as $2 \times 10^{15}$/cm$^3$ (measured by C-V method) was achieved by using normal growth conditions at low temperature (720°C). In addition, a special purification method was employed to purify source materials. After using this purification method, the background concentration becomes very low (less than $3 \times 10^{15}$/cm$^3$, estimated) and it is too low to be measured by CV method.

2:20 pm **EM-WeA2** Ohmic Contacts to Intrinsic N-Type GaN,* S. Miller, P. H. Holloway, Department of Materials Science and Engineering, University of Florida, P.O. Box 116400, Gainesville, FL 32611, J. Pankove, Department of Electrical Engineering, University of Colorado at Boulder, Box 425, Boulder, Colorado 80309-0425.
Ohmic contacts by In, Ti, Ti/Au, and Ti/Si/Au were made to intrinsically n-type CVD grown GaN (n = 2 x 10^19/cm^3) films 2 μm thick. The contacts were deposited by thermal evaporation or planar magnetron sputter deposition. Contact properties were investigated by current-voltage analysis, scanning Auger microscopy, scanning electron microscopy, and secondary ion mass spectroscopy. It was found that as deposited In formed an ohmic contact with a resistance of 0.5 Ω cm^2. No interfacial compounds were found after heat treatments of up to 200 C for 30 minutes. Ohmic behavior will be explained by an analysis of the Fermi level and the band alignment between In and GaN. The Ti/Au contacts, annealed for 15 seconds in N_2 gas at 900 C, formed an excellent ohmic contact. Thermodynamic data suggest that formation of a Ti/N layer at the interface is the mechanism for the ohmic behavior. This will be further explained by an examination of the Fermi levels and band alignments. The best ohmic contacts have been observed for the previously unreported metalization scheme of Ti/Si/Au after annealing for 15 seconds in N_2 gas at 900 C. The superior performance will be explained on the basis of an interfacial reaction and diffusion, supported by SIMS and Auger data.

*This work supported by ONR/ARPA Grant N00014-92-J-1895.

2:40 pm EM-Wea3 InGaN/AlGaN Blue-Light-Emitting Diodes, Shiji Nakamura, Department of Research and Development, Nichia Chemical Industries, Ltd., P.O. Box 6, Anan, Tokushima 774, Japan.

Much research has been done on high-brightness blue light-emitting diodes (LEDs) for the use of full-color display or full-color indicators with the characteristics of high-reliability and high-speed. However, it has been impossible to obtain high brightness blue LEDs with the luminous intensity over 1 cd. Recently, we have succeeded in commercializing InGaN/AlGaN double heterostructure (DH) blue LEDs with the luminous intensity over 1 cd for the first time. In this report, the characteristics of the InGaN/AlGaN DH blue LEDs are described. Figure 1 shows the structure of the InGaN/AlGaN DH LEDs. The peak wavelength of the electroluminescence is 450 nm and the FWHM of the peak emission is 70 nm. The typical output power of the InGaN/AlGaN DH LEDs is 1.5 mW at 20 mA. The external quantum efficiency is 2.7% at 20 mA. A typical on-axis luminous intensity is 1.2 cd at 20 mA. The forward voltage is 3.6 V at 20 mA.

![Fig.1 InGaN/AlGaN DH LEDs.](image)


We have studied an atomic layer growth process using amine-alane adducts as a precursors for depositing aluminum nitride thin films at temperatures between 300–700 K. Adsorption and decomposition of these precursors were studied in UHV using Auger electron spectroscopy, temperature programmed desorption and Fourier transform infrared spectroscopy. The atomic layer deposition process was carried out in a MOCVD reactor at atmospheric pressure using H_2 as a carrier gas. Dimethylaluminalane (Me_2AlH_3) and ammonia (NH_3) were used to deposit AlN thin films at temperatures as low as T = 610 K. The source gases were sequentially flowed into the reactor with hydrogen flush steps between each source gas step. Thin films were deposited onto Si(100) and 6H-SiC(0001) substrates that were heated on a susceptor by rf-induction. Analysis by x-ray photoelectron spectroscopy and electron microscopy reveals the deposition of AlN at a temperature of 610 K. A high resolution scan of the N(1s) region shows a peak component at a binding energy 396.8 eV corresponding to AlN; the spectrum contains a second component at 398.3 eV which is associated with surface NH_2 species. Processing at a lower temperature (T = 510K) results in no detectable AlN deposition. Processing at higher temperature (T = 723 K) results in nitridation of the Si surface to Si_3N_4 but no detectable AlN deposition.

3:40 pm EM-Wea6 Growth of Epitaxial Aluminum Nitride Films on Hydrogen Terminated Si(111) Surfaces, F. Ahmad, T. Lenane, G. Auer, Department of Electrical and Computer Engineering and R. Naik, Department of Physics, Wayne State University, Detroit, Michigan 48202.

Thin films of aluminum nitride (AlN) were grown using the technique of plasma source molecular beam epitaxy (PSMBE). The deposition method uses a magnetically enhanced hollow cathode lined with aluminum as the target material. The films were grown on hydrogen terminated Si(111) substrate. Epitaxy was achieved at substrate temperatures ranging from 400°C to 600°C. Several bias settings ranging from 0 to –20 V were used to control the energy of the depositing ions and the flux. Structural characterization of the films were performed by x-ray diffraction (XRD) and high resolution electron microscopy (HREM). The XRD pattern shows only AlN (0002) reflection peak. The absence of other reflections indicate complete film texture with AlN[0001] // Si[111]. Cross-sectional transmission electron microscopy reveals epitaxy of AlN on Si(111) with orientation relationships of AlN[2110] // Si[011]. Furthermore, HREM micrograph of AlN/Si(111) interface shows parallel lattice fringes of Si(111) and AlN(0001) extending up to the interface, indicating sharpness and the absence of interfacial dislocations. Although it is commonly believed that below 600°C polycrystalline growth occurs, our observations clearly indicate epitaxial growth in the temperature range of 400°C to 600°C. Low temperature epitaxial growth of AlN probably occurs as a combination of the controlled kinetic energy of the depositing species from the PSMBE source and the change in surface energy of the hydrogen terminated silicon substrate.

4:00 pm EM-Wea7 Growth of In_{1-x}Al_{x}N and In_{1-x}Ga_{x}N by Metalorganic Molecular Beam Epitaxy, C. R. Abernathy, S. J. Pearton, J. D. MacKenzie, S. Bharathan, and K. S. Jones, Department of Materials Science and Engineering, University of Florida, Gainesville, FL 32611.

II-N devices such as heterostructure lasers or heterojunction bipolar transistors require development of the ternary materials. To date, most attention has been focused on the binary compounds GaN, AlN and InN. In this work, growth of the In-containing ternaries, In_{1-x}Al_{x}N and In_{1-x}Ga_{x}N, on GaAs (100) substrates by metalorganic molecular beam epitaxy (MOBE) has been investigated. In order to suppress the absorption of In from the surface, the growth temperature was kept below 540°C. As the In mole fraction, x_{In}, is increased in either material, the polytype changes from cubic to a mixture of cubic and hexagonal and becomes increasingly defective. Electrically, the carrier concentration decreases with decreasing In content from n ~ 10^{18} cm^{-3} in the InN to semi-insulating in the GaN and AlN. The decline is more rapid for In_{1-x}Al_{x}N than for In_{1-x}Ga_{x}N, probably due to the larger bandgap of the In_{1-x}Al_{x}N. The rate of the drop with x_{In} in either material system is increased if hydrogen is used as the carrier gas rather than HCl. Presumably this effect is due to passivation of the native defect or impurity responsible for the electron concentration. TEM, XRD, growth rate and morphology as a function of material composition will be given. Material grown on sapphire substrates will also be discussed for comparison.


Deuterium lasers in the III-N materials system require achievement of high p-type doping levels. It is found that hydrogen passivation occurring during MOVPE growth of GaN and related alloys limits the acceptor concentrations in as-grown samples and that post-growth annealing at ≥550°C is necessary to reactivate the dopants. We have quantified the outdiffusion using D-splasma treated (250–400°C; 30 min) of H₂ implanted GaN, AlN and InN annealed from 300–900°C, and SIMS profiling to measure the resultant redistribution. In plasma treated samples two populations of D are found — (i) a high-concentration (> 10^{19} cm^{-3}) near-surface (< 0.2 μm) region probably due to the formation of platelet defects and (ii) a lower concentration (~ 10^{17})...
MANUFACTURING SCIENCE AND TECHNOLOGY

Room A110 – Session MS-WeA

Diagnostics, Sensors, and Control

Moderator: L. M. Cecchi, Sandia National Laboratories.

2:00 pm MS-WeA1 Process Control in Semiconductor Manufacturing, S. W. Butler, Semiconductor Process and Device Center, Texas Instruments, Dallas, TX 75205.

The goal of this presentation is to propose what level of process control in semiconductor manufacturing is required for current device technology and future device technology. The current level of process control implemented in the industry will be highlighted and areas requiring further development will be suggested. Some of the benefits of process control that Texas Instruments already experienced will be related. The major concepts of process control will be discussed. The technical need for process control, such as machine aging, will be presented, along with the appropriate control implementation schemes. The basic requirements for control to be possible will also be introduced. The role of metrology, specifically in situ vs. ex situ, as pertaining to process control methods will be presented. Examples of process control applications in semiconductor manufacturing will assist in explaining the concepts introduced during this presentation. The various control methods will be linked to facilities doing research and development of process control.


We describe an adaptive nonlinear controller for water-to-wafer plasma gate etch for 0.9 micron CMOS technology. The typical process requires constant human intervention to estimate the over-etch time. The algorithm uses in situ process signatures and information from a historical database to compute, in real time, the ideal over-etch time for the current wafer etching within the reactor. Our adaptive process gives a comparable standard deviation (about 10 Angstroms) for the remaining gate oxide without human intervention. Since humans have effectively been removed from the feedback loop the process throughput has improved. The production worthy controller is being installed on two reactors in our MOS line in Orlando, Florida.

3:00 pm MS-WeA4 Real-Time Process Control Method Using Ellipsometry Applied to Gate Etching, J. T. C. Lee, N. Blayo, A. Grevoz, H. L. Maynard, and D. E. Ibboton, AT&T Bell Laboratories, Murray Hill, NJ 07974.

Current trends toward single wafer processing using advanced low pressure, high density plasma sources require rapid etching rates that place higher demands on the accuracy of process control algorithms and diagnostic techniques. In particular, etching of multilayer gate stacks for ULSI applications requires an extremely anisotropic and rapid process that exhibits unprecedented selectivity to the gate oxide. The high cost of wafers precludes extensive use of time consuming, destructive post-process diagnostics for process development. Optically based diagnostic techniques such as interferometry, optical emission and ellipsometry offer non-invasive and non-destructive means for obtaining real-time information during etching.

We have discovered that with judicious selection of the probing wavelength, ellipsometry signals remain highly sensitive over patterned features, and thus can be used for real-time control of manufacturing processes. The ability to use ellipsometry over patterned structures was unexpected. In this paper, we present the real-time traces obtained at energies from 1.5 to 4.0 eV during the gate etching of sub 0.35 μm CMOS devices using a helium plasma source. We quantify the effect of mask patterns and underlying topography on the effective band of "useful" wavelengths, i.e., wavelengths that provide characteristic signatures of the endpoints of the various films being etched.

3:30 pm MS-WeA5 The Application of Real-Time Optical Monitors to Semiconductor Manufacturing, James A. O'Neill, IBM Microelectronics, Semiconductor Research and Development Center, Hopewell Junction, NY, 12533.

The manufacture of advanced microelectronic devices is testing the limits of dry process technology. Emerging technologies such as high density etching plasmas and high pressure chemical vapor deposition systems are being developed to meet the fabrication demands of advanced devices. Also, the utility of conventional processing tools is being extended to reduce development and manufacturing costs. In both efforts, optical sensors have received increased attention as a means to characterize new technologies and to improve the productivity of existing tools. Optical technologies are particularly appropriate for semiconductor manufacturing applications since they provide a sensitive, non-perturbing means to monitor and control dry processes in real-time. This talk will discuss the novel application of two optical sensing techniques to semiconductor device fabrication equipment. First, an infrared-based sensor is employed to control the delivery of condensable feedgases to CVD reactors used to deposit dielectric films. This sensor continuously monitors the flow of reactants to the chamber and reduces the need for test wafers typically used to verify film thickness and dopant concentration. Second, an ultraviolet absorption technique is employed to monitor reactants in a high density plasma reactor used for the selective etching of SiO2 films. This sensor aids process development by identifying reactor parameters which affect the concentration of species which control etch selectivity.

INVITED


Reactive chemical processes play a critical role in semiconductor manufacturing, but concentrations of reactive gas species are rarely measured during the process, nor used for process control. Mass spectroscopy, already in use as residual gas analysis for contamination control in manufacturing, has been exploited here for rapid real-time sensing of both reactant and product species in single-wafer CVD reactions, including RTCVD polySi from SiH4 and LPCVD SiO2 from TEOS. Active mass spectrometric sampling at pressures to 5 Torr is achieved using two-stage differential pumping of a sampling aperture in the exhaust gas stream, permitting response times as short as 5
The effects of incident ion/metal flux ratio $J_i/J_T$ on the microstructure and texture of TiN films produced by ultra-high vacuum reactive magnetron sputtering have been investigated using x-ray diffraction and cross-sectional transmission electron microscopy (XTEM) including high-resolution XTEM. The films, typically 1.0 μm thick, were deposited at a pressure of 20 mTorr in $N_2$ on thermally oxidized SiO$_2$ substrates at 350°C. The flux ratio $J_i/J_T$ at the substrate was controlled between 1 and 15 by means of a variable axial magnetic field superimposed on the permanent field of the magnetron. The potential difference accelerating the ions to the floating substrates remained relatively constant (≈ 20 V) as the ion flux (primarily $N_2^+$) was varied. Films deposited at $J_i/J_T = 1$ nucleated with a mixed texture, but subsequent film growth resulted in a porous columnar structure in which the preferred orientation evolved to complete (111) texture at film thickness above 0.2 μm. In contrast, films deposited at $J_i/J_T = 14$ nucleated with a complete (002) texture which was retained during growth to yield a dense columnar structure. Once film texture fully evolves, however, changing ion flux has little further effect on preferred orientation, as pseudomorphic forces dominate, but still controls intercolumnar porosity.

2:20 pm VM-WeA2 The Effect of 20-95 eV Ar Ion Bombardment on Molecular Beam Epitaxy of GaAs(100), J. Mirecki Milliehannick and S. A. Barnett, Department of Material Science and Engineering and the Materials Research Center, Northwestern University, Evanston, Illinois, 60208, L. Hultman, Physics Department, Linköping University, Linköping, S-581 83, Sweden.

Low-energy ion-assisted molecular beam epitaxy (IAME) is useful for suppressing three-dimensional island nucleation and phase separation in III-V semiconductor alloys. However, little is known about ion damage in this energy range. Defect generation in GaAs(100) has been studied in situ by Reflection High Energy Electron Diffraction (RHEED) and ex situ by cross-sectional Transmission Electron Microscopy (XTEM). Ar$^+$ bombardment was performed at energies $E =$ 20 to 95 eV and current densities $J =$ 0.1 to 0.75 mA/cm$^2$ both on static surfaces and during homoepitaxy. RHEED patterns exhibited 2 × 4 reconstruction and strong Kikuchi lines at all times, indicative of flat and ordered surfaces. XTEM showed that subsurface dislocation loops and stacking faults were generated when $E > 50$ eV, for example, during bombardment of a static surface at $E = 75$ eV and $J =$ 0.5 mA/cm$^2$, the density of the defects was on the order of $5 \times 10^{10}$ cm$^{-2}$. Films grown under $E < 50$ eV and $0.25 < J < 0.75$ JV/cm$^2$ irradiation did not exhibit any defects. While there was no measurable ion etching for $E < 30$ eV, the deposition rate of films grown at $E =$ 50 eV and $J =$ 0.5 mA/cm$^2$, for example, was decreased by 0.16 nm/s. These results show that the beneficial effects of IAME can be achieved without causing ion damage or sputtering for $E < 50$ eV.

2:40 pm VM-WeA3 Theoretical Considerations on Stress Effects on Thin Film Microstructure, David J. Strolovitz, Dept. of Materials Science & Eng., University of Michigan, Ann Arbor, MI 48109-2136 USA.

Stresses affect the microstructure of thin films in several ways. These include texture effects, island formation, film morphology, and the nature of defects (such as grain boundaries) that make up the microstructure. The microstructure can also play a key role in determining the magnitude of the stresses in a thin film by controlling stress relaxation mechanisms. This talk will review several important areas of stress/microstructure interaction and will focus primarily on how stresses modify texture, grain size, grain boundaries and other planar defects in thin films.

3:20 pm VM-WeA5 Surface Thermodynamics effects on Thin Film Microstructure, R. C. Cammarota, Department of Materials Science and Engineering, The Johns Hopkins University, Baltimore, MD 21218.

Associated with every surface (interface) are surface thermodynamic quantities referred to as surface (interfacial) tension and stress. It is well known how surface tension influences such processes as normal grain growth in thin films. However, there are several other less well known mechanisms of microstructural development where surface thermodynamic properties play a central role.

Surface and interfacial tensions and stresses will first be defined and related to thin film systems. This will be followed by a description of how these quantities can affect processes involving texture development, epitaxial growth, surface structure, and internal stresses. In particular, the work of Thompson et al. on surface tension effects on
texture development by abnormal grain growth as well as recent developments concerning the influence of surface and interface stresses on film growth will be presented. INVITED

4:00 pm VM-WeA7 A Molecular Dynamics Study of Transient Processes During Deposition on (001) FCC Metal Surfaces, C. M. Gilmore* and J. A. Sprague* (1) School of Engineering and Applied Science, The George Washington University, Washington, D.C. 20052, (2) Surface Modification Branch, U.S. Naval Research Laboratory, Washington, D.C. 20375-5345.

Strong RHEED oscillations have been observed during growth of Cu onto (001) Ag substrates at 77 K. This result indicates layer by layer film growth and surface mobility of the deposited atoms. This unexpected result was explained by proposing a "transient mobility" to account for the atom mobility normally required for layer by layer growth. We have utilized molecular dynamics simulations with the embedded atom method to study transient processes that occur over hundreds of ps. Transient processes were observed that promoted the formation of clusters, and other processes were observed that resulted in spreading atoms across the surface.


4:20 pm VM-WeA8 Synthesis of Epitaxial Sn,Ge$_x$ alloy Films by Ion Assisted Molecular Beam Epitaxy and Pulsed Laser Deposition, Gang He, Maggie E. Taylor, Harry A. Atwater, California Institute of Technology, Pasadena, CA 91125.

The metastable Sn,Ge$_x$ alloy system is an interesting group IV semiconductor material with potential applications in the fabrication of Si-based high performance heterojunction devices and long wavelength infrared optoelectronic devices. Band structure calculations have suggested that the Sn,Ge$_x$ alloys may have direct energy gaps continuously tunable from 0.55 eV to 0 eV for compositions x from 0.2 to 0.6 with very low electron effective masses and hence high mobilities. However, syntheses of Sn,Ge$_x$ alloy films in this composition range by conventional epitaxial or polycrystalline film growth techniques have not been successful due to the severe surface segregation of Sn during the film growth. In this work, we report the synthesis of epitaxial Sn,Ge$_x$ (Ge(001)/Si(001)) with compositions up to x = 0.35 by ion assisted molecular beam epitaxy with 20-40 eV Ar$^+$ ions produced by an electron cyclotron resonance ionization source with ion to atom flux ratios of the order of unity in the substrate temperature range of 140 to 170°C. High flux low energy ion beam irradiation greatly inhibits Sn segregation without interrupting epitaxy. In situ reflection high energy electron diffraction during the film growth indicated that the Sn,Ge$_x$ films were epitaxial up to a thickness of 10-20 nm and subsequently undergo a gradual epitaxial to polycrystalline transformation. Rutherford backscattering spectra confirmed the Sn,Ge$_x$ alloy compositions and indicated an absence of Sn segregation. Comparison of ion assisted molecular beam epitaxy and pulsed laser deposition in the same chamber, as well as ion channeling, x-ray diffraction, transmission electron microscopy, and optical absorption studies will be discussed.


We have studied systematically thermal annealing effects on the surface morphology of 800 A thick gold films, using a scanning tunneling microscope (STM). The gold films were thermally evaporated onto glass and mica substrates, and were then measured with the STM at room temperature before and after annealing. The annealing treatments were done at temperatures between 200 to 500°C and for times from 3 to 60 hours. The topographic images were analyzed using various statistical methods and image processing techniques. We present data showing the evolution of the surface-grain size distribution and roughness amplitude of the gold films as a function of annealing temperature and duration. The typical grain size was found to increase with time for all annealing temperatures, whereas the roughness amplitude shows a more complex dependence on the annealing parameters. We are now developing a theoretical model to describe quantitatively the surface processes taking place during annealing. Computer simulations based on our model will be performed and compared with experiment. The possible fractal nature of the film surfaces and its change upon annealing will also be discussed.

5:00 pm VM-WeA10 Structure-Property Relationships in Compositionally Modulated Titanium-Aluminum Thin Films, Rajiv Ahuja and Hornish L. Fraser, Department of Materials Science and Engineering, The Ohio State University, 116 W 19th Ave., Columbus, OH 43210.

In a previous study by the authors, on compositionally modulated Ti-Al thin films, evidence was presented for a series of structural transitions that occur as a function of the bilayer thickness of the multilayer. There is evidence which indicates the occurrence of a titanium based fcc structure in these films, at a bilayer thickness of 9.8 nm. Upon reducing the bilayer thickness further to 5.2 nm, both the Ti and Al layers adopt the hcp structure and are coherent with each other. This paper aims at examining the influence of crystallographic structure, bilayer thickness and growth morphology on the electrical resistivity and some of the mechanical properties of Ti-Al multilayers. The resistivity is essentially determined by interface scattering and to some extent by grain boundary scattering in the multilayers with small bilayer thicknesses. There is no evidence of the 'supermodulus effect' in these multilayers, but there is a significant increase in the multilayer hardness, as a function of the bilayer thickness. There appears to be a Hall-Petch type of relationship between the hardness and the bilayer thickness, indicative of the fact that the mode of plastic deformation in these multilayers appears to be influenced not only by the presence of interfaces, but by their nature too.
**Surface Science**
Room BR4 - Session SS-WeP

**Aspects of Surface Science**
Moderator: J. C. Hemminger, University of California, Irvine.

**SS-WeP1 Elevated Temperature Structures of the Ag on Si(111) System at Submonolayer Coverages**, G. Navrotsky and J. M. Blakely, Cornell University, MS&E, Ithaca, N.Y. 14853-1501.

Adsorption of Ag on the Si(111) surface has been studied by almost every available surface science technique over the last 27 years, yet its structure remains an enigma. We study here the effects of three experimental variables on the atomic positions in the Ag/Si(111)(\(\sqrt{3} \times \sqrt{3})R30^\circ\) Ag and (3 \(\times\) 1) Ag phases at temperatures up to 560°C. The average location of Ag atoms for each phase and the magnitude of the Si surface contraction are determined by combining the results of two complementary synchrotron x-ray techniques; the x-ray Standing Wave (XSW) technique and the polarisation dependent Surface Extended X-ray Absorption Fine Structure (SEXAFS) technique, supplemented by Rutherford Backscattering (RBS) and AES/LEED. We find that with sub-monolayer levels of carbon, the distance of the Ag (in the \(\sqrt{3}\) phase) along the surface normal is increased by about 0.36d_{Si}, and is more narrowly confined. Carbon apparently occupies sites normally taken by Ag in the 3 \(\times\) 1 phase. At C coverages of greater than 1/3 monolayer, all the 3 \(\times\) 1 adsorption sites are occupied and the adsorbed Ag in the \(\sqrt{3}\) phase moves outward from the surface to (0.68 \pm 0.016)d_{Si}, with a Ag-Si nearest neighbor bond length of (2.5 \pm 0.05) Å. We find there is no measurable effect from incident surface contamination by boron introduced during wet chemical sample preparation. At low coverages on clean surfaces, we infer that Ag in the 3 \(\times\) 1 phase adsorbs at a different site from that in the \(\sqrt{3}\) Ag: this makes Ag multi-positional when grown at temperatures below 400°C. Clean Si(111) binds the Ag 3\(\sqrt{3}\) phase at (0.33 \pm 0.020)d_{Si}, above the surface with a Ag-Si nearest neighbor bond length of (1.6 \pm 0.05) Å.

**SS-WeP2 Surface Morphology Induced by Ga and Sn Overlayers on Si(100) and Si(311) Surfaces**, L. Li, Y. Wei and I. S. T. Tsong, Arizona State University, Tempe, AZ 85287.

The evolution of surface reconstructions and facets on Si(100) and Si(311) as a function of Ga and Sn coverages \(2 \times\) monolayer (ML) is presented. The changes in surface morphology with annealing temperature were followed by scanning tunneling microscopy (STM). When the Ga coverage on Si(100) exceeded 1 ML, square depressions shaped like inverted pyramids were formed on the Si(100)-(4 \(\times\) 8)Ga surface. The inverted pyramid walls were (311) facets. Deposition of Sn on Si(100) at coverages exceeding 2 ML also produced (311) facets, but they formed facets of large orthogonally cut unit cells, giving rise to a large scale maze structure on the surface. As expected, deposition of Sn on Si(311) did not produce facets. Layer-by-layer of epitaxial (1 \(\times\) 1)Sn growth was observed on the Si(311) surface up to 2 ML. On the other hand, deposition of Ga on Si(311) produced highly ordered (211) and (611) facets at coverages above 1 ML.

**SS-WeP3 STM Studies of Benzene Adsorption on Both Clean and O-preadsorbed Ni(110)**, L. Ruus, J. Stensgaard, F. Besenbacher, and E. Lægsaard, Institute of Physics, Aarhus University, DK-8000, Aarhus C, Denmark.

Interaction between benzene and both clean and O-preadsorbed Ni(110) has been investigated using STM in ultra-high vacuum. The benzene adsorption on clean Ni(110) at room temperature and low temperature produces disordered and short-range ordered structure, respectively. Most of the molecules are observed as flat rings lying on the substrate and this result is consistent with other studies. The rest observed as tilted rings are ascribed to the benzene molecules losing one hydrogen. On O-preadsorbed Ni(110) the benzene adsorption results in a structural transformation from p(3 \(\times\) 1) to p(2 \(\times\) 1) by compressing the Ni-O- chains.

**SS-WeP4 Determination of the Reconstruction of Pt(100) Surface on Atomic Scale by Scanning Tunneling Microscope**, C. S. Chang, W. B. Su, and T. T. Tsong, Institute of Physics, Academia Sinica, Taiwan, ROC.

The rotation phases of the stable reconstruction for the clean Pt(100) surface have been identified on an atomic scale by the scanning tunneling microscope. The slightly rotated buckled hexagonal close-packed model is strongly substantiated by our STM results. Our data also provide information about the atom positions in the top hexagonal layer with respect to the square substrate via the determination of the registry. These data can be useful for the further theoretical study of the nature of the distortion waves originated from a high order commensurate hexagonal layer over a square substrate.

*Supported by the National Science Council of ROC under grant No. NSC3-0208-M001-038 and the special surface science project of Academia Sinica.

**SS-WeP5 Multiple Tunneling Gap Resistance Imaging for Surface Structure Determination with the STM**, J. C. Dunphy, P. Sau
t*, D. F. Ogletree, and M. Salmeron, Material Science Division, Lawrence Berkeley Laboratory, Berkeley, CA 94720 USA. *Contact author: 11 Rue de la Catalpa, Villeurbanne, France and Laboratoire de Chimie Théorique, CNRS, Lyon, France.

We have used Electron Scattering Quantum Chemistry (ESQC) STM theory for surface structure determination of adsorbates on metal surfaces from STM images. The theory indicates that the contrast in STM images is sensitive to changes of order 0.1 Å in surface, adsorbate, and tip geometry. Additionally, the decay of the tunneling current with tip-surface separation is found to be non-uniform across the unit cell. This causes the image contrast to change with the gap resistance. Therefore, we have implemented an image acquisition mode where images are acquired at up to eight different tunneling gap resistances. To overcome the experimental problems of thermal-drift, piezo-hysteresis, and unknown tip changes, each scan line of an image is acquired at a range of gap resistances before proceeding to the next scan line. We have applied this technique to determine the structure of adsorbate on metal systems (S and C on Mo and Cu) using Pt oxidation W, and clean W tips.

**SS-WeP6 Cu Deposition on Ru(0001) by Low Energy Li+ Ion Scattering**, Y. G. Shen, D. J. O'Connor, J. Yao, R. J. MacDonald, H. van Zeel, R. H. Roberts, and W. Wanders, 1Department of Physics, University of Newcastle, New South Wales 2308, Australia, 2Department of Physics, Eindhoven University of Technology, 5600 MB Eindhoven, The Netherlands, 3Institut für Physikalische und Theoretische Chemie der Universität Bonn, Wegelerstrasse 12, D-5300 Bonn 1, Germany.

The surface structure of Cu thin films on Ru(0001) has been studied by low energy Li+ ion scattering, AES and LEED. It was found that Cu formed pseudomorphic layers for the first two layers. Experimental results obtained by analyzing the incident angle dependence associated with shadowing of Ru by Cu confirmed that the first layer Cu atoms were in a normal Ru registry position, i.e. there was a continuation of the hcp stacking sequence. The second layer Cu atoms have also been determined to be at the fcc sites by analyzing the shadowing critical angles for the second layer Cu focusing onto the first layer Cu atoms in the [1010] azimuth. The structure of the Cu thin film up to 6 ML was also been probe with LEED measuring the incident and azimuthal angle dependences. The results were consistent with an epitaxial Cu(111) structure by comparing with the results of standard Cu(111) structure.

**SS-WeP7 Morphological Effects of Ultrathin Metal Films on Mo(111)**, Jie Guan, Robert A. Campbell and Theodore E. Moday, Department of Physics and Chemistry and Laboratory for Surface Modification, Rutgers, The State University of New Jersey, Piscataway, NJ 08855.

The interaction of Pd, Rh, Pt, Au, Ti, Mn, Fe, Co, Ni, Cu, Ag and Gd ultrathin films with a Mo(111) substrate has been investigated by means of Auger electron spectroscopy (AES), low energy electron diffraction (LEED) and temperature programmed desorption (TPD). The deposition of >1.0 ML of Pt, Au, Pd and Rh results in the Mo(111) surface reconstructing to form pyramidal facets with [211] orientation upon sample annealing. Average facet dimensions are
- 10Å. The remaining metals are not effective in inducing the morphological reconstruction. An analysis of the electronic, physical and chemical properties of the metal overlays strongly suggests that the electronic structure plays a dominant role in causing surface instability on Mo(111). Metals with a Pauling electronegativity of >2.0 are effective for inducing faceting. These results are consistent with the previous investigation of the W(111) surface.

*Supported by DOE, Office of Basic Energy Sciences.

**SS-WeP8** Highly Angular Resolved Patterns of Photo- and Auger Electron Diffraction from MgO(001), Y. Nishio, H. Ishii, M. Owarri and Y. Ichinose, Inst. of Ind. Sci., University of Tokyo, Roppongi 7-22-1, Minato-ku, Tokyo 106, Japan.

In an attempt to understand the nature of photoelectron and Auger electron diffraction from a single crystal, the two dimensional diffraction patterns of the emitted electrons from a cleavage MgO(001) were precisely measured with high angular resolutions. In the electron diffraction patterns with the angular resolution of 0.6° (an angular step size was 0.5°), several interesting features were clearly observed: bands, lines, bright spots and "dark" spots at the crossing points of those lines, and circular diffraction patterns. The band and line structures were closely related to the low-index crystallographic planes. However, to our knowledge, this is the first observation of these types of spots in photoelectron and Auger electron diffraction. With a highly angular resolved measurement, these fine structures described above were clearly observed.

**SS-WeP9** Structural Surface Phase Transitions During Segregation Competition, M. Mittlner* and S. Hoffmann, *The Centre for Metallurgical Process Engineering, The University of British Columbia, Vancouver, B.C. V6T 2Z4, "Max-Planck-Institut für Metallforschung, D-70174 Stuttgart, Germany.

The kinetics of surface segregation competition, when the segregants form different saturation structures, is investigated for three cases: Si-P at the (110) surface in Cu, Si-Si and Sn-Si at the (100) surface in FeSi. In these cases the weaker segregant (P, Si) first forms a c(2x2) structure before being replaced by the stronger segregant (Si, Sn). With a p(1x1) saturation structure. The surface phase transition, c(2x2) -> p(1x1), indicates complex site competition mechanisms; these are described with phenomenological models. Further, enrichment by bulk as well as pipe diffusion is taken into account. The changes in the enrichment rates associated with the surface phase transition can be explained within the proposed approach. The S segregation is markedly reduced by the phase transition since the contribution from pipe diffusion vanishes when the new surface phase nucleates at the pipes. For Sn, on the other hand, the surface phase transition provides a higher driving force for segregation by removing the retardation effect of Si, allowing the Sn segregation rate to increase.

The models are in good agreement with results from AES measurements.

**SS-WeP10** Direct Determination of Adsorbate Positions with Energy-Dependent and Angle-Resolved Photoelectron Diffraction, Huasheng Wu and G. J. Lapeyre, Physics Department, Montana State University, Bozeman, MT 51717.

While we successfully implemented a method to directly determine the adsorbate position using energy-dependent and angle-resolved photoelectron diffraction for Cu(111), we also found large artifacts in the atomic image. To reduce the artifacts, the angular behavior of the effective scattering factor was examined. The result suggests that the artifacts are from the large variation in factor's phase in the cases where the emission directions are far from the emitter-scatterer direction. As a consequence, we proposed a new transform scheme—the small cone method where the cone size is from the data. To calculate the image function intensity at a given position vector in real space, we use only those diffraction curves with their emission directions within a small cone, the axis of which is opposite to the given position vector. The small cone method dramatically reduces the artifacts and interesting results are obtained from all measured structures. In Si(111) 5x5-V3Si, the image reveals that the Si atoms occupy the T4 site. The image for the Si(111) 1x1-A structure shows that the As atoms substitute the top layer Si atoms. The result for the more complicated two-invariant-site structure Si(100) 2x1-A clearly shows an adatom dimer: the first case for a two site image. The atomic position values obtained with the small-cone method are in good agreement with other values where they exist.

Research and SRC supported by NSF.

**SS-WeP11** Magneto-Optical Kerr Effect of Exchange Coupling in Co/Cr Films on Pd(111) and Pd(100) Substrates, J. D. McKinley, C. Rau, Department of Physics and Rice Quantum Institute, Rice University, Houston, TX 77251-1892, USA.

Oscillatory exchange coupling between Fe or Co layers through variable thickness transition/noble metal spacer layers (Cr, Au, Cu) may induce spin polarization in the spacer layers. Previous studies only examine exchange coupling between in-plane magnetized layers. Films of Co on Pd(111) are known to be magnetized out of plane, in contrast to the in plane magnetization of Co films on Pd(100). Co/Cr films grown on Pd(111) constitute a model system for exchange coupling between out of plane magnetized layers through a spacer layer. Identical Co/Cr/Co films are grown on Pd(100) for comparison to in-plane exchange coupling. Results are reported for polar and longitudinal magneto-optical Kerr effect (MOKE) of Co/Cr/Co/Pd(111) and Co/Cr/Co/Pd(100) films.

*Supported by the National Science Foundation, the Welch Foundation, and the Texas Higher Education Coordinating Board.

**SS-WeP12** Thickness- and Temperature-Dependent Spin Reorientation Transition of Epitaxial Ni Films on Single Crystal Cu Surfaces, S. Z. Wu, G. J. Monkey, and R. F. Willis, Department of Physics, The Pennsylvania State University, University Park, PA 16802.

We have studied the ferromagnetism of Ni films deposited on Cu(100), Cu(110) and Cu(111) surfaces in situ using the surface magneto-optic Kerr effect (MOKE). For ultrathin Ni films grown on Cu(100) and Cu(110), the easy axis of magnetization lies in the plane of the film. Above a switching thickness, the easy axis of magnetization is perpendicular to the film. In addition, the magnetization increases dramatically by about an order of magnitude. This behavior is due to the conventional spin reorientation transition where ultrathin films exhibit perpendicular magnetization and thicker films exhibit in-plane magnetization above the switching thickness. We will report MOKE measurements of the magnetic anisotropy as functions of both film thickness and temperature. The switching occurs at 8 ML film thickness for Ni on Cu(100) and at 16 ML for Ni on Cu(110). For Ni on Cu(111) no switching is observed. These results are discussed in terms of competing interfacial, volume and surface anisotropy energy terms in a complex Hamiltonian.

**SS-WeP13** Experimental FermiSurface of Magnetic Multilayers, C. Casado, J. Avila, A. Vajravin, F. Sorra*, M. C. Asensio, LURE, bat. 209D, Université Paris-Sud, 91405 Orsay cedex, FRANCE, Fax(34)16446148. *Instituto de Ciencia de Materiales de Madrid, CSIC, c/Serrano 144, (28006) Madrid, SPAIN.

The behavior of the Fermi Surface (FS) has a decisive influence on the understanding of a wide variety of phenomena such as magnetic thin film multilayers and high Tc superconductors. Traditional methods of FS measurements demand strong restrictions on the crystalline quality; therefore they can be hardly suited by low-dimensional systems. In these cases, Angle-resolved Photoelectron Spectroscopy (ARUPS) is one of the most convenient tools to define FS contours. In ARUPS, the momentum parallel to the surface and the energy are rigorously conserved, such as the Fermi-level crossing by the bands of systems localized at a few layers can be easily sampled.

In this contribution, we present the evolution of the FS of the Co/Cu(100) interface as a function of Co coverage. The FS 2-D images show the Co transition from a thin two-dimensional metallic layer to a three-dimensional one with a FCC structure. The FS of sandwich structures as Cu/Co/Cu and Cu/Fe/Cu were also investigared as a function of the non-magnetic metal coverages. In order to perform the full mapping of the FS, 2r angular distributions of density of states at the Fermi level were recorded along all directions of the Brillouin Zone. Typical energy and full angular resolution were less than 100 meV and 1°, respectively.

In addition, ARUPS was combined with a synchrotron radiation source in order to obtain well defined cuts through the three-dimensi- onal FS at different k values. Polarized and unpolarized radiation with photon energy of 21.2, 33, 50 and 55 eV were employed to study the symmetry of the states sampled.

**SS-WeP14** Combined XPD, STM, and LEED Study of Iron Oxide Films on Pt(111), Y. J. Kim,* a Westphal,* c Westphal,* c Westphal,* c Westphal,* c Westphal,* c Westphal,* c Westphal,* c H. Xiao, a, Z. Wang, a, H. C. Galloway,* a, M. Salmeron,* a M. A. Van Hove,* a and C. S. Fadley,* a Lawrence Berkeley Laboratory, Berkeley, CA 94720, a Dept. of Chem., University of Hawaii, Honolulu, HI 96822, a Dept. of Phys., University-GHS-Essen, 45141 Essen, Ger-

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many, "Dept. of Phys., Univ. of California-Davis, Davis, CA 95616, Dept. of Phys., University of California-Berkeley, Berkeley, CA 94720. For the first time, three complementary surface structure probes, x-ray photoelectron diffraction (XPD), scanning tunneling microscopy (STM), and low-energy electron diffraction (LEED) have been combined in a single system. This system has been utilized to study the structure of iron oxide films grown on Pt(111). XPD is a near-surface structure probe of the short-range order in the first 3-5 shells of neighbors around each emitter. STM probes both short- and long-range order of the top-most surface layer(s). LEED is primarily sensitive to long-range two-dimensional order. XPD is atom-specific, while STM and LEED are not. For ~1 ML of FeO grown on Pt(111), XPD shows a topmost oxygen layer relaxed inward by ~0.6 Å compared to bulk FeO(111), while STM and LEED show an incommensurate oxide film with short- and long-range periodicities of ~3.1 Å and ~26.0 Å. The dependence on oxide layer thickness of both short- and long-range structures of FeO grown on Pt(111) will be discussed.


We have used Monte Carlo simulations on simple cubic Ising lattices with modified surface interaction parameters to model phenomenologically the temperature dependence of magnetic order near ferromagnetic and antiferromagnetic surfaces. These results are also discussed in connection with previous experiments suggesting surface-specific magnetic transition temperatures for semi-infinite systems, with special emphasis on spin-polarized photoelectron diffraction as a probe of short-range magnetic order. The calculated spin-spin correlation functions show no evidence of a high-temperature transition in short-range magnetic order. However, near the crossover from ferromagnetic to antiferromagnetic surface magnetic order transitions at T_Nsurf of 2.7 and 4.5 times T_Nbulk respectively.


We propose extending spin-polarized photoelectron diffraction (SPPD) [1,2] to direct holographic imaging of magnetic spin configurations. If a spin-polarized photoelectron is somehow emitted from a core level (e.g., as resolved in a final-state multiplet splitting), it can then undergo spin-dependent scattering from neighboring magnetic atoms to produce SSPD effects. Measuring such SSPD patterns over a large solid angle and/or over several energies should yield a data set of sufficient size to be analyzed using holographic Fourier transform inversion methods. We discuss the potential of this approach based on multiple-scattering simulations of SSPD patterns for small clusters of Mn^3+ ions and of Mn^2+ and O^2+ ions, with the latter simulating the MnO lattice. Two spin-sensitive holographic image functions Δ and Δ' are considered, with both suppressing the effects of non-magnetic scatterers, Δ' providing information on the location of magnetic scatterers, and Δ being sensitive to both the position and orientation of magnetic scatterers. Although some image artifacts and distortions are seen, multienergy holographic imaging appears capable of determining magnetic spin configurations near surfaces.


The Fe 3p angle-resolved soft x-ray photoemission spectra reveal a large magnetic dichroism for both linearly and circularly polarized light incidence which can be quantitatively explained within a single-particle picture. The obtained spin-orbit and exchange splittings (1.0-1.2 eV and 0.9-1.0 eV), via comparison between the theory and experiment, are 50-100% larger than those hitherto reported [1,2]. We also predict numerically three independent spin-polarizations, which are induced only by exchange, by spin-orbit and by their interference effects in the case where the magnetic moment vector, the electric field vector of p-polarized light and the wavevector of photoelectrons lie simultaneously in a crystallographic symmetry plan. Comparison will be made between theoretical predictions and an extensive magnetic x-ray circular dichroism photoemission data set from Fe/Cu(001). Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.


SS-WeP18 Investigation of the Curie Temperature of Iron Bilayer Components of Fe(110)/Ag(111) Multilayers Grown by Molecular Beam Epitaxy, D. F. Storm, D. F. C. Lens and W. Freeland, I. Gregorov, and J. C. Walker, Department of Physics and Astronomy, Johns Hopkins University, Baltimore, MD 21218.

The temperature dependence of the magnetization of ultrathin films, surfaces, and multilayers is well-documented for a variety of systems when the temperature T is well below the Curie point. Due to the metastability of these structures, however, almost no reliable measurements have been made at temperatures significantly above room temperature. Hence Tc for ultrathin films is still a matter of great uncertainty. We have used fast-passage zero-velocity Mossbauer spectroscopy in the transmission geometry to investigate the behavior of the magnetic ordering of ultrathin Fe bilayers in Fe(110)/Ag(111) multilayered systems, where x = 3, 4, or 5 monolayers (ML). The Ag thickness in each multilayer is 50 ML. We have observed reversible behavior, and our results are indicative of a discrete set of Curie temperatures for each sample and not a continuous distribution. The behavior we observe is consistent with computer simulations. We have made of a magnetically-ordered Fe film composed of multiple domains, each with its own Tc, critical exponent, and temperature-dependent isomer shift. The multilayers exhibit no measurable oxidation, even after exposure to temperatures as high as bulk Tc (1043 K), although there is evidence of an annealing transition, particularly during the initial heating ramp. However, Mossbauer spectroscopy before and after heating suggests that annealing improves the film quality rather than degrades it. We have used X-ray diffraction to confirm the structural integrity of the as-grown multilayers and after two temperature ramps.

SS-WeP19 Reaction of HN3 with Al(111): Growth of an AlN Film, J. N. Russell, Jr., Chemistry Division, Naval Research Laboratory, Washington, DC 20375-5342.

Wide energy bandgap III-V semiconductors, such as AlN, have received increased attention due to their optical and electronic properties. Hydrazic acid, HN3, is a potential precursor for low temperature nitridation of aluminum. The chemistry of HN3 on an Al(111) surface was investigated between 100 and 780 K under ultrahigh vacuum with temperature programmed desorption (TPD), low energy electron diffraction (LEED), Auger electron spectroscopy (AES) and electron energy loss spectroscopy (ELS). Molecular HN3 desorption was observed at 125 K from multilayer coverages of HN3. A broad N2 desorption feature was peaked at 295 K. H2 desorption had a peak maximum at 615 K, well above the temperature for H3 desorption from the hydrogen covered surface. These observations imply the presence of an NH surface reaction intermediate. The AlN Nguyen line shape was monitored after exposing the 300 K Al(111) surface to HN3 and annealing at 780 K. A series of exposure/anneal cycles revealed the sequential conversion of the metallic Al AES feature at 68eV into a feature at 58 eV which is representative of an AlN film. A new N2 desorption state at 580 K appeared as AlN surface became more nitrided. The nitridation mechanism is discussed.
SS-WeP20 Tailoring Sensing Film Selectivity and Sensitivity Using Principles from the Hard/Soft Lewis Acid-Base (HSAB) Paradigm, V. Thomas, J. W. Schwank, J. L. Grant*, University of Michigan, Department of Chemistry*, Department of Chemical Engineering, Ann Arbor, MI 48109-2136.

There are two primary factors restricting the further advancement of gas sensor technology today, sensing film selectivity and sensitivity. The sensing event is a manifestation of the interaction between the gaseous species present at the time and the sensing film. A fundamental and directive approach that addresses these issues is needed. In this research, HSAB principles are used to tailor platinum sensing films to selectively chemisorb aromatic cyclic hydrocarbons amongst other cyclic hydrocarbons. The HSAB Paradigm is a predictive approach that can be used to tailor sensing films for selective chemisorption. This research illustrates the effective use of these principles to understand adsorbate-adsorbent chemical interaction occurring during a sensing event.

A prototypical polycrystalline platinum sensing film, deposited on a 1 cm² piece of silicon wafer equipped with a tantalum heater on its backside, is used. Chemical species coadsorption and thermal treatment are used to tailor the sensing film for selective aromatic cyclic hydrocarbon chemisorption. The effect of prescribed modification protocols on the sensing film is studied by characterizing the sensing film, before and after modification, using temperature programmed desorption, work function measurements and both ex situ X-ray diffraction and X-ray photoelectron spectroscopy. Correlations between particle size and both particle and electron distributions are discussed from the perspective of the HSAB paradigm.

SS-WeP21 Differential-Conversion Temperature Programmed Desorption: A New Method for Obtaining Binuclear Surface Rate Constants, R. P. Southwell and E. G. Seebauer, Department of Chemical Engineering, University of Illinois, Urbana, IL 61801.

For binuclear surface reactions of the form A + B → AB, temperature programmed desorption (TPD) cannot be used efficiently to detect AB unless one of the reactants desorbs before AB. A modification of TPD (differential-conversion TPD, or DCTPD) that circumvents this problem is described in this paper. The reactant desorbing at high temperature is first adsorbed as in normal TPD. The surface is then exposed to a continuous flow of the other reactant, and the rate of product AB desorption is monitored at the same time. The flux is kept so low that the coverage of the reactant adsorbed initially scarcely changes. The rate constant is then determined using this measured coverage and that calculated for the impinging species from its previously-determined adsorption/desorption kinetics. Application of DCTPD is demonstrated in the particular case of HCl production from SiH₄ and TiCl₄ on TiSi₂.

SS-WeP22 Effect of Fluorination on the Reaction Kinetics of the Phenyl Coupling Reaction on Cu(111), Jerry M. Meyers, University of Illinois, Urbana, IL 61801 and Andrew J. Gelman, Carnegie Mellon University, Pittsburgh, PA 15213.

Understanding surface reaction kinetics and the transition states which determine these kinetics is of fundamental importance in numerous fields of science and technology. The amount of charge separation in the transition state can be determined by comparing activation energies of reactions of fluorinated and hydrogenated molecules. Fluorination has been shown to have a significant effect on the activation energies of reactions in which there is charge separation in the transition state. Fluorination of the iodobenzene at the ortho, meta, and para positions has been used to study transition states of the phenyl coupling reaction. On the Cu(111) surface, iodobenzene (C₆H₄I) dissociates into adsorbed phenyl groups (C₆H₅,ads) and adsorbed iodine (Iₐds) atoms at approximately 175 K. Upon heating, the phenyl groups couple to form biphenyl (C₆H₅-C₆H₅), which desorbs molecularly at 400 K. Temperature Programmed Desorption (TPD) has been used to determine the activation energies and Fourier-Transform Infrared Reflection-Absorption Spectroscopy (FT-TRAS) has been used to determine the geometry of the adsorbed molecules. The evidence shows that there is little charge separation in the transition state for phenyl coupling.

SS-WeP23 Thermal and Electron-Induced Chemistry of CF₃ on Ni(100), M. B. Jensen and P. A. Thiel, Department of Chemistry and Ames Laboratory, Iowa State University, Ames, IA 50011.

We have used thermal desorption spectroscopy (TDS) to study the reactions of CF₃ following adsorption at 100 K on Ni(100). At lowest exposures no molecular CF₃ or CF₂ species are observed, although atomic iodine is visible at 1100 K, indicating C-I bond cleavage. At higher exposures molecular CF₃ is observed at 200 K, along with etched Ni desorbing as NiF₂ in a peak at 900 K. This is followed by a small amount of CF₂, and finally multilayer CF₂, with increasing exposure. The interaction of low-energy electrons (E < 60 eV) with CF₃ multilayers shows very interesting chemistry. Electron-induced desorption of the parent molecule occurs with a cross-section of approximately 1 × 10⁻¹⁶ cm². Atomic fluorine is the major product detected in electrons of electron energy loss spectroscopy. This shows CF₃, CF₂, and CF₂ as well as carbon-carbon bond formation. Multiple-carbon species (as high as C₆F₆) desorb in a peak centered at 400 K. This process may be analogous to the electron-induced cross-linking of polytetrafluoroethylene.

SS-WeP24 Thermally Stimulated Desorption of Neutral CF₃ from Cu(111), K. H. Junker, Z.-J. Sun, T. B. Scoggin, and J. M. White, Department of Chemistry and Biochemistry, University of Texas at Austin, TX 78712.

The low temperature thermal chemistry of CF₃ on Ag(111) presents an example of competing catalytic reaction pathways: molecular desorption versus desorption of radical CF₂. Temperature programmed desorption and angle resolved temperature programmed desorption, complemented with Auger electron spectroscopy and low energy electron diffraction, were used to discern the mechanism of the CF₃ radical desorption channel. Isothermal scans, desorption yields, and angular distributions at several coverages are presented. Initially, CF₃ desorbs onto Ag(111) and breaks the C-I bond. The C-F₃ radical desorbs as a radical at high temperatures (~350 K) and saturates at a coverage of ~0.5 ML. As the coverage increases, CF₂ adsorbs molecularly toward the iodine surface. On the other hand, a low coverage of ~0.6–0.9 ML, a low temperature (120–160 K) radical desorption channel appears. Results indicate that low temperature C-F₃ thermal desorption occurs via direct dissociation of molecular CF₃, yielding radical CF₂ and adsorbed iodine.

SS-WeP25 Cyclopropyl on Cu(111): Area Selective Preparation and Vibrational Analysis Using a Dispersion-Compensation HREELS Spectrometer, R. Martel, A. P. Rochefort and P. H. McBreen, Département de chimie, Université Laval, Québec, G1K-7P4, Canada.

The preparation of a chemisorbed cyclopropyl species localized on a small area of the Cu(111) surface at 90K and the subsequent vibrational analysis has been carried out using a high throughput Dispersion-Compensation (DC) HREELS spectrometer. We describe the application of this novel approach to excite physisorbed alkanes on a small area of the surface using low energy electron impact produced by the DC-HREELS monochromator. The activation and the vibrational analysis were achieved in two different operating modes of the spectrometer and hence it is not a simple microscopic experiment. Energy dependent excitations of physisorbed cyclopropane showed a resonant enhancement of ~10 eV and yielded the cyclopropyl on Cu(111) with a cross section of $\sigma = 1 \times 10^{-15}$ cm². A careful energy and angular dependent vibrational analysis of the physisorbed cyclopropane at 90K did not indicate a vibrational enhancement due to the resonance state at 10 eV. The chemistry of the cyclopropyl species on Cu(111) has also been studied using vibrational measurements. A CH bond vibration of the metal at ~250 K has been observed just before a desorption at around 320K.


Studying the interaction of electrons with surfaces and adsorbates will offer better understanding of electron beam lithography, photochemistry on surfaces, and electron damage to adsorbates. Temperature programmed desorption spectroscopy (TPD) has been successfully used to probe the molecular fragmentation of methanol multilayers adsorbed on a Mo(110) single crystal. The low energy electrons (~55 eV) under ultrahigh vacuum (UHV) conditions. Electron induced dissociation (EID) of methanol in the multilayers followed by heating of the Mo(110) single crystal results in the formation of CH₃ species on the surface. These CH₃ species remain stable at 350 K and yield ethene which desorbs immediately upon formation. No evidence was found for the desorption of ethene, formaldehyde or dimethyl ether. Isotopically labeled experiments reveal that the carbon atoms of the ethene originate exclusively from the methanol multilayers upon electron irradiation. The cross section for EID of methanol in the multilayers is 4 × 10⁻¹⁵ cm² at an electron energy of ~55 eV. The above value for the cross section is higher than typical values.
measured previously for EID of monolayer adsorbates. The higher
cross section in the multilayers is attributed to the diminished role
of the substrate in quenching electronic excitations occurring in the
multilayers. This novel phenomenon offers the possibility of synthesizing
catalytically interesting surface intermediates in significant quantities.

SS-WeP27 Kinetics of Propene Desorption from Pd(111), N. A.
Thornburg, I. M. Abdeileihim, C. M. Gerth, E. A. Picciotto, D. P.
Land, Department of Chemistry, UC Davis, Davis, CA 95616.

A new surface science instrument which incorporates several tech-
niques, including Fourier transform infrared spectroscopy (FTIR), ther-
mal desorption spectroscopy (TDS), laser-induced thermal desorption
(LITD), low energy electron diffraction and Auger electron spectro-
cyroscopy has been built at UC Davis to study surface processes. In par-
cular, the interaction of propene with Pd(111) has been investigated to
model palladium catalysis. Thermal desorption spectroscopy was used
to determine exposure versus coverage dependence for propene
adsorbed to Pd(111). TDS showed that propene desorption was first order
decomposition at a near monolayer coverage, the peak intensity of the
desorption current being integral to the amount of propene adsorbed.

SS-WeP28 Adsorption and Film Growth of BTA on the Clean
and Oxygen Adsorbed Cu(110) Surfaces, K. Cho*, J. Kishimoto, T. Hashi-
zeume, H. W. Pickering and T. Sakurai, Institute for Materials Re-
search (IMR), Tohoku University, Sendai 980-77, Japan.
The benzotriazole (BTA) is widely used as a corrosion inhibitor of
the Cu surface. We have performed the detailed experiment of ad-
sorption, film growth and polymerization of the BTA on the clean,
O2 x 1-O and O2 x 2-O surfaces of Cu(110) in situ of the UHV FIB-
STM (Field-ion scanning tunneling microscopy).

The BTA molecules adsorbed on the clean Cu(110) surface are
mobile at the initial stage desorption, segregate to the steps, and form
the c(4 x 2) phase. Another type of the c(4 x 2) phase growth at
brightly imaged double line structures of BTA molecules are also
observed, which may be related to the polymerization of BTA at the
surface Cu defects. After completion of the first layer of the c(4 x
2) phase, the dual string shaped second layer forms.

On the oxygen adsorbed Cu(110) surfaces, BTA molecules bond to
the surface strongly from the initial stage, resulting in random ad-
sorption and clustering. By the further deposition of BTA, the surface
is covered by the amorphous-like three dimensional islands. The elec-
tronics structure of BTA studied by photoemission spectroscopy will
be discussed.

NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room BR4 – Session NS-WeP

Moderator: R. C. Colton, Naval Research Laboratory.

NS-WeP1 The Nanometer-Scale Structure of Isocyanide Functional-
ized Polyamine Thin Films, T. L. Porter, A. G. Sykes, Y. Shi,
G. Caple, Northern Arizona University, Flagstaff, AZ 86011.

The functionalization of conductive polymers allows one to tailor
the chemical and electrical properties of these synthetic metals for a
wide range of needs. Isocyanide functionalized polyamine may show
promise as a material that can bind to metal ions, with many possible
uses in a variety of important catalytic reactions. The electronic and
chemical properties of films of isocyanide functionalized polyamine
are determined in large part by the nanometer-scale film structures.
Other polyamine films have displayed a rich nanometer-domain scale
structure which inhibits the overall macroscopic conductivity of these
materials. We have used the techniques of contact and non-contact
scanning probe microscopy (SPM) to study the surface nanometer-
scase structure of new poly-isocyanaoamine thin films. We have also
investigated the structural changes that occur within these films upon
exposure to Ir" cations in solution. We will discuss these nanostruc-
tures and their changes as they apply to conduction, catalysis and
sensing applications for this material.

NS-WeP2 Structural Characteristics of a Uniquely Nanocrystalline

The red pigment N,N-Di(3,5-xyllyl)perylen-3,4,9,10-bis(dicarboximide)
is easily vacuum deposited and when subsequently vacuum annealed, a
1500Å film transforms to an extremely dense (-50 nm x 2 μm)
uniformly oriented with their long axes normal to the substrate. We have previously reported on growth kinetics of the whiskers by surface diffusion to screw dislocations. Here we present a structural characterization of the whiskers and a minority platelet phase, using TEM and selected area electron diffraction. The whiskers’ dimensions, size distributions, specific surface area and lattice structure are determined. Surprisingly, the lattice is determined to be hexagonal with a 14.5 Å lattice constant. The growth tips are in the [-211] direction, bounded by (111) and (001) side planes. The latter side plane surfaces are decorated with steps lying parallel to the long axis, and spaced ~4 Å apart. The pigment whiskers can be readily sputter or vapor coated over large areas to provide conformally coated nanostructured sized features with numerous novel chemical and physical properties.

NS-WeP3 Nucleation and Growth Kinetics of Monolayer Organic Films Studied with Graphite Etch Pits and STM, Victor J. Cee, David L. Patrick, and Thomas P. Beebe, Jr., Department of Chemistry, The University of Utah, Salt Lake City, UT 84112.

Etch pits on the (0001) surface of highly oriented pyrolytic graphite (HOPG) were used to study the nucleation kinetics of self-assembled monolayer organic films. High temperature oxidation of HOPG creates circular, monolayer-deep pits in the basal layer. These etch pits function as small “nanosamples” which are semi-independent from the surrounding terrace. Nucleation rates for self-assembly can be calculated for thin films of molecules on graphite by measuring the changing distribution of pits containing ordered molecules vs. those containing disordered molecules with time. By varying the pit size, the relative influence of pit edges and pit area on nucleation and growth can be determined. Self-assembled films of the liquid crystal 4′-(octyl-4-cyanobiphenyl) noluate and grow from open terrace sites rather than edge sites, in contrast to the growth modes exhibited by most adsorbate surface systems. The temperature dependence of nucleation rates can also be measured using etch pits, yielding values for nucleation activation energies.

NS-WeP4 Scanning Force Microscopy of Quasi-Single Crystalline Polyethylene and Nylon 6 Produced by Channel Dye Compression, C. Wang, R.-E. Riemann, M. Drechsler, H.-J. Cantow, Freiburg Materials Research Center, D-7800 Freiburg, Germany; Albert-Ludwigs University, Stefan-Meier-Str.21, 79104 Freiburg, Germany.

Oriented semicrystalline polymers form a class of advanced materials with high tensile strength in the direction of orientation. Such materials are available by different processes, which are accompanied by morphological changes. Thus, the study of relationships between structure, morphology and properties of such polymers is of significant interest. In our studies, PE and Nylon 6 were oriented in a channel direction from initially spherulitic structure into a quasi-single crystalline perfection. SFM in the lateral mode was employed to examine the morphology of the oriented polymers at three perpendicular surfaces, compression, constraint and flow direction. The images show that nano-fibrils are the main structural elements. The visualized long periods of alternating lamellar crystals in both amorphous layers are completely identical to those derived by SAXS. The molecules can be distinguished between the fibrils. The molecular resolution images of PE show interchain distances of 4.6 Å and 7.3 Å, which agrees fairly well with the d_{100} and d_{002} interchain distances in orthorhombic PE. With 8.7 Å the interchain distance by STM show a mean value of 9.42 Å of α-monocrystalline Nylon 6.

It is evident that, at the first time, SFM permits a direct visualization of nano-scale morphological elements in oriented polymer structures, PE and Nylon 6, which are unaccessible by electron microscopic methods. This leads to a deeper understanding of plastic deformation mechanisms.

NS-WeP5 Nanoclusters of Polyethylene at Au(111) Surfaces, K. Seshadri, S. Aire, Y. T. Tso* and D. L. Allara, Departments of Chemistry and Materials Science, Pennsylvania State University, University Park, PA 16802; and Academia Sinica, Taipei, Taiwan, ROC. Nanometer scale clusters of polyethylene have been formed on evaporated gold surfaces of high (111) texture by the decomposition of diazonethane. Atomic force microscopy shows that the clusters form in grain boundary regions. Characteristic crystal field splitting of vibrational modes as seen by infrared spectroscopy shows the polyethylene to be crystalline. Further interpretation of the infrared spectra shows that the polymer chains run parallel to the gold surface plane. Thermal annealing of the clusters improves the crystallinity and the melting point of the material appears similar to that of the bulk form. The polyethylene is highly hydrophobic and can serve to protect the grain boundary regions while chemical manipulation of the remaining gold surface takes place.

NS-WeP6 Amorphous and Ordered Layers of Oligothiophenes: A Combined AFM-, UPS-, HREELS-, and Conductivity Study, Oliver Böhm, Dietmar Oeter, Christiane Ziegler, and Wolfgang Göpel, Institute of Physical Chemistry, University of Tübingen, Morgenstelle 8, D-72076 Tübingen, FRG.

Amorphous layers of oligothiophenes with chain lengths between 4 and 8 monomer units (o4T-o8T) were deposited and characterized under UHV conditions. With increasing chain length the development of the π-band was studied by means of high resolution electron energy loss spectroscopy (HREELS). This makes it possible to determine the ideal band-gap (2.1 eV) of an infinitely long and defect-free polythiophene. Because of the resulting low conductivities, oligothiophenes (and polythiophene) have to be doped for future applications as semiconducting molecular material. A new method of coulombation of the dopant (here: FeC14) was developed in this context, which allows for an ultrapure UHV-preparation of doped thin films. The success of the p-type doping process of α5T, α6T, and α7T was checked by UV-photoemission spectroscopy (UPS) and HREELS. The DC-conductivity of α6T at room temperature could be increased upon doping from less than 10^-10 S/cm to 10^-7 S/cm (four-point measurement).

For α5T two coherent and intact monolayers could be prepared under UHV-conditions on SiO2 and SiO substrates. In these layers the molecules are oriented with their long axis perpendicular to the substrate surface.

NS-WeP7 Substrate Effects on Two-Dimensional Ordering of Self-Assembled Layers on TMDs by STM, David L. Sampson, and Bruce A. Parkinson, Department of Chemistry, Colorado State University, Fort Collins, CO 80523.

A large body of research has centered on the imaging of self-ordered films by scanning tunneling microscopy. The majority of this work has been done with graphite as a substrate. We have studied the ordering of various liquid crystals, alkanes and hydrogen bonded systems on a series of transition metal dichalcogenide (TMD) compounds. TMDs offer the cleanness of graphite but with lattice constants that vary from 3.16Å for MoS2, to 3.80Å for MoSe2. At high temperature, the interfacial order must adjust to maintain commensuration with the substrate while maximizing surface coverage. We have observed a wide variety of structures including several structures never before reported.

NS-WeP8 Scanning Force Microscopy of Stearic Acid LB Film Deposited on Mica, Jing Li, Wang Yan Shi, Quan Xi Erkang Wang*, The Laboratory of Electroanalytical Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, 130022, CHINA.

In this paper, the surface morphology of monolayer stearic acid LB film and their intrinsic defect structures as well as the modifications of these films induced by the image process were investigated using SFM(TMX 2000 mode SFM, Santa Clara, CA, USA). Especially the interaction between the tip and the stearic acid molecules was discussed in detail. Nanowriting on the LB film can be performed through controlling SFM force sensor. But the interaction between the tip and the surface molecules is so small that it is impossible to write some letters on the fresh mica and aging LB film/mica. Some letters can be only written on the fresh LB film/mica. If the LB film has stored for two and three months, many pinholes occurred on the LB film surface due to aging. When the nanowriting was performed, where did those molecules go? We first proposed a theoretical model of LB film to explain it was dependent on the experimental results. High resolution SFM images showed that stearic acid molecules freshly deposited on the mica was basically perpendicularly to the substrate surface to form order stains, which width corresponds to the molecular size in 0.4 nm. When a relative stronger force exerted on the molecules to scan along TV letters, the orientation of these molecules were really changed. After that, you could see the TV traces due to the different height of molecules when you enlarged the original range to scan. This is very significant for fabrication of biosensor and micro machining of single molecules.

NS-WeP9 Atomic Force Microscopy Investigation of the Structure and Orientation Ordering in C60 Single Crystal, L. Jiang, L. A. Nagahara, J. Li, N. Kino, T. Iyoda, K. Hashimoto, K. Kitazawa, and A. Fujishima, Department of Synthetic Chemistry and Department
of Industrial Chemistry, Faculty of Engineering, University of Tokyo, Tokyo, Bunkyo-ku, Tokyo 113, Japan.

C₆H₅ single crystals which are free from solvent contamination were grown via vapor phase transport. Large C₆H₅ crystals, typically 1.5 × 1 × 1 mm² in size, can be grown with this technique. The (0001) face of the crystal was imaged using atomic force microscopy (AFM) under ambient conditions. For a freshly made crystal, low resolution images revealed well-ordered faceted regions with multiply terraces extending for several hundred square nanometers. At higher resolution, individual C₆H₅ molecules packed in a hcp structure could be resolved with an average diameter of 11 Å. After the crystal was kept for about one month at room condition, the molecules reoriented into a mixed phase of both hcpf and fcc packing. Molecular images of the phase transformed surface was directly observed with the AFM.

NS-WeP10 Force Microscopic Studies of Langmuir-Blodgett Film of Complex TF₄C₆B₃, P-C. Zhang, C. Bai, D. Zhu, M. Han, Y. Xu, Y. Liu, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, CHINA.

By rection of tetraethylfulvenetetrafluoride(TTF) with C₆B₃, a donor-acceptor complex of TTF-C₆B₃ was synthesized. The preparation of LB films containing the complex was performed in a computer-controlled KSV instrument at room temperature. The multi-layer LB films were transferred to a slide substrate at a constant pressure with a suitable dipping speed. The topographic and the frictional properties of the LB films were studied with atomic force microscopy (AFM) and lateral force microscopy (LFM). LFM is a contact mode in air and ambient temperature. Furthermore, the LB films were imaged with the AFM in the tapping mode and magnetic force microscopy (MFM) in the lift mode. The MFM images of this TTF-C₆B₃ LB films show characteristic magnetic domains in many regions. These magnetic properties are consistent to the results of ferromagnetic measurements of the bulk. The first report on the magnetic behavior on the C₆H₅ complex LB film. Structural and magnetic features of the LB films are discussed.

NS-WeP11 Structure and Electrical Property of C₆H₅ Ag Ultrathin Particle-Polymer Thin Films, H. J. Gao, Z. Q. Xue, and Q. D. Wu, Dept. of Radio Electronics, Peking University, Beijing 100871, P.R. China, S. Pang, Beijing Laboratory of Vacuum Physics, Academia Sinica, Beijing 100080, P.R. China.

The electrical conductance of ICB fabricated C₆H₅-Ag-NM thin films is presented, wherein the NM is a newly synthesized conducting polymer which shows the very strong third non-linear optic properties. The thin films are characterized by transmission electron microscopy, STM/AFM and electronic spectroscopy. The TEM results indicate that the thin films contain ultrafine Ag and C₆H₅ particles which may play an important role in the conductance of the films. The thin films possess a relatively uniform surface structure and the periodic arrangement of the molecular structures at the surface of the films is also reported. Moreover the electronic spectroscopic results evidenced the formation of the charge transfer in the films. The thickness of the film is of ~100 nm. The possible mechanism of the electrical bistability of the films is discussed in the paper.

NS-WeP15 The AFM as a Tool for Metal Surface Modification, H. Göbel, P. v. Blankenagen and W. Schommers, Kernforschungszentrum Karlsruhe, Institut für Materialforschung I, Postfach 3640, 76021 Karlsruhe, Germany.

The AFM has been widely used as a tool for surface imaging and more recently also for surface machining [1]. We have applied an AFM for structurization of polycrystalline metal surfaces (Au, Cu). Programmed periodic structures with a depth of 10-30 nm were produced using a cantilever with a sufficiently high spring constant. By analyzing surface profiles as a function of time surface diffusion constants have been determined. The applicability of the AFM for nano-hardness measurements will be discussed.


It has been shown that one can modify surfaces in nanometer scale by using local probe methods like scanning tunneling microscopy (STM) and atomic force microscopy (AFM). Depositing gold from gold tips by applying voltage pulses was demonstrated and the mechanism of field evaporation was proposed by Mamin et al. [1]. Recently Pascual et al. [2] showed the neck formation during the process. We have investigated the field-induced surface modification under various conditions in UHV.

By applying negative pulses to the tip, a hole or a mound was produced on the sample surface. With a tunneling resistance of about 20GΩ where the separation between the tip and the surface is sufficiently large, a hole of typical size <15nm and depth <2.5nm was produced without the tip contact, indicating that the positive gold ions are transferred from the surface to the tip. On the other hand, as the resistance was decreased down to about 0.2GΩ, the mound was created.

According to the observed current during applying pulses, it seems that the tip contacted to the modified surface when the mounds appeared. The threshold voltage for the modification indicates the critical field of about 5V/nm, comparable to 4V/µm by Mamin et al. [1]. In the case of positive pulses, sizes of created mounds varied widely from several hundreds nm to tens nm.

We have concluded that the mechanism of gold surface modification is due to the field evaporation of positive Au ions and the formation of mound in the case of negative pulses is a result of necking due to the tip contact.


This paper describes micromachining of metal surfaces in air by a scanning probe microscope (SPM) which has a very sharp diamond tip mounted on the end of a cantilever beam. The sample surface was scanning-scratched by a diamond tip with a scan width of 2-3µm and a loading force of 500-10000nN, and topographies of the scratched area were observed by the same SPM with a loading force of less than 100nN.

In the case of Ni, flat square hollows of nanometer-scale depth were obtained. The scratched depth was increased with increasing loading force and repetition numbers of scratching. The effects of x direction scan rate, y direction feed, and scan width on the scratched depth were also revealed. Scratched surface of other materials was discussed comparing with the case of Ni.

Results obtained indicate that the nano-scale thickness material removal can be controlled and that the method presented here will be applied for micromachining of various materials.

NS-WeP15 Nanofabrication of Gold Surface with Scanning Tunneling Microscope by Combination of Mechanical and Electrical Method, Yoshihisa Ishikawa, Norihiro Umeda, and Atsuo Takayanagi, Faculty of Technology, Tokyo University of Agriculture and Technology, Tokyo, 184 JAPAN.

Scanning Tunneling Microscope (STM) is known as an instrument for probing an image of surface structure with atomic scale. Recently, it has proven to be a powerful tool for nanoscale fabrication technique. This paper describes a mechanical and electrical fabrication technique by using the STM in air environment. In mechanical technique, an indentation to gold surface with the STM tip is performed. It is shown that the nano-indentation was caused at some threshold value of indent displacement of the tip. In electrical technique, the electrical pulse voltage is applied to surface structure after indentation. In contrast of applying a pulse voltage only, the nanoscale pit with good shape is formed by the pulse voltage after indentation. Finally, the damage of tip apex by the fabrication is evaluated by the SEM micrograph.


To determine the possible mechanisms of ordering, nanostuctures prepared by the graphite surface treatment with oxidizing agents (vanadium (IV) and phosphorus (V) oxichlorides) were studied with STM and angle-resolved XPS. It was shown that two types of the ordered structures with the periods near 0.32 nm and 0.42 nm appeared after the treatment with vanadium oxichloride depending on the synthesis conditions. Their formation is explained in terms of direct oxidation
of the substrate or the reagent interaction with the oxygen-contained surface species. Mixing of vanadium oxichloride with oxygen stimulated an increase of vanadium concentration at the surface and resulted in appearance of the goethite-like layer over the vanadium-contained layer. The latter fact can be attributed to intercalation process. The graphite surface treatment with phosphorus oxichloride did not cause any ordering. However, after combined treatment with both the oxichlorides, the nanostructures were formed which could change easily the surface related pulse interaction. The secondary X-ray photoelectron spectroscopy (XPS) and tietochemicals of the nanostructures observed were proposed. Possibilities of application of the ordered nanostructures on graphite are discussed.

NS-WeP17 Nanostructure of Cleaved Surface of Bi Crystal, A. M. Troianovskii and V. S. Edelman*, Institute for High Pressure Physics, *Institute for Physical Problems, Academy of Science of Russia, Moscow.

We investigated by STM the cleaved surface of a Bi-crystal. The crystal was placed in a sample holder of a STM and was cleaved 'in situ' at a working pressure low than 10^-9 torr. The sample holder with a sample can be heated up to melting point of Bi. On the STM images of Bi surface with atomic resolution there were clearly seen atomically smooth terraces with dimensions of a few hundred nm and 'islands' with densities 1.5 x 10^14 cm^-2 and an height ~ 3.6 Å. We found that boundaries of such 'nanoislands' which coincides with the directions of the atomic rows are stable. But other boundaries can move randomly on 2 to 3 interatomic distance around equilibrium position. The amplitude of these movements increases with increasing of a temperature and can reach several nm at ~ 520 to 530 K. Moreover we observed the drifting of 'nanoislands' from one place of the terrace to the other on a distance ~ 50 Å without visible changing of their size. The velocity of drifting was ~ 0.04 Å/s. Formation of the observed 'nanoislands' can not be connected with impurities, because the density of impurities was 3 orders smaller than density of 'nanoislands'. We can suppose that they are formed due to dynamically deformations while cleaving the sample. After annealing of the sample at ~ 530K they were formed on the sample wide atomically smooth terraces without any small details.

NS-WeP18 Scanning Tunneling Microscopy of Transition-Metal Clusters on Nonmagnetic Supports, P. E. Quezenberry, T. A. Hann, and P. N. First, Georgia Institute of Technology, Atlanta, GA 30332-0430.

A great deal of previous research has addressed the question of how to grow uniform thin films of magnetic materials. Whether for fundamental studies or for technical applications, this goal is often quite difficult to achieve, since it involves details of both the energetics of the film/substrate couple and the growth kinetics. Thus the initial stages of transition-metal film growth often involve the formation of monolayer islands as well as clusters of the magnetic metal. This initial growth influences both the ultimate film morphology and the magnetic properties of the film. Recognizing that the clusters are often superparamagnetic, this presentation will describe a technique by which the STM might be used to determine magnetic properties of superparamagnetic clusters, up to the thin-film limit. The measurements rely upon spin-polarized tunneling from a ferromagnetic tip. Initial results will be presented from transition metal clusters grown by vapor deposition on Au(111).

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Porous silicon has recently become an important optoelectronic material. We have for the first time observed lateral directionality of anodization for Si during porous silicon formation. The starting material was (100) n/n' n 7/n silicon wafers doped with phosphorous. The dopant concentrations of top n-type silicon layer, n' middle layer, and n-type substrate were 10^17/cm^2, 10^15/cm^2, and 10^17/cm^2, respectively. The thicknesses of the top n-type silicon layer and the n' middle layer were 4μm and 20μm, respectively. After the top n-silicon window layer was etched in a 147 HNO3: 3 HF solution, anodization was carried out in a 20% HF solution. Only the n' middle layer was anodized to become porous silicon. The porous layer was removed in a 5% NaOH solution and the size of the resulting silicon cavity was approximately 150μm x 150μm. Even though the shapes of top silicon windows were different, the shapes of reacted porous silicon area under the top silicon layer were roughly similar and the sizes were almost identical. The ratio of the horizontally anodized distance for [100] direction to that for [110] direction was about 1.2. This means that the porous silicon layer is formed anisotropically. This result is contrary to the conventional concept that porous silicon formation is isotropic. A model explaining this behavior will be presented in this paper.

NS-WeP20 Doping and Photoeffects in Layered Semiconductors Studied with Scanning Tunneling Microscopy, D. R. Louder and B. A. Parkinson, Department of Chemistry, Colorado State University, Ft. Collins, CO 80523.

The scanning tunneling microscope (STM) has become a valuable tool in studying not only the topography of a sample, but also its electronic properties. Layered dichalcogenides, such as WSe2 and MoS2, exhibit spots on their surfaces when viewed with the STM. These features can have the appearance of hills or depressions on the sample surface depending on the sample bias. It was concluded that the spots were electronic effects because the number of spots seen in a certain area relates to the bulk doping density of the sample, and they are not seen in atomic force microscopes images of the same samples. Dopants on layers below the top layer can also be seen.

The STM has also been used to investigate materials using photoinduced tunneling current (PTC). This tunneling current is separated from the normal tunneling current by using a lock-in technique. PTC can be used to measure the effect of defects, such as steps, on charge carriers just as electron beam induced current (EBIC) is used in scanning electron microscopy. Islands of another semiconductor grown on top of such as SnO2, can be distinguished by the appearance of PTC due to SnO2's higher bandgap. This method surpasses EBIC in resolution, is nondestructive and can be done in atmosphere with no sample preparation.


Cross-sectional scanning force microscopy (X-SFM) and secondary ion mass spectrometry (SIMS) was used to find study Mg-Co/SiO2 and Co-Mg/SiO2 catalysts prepared from acetate and nitrate precursors on silica. It was shown that the SFM scanning altered markedly some surface structure of the catalyst prepared from nitride precursor. On the contrary, the scanning seemed to have effect on the surface structure of the catalysts prepared from the acetate precursors. The reasons for this fact and the possible structural changes occurring on the catalyst surface activated by the SFM scan will be discussed and compared with the XRD and XPS measurements. In addition, the cross sectional sample preparation method used will be discussed in this paper.

NS-WeP22 Observation of Atomic Ordering in GaInP, by Scanning Probe Microscopy, Y. Leng, Y. J. Huang, and C. C. Williams, Dept. of Physics, L. C. Su and G. B. Stringfellow, Dept. of Material Science, Univ. of Utah, Salt Lake City, Utah 84112 USA.

Under certain growth conditions, GaInP spontaneously orders on the InP III sublattice with gallium and indium atoms occupying alternating [111] planes. The atomic ordering of GaInP has been established and studied by a variety of methods, including transmission electron diffraction (TED), cathodoluminescence (CL), and photo luminescence (PL). In this work, Kelvin Probe Force Microscopy (KPFM) and Scanning Capacitance Microscopy (SCM) have been employed to image several GaInP samples previously characterized by the established techniques mentioned above. Images have been performed both on as grown GaInP surfaces and cleaved cross sectional surfaces. KPFM and SCM images of epitaxially grown GaInP layers on planar substrates (multiple samples) have been directly correlated with TED and CL in attempts to establish their sensitivity to ordering in GaInP. The results of our study clearly show that KPFM and SCM are capable of distinguishing between ordered and disordered regions in GaInP. Local electronic spectroscopies, such as capacitance vs. voltage, reveal that atomic ordering influence the measured surface potential, surface pinning, and band bending. The mechanism of image contrast by KPFM and SCM and the influence of ordering on electronic property of surface will be discussed.
PLASMA SCIENCE
Room BR4 – Session PS-WeP

Plasma Etching and Inertial Confinement Fusion Targets
Moderator: J. L. Cecchi, University of New Mexico.

PS-WeP1 Extraction of Oxygen from CO₂ Using Glow-Discharge and Permeation Techniques, D. Wu, R. A. Outlaw, R. L. Ash, Old Dominion University, Norfolk, Virginia 23508.

A promising method to extract oxygen from CO₂, which constitutes more than 95% of the Mars atmosphere, is by using glow-discharge dissociation of CO₂ combined with the permeation of the generated oxygen through a silver membrane. A quadrupole mass spectrometer on the downstream side of an ultra-high vacuum permeation system was used to measure the oxygen flux through a 0.34 mm thick silver separation membrane. On the upstream side of the membrane, with 400 V, 5 mA dc glow-discharge, it was found that as much as 70% of the CO₂ was dissociated to form CO and O (which recombined to form O₂). The downstream oxygen flux as a function of upstream CO₂ pressure, discharge current, discharge probe to membrane distance, and membrane temperature was also studied. It was found that a small amount of atomic oxygen, generated by the glow-discharge, bypassed the dissociative adsorption step and produced much higher flux through the membrane than with molecular oxygen.

An estimation of the atomic oxygen sticking coefficient is also presented and suggests that the value approaches 1 at a membrane temperature of 300 K. The total oxygen flux through the membrane with a 5 torr CO₂ glow-discharge and a membrane temperature of 450°C was found to be greater than 10⁻⁶ cm⁻² s⁻¹. Since the oxygen permeation flux varies inversely with membrane thickness, a membrane of 1 μm is being developed which will increase the flux by two orders of magnitude. This method can be used to continuously and efficiently supply oxygen to astronauts in a future manned mission to Mars.


In a UHV beam surface experiment (J. Vac. Sci. Techn. A, 12 1994 in press) we studied the interaction of XeF₆ molecules with the Si(100) surface in a quantitative manner. Although this system has been studied frequently in the past as a model for plasma etch interactions, most data are qualitative and a consistent model has not yet been developed. The question addressed in this study is the temperature dependence of the steady state reaction kinetics. The reaction of XeF₆ and the production of SiF₄ species were measured using XeF₆ fluxes from 0.1 to 5.0 ML/s and for sample temperatures ranging from 100 to 1000 K. At room temperature 20% of the XeF₆ reacts with the silicon; this increases to 50% at 900 K. At 300 K SiF₄ is the dominant etch product. For higher temperatures it is gradually replaced by SiF₄ up to 90% of the total production at 900 K. In the regime studied the reaction coefficient and product distribution are independent of XeF₆ flux, the reaction is limited by the XeF₆ flux. For temperatures below room temperature we also observe an increase in the XeF₆ reaction and corresponding SiF₄ production. This is due to an increasing XeF₆ precursor concentration. At 150 K this even leads to the formation of a SiF₄ overlayer which blocks the reaction. By TDS- analysis of the layer the adsorption energy of the precursor could be determined. Combining this with the high temperature data we were able to model the reaction kinetics of the Si/XeF₆ system as a function of temperature.

Experimental research and theoretical analysis of current-voltage characteristics and current-distance dependencies have shown that adsorbed films can be formed by the current of STM operated in ambient air. These current-induced adsorbed films can cover both W tip and Si surface under the tip causing the transitions of the conductivity process from direct tunneling to Schottky emission. It appears as extremely large gap distances and low potential barrier heights in STM experiments on HF-treated Si surfaces in air. To our mind hydrocarbons and water are most likely candidates to form these mobile (fluid-like) films.
PS-WeP3 Reactive Ion Etching-Induced Damages in GaAs/AlGaAs Quantum Wells Structures and Recovery by Rapid Thermal Annealing and Hydrogen Passivation, Byung-Su Yoo, Seong-Ju Park, and Kyung-Ho Park, Electronics and Telecommunications Research Institute, Yusong Post Office, P.O. Box 106, Taejon, 305-600, Korea.

In this study, we have investigated the spatial distribution of RIE-induced damages using low temperature photoluminescence (PL) of a GaAs/AlGaAs quantum well structure. We have also studied the recovery of defects by rapid thermal annealing (RTA) and passivation of defects in a hydrogen plasma. A typical sample structure consisted of four quantum wells of widths 2, 4, 6, and 10 nm with barriers of 30 nm thickness. A mixture of He and CCl₄F₄ gases were used in RIE experiments. The PL intensities from quantum wells in etched samples decreased uniformly across the depth of 150 nm without any line shift or broadening of PL spectra. This indicates that the damages are created in quantum wells by penetration of energetic ions into substrate during the reactive ion etching process. The results obtained under the various etching conditions suggest also that the physical sputtering induces more damages in the quantum wells and reduces selective etching ratio of GaAs over AlGaAs than the chemical etching process. Furthermore, the RIE-etched quantum well structures were annealed by rapid thermal annealing or passivated in a hydrogen plasma to understand the nature of defects. For both cases, the PL intensities from quantum wells situated at the deep region from surface were drastically recovered. These results suggest that the origins of defects created by energetic ions in the deep region are quite different from those in the near surface region. Moreover, the results indicate also that the damages in deep region are closely related to the vacancies of sublattice, particularly when light He ions are involved in the etching process.


The composition and energy distributions of both ions and neutrals from electron cyclotron resonance (ECR) plasmas were studied by a quadrupole mass spectrometer and cylindrical mirror energy analyzer. These characteristics were found to be very sensitive to the plasma parameters including the power input, magnetic field strength, and gas pressure for plasmas such as the hydrogen, nitrogen, ammonia, water, and methane systems. It was also found that the energy spread of ion beams extracted from such ECR plasmas could be controlled to about 2 eV. We believe that this is the lowest energy spread ever shown for ECR ion beams. With such a low energy spread, the measurements of energy distributions as a function of the plasma parameters were found to be informative about the plasma reactions. The implications of these and the beam energy control on ECR plasma induced etching/deposition will be discussed.

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An understanding of sheath evolution and ion current incident on the target in the presence of negative ions is important for the development of a tool for semiconductor thin film growth based on the plasma immersion ion implantation technique. This compact, inexpensive, high throughput implanter allows separate control of dose rate, ion energy, and substrate temperature for mitigating radiation effects. The time variation of sheath position around a high voltage (1-10 kV) pulse biased electrode in an oxygen plasma (~10¹⁴ cm⁻²) has been measured using Langmuir and capacitive probes. The pre-sheath existing prior to the pulsed bias was measured using an emissive probe. Sheath evolution and collected current has been measured for both positive and negative pulse biases, while varying the fraction of negative ions present in the oxygen plasma as measured with an ion mass spectrometer.

*Work supported by U.S. Department of Energy.

PS-WeP6 Power Versus Time in Pulsed, Parallel Plate rf Discharges, Lawrence J. Overzet, University of Texas at Dallas, Plasma Applications Laboratory, PO Box 830688, Richardson, TX 75083-0688.

While it is now easy to make measurements of the power delivered to a steady state discharge as a function of frequency; it is more difficult to measure the power delivered to the discharge as a function of both frequency and time during the transient turn on and turn off of the discharge. We have now measured the power transients to pulsed rf discharges in the Gaseous Electronics Conference Reference Reactor on a microsecond time scale. There are large swings in the power delivered to the cell as a function of time during the first few microseconds of the transient turn on and turn off. These power swings are in large part caused by the passive circuitry between the current-voltage probes and the electrode assembly of the cell and have been modeled using an equivalent network with reasonable accuracy. This circuitry can store rf energy and release it both to the plasma and to the rf generator at plasma turn over. The power delivered via a 500 mTorr argon discharge does not reach steady state until approximately 500 microseconds after the discharge turn on even though the initial power swings end within the first 10 microseconds. This second power variation appears to be related to a contraction of the electrode sheath as well as an electron density increase during this time. The method used in making time resolved rf power measurements will be presented along with results obtained from a 500 mTorr argon discharge.


Langmuir probes (LP) have been widely used in characterizing the properties of sputtering magnetron plasmas. Due to probe interference and coating problems, however, LP were limited to most of the discharges and not suitable for high power commercial magnetrons. On the other hand, optical emission spectroscopy (OES) which was also used in the study of magnetrons was more suitable for the monitor and control of the discharge in manufacturing applications. The major drawback of OES is the difficulty in extracting plasma parameters from the measured light intensities. We have developed an analytic model to describe light emission in a magnetron discharge. In this model, the amount of emitted light can be related to the maximum electron excitation cross section and the excitation energy of the argon species. By measuring the light emissions of argon neutrals at selected wavelengths, the electron temperature in a magnetron plasma can be determined. With this technique a real time electron temperature monitor for argon plasmas can be developed. The accuracy of this method has been verified using LP at low discharge power. The electron temperature was found to vary inversely with pressure and increased from 5 eV at 2 mTorr to as high as 15 eV at 0.1 mTorr in a commercial sputtering magnetron. The dependence of electron temperature with distance from cathode surface, and discharge power have also been determined using this diagnostics. The aspect of extending this technique to other species like argon ions and sputtered neutrals will be discussed.


Plasma excitation using microwave electron cyclotron resonance (MW ECR) has been used extensively not only in plasma ion assisted etching, but also in thin film chemical vapor deposition. The gas (both ion and neutral) temperatures (Tᵢ and Tₑ) are crucial parameters in determining etching and thin film quality.

In this paper, we evaluate a widely used technique for obtaining the Tₑ's; measuring the thermal Doppler broadening of emission lines. Here, we focus on Ar lines from MW ECR plasmas. In particular, we are interested in the most relevant factors in achieving the greatest accuracy and precision in the determination of Tₑ. We emphasize using the same technique for both ion and neutral species. We identify three important factors that contribute to obtaining reasonable temperatures: (1) knowing the correct instrument response or width (Γₑ) for each ion and neutral line; (2) understanding all the factors that lead to variations in the measured widths, i.e., the noise; and (3) knowing how the magnetic field affects the spectra (Zeeman processes). Ion temperatures cannot be directly observed from the widths (Γᵢ) unless Γᵢ at the same spectral position is known. Also, because Γᵢ is greater than the Doppler width (Γₑ), knowing Γᵢ accurately, i.e., reducing the noise ΔΓₑ, is critical. An analytical expression for ΔΓₑ is derived consisting of two sources, the signal counting and a constant instrument noise related to mechanical aspects of the spectrophotograph. These sources fully account for the size of ΔΓₑ. With proper signal averaging, we estimate that changes in Tₑ of ~10 K are detectable.

SiO2 to Si selectivity is an important control parameter in etching process. The goal of this study was to determine how changes in the chemistry at the wafer influence the SiO2 to Si selectivity in high density fluorocarbon plasmas. To do this, Si and SiO2etch rates in an ECR source were measured with an in-situ HeNe interferometer under a variety of etching conditions. The source gas chemistry for these experiments included CHF3 and C2H2F2. The microwave power, RF bias, pressure, and chemical composition were varied to determine a parameter space producing significant changes in the SiO2 to Si selectivity. These data were correlated with ion chemistry data taken over the same parameter space which was obtained using an in-line, differential pumped quadruple mass spectrometer. The spectrometer orifice was mounted directly beneath the substrate holder with a sample ring holder. The data to some of the ions and neutrals incident at the substrate surface into the spectrometer. The effects of chemical species present at the wafer surface on selectivity control in high density plasmas will be discussed.

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PS-WeP10 Determining the Neutral Radical Composition of Plasma Environments: Pulsed, Supersonic, Plasma Sampling Mass Spectrometry, Hongbin Zhu and Rik Blumenthal, Department of Chemistry, Auburn University, Auburn, AL 36849.

Over the past decade, the use of plasmas in materials processing has become almost commonplace. However, a chemical understanding of these plasma processes has lagged far behind their application in both thin film etching and deposition, as a result of the difficulty in obtaining direct experimental information from the energetic and hostile environment of the plasma. In this talk, a new mass spectrometric technique will be introduced which neutral radical species are extracted from the plasma by a supersonically expanding pulse of noble gas atoms. By limiting the interactions of the “plasma-born” reactive species, collisions with the noble gas atoms of the cooling pulse, the radical species are removed from the plasma with virtually no likelihood of reactions with walls or other chemical species. Experimental results relating to the deposition of diamond films and growth of the III-V Nitrides will be presented.

PS-WeP11 Fluid Simulations of Particle Contamination in Afterglow of Plasma Processes, Mary P. Garry and Thomas Peterson, Department of Chemical Engineering, Lisa Garrett and John F. O'Hanlon, Department of Electrical and Computer Engineering, University of Arizona, Tucson, AZ 85721.

Particles are problematic in plasma. Once generated, either by gas phase processes or by fragmentation from chamber surfaces, particles acquire a negative charge and are trapped at the plasma sheath interface. In the absence of the electric field at the sheath upon rf extinction, the trajectories of the particles depend on their mass and the local gas velocity and pressure. Using FIDAP, a commercially available finite element code, we have simulated fluid flow patterns and particle trajectories in the afterglow of a single wafer, parallel plate reactor. Particle trajectories are calculated using a Lagrangian technique and the viscous drag on the particle has been modified to account for noncontinuum effects at low pressures. Two dimensional axisymmetric simulations as a function of pressure, flow rate, initial particle location, and reactor configuration are presented.

PS-WeP12 Simulations of Damage Profiles Due to Reactive Ion Etching and Ion-Assisted Etching, R. J. Davis and P. Jha, Microelectronics Sciences Laboratory, Columbia Univ.(101) Schapiro Research Center, 350 W. 120th Street, New York, NY 10027.

Dry etching of surfaces with low-energy ions is important to the fabrication of electronic and optoelectronic devices, yet these techniques can cause severe damage to semiconductors. There still is debate regarding the separate contributions of ion channeling and damage to the anomalously deep damage which is observed after dry etching. Recently, we have theoretically modeled steady-state damage profiles, and shown that such profiles are quite sensitive to etch rate and etch yield effects, regardless of the contributions of channeling and diffusion. The parameter D/ε, where D is the damage diffusivity, the 1/e depth of inherent ion damage, and ε the etch rate is an important parameter in determining the relative contribution of diffusion and range (channeling) effects to the final damage profile. In this work, we simulate the evolution of dry etching damage profiles from inception to steady-state, and establish characteristic settling times for realistic dry etching situations. We also simulate profiles using Gaussian and more realistic damage distribution functions, and examine the validity of theoretical predictions and guidelines to more complex and realistic dry etching damage situations.


Development of a BiCMOS ASIC process encountered MOS device failure behaviors that were traced to a single processing plasma source. Despite the fact that the process employed relatively thick 500Å MOS gate oxides and was made in a manufacturing line that also produces CMOS product with gates below 200Å, significant passivation degradation effects were identified. Four distinct failure signatures were observed: shorted NMOS gates, leaky NMOS gates with functioning channels, parasitic depletion mode conduction at the ends of the enhancement NMOS device channel, and PMOS threshold shift as observed in a differential amplifier input offset. Test devices based on work originating at SEMATECH and specifically designed to monitor plasma damage through various gate antenna configurations were used to identify the cause of the failures as electrical damage induced during the backend of wafer processing. Although the methodology was successful, the experience points toward significant improvements that can be made and will be employed in future development and characterization efforts on a routine basis. Sequential characterization of device performance from first metal out quickly identified the FECDV tool used for oxidation as the primary contributor to the plasma damage. Subsequent process flow changes did eliminate this source of plasma damage, and continuing efforts center on correcting the basic tool problem.

PS-WeP14 Modeling of Wafer Charging and Damage in High Plasma Density Etching Tools1, Michael Grapperhaus, and Mark J. Kushner, University of Illinois, Dept. Electrical and Computer Engineering, Urbana, IL 61801.

Wafer charging and nonuniform fluxes of ions to the wafer have been identified as damage mechanisms to thin oxide layers in conventional RIE and magnetically enhanced RIE etching systems. The propensity for ion impact damage in high plasma density tools [inductively coupled plasmas (ICP), electron cyclotron resonance (ECR)] is less than in RIE tools due to the lower substrate bias. However, the higher plasma densities in ICP tools and the high density of their ion fluxes still leave open questions regarding damage caused by charging and oxide tunneling currents. We have developed a computer model of ICP reactors, oxide charging and current transport through simple MOS devices with the goal of assessing plasma induced damage. The integrated tunneling current through oxide layers during etching has been correlated with low breakdown voltages, and is an unmeasured source of plasma damage. The ICP reactor model is a 2-dimensional hybrid model which produces spatially resolved electron and ion fluxes. A second integrated microscopic model examines the charging of thin oxide layers while accounting for device conduction and tunneling currents in MOS structures. We will discuss the differences in oxide charging and tunneling currents caused by nonuniform ion fluxes in typical ICP reactors.

1Work supported by NSF, SRC, Sandia/Sematech and Univ. of Wisconsin ERC for Plasma Aided Manufacturing.


The main objective of these studies is to determine the relationship between plasma parameters and charging damage to polysilicon-gate metal-oxide-semiconductor (MOS) capacitor test structures upon exposure to electron-cyclotron-resonance (ECR) plasmas. The test structures are designed as “antennas” with a gate-oxide thickness of 10 nm and antenna ratios of from 16 to 11,000. After plasma exposure,
the damage is quantified using a number of damage-sensitive electrical measurements, including ramp-voltage oxide breakdown. Langmuir and emissive probe measurements of O₂ plasmas in the ECR system have been made as a function of microwave input power, neutral pressure, magnetic field configuration, and radial position above the wafer stage. Contour plots of the plasma parameters (plasma potential, electron temperature, plasma density, etc.) have been generated from this data. We have concentrated on determining the effects of plasma potential uniformity and exposure time on the ramp-voltage oxide breakdown of the MOS capacitors. Results show that early breakdown of the antenna structures at below 5 volts increases as the antenna area increases, indicating that charging is the primary damage mechanism. Experiments in plasma with a uniform plasma potential across the wafer revealed that processing time did not have a significant effect on the early breakdown distributions, indicating that damage induced during either plasma ignition or turn-off may be the dominant damage mechanism. Results of the role of plasma-induced damage in non-uniform plasmas will be presented.

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PS-WeP16 Impact of High-Z Limiters on Ion Fluxes in the Plasma Edge of the Textor Tokamak, M. Rothb, B. Emmoth, L. Köven, V. Philippa, A. Pospieszczka, J. von Seggern, T. Tanahe, Y. Udeta, and P. Weinhold, Physics Department—Frescati, Royal Institute of Technology, Association EURATOM—NFR, 24, S-104 05 Stockholm, Sweden, Institute of Plasma Physics, KFA, D-52425 Jülich, Germany, Department of Nuclear Engineering, Osaka University, Japan.

Heavy metals of high melting point are considered as plasma facing materials in controlled fusion devices. Recently, extensive tests of molybdenum and tungsten limiters were performed in the TEXTOR tokamak. It was important to recognize the erosion of the limiters and its impact on the transfer of heavy metals to the plasma and scrape-off layer (SOL). Among many diagnostic techniques active during the experiment, the surface collector probe measurements were also done in order to trace ionic fluxes in the SOL. Probe exposures were made under different conditions (density, position of limiters, edge cooling) during ohmic and NBI heated discharges. The species deposited on the probe surfaces were qualitatively and quantitatively determined by means of high energy ion beam techniques. The operation with the high-Z limiters did not increase the fluxes of other impurity atoms (O, Si, N) when compared to the operation without those limiters in the boronized or siliconized TEXTOR. The results of the collector probe measurements for the fluxes in the SOL are compared with the spectroscopic observations of impurity atoms in the plasma.

PS-WeP17 Mass Spectrometric Analysis of the Gas Phase Coating Environment in Plasma Polymerization, S. Lotts and R. Brusasco, Lawrence Livermore National Laboratory, Livermore, CA 94550.

Plasma polymerization is used to prepare highly crosslinked organic polymer layers on shells used as targets in Inertial Confinement Fusion experiments. Optimization of the plasma polymerization process is desirable to make the coating process more reliable and to produce smoother coatings at higher deposition rates. To begin investigating the gas phase and surface chemical reactions and their kinetics, it is necessary to identify the gas phase species impinging on the substrate. A novel, close-coupled coating chamber combining a quadrupole mass spectrometer with a helical resonator that allows one to identify the gas phase species formed in the plasma has been built. The mass spectrometer shows that gas phase oligomerization occurs to create organic species of molecular weight greater than the C₆H₆ starting material. The co-axial design of the reaction chamber allows one to probe the ions in the gas phase as well as neutral species. The gas phase at the point of contact with the substrate is rich in hydrogen ions but ionic hydrocarbon species concentrations are very low. It is possible to alter the distribution of gas phase species present by blending precursor gases. Work to correlate the effects of changes in deposition conditions on coating properties is ongoing and will be discussed in terms of residual coating stress and surface roughness.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.


Polyvinyl alcohol (PVA) is used as a gas-barrier to hold deuterium and diagnostic gases (such as argon) inside the hollow, spherical shells used as laser fusion targets. A PVA layer several microns thick will, at room temperature, leak on the order of 50 atmospheres of deuterium in the shells for several hours, a time sufficient for laser experiments. However, this thickness of PVA permanently entraps the argon. Normally, the argon is entrapped when the PVA coating is applied. We have developed a procedure whereby argon is permeated through the PVA layer of completed shells to fill them to specified fill pressures (typically 0.1 to 1.0 atm). The shells are placed in a sealed fill tube pressurized with the desired amount of argon. The tube is then heated to approximately 100°C and kept at temperature until the argon pressure in the shell reaches equilibrium. An X-Ray Fluorescence (XRFl) system is used to measure the argon content of the shells so that permeation coefficients can be calculated. We have found that half-millimeter thick coated shells with 3 µm of PVA can be filled with argon in approximately ten days. This procedure offers the prospect of filling any shell thin in excess of 1 atmosphere, uniform shell-to-shell argon content, removal of residual gases (i.e., air and water vapor) from the shells, and calibration standards for XRF measurement of shell argon content.

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PS-WeP19 Preparation of Germanium Doped Plasma Polymerized Coatings as ICF Target Ablators, R. Brusasco, M. Saculla and R. Cook, Lawrence Livermore National Laboratory, Livermore, CA 94550.

Targets for Inertial Confinement Fusion (ICF) experiments at Lawrence Livermore National Laboratory (LLNL) utilize an organic (CH) ablator coating prepared by plasma polymerization. Some of these experiments require a mid-Z dopant in the ablator coating to modify the opacity of the shell. Bromine had been used in the past, but the surface finish of brominated CH degrades rapidly with time upon exposure to air. This paper describes the preparation and characterization of plasma polymerized layers containing germanium as a dopant at concentrations of between 0.9-1.8 atom percent. The coatings are stable in air and have an rms surface roughness of 30 to 75 nm which is similar to that obtained with undoped coatings. High levels of dopant result in cracking of the inner mandrel during target assembly. Possible explanations for the observed cracking behavior will be discussed.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.


Advanced inertial confinement fusion targets require uniform condensed cryogenic hydrogen fuel layers with inner surface smoothness as low as 1000 Å rms for some designs. For a liquid layer stabilized against gravity, surface tension would provide this surface smoothness. Liquid layers less than 10 µm thick have been created by a thermal gradient layering technique in glass shells with a few hundred micron diameter. Advanced targets require layering of 1-2 mm thick with fuel layers up to 100 µm thick. Current experiments are being conducted in this size range. Liquid oscillations as well as stable liquid surface configurations have been observed. We will report on our progress towards creating stable uniform liquid layers.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48 and by W. J. Schafer Associates under Contract DE-AC03-91SF18601.


Foam shell targets are being considered as a liquid-layered cryogenic target for the Omega Upgrade and the National Ignition Facility. For
Omega Upgrade the target consists of a 1 mm diameter and 100 mm thick spherical, 50 mg/cm² foam shell surrounding a central void. The foam is overcoated with a full density polymer fuel barrier which must be topologically smooth. The foam will be slightly overlaid with liquid D₂ or DT, the overlaid excess being symmetrically distributed on the inside of the shell using thermal gradient techniques. The technology for producing this style of foam shell involves microencapsulation techniques that have been developed at Osaka University's Institute for Laser Engineering. Our goal has been to adapt this technology to meet US ICF objectives. We will report on our progress in accomplishing this goal.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

PS-WeP2 Determination of the Wall Thickness and Uniformity of Inertial-Fusion Capsules Using the Self-Interference Fringes Produced with Narrow-Bandwidth Illumination, Mark D. Wittram, Hye-goon Kim, and Albert S. Chow, Laboratory for Laser Energetics, University of Rochester, Rochester, NY 14623-1299.

Direct-drive inertial-fusion capsules must possess a high degree of spherical symmetry including concentricity of the inner and outer wall surfaces. When viewed with a compound microscope using transmitted narrow-bandwidth light, such as that produced with a thin-film interference filter, self-interference fringes appear within the image of a capsule that are localized to its equatorial plane. The fringes are distinct and concentric when the capsule is uniform, whereas faint, distorted, or discontinuous rings indicate nonuniformity. We have formulated a model that predicts this self-interference pattern as a function of capsule parameters, and it agrees well with actual measurements from the image of a well-characterized capsule. The wavefront directly transmitted through the capsule and wavefronts that are multiply reflected within its walls interfere to produce these rings. These wavefronts are constructed by tracing a single paraxial ray through the capsule and including reflections and third-order spherical aberration. The diameters of the rings are predicted from the intersection of the wavefronts. Changes in wall thickness as small as 10 nm affect the self-interference rings' diameters by several micrometers, whereas the ring diameters are independent of both the internal gas-fill pressure and the angle that rays impinge on the capsule.

TF-WeP2 Protecting Silver Polymer Mirrors with Oxide Coatings for Solar Applications, C. E. Kennedy, National Renewable Energy Laboratory, Golden, CO 80401.

Large mirrors are used to concentrate sunlight for renewable power generation for the solar thermal technologies. The reflector materials must be low in cost and maintain high specular reflectance for extended lifetimes under severe outdoor environments. Compatibility with mass production techniques is necessary to meet the low-cost requirements. Currently the best candidate materials for solar mirrors are silver-coated low-iron glass and silvered polymer films. Polymer reflectors are lighter in weight, offer greater system design flexibility, and have the potential for lower cost than glass mirrors. A promising low-cost construction uses an inexpensive polymer substrate, a silver reflective layer, and an optically clear protective top coat. Test samples were fabricated by magnetron sputtering. SiO₂, Al₂O₃, and Si₃N₄ were used as the protective layer. The samples were characterized by optical and surface analysis. The optical durability of the reflector materials was evaluated by accelerated weathering in controlled laboratory environments and real time exposure at outdoor test sites.

TF-WeP3 UHV E-Beam Deposition of Pt and Ti on Single Crystal Si and SiO₂ Substrates for the Preparation of PtSi, TiSi₂ Thin Films by Laser Ablation, M. Duben, T. Wilber, L. Caza, R. Lareau, K. J. Jones, Army Research Lab, EPSD, Fort Monmouth, NJ 07703.

The use of PZT, Pb(Zr, Ti)O₃ films in electronic applications requires the preparation of PZT on a conducting substrate. In this experiment, the Pt or/and Ti (100-200 nm) were deposited at a rate of 1.2 Å and at different temperatures (100-560°C) on Si (100), (111) and silicon oxide (50-200 nm) surfaces using UHV (10⁻⁸-10⁻¹⁰ Torr) E-Beam evaporation. The films were deposited by laser ablation onto the metallized substrate at a temperature of approximately 600°C under 100 mTorr oxygen atmosphere. The substrate-metal interface reactions, grain size, orientation and structure of Pt, Ti, Pt/Ti, and the PZT films grown on them, with and without oxide on silicon substrates, were analyzed by X-ray, STM, SIMS and AES. The PZT films were analyzed electrically for resistivity and hysteresis.

THIN FILM

Room BR4 – Session TF-WeP

Aspects of Thin Films
Moderator: F. Sequeda, Conner Peripherals.

TF-WeP1 A Novel Approach to the Calculation of ITO Films’ Figures of Merit, S. A. Klaubert, A. K. Kulkarni, Michigan Technological University, Houghton, MI 49931.

As flat panel display devices gain in popularity, the need for highly conductive transparent films will increase. Indium tin oxide (ITO) is just such a film and it presently exhibits some of the most impressive qualities with regards to electrical conductivity and optical transparency. These qualities have competing mechanisms which give rise to the need for a compromise between the transmission and conductivity. To allow films with various properties to be compared, several figures of merit have been developed to relate the transmission (T) and electrical sheet resistivity (Rₐ) of ITO thin films. Of these figures of merit, two are based on ratios of the transmission and the sheet resistivity and these are thickness dependent quantities. The other two are based on the product of the parameters and yield thickness independent formulas.

In order to optimize the conductivity and the transmission as a function of doping, it is necessary to determine the figure of merit as a function of the carrier concentration. We have compared different figures of merit as a function of carrier concentration on the basis of theoretical concepts instead of experimental values of conductivity and transmission. Estimates of the refractive index (n) and the extinction coefficient (k) are made and used to predict the optical transmission as a function of the carrier concentration in the films. These values are then used in four separate figures of merit Fₘ, Fₚ, Fₛ and Fₑ. From these plots it appears there is an optimum value of doping in ITO thin films.


Strontium sulfide doped with europium and samarium is a stimulable phosphor that is being investigated as the active layer of a photonic data storage disk. Lithium and fluroine are added to the Sr₅Eu:Sm in order to fully activate the photonic properties of the material. Thin films are grown on glass substrates above 650 K by ion-assisted e-beam evaporation and RF magnetron sputtering. The resulting films may have a preferred orientation which is dependent on substrate temperature. To obtain high performance films, care must be taken to assure sufficient crystallite size, minimize structural defects within crystal grains, and maintain a uniform chemistry throughout the thickness of the films. We present results on film performance in which we show the relationship to these latter properties employing X-ray diffraction (XRD), transmission electron microscopy (TEM), and secondary ion mass spectroscopy (SIMS). Structural defects within grains have a major impact on film performance. The relative influence of the difference in the coefficients of the thermal expansion and deposition-induced defects will be discussed.

TF-WeP6 The Structure and Magneto-Optical Properties of Ta/Fe and Ta/Co Bilayers, E. E. Shaltilin, Y. V. Kozlovsky, University of Daugavpils, Daugavpils, Latvia, LV-5407.
Magnetic films have received much attention in recent years. The purpose of the present study is to investigate the influence of non-magnetic layer between substrate and magnetic film on crystallographic structure and magneto-optical properties of the film.

The films were prepared by sputtering in Penning discharge at Xe pressure of 7×10⁻² Torr and substrate temperature near to room temperature. Before each run the chamber was evacuated to 5×10⁻¹ Torr. 40 nm Ta films were deposited on glass substrates. The thickness of Fe and Co films was varied between 0.5 and 200 nm. The X-ray measurements were performed on a X-ray diffractometer. The magnetic measurements were performed on the magneto-optical micro-magnetometer and the spectral technique by means of transverse Kerr effect (TKE).

40 nm Ta films could be characterized as a mixture of α and β-Ta crystalline phases and were textured. Fe and Co films had preferred orientation of {110} and {001} lattice planes respectively parallel to the surface. The degree of crystallographic texture of Fe and Co films on textured Ta layers was much higher than in the case of these films on glass substrates.

It is described how the magnetic films with the thickness varying between 2 and 20 nm have an in-plane easy axis. The magnitude of coercivity Hc decreases with reducing magnetic film thickness. The coercivity of the samples with Ta layer was 2-3 times more than Hc of ones without non-magnetic layer. The investigations of dispersion dependencies of TKR were performed over the spectral range 2-4.5 eV. TKR was absent for the samples with magnetic film thickness smaller than 1 nm.

**TF-WeP7** Surface Compositions of Chemically Treated CdTe Thin Films for Photovoltaic Applications: an XPS and AES Study, D. W. Niles, X. Li, P. Sheldon, National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden, CO 80401.

We used X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy to investigate chemical treatments used to form back contacts in CdTe-based photovoltaic devices. We produce photovoltaic grade thin film CdTe by closed space sublimation (CSS). Because CSS CdTe is highly resistive and not amenable to low resistance back contact formation, one must treat the CdTe to form a highly p-type conductive layer on which to form the back contact. We etch our CSS CdTe in H3PO4 + HNO3 to form Te-rich, p-type CdTe surfaces, and show that chemically etching the CdTe thin films leaves a highly conductive, pure Te layer. Auger depth profiles show a Te-rich region extending up to a depth of 1000 nm for CSS grown CdTe. In comparison, the same etch applied to single crystal CdTe leaves a 50 nm Te layer, implying enhanced etching along grain boundaries for CSS CdTe. The Te layers oxidize in air at a rate of 0.3 at. % per minute, and saturate at TeO2/(Te + TeO2) = 60% after an overnight exposure. We will discuss compositional analyses of CdTe after H3PO4 + HNO3 etches and KOH treatments, the implications in forming shunt paths that inhibit efficient photovoltaic performance, and the formation of low resistance back contacts to CdTe.

**TF-WeP9** Low Temperature Formation of Textured ZnO Transparent Electrodes by Magnetron Sputtering, T. Minami, H. Sonohara, S. Takata and I. Fukuda, Kanazawa Institute of Technology, 7-1-1 Ogiharaoka, Nonoiichi, Ishikawa 921, Japan.

Milky transparent conducting ZnO films with a textured surface have attracted attention as low-cost transparent electrodes for thin-film solar cells such as a-Si and CuInSe₂. Although these applications require film deposition at temperatures below 250 °C, large-area film coatings produced by magnetron sputtering normally involve high-temperature treatments to obtain a low resistivity. In this paper, we demonstrate a dc magnetron sputtering technique that results in highly conductive ZnO films with a textured surface prepared on substrates at a temperature of 200 °C. Large-area transparent conducting impurity-doped ZnO films with a texture formed at a sputter pressure of 60 to 80 Pa and temperature above 200 °C were prepared by conventional dc magnetron sputtering using sintered ZnO powder doped with impurity as the target. A resistivity of 4.6×10⁻⁴ Ω·cm and a total transmittance of 84% were obtained for textured ZnO:Al films prepared at a temperature of 200 °C. Textured ZnO:Al transparent electrodes with a sheet resistance of 40 Ω/sq and a haze ratio of 54% at a wavelength of 550 nm were formed with large-area 1.5-mm-thick ZnO:Al films.

**TF-WeP10** Vacuum Deposition Parameters for Thin Film Shape Memory Alloys, A. Peter Jardine and Rand Dunnenberg, Dept. of Materials Science and Engineering, SUNY at Stony Brook, Stony Brook, NY 11794-2275.

Thin film Shape Memory Alloys are an interesting new material, both from fundamental physical properties and from technological interest. When in their highly twinned, low temperature martensitic state, these alloys are ductile due to the easy motion of twin boundaries. The high temperature austenitic state does not possess these properties and so the internal stress of the thin film changes dramatically with state. Technological interest is due to their comparatively high energies for actuation, making them interesting as linear drive elements for Micro-Electro-Mechanical Machines, for example.

There has yet to be a definitive study on deposition parameters for these alloys. In particular, there is a need for better determination of vacuum conditions/processing parameters for this thin film SME materials. In particular, the effect of base pressure, gas cleanliness, target to sample distance, substrate material and post-processing of the material all affect the transformation properties of the alloy. Using a UHV deposition chamber with in-situ heating and two DC sputter deposition sources, thin film TiNi and CuAlNi were deposited onto Si and ceramic substrates. In this paper, we correlate the transformation properties using Resistance-temperature profiles and the microstructural properties of thin film TiNi using XRD and SEM with these mentioned deposition parameters.


Low temperature processing is essential for the fabrication of semiconductor devices of decreasing dimensions and increasing complexity. The energy necessary for the oxidation reaction to proceed can be supplied without heating the substrate by using low energy ions. A two-step process using deposition from low-energy ion beams produces dense SiO2 films of any desired thickness. The experiments are performed at the unique dual-source, fast-switching, mass-selected low-energy ion beam system at the University of Houston. First, ion beam oxidation of the Si substrate is carried out, forming the SiO2/Si interface below the original Si surface. This is followed by deposition of silicon dioxide using alternating Si⁺ and O⁺ ion beams. The films are deposited at temperatures between 20 and 350°C using ions with energies between 5 and 50 eV. The first step of the process leads to self-limiting oxide film growth with a steady-state thickness determined by the substrate temperature and, to a lesser extent, by the O⁺ energy. The deposition of the second phase is carried out by atomic force microscopy (AFM) and Auger electron spectroscopy (AES) and ex situ X-ray photoelectron spectroscopy (XPS). The films are stoichiometric and show no traces of suboxides as indicated by analysis of the Si 2p XPS peaks of, e.g., a film grown at 350°C by 25 eV O⁺ ion beam oxidation. This instrument provides individual control over the experimental parameters which in essence allows modelling and possibly optimization of commercially viable batch-scale plasma processing.
A compact negative metal ion beam source for various direct low energy ion beam depositions in high vacuum environment, has been developed. The ion source is based on the state-of-the-art solid state cesium ion technology. The negative ion beams produced by cesium ion bombardment (negative sputter ion yield varies in 0.2-0.5 for various metals). The ion source produces negative metal ion currents of up to 0.5 mA/cm² for the beam diameter of 1 °. The ion beam energy can be independently controllable in the range of 10-2000 eV. The ion source is very compact, stable, and easy to use. Due to the complete solid state ion technology, 10^-10 Torr of chamber pressure can be maintained while operating the source. The source can be used for various species of metals such as C, Ag, Au, Pt, Al, W, Ta, Mo etc. The source has been successfully used for the deposition of the amorphous diamond carbon and carbon nitride films.

Bias Sputter Deposition of Dense Ytria-Stabilized Zirconia Films on Porous Substrates. T. Tsai and S. A. Barnett, Department of Materials Science and Engineering, Northwestern University, Evanston, Illinois, 60208.

In thin-film solid oxide fuel cells (SOFC), yttria-stabilized zirconia (YSZ) electrolyte thin films deposited on porous substrate/electrodes must be electrically isolating and gas impermeable. In this paper, we describe YSZ film deposition by reactive magnetron sputtering of Zr-Y targets in Ar/O₂ mixtures on porous substrates, typically La₀.₇SrₓMnO₃ (LSM) with pore size <0.5 μm. Without substrate bias, SEM images of the films showed rough surfaces with evidence of nodular growth and porous structure; these films were too dense for SOFC applications only for thicknesses >20 μm. SEM fracture cross-section images showed that a negative DC substrate bias Vₛ of 150V produced an apparently dense structure with columnar features, while Vₛ = 300V produced a dense featureless morphology. Residual surface roughness was still present, showing dune-like morphologies. Both -150V and -300V biased samples have same peak to valley roughness values, ~0.5 μm, but different lateral period, ~1 μm for Vₛ = 150V and ~5μm for Vₛ = 300V. The stress field in the films increased with increasing bias. Cracking was observed for highly biased samples. Using optimal conditions, dense and leak-tight YSZ films were obtained at a thickness of only 3 μm, with measured SOFC open circuit voltages in 15% of theoretical values.

Modeling and Experimental Studies of Multi-Phase Formation in Reactive Sputtering. C. Neder, T. Nyberg and S. Berg, Inst. of Technology, Uppsala University, Box 534, S-751 21 Uppsala, Sweden, K. O. Legg, M. Graham, P. J. Rudnik, M. S. Wong and W. D. Sprout, BIRL, Northwestern University, 1801 Maple Ave., Evanston, IL 60201-3135, USA.

Reactive sputtering is a complex thin film deposition technique. Several material properties (mechanical, electrical and optical) of the deposited film are highly dependent on the process conditions. Due to instabilities (hysteresis effects) it may be necessary to use a real-time advanced feedback control loop in order to produce compounds with correct properties. In the following paper it is experimentally verified for a number of materials that different phases of the reactively sputtered compound can be formed at different reactive gas pressures. Since the deposition rate varies with reactive gas pressure, one phase may be deposited at a much higher rate, which may be of high importance for the cost of industrial processes. In order to better understand the process behavior of reactive sputtering and to be able to better predict the deposited film properties, computer models are becoming an important tool. This enables a much faster process development. An extended reactive sputtering model for the deposition of two compound phases is presented. The model enables predictions of the compositional changes of the deposited compound and the relative deposition rates versus the reactive gas pressure. A comparison with experimental result is also presented.


Carbon nitride thin films were prepared using an ionized magnetron sputtering system. An inductively coupled rf plasma was generated in the region between the sputtering source (high purity graphite) and the substrate table. An argon and nitrogen mixture was used as the sputtering gas. Sputtered atoms which pass through the high density plasma may be ionized, and the degree of ionization depends on several processing variables, such as the gas total pressure, reactive gas partial pressure, and applied rf power. Pulsed dc bias voltage, applied to the substrates, was varied to -500V. The chemical bonding, composition, and microstructure of the deposited films were studied by infrared analysis, Raman spectroscopy, Auger electron microscopy, and analytical transmission electron microscope. Optical properties were evaluated using ellipsometry. An ultra-micro indentation system was used to measure the hardness. The preliminary results showed that nitrogen is bonded to carbon in various configurations, that the nitrogen to carbon ratio in the films can be up to 0.8, and that the optical band gap is as high as 1.7 eV.


Ionized cluster beam deposition uses a beam of ionized, accelerated atom clusters to grow thin films. Late in 1991, following nearly 20 years of development, synthesis of large clusters of zinc by homogeneous nucleation was achieved through significant changes in two source parameters. Crucible pressure was increased from 2 Torr to over 1000 Torr and the nozzle was changed from cylindrical (1 x 1 mm) to converging-diverging (18 mm long and 0.4 mm in diameter at the throat). Such high vapor pressure is presently impractical for materials of industrial interest such as gold, silver, copper, and aluminum. The vapor pressure of Zn, Au, and Ag as a function of temperature are shown below. Recent results using gold with the converging-diverging nozzle at a pressure of just under 1 Torr showed no significant synthesis of large clusters. The work presented here describes results using silver at an intermediate pressure of around 100 Torr. The deposition rate was intermediate (1.7 mm s⁻¹) between gold (0.2 mm s⁻¹) and zinc (4.3 mm s⁻¹). Silver purity influenced the appearance of the beam. X-ray analysis of deposited films was presented. Ongoing work in real time, in-situ cluster size measurement will be reported. Preliminary results of ICB for trench filling will also be shown.


Review summarizes the development of vacuum arc ion sources from the first version made in 1984 which generated any metal ion beams of 20 cm diameter with current up to 1 A at an accelerating voltage up to 130 kV, pulse duration of 300 μs and repetition rate up to 50 Hz, for doing high dose implantation (HDI), to the creation of the Technological Accelerator of Metal Ions and Electron Kilo—TAMEK source which can produce, without switching of the source, the regimes HDI, ion deposition, ion beam mixing, and ion beam assisted deposition of the same metal ions, as well as electron beam generation. Ion beam (S = 300 cm², E < 100 keV) sources with vacuum arc current of several amperes (Iarc > 2 A, ill > 0.1 A) and milliseconds duration, and with arc current up to 100 kA (I = 1 to 10 kA) and microseconds duration, are described. Application of these sources for improving
the properties of surface layers of tools, construction materials, electrical contacts and electrodes, biomaterials, optical, dielectric, glass are discussed.


One of the primary factors believed to determine the hardness enhancement in superlattice (multi-layered) coatings, is the difference in layer shear moduli. The CrN/TiN superlattice system, with a larger modulus difference than previous nitride superlattices, was investigated in order to determine whether the modulus difference is the main factor determining the hardness. Polycrystalline CrN/TiN superlattices were deposited on M1 tool steel in an opposed-cathode unbalanced magnetron sputtering system with a rotating substrate holder. In initial experiments, the individual layers (CrN and TiN) of the superlattice were made of equal thickness. During reactive sputtering cubic CrN requires a N₂ partial pressure that is about one order of magnitude more than that required to form cubic TiN. In order to deposit both CrN and TiN in the cubic phase in the same deposition chamber, we attempted to produce a nitrogen pressure gradient. Semi-quantitative analysis of Auger electron spectroscopy data from CrN, coated samples, was used to estimate this pressure gradient, as well as to study the stoichiometry of the CrN and TiN films. The superlattice structure was characterized by X-ray diffraction which showed first order satellite peaks around the (111) Bragg peak of the material. The superlattice periods were varied from 1.8 nm to 16 nm. For 2 μm CrN/TiN coated samples, the results of the UMIS 2000 Ultra-Micro indentation hardness testing showed an increase in hardness of more than 1000 kg/m² over the range of mixtures for CrN and TiN, and the scratch testing showed critical loads that exceeded 6 kg.

**TF-WeP19** Neutralized Beam Assisted Evaporation of Cubic Boron Nitride on Si(100), Ming Lu, A. Bousett and A. Bensousa, SVEC, University of Houston, TX 77204 and K. Waters, A. Schultz Ionwerks.

We report the growth of cubic boron nitride (CBN) films on Si(100) substrates using electron beam evaporation of boron and a neutralized nitrogen beam. The beam neutralizer was adapted to 3 cm Kaufman-type ion source. The films were grown at substrate temperatures in the 400–500°C range and a boron evaporation rate of 2–3 nm/sec. Using this new approach, we show that highly stoichiometric BN films can be obtained with up to 80% cubic phase. We will present data from a variety of thin film characteristic techniques including FTIR, RBS, EPMA and TEM.

Supported by NASA NAGW-977, ARP under grant #9-00365-2224, and Ionwerks under SDIO Grant #L0391L0804.

**TF-WeP20** An X-ray Diffraction Study of Epitaxial Nitride Superlattices, A. Madan, Department of Physics and Astronomy, Northwestern University, Evanston, IL 60208. M. Skolnick, and S. A. Barnett, Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208.

Thin film superlattices generally exhibit substantial strength and/or hardness enhancements relative to the corresponding homogeneous materials. For example, epitaxial TiN/NbN superlattices exhibit a maximum Vickers microhardness value of 4900 kg/mm² at a superlattice period A = 4.6 nm, compared with ≈2000 kg/mm² for TiN/NbN alloys. Recent calculations suggest that there is a strong dependence of the strength and hardness enhancements on interface widths and concentration gradients. X-ray diffraction measurements and simulations were thus carried out to determine the nature of the superlattice composition modulation. A trapezoidal form of the compositional profile was assumed. Layer thickness fluctuations and random d-spacing variations were included to explain the broadening of the diffraction peaks. For cases where the layer materials were miscible, the composition within each layer and the interface widths was varied. Best fits to the experimental data for nitride superlattices showed that considerable interdiffusion was present. In TiN/NbN, for example, the layers contained up to 20 at% of the other metal constituent and the interface widths were found to be ≈1 nm. This latter value, when used in calculations of TiN/NbN hardness, provides reasonable agreement with the measured hardness values.


The surface defect structures on diamond (100) surfaces induced by 500 eV neon ion bombardment and by subsequent annealing were studied in situ with x-ray absorption near-edge structure (XANES) spectroscopy using 250 to 800 eV synchrotron radiation and with low energy electron diffractions. *Ex situ* x-ray photoemission spectroscopy (XPS) was also used to characterize the defective layer. Significant changes in the XANES spectra were identified for the defects induced by ion bombardment and subsequent annealing. The diamond discrete excitation absorption at 289.0 eV was clearly suppressed even at the lowest ion fluence used in this study, i.e. 3 x 10¹⁹/cm², and no such excitation could be observed at 7 x 10¹⁸/cm². However, the changes in the multi-maxima shape-resonance absorption structure in the range of 290-340 eV indicated that amorphization of the diamond surface required a fluence of 1 x 10¹⁹/cm². The structural changes were also manifested by the transformation of gap state absorption typical of clean (2 x 1) surfaces to the *s* absorption typical of amorphous carbon. Calculations from the XPS data showed that the defective layer was about 2 nm thick. Both the XANES and XPS data also indicated that the defects were stable upon annealing up to a temperature of 1100°C with no phase transformation to graphite.

**TF-WeP22** The Effect of the Physical Vapor Deposition Techniques of Sputtering and Evaporation of Ti on the Formation and Transformation of C49 to C54 TiSiN, C. Cabral, Jr., R. A. Roy, L. A. Clevenger and K. L. Saenger, IBM T. J. Watson Research Center, P.O. Box 218, Yorktown Heights, NY 10598.

It has been demonstrated that sputtered Ti thin films on polycrystalline substrates undergo the C49 to C54 TiSi phase transformation at lower temperatures than evaporated films. In particular, sputtered Ti films deposited at room temperature in the presence of 10 mTorr of Ar undergo the C49 to C54 phase transformation at a lower temperature than those deposited using alternate deposition conditions. High substrate bias and sputter deposition pressure cause the C49 to C54 transformation to be delayed by as much as 80°C respectively. Sputtered and evaporated Ti films, 45 to 63 nm thick, were deposited on polycrystalline Si substrates. The sputtering parameters of temperature, pressure and substrate bias were varied. The films were annealed in He either in a furnace or rapid thermal annealing system while *in situ* resistivity and/or stress as a function of temperature were monitored. To confirm TiSi phase formation isothermally annealed films were analyzed using x-ray diffraction (XRD).

**TF-WeP23** Residual Stress in Ion Implanted Titanium Nitride Films by Parallel Beam Glancing Incidence X-ray Diffraction, Anthony J. Perry and James R. Treglia, ISM Technologies Inc., 9965 Carroll Canyon, San Diego, CA 92131; Daniel E. Geist, Martin Marietta, Mail Stop 9683, P.O. Box 179, Denver, CO 80201.

It has long been thought that the improvements in the wear behavior of titanium nitride TiN after ion implantation with gas or metal ions is due to an increase in the compressive residual stress. Previous work using Seemann-Bohlin glancing angle focusing x-ray geometry [1] has shown that implanting TiN with argon or krypton ions has a negligible effect on the residual stress. In the present study, the work is extended to implants with ions of practical importance, namely nitrogen or a nickel-titanium dual implant in coatings made by both chemical and plume vapor deposition, CVD and PVD respectively. The coatings are in a state of low and high compressive residual stress respectively. Parallel beam glancing angle x-ray diffraction at incident angles in the range 0.5–10° is used to study the state of stress at depths of penetration in the range 0.1–1.6 μm, before and after implantation. The effect of nitrogen implantation or the nickel-titanium dual implant is discussed in this work. Further, the effect on the resulting data of using the x-ray elastic constants for titanium nitride in place of the bulk values is discussed.

VACUUM TECHNOLOGY
Room BR4 – Session NP-WeP

New Products
Moderator: W. Weed, Sandia National Laboratories.

NP-WeP2 Latest Developments on Valve Seat-Seal Assembly, R. De Villepoix, M. Lefrançois, J. Montuclard, C. Rouaud, Carbone Lorraine/Céladon-Francheville & Helicofox Saint-Etienne, FRANCE/Columbia, SC USA.

The Helicofox® metallic seal initially designed for static assemblies is presently used as a valve primary seal for some specific applications where the elastomer seal commonly used in such configuration becomes totally unsuitable due to low resistance to radiation and high temperature, and incompatibility with ultra-high vacuum conditions.

The hydrogen injection quick valves on IET fusion reactor, as well as the aluminium ultra-high vacuum valves on TRISTAN accelerator already operate using this technique over a rather narrow range of working conditions.

In order to widen the field of applications and establish the basic principles of the sealing function applied to a valve seat-seal assembly, tests were carried out in the Sealing Techniques Laboratory of the French Atomic Energy Commission (CEA).

Seal configuration and seal holding device have been reconsidered. Seals have been tested using different grades of sealing lining material i.e. successively aluminium, silver and copper. In that respect, such assembly configuration was found in full compliance with the requirements of the new types of valve, those which are being considered for applications where ultra-high vacuum is combined with a leak rate requirement of 1.10e-10 Pa.m³.s⁻¹ and a high bake-out temperature.

NP-WeP3 Reliable In-Situ Gas Analysis for Aggressive Gas Processes, W. Eisinger, Leybold Infinon Inc.

The Transpector® AGM is a mass spectrometer designed for process monitoring of aggressive and reactive gas environments. It employs a patented gas shield sampling method (the Virtual Valve), minimizing the exposure of the sampling system and gas analyzer to reactive gases and particulates in the process vessel. The Virtual Valve uses a small stream of the process' carrier gas (typically 10 sccm) to shield the sampling orifice. A laminar flow of this gas creates a barrier in front of the sampling orifice, essentially stopping the flow of particulates and process gases into the mass spectrometer. To begin analysis the diverted flow of the gas is stopped.

Applications include CVD processes between .1 and 100 torr. Specifically, the Transpector® AGM has been used to monitor impurities such as water and oxygen during a thermally activated LPCVD blanket tungsten process run of several wafers over 10 minutes at 40 torr. The process used tungsten hexafluoride (WF₆) as the precursor, hydrogen (H₂) as the primary reducing agent, silane (SiH₄) as an initiator and argon (Ar) as a diluant (also used as the shield gas).

The Transpector AGM has demonstrated extended life in an extremely hostile environment, fast time response to follow rapid events (due to its close coupling to the process and absence of mechanical parts in the Virtual Valve), and detection limits for impurities in the ppb range, all of which contribute to reliable sampling in aggressive gas environments relative to conventional sampling techniques.

NP-WeP4 Non-Reclosing Pressure Relief Device for Vacuum Systems, W. A. Swanson, Sandia National Laboratories, Livermore, CA 94550.

Many vacuum systems have components which cannot withstand pressures greater than 1 or 2 psig. Relief valves do not have the leak-tightness required for high vacuum applications and existing rupture discs do not relieve at sufficiently low pressures. Sandia National Laboratories was recently granted a patent for a non-reclosing pressure relief device for vacuum systems that is UHV-compatible and relieves at precisely settable pressures ranging from 0.5 psig to 2 psig. The device consists of a welded metal bellows with conventional vacuum flanges on both ends. One end is attached to the vacuum system in a vertical position; the other end is closed by a thin metal membrane. A four-bladed cutter is positioned so that the extending bellows will drive the membrane into the cutter blade when the system reaches the desired relief pressure. Relief pressures above 0.5 psig can be set by adding weight to the bellows. This has the effect of increasing the reference pressure (normally atmospheric) and allows one bellows to be used for a range of pressures. The rupture element (a 0.001" thick stainless steel disc brazed to a copper Mini-Confal™ gasket) can be easily and cheaply replaced without removing the device from the vacuum system and without altering the relief pressure of the device.

NP-WeP5 New Compact Total Pressure Gauges, A. Schmid, R. Stöcker, BALZERS Limited, Vacuum Technology Instruments, FL-9496 Balzers, Liechtenstein.

The design criteria and applications of the new gauges are discussed. The new unique FullRange™ gauge is the combination of Pirani and cold cathode ionisation gauge with on-board electronic, giving lin/log voltage to pressure response with overrange, underrange and failure indication. This new compact gauge combines two gauges in one housing and provides significant advantages compared to conventional gauges:

- The salient feature is a linear output as a function of the logarithmic pressure over the very large pressure range of 1000 to 10⁻⁹ mbar without discontinuities.
- The user just needs one formula to easily convert from voltage to pressure in any desired unit.
- The rugged, space-saving compact design makes these intelligent gauges ideally suited for cost-saving integration into any computer-controlled vacuum system with no separate gauge controller required.
- The extremely low power consumption of max. 2 VA and the power range from 15 to 30 VDC without stabilisation allows multiple use in existing vacuum systems with their own power supply.
- The output includes in addition to the pressure readout an overrange, underrange and failure indication.

The gauge head design will be presented and the properties, such as the large measuring range, accuracy, up time, ultra small size and fail-save construction will be discussed.

NP-WeP7 Combination Valve Enables In-Situ Zeroing and Calibration of Capacitance Manometers, Jonathan Skuba, HPS Division of MKS Instruments, Boulder, CO, John Dunn, MKS Instruments, Santa Clara, CA.

Thin film process repeatability involves the predictable control of a variety of parameters, including pressure measurement. As with most process and gauge technologies, proper use of capacitance manometers requires periodic verification to ensure calibration integrity. The In-Situ Diagnostics Access Valve (I.D.A. Valve™) from HPS Division of MKS Instruments is a compact, high conductance combination gauge isolation valve with a secondary, manually valued port for calibration and diagnostic access. The I.D.A. Valve™ fulfills the following: It functions as a gas isolation valve. While isolated from the process chamber, the gauge may be zeroed or calibrated without its removal from the system, eliminating system dependent variables. With both valves open, leak check and system related diagnostics can be performed. Used regularly, the I.D.A. Valve™ reduces labor time requirements and enhances system uptime and availability.
SURFACE SCIENCE
Room A205 – Session SS1–ThM

Nanoscale Measurements
Moderator: J. E. Reut–Robey, University of Maryland.

8:20 am SS1-ThM1 Nanostructuring of Surfaces by Diffusion Controlled Growth, Klaus Kern, Institut de Physique Experimenterel, EPF Lausanne, CH-1015 Lausanne, Switzerland.

We demonstrate a novel technique for the synthesis of densely packed planar nanostructures: diffusion controlled aggregation on surfaces. By exploiting the dependence of the mobility of adsorbed atoms on substrate crystal face and temperature, we are able to grow linear, two-dimensional compact or tenous fractal aggregates of nanometer dimensions. The high number density (10^13-10^16 cm^-2) of these structures means that their physical and chemical properties can be easily measured with conventional surface spectroscopies. The underlying kinetic surface processes, like diffusion, nucleation and aggregation are discussed and their implication for self-organized two-dimensional growth is outlined.

INVITED

9:00 am SS1-ThM3 Observation of Quantum Size Effects at Room Temperature on Metal Surfaces with the STM, Ph. Avouris, J.-W. Lyo and Y. Hasegawa, IBM Research Division, T. J. Watson Research Center, Yorktown Heights, NY 10598.

The study of the physics of electrons confined in low-dimensional structures, such as the two-dimensional electron gas (2DEG) systems provided by semiconductor heterostructure interfaces, is a very active area of research, the results of which have an important impact on basic science and technology.

A different, higher density 2DEG system is provided by electrons in Shockley metal surface states. Surface steps and adsorbrates act as potential energy barriers reflecting the electron de Broglie waves. We use this property to confine the metal surface electrons in low-dimensional structures which, because of their size and stability show quantum-size effects even at room temperature. The structures involve narrow terraces or single-atom-high islands and are formed by the deposition and annealing of metal films. A variety of different size and shape structures which are stable over a wide range of temperatures can be produced. Scanning tunneling spectroscopy is used to image the probability distributions of the confined states and determine their energies. The results allow us to estimate the confining barriers and provide evidence for the existence of 1D step-edge states. Steps and point defects are also found to mix surface and bulk states and lead to the scattering of surface electrons to bulk states, limiting the extent of confinement.

The charge-density distributions and field-gradients of the confined states formed during film growth are different from those at an extended terrace. These factors are likely to influence surface processes that depend on them, such as the sticking, diffusion and spatial distribution of adatoms and the growth of islands.

*Present address: Mesoscopic Materials Center, Kyoto University, Kyoto 606-01, Japan.

9:20 am SS1-ThM4 STM of Thin Film MgO Grown on Mo(001), M. C. Gallagher, M. Fyfield, James P. Cowin, and S. A. Joyce, Molecular Science Research Center, Pacific Northwest Laboratory, Richland, WA 99352.

Recently the surface properties of metal oxides have attracted a great deal of attention. The rocksalt crystal structure and the single valence of the Mg ion makes MgO an attractive model system. Although Scanning Tunneling Microscopy is a powerful probe of surfaces, the technique is restricted to conducting samples. One approach to extend STM to insulating materials is to study well defined thin films on metal substrates. To this end we have used in-situ STM to investigate the growth and electronic properties of MgO thin films deposited on Mo(001). The films were grown by evaporating Mg metal in a background pressure of oxygen. Correct stoichiometry of the films was ensured using Auger electron spectroscopy. To date we have successfully studied films as thick as 25 A clearly demonstrating the feasibility of this method. Growth temperatures from room temperature to 750°C have been studied. Drastic changes in film morphology were observed. Films grown at 750°C exhibited a higher degree of microscopic roughness than those grown at room temperature. Post growth annealing reduced the overall roughness. Quantitative results for films grown under a range of growth conditions will be presented.


* Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RL0 1830.

9:40 am SS1-ThM5 Nanosphere Lithography, John C. Hulteen and Richard P. Van Duyne, Department of Chemistry, Northwestern University, Evanston, IL 60208.

The fabrication of nanoparticles with diameters, d < 100 nm, is usually carried out by e-beam and/or x-ray lithography. In this presentation, we discuss the implementation of an inherently parallel nanofabrication technique based on the self-assembly of polymer colloid nanospheres. This process is termed nanosphere lithography (NSL). Using NSL, periodic particle arrays (PPAs) of Ag and Au on freshly cleaved mica substrates have been fabricated with particle diameter, 20 nm < < 200 nm and interparticle spacing, d - dpp. PPAs with tunable structural characteristics d, dpp, and particle height, h, can be fabricated by: (i) varying nanosphere diameter, dpp; (ii) angle, θ, between colliimated vapor deposition beam and the nanosphere mask surface normal; and (iii) mass thickness, dpp, of particle material, M. The structural characteristics of NSL-derived PPAs are determined by atomic force microscopy (AFM). It will be shown that the standard deviations of d and dpp histograms are entirely determined by the original size distribution of dpp. (Var., < 2-3%). NSL processing is essentially independent of the choice of M and substrate material, S. Any M capable of gas phase introduction and any S on which nanospheres will coherently self-assemble may be used. Preliminary optical property vs. particle size investigations have been carried out using UV-VIS absorption spectroscopy and surface-enhanced Raman excitation spectroscopy (SERES) demonstrating that these properties are extremely sensitive to dpp, d, h, and particle shape.

INVITED

10:00 am SS1-ThM6 Nanofabrication on Electron Beam Resist Using Scanning Tunneling Microscopy, M. H. Naghesh, A. Archer, J. M. Hetrick, and I. Adeidas, University of Illinois at Urbana-Champaign, Urbana, Illinois 61801.

Recently, techniques have been developed which use a scanning tunneling microscope (STM) as a tool to fabricate nanometer-scale structures. Using electron beam resist in the nanometer-scale fabrication, structures have the potential for pattern transfer, since resists exposed to electrons can be developed and used as masks for further processing. Standard electron beam lithography on resists at energies of approximately 50 KeV has already been used for the fabrication of structures in the nanometer range, yielding line widths of approximately 95 nm. Investigations of STM fabrication on electron beam resist by Marrian et al. have already been shown that this technique has potential in both the achievable resolution and the dimensions of the fabricated structures. We report here the results of nanofabrication using a scanning tunneling microscope on silicon coated with SAL 601 electron beam resist. The resist was exposed to an electron beam with a range of energies between 1 and 50 eV approximately. The results of the exposure were found to be complex, switching over from producing structures that appear as mounds to structures that appear as grooves as the biasing voltage drops below 6 V. Mound-like structures of width as small as 18 nm with a 2 nm corrugation, and structures like grooves as small as 15 nm in width and 0.5 nm in depth were fabricated. Somewhat narrower but shallower structures can be produced on the resist, but we believe that these results are reaching the practical limit of fabrication. If pattern transfer can be achieved, STM-based techniques will be able to compete and even surpass the capabilities of more conventional approaches.

INVITED


Metals (e.g., Ag, Cu, Cd), semiconductors (e.g., CdS), and organic
conductors (e.g. TTF-Br) have been synthesized in single nanoscopic batches on the van der Waals surfaces of graphite and the semiconductor MoS2 using the scanning tunneling microscope (STM). The synthesis reaction is localized at a shallow, circular pit approximately 100 Å in diameter which is induced by the application of a 4-6 V bias pulse—applied between the STM tip and the surface while tunneling—with a duration of ~2 μsec. This pulse can be extended to longer times (from 3-50 μsec), generally at a lower amplitude (~300 mV-3V), to effect a single electron synthesis reaction of interest which then occurs selectively in the freshly formed pit. The dimensions of the synthesized products are typically 100-500 Å in diameter and 20-100 Å in height. The characterization of the deposited materials is accomplished using TEM e-diffraction, scanning Auger probe, elemental analysis, and laser-induced fluorescence (for semiconductor nanostructures) in addition to STM. In-situ STM investigation of nanofabrication reactivity and chemical passivation will be reported.

**INVITED.**

11:00 am SS1-ThM9 Self-Assembled Monolayers of Pendant Monomers: A Basis for Fabrication with the Scanning Tunneling Microscope, R. J. Wilicutt and R. L. McCarley, Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803.

We are interested in nanofabrication with the STM using well-defined electrochemical reactions on surfaces. Our approach is to attach monomeric species onto smooth surfaces and polymerize them using the STM. We describe here the synthesis and characterization of a series of electropolymers on surfaces which are covalently attached to electrode surfaces via self-assembly. We have taken advantage of the well-known interaction of organolithanes with Au and Pt surfaces in order to form monolayers of N-(ω-mercaptoalkylthio)pyrroles on solid electrodes. The voltammetry of the surface-confined pyrroles in various electrolyte systems will be presented in order to establish whether or not the electopolymerization into the monolayer occurs and what effects monolayer disordering have on possible coupling reactions between neighboring pyrrole sites. Investigations of orientation and chain length effects will also be presented. The adhesion of poly(pyrrole) films to the electrode surface has been increased dramatically by use of the monomer monolayers. We will also present SEM and SFM images of poly(pyrrole) films deposited on the monolayers.

**INVITED**

11:20 am SS1-ThM10 STM-Induced Etching of Ultra-thin Organic Resists: Structure, Mechanism, and Post-etching Elaboration, J. K. Schorr and R. M. Crooks, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255; T. Corbit and M. J. Hampden-Smith, Department of Chemistry, University of New Mexico, Albuquerque, NM 87131.

We have prepared ultra-thin organic resists by spontaneous adsorption of organometallic molecules onto Au (111) substrates. The resist can be patterned by scanning tunneling microscopy (STM). To better understand the resist etching mechanism, we varied the tip bias, tunneling current, resist composition, the environmental exposure, scan rate, and the environmental conditions. The results indicate that organometallic monolayers form excellent STM resist materials. However, we found a strong dependence of etching efficiency on the applied tip bias. For example, we observed a distinct bias threshold below which etching did not occur. The tunneling current, resist composition, and other variables also affect the pattern formation, but less dramatically. In addition to fabrication and characterization of patterns as small as 10 nm, we have also recently shown that patterned surfaces can be further elaborated by subsequent selective Cu chemical vapor deposition. Results of these studies will also be discussed.

11:40 am SS1-ThM11 NEXAFS Spectroscopy, A Tool for Studying Metal-Organic Molecule Interfaces, G. Tournier, LURE-Bât. 209D, 91405 Orsay, France.

The interaction of unsaturated molecules with a metallic surface is of great interest both from a theoretical point of view (charge-transfer mechanisms between the substrate and the compound, modifications of the orbital distribution) and from a practical point of view (surface chemistry, catalysis). A considerable amount of work has thus been devoted to the adsorption of benzene, pyridine, CO or ethylene on various substrates. However, questions regarding the nature of the interaction and/or the chemical bond between the organic compound and the metal and ii) the organization of the monolayer on the surface need a better knowledge. Near edge X-ray absorption fine structure spectroscopy (NEXAFS) is well adapted to probe these parameters, since it allows the determination of the unoccupied density of states, the bond lengths and the orientation of the organic molecule. Our aim is here to get a better understanding of the chemical structure influence of ethynyl derivatives on the interactions modes with platinum, particularly when a hydrogen atom is replaced by an acid, ester or aldehyde function (acrylic acid: CH2=CH-COOH), acrolein (CH2=CH-CHO) and methylnitrocatechol (CH3=CH-CO-OCH3). When a monolayer of acrylic acid is adsorbed on Pt(111) at 95K, the C K edge NEXAFS spectra reveal that i) the molecule is flat on the surface and ii) interacts strongly with the Pt d band configuration (hybridization effect between the C orbital and the Pt d band, formation of Pt-C bond). Similar results are obtained with methylnitrocatechol. On the contrary, a c-olefin is physisorbed on Pt (111) at 95K, the molecule lying parallel to the surface. These differences in the interactions modes must be linked to a more or less important delocalization of the C = C or C = O orbital with the chemical group which induces molecular levels able or not to hybridize with the Pt d band. XPS, UPS and theoretical multiple scattering calculations will be presented to support the NEX-IFS results.

**SURFACE SCIENCE**

**Room A201 - Session SS2-ThM**

**Surface Interactions**

**Moderator:** D. N. Belton, General Motors Research and Development Center.

8:20 am SS2-ThM1 Coadsorption of Alkalds and Hydrogen on W(100), W. Hagoë* and P. J. Estregup, Department of Chemistry, Brown University, Providence, Rhode Island 02912.

Surface reconstruction is known to be essential to understanding the structure and desorption features of H/W(100) and H/Mo(100), as well as the effective repulsion of coadsorbed H and O on W(100) and Mo(100). Similar effects are expected for other coadsorbates on these surfaces. In the present study we have investigated the effect of substrate distortions on the interactions between alkalis (Li, Na and K) and hydrogen on W(100). We have examined the desorption, work function and LEED behavior of the coadsorbed systems Li + H/W(100), Na + H/W(100) and K + H/W(100). We find that the desorption energy of the most strongly bound state of hydrogen is raised by 500, 100 and 0 meV in the presence of Li, Na and K, respectively. We derive from the work function behavior that alkalis significantly reduce the effective dipole moment of hydrogen on the surface. LEED observations reveal that alkalis strongly affect the surface phases of H/W(100), except in the case of Na. For the systems Li + H/W(100) and Na + H/W(100), the substrate does not significantly alter the adatomatom interactions. For K + H/W(100), a strong effect is seen. Contrary to expectations, for all three coadsorbed systems, we find evidence of attractive interactions between the alkalis and hydrogen at early coverages.

*Present Address: Department of Chemical Engineering, University of California, Santa Barbara, California 93106.

8:40 am SS2-ThM2 Comparative HREELS Study of H and CO on Pt(335), Hong Wang, R. G. Tobin, David K. Lambert, Galen B. Fisher, and Craig L. DiMaggio, Dept. of Physics and Astronomy and CFMR, Michigan State University, East Lansing, MI 48824-1116.

*Physics Department, *Physical Chemistry Department, *General Motors Research & Development Center, Warren, MI 48090-9055.

The interactions of CO and H on Pt surfaces affect the operation of catalytic converters, exhaust gas sensors and fuel cells. We studied the coadsorption of CO and H on Pt(335) [Pt(6)[4(111)×(100)] by high resolution electron energy loss spectroscopy and temperature programmed desorption. Both CO and H adsorb preferentially at edge sites. Along the edge, islands of mixed H and CO coexist with regions of pure CO, in sharp contrast with the complete segregation found on structurally similar Pt(112) surface. These results suggest a dependence of the CO-H indirect interaction on terrace width. Within the mixed islands coadsorbed H locally shifts atop CO to bridge sites; at saturation H coverage the shift is almost complete. On the terrace, however, the intensity and frequency of the atop CO vibration are unaffected by coadsorbed H. The EELS cross sections of bridge and...
atop CO on the step edge are equal, whereas on Pt(111) atop CO’s cross section is 1.8 times larger than that of bridge CO.

Supported in part by the Petroleum Research Fund and NSF Grant No. DMR-9201077.

9:20 am **SS2-ThM4** Thermal Desorption Studies of High-Coverage Hydrogen Overlayers Created with Gas-Phase Atomic Hydrogen on Ru(001)**, T. A. Jachimowski, B. Meng, D. F. Johnson, and W. H. Weinberg, Department of Chemical Engineering, University of California, Santa Barbara, CA 93106.

The adsorption and desorption of high-coverage hydrogen overlayers on Ru(001), created at 100 K using gas-phase atomic hydrogen, has been investigated using thermal desorption mass spectrometry. Hydrogen surface coverages up to 1.42 hydrogen adatoms per primitive Ru(001) unit cell have been obtained, in excess of the saturation coverage of unity obtained by dissociative chemisorption of molecular hydrogen. Furthermore, the desorption spectra are characterized by a peak, previously present for dissociative adsorption of molecular hydrogen, which increases in area but does not change in peak temperature (320 K) for coverages greater than unity. Impinging upon this atomic hydrogen (deuterium) was also found reacted with chemisorbed deuterium (hydrogen) and desor molecularly. The abstraction of surface hydrogen by gas-phase atomic hydrogen occurs at more than 150 K below the associative thermal desorption temperature of hydrogen from this surface. The ratio of the cross section for abstraction to the cross section for desorption was found to be 0.4. These experimental results suggest that the abstraction reaction is occurring via an Eley-Rideal mechanism.

*This work was supported by the National Science Foundation (grant CHE-930020).*

9:40 am **SS2-ThM5** The Adsorption of Hydrogen on the Mo(001) and Ru(001) Surfaces, M. Okada, The University of Tennessee, Knoxville, TN, and D. B. Porter and D. M. Zehner, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6057, USA.

From low-energy electron diffraction (LEED), low-energy ion scattering, and surface core-level photoemission results, it is known that the relative distribution of Mo and Re in the outermost layers of the (001), (110), and (111) surfaces of the random alloy Mo75Re25 is orientation dependent. As a consequence, there is a distribution of inhomogeneous arrangements of both species that participate in any adsorption process. The adsorption of hydrogen (deuterium) was studied with LEED, thermal desorption spectroscopy (TDS), and nuclear reaction analysis to investigate this distribution. Following exposure to H2, only (1×1) patterns are observed with LEED for all coverages, indicating no adsorbate-induced reconstruction. Using deuterium and the D(H, p)He reaction, the saturation coverages for the (100), (110), and (111) surfaces were determined to be 2.0, 1.0, and 2.9 respectively. A coverage of 2.0 for the (100) surface is in excellent agreement with a model determined with a LEED-IV analysis in which all the H-adsorption sites on the predominantly Mo-terminated surface are occupied at saturation coverage. The TDS spectra from the saturated (110) and (111) surfaces show two peaks, whose relative intensities are different. These may correspond to desorption from inhomogeneous rich Mo and Re regions respectively, providing additional evidence for excess Re in the outermost layer of the (110) surface. Exposure of the clean surfaces to atomic deuterium results in the same set of observations with no indication of adsorption into the bulk.

*This work was supported by the U.S. Department of Energy, under contract DE-AC05-84OR22400 with Martin Marietta Energy Systems, Inc.*

10:00 am **SS2-ThM6** Separating Ensemble and Electronic Effects on Bimetallic and Alloy Surfaces, Chen Xu and Bruce E. Kooi, Department of Chemistry, University of Southern California, Los Angeles, CA 90089-0482.

We have recently compared the chemistry of ordered surface alloys formed by Sn on Pt(111) and Ni(111) surfaces. CO adsorption is strongly suppressed on the (133)/(133)R30° Sn/Pt(111) surface alloy, where a similar saturation coverage, adsorption site population, and adsorption energy to that on Pt(111) was observed. These Ni-Sn and Pt-Sn alloys have similar surface roughness, but have different buckling distances for the Sn atoms with respect to the surface plane. We attribute the dramatic differences in the chemistry of these similar surfaces to the vertical position of the Sn in the surface layer. The balance between direct interactions of Sn with adsorbed molecules and the interactions of the transition metal atoms with adsorbed molecules controls the effectiveness of Sn as a site-blocking surface modifier, and this balance is affected by the vertical position of the Sn. These results reveal new insight into the influence of surface modifiers on chemical reactions at surfaces and force us to reevaluate simple concepts of site-blocking and ensemble sites on alloy surfaces.

10:20 am **SS2-ThM7** Adsorption of Sulfur on Bimetallic Surfaces,* M. Kuhn and J. A. Rodriguez, Chemistry Department, Brookhaven National Laboratory, Upton, NY 11973.

The coadsorption of S and a noble metal (Cu, Ag or Au) on Ru(001) has been investigated using LEED, TDS, XPS and XAES. The Au-S, Cu-S, and Ag-S interactions are repulsive. In the Cu/S/Ru(001) system, the Ru-noble metal bond is stronger than the Ru-S bond, and the presence of Au leads to a significant change in the kinetics of sulfur desorption. On Ru(001) sulfur desorbs in a flat feature between 1100 and 1500 K, whereas on Au/Re(001) a sharp desorption peak is seen at 1100 K. The Au atoms compress S into 2D islands, keeping the local coverage of S large. In the Ag/S/Ru(001) system, the Ru-S bond is stronger than the Ru-Noble metal bond, and the presence of S reduces the local desorption temperature of Ag from Ru(001) by almost 150 K. Results of XPS and XAES indicate that sulfur and copper atoms coadsorbed on top of Ru(001) react to form a sulfide. The Cu atoms in this compound exhibit Co-desorption temperature smaller than those of Cu/Ru(001) or pure Cu.

*This work was supported by the US Department of Energy (DE-AC02-76CH00016).*

10:40 am **SS2-ThM8** ‘Nanocaltasis’ by the Tip of a Scanning Tunneling Microscope Operating Inside a Reactor Cell, B. J. McIntyre, Uwe Schroder, Maris Salmson, and G. A. Somorjai, Lawrence Berkeley Laboratory and Department of Chemistry, University of California, Berkeley, CA 94720, USA, 1Institut für Physikalische Chemie der Universität Bonn, Wegelerstr. 12, 53115 Bonn, Germany.

The platinum tip of a Scanning Tunneling Microscope that operates inside an atmospheric pressure chemical reactor cell, has been used to locally rehydrogenate carbonaceous fragments deposited on the surface of Pt(111). The Pt tip acted as a catalyst after activation by short voltage pulses when in atmospheric pressures of hydrogen. In this active state the clusters in the area scanned by the tip were reacted
away with nanometer spatial resolution. This effect did not occur when using gold tips, or in CO environments. The tip catalysis was observed to be strongly dependent upon tip proximity to the surface, except for hydrogenation decomposition, reactant gas composition and reactant pressure.

11:00 am SS2-ThuM9 SPA-RHEED—Spot Profile Analysis for in situ Characterization, B. Miller and M. Henzler, University of Hannover, Appelstr. 2, D-30167 Hannover, Germany.

The morphology of the surface dominates the quality of the epitaxial growth. In order to tailor low dimensional artificial structures the surface must be characterized during growth. One of the most powerful methods for in situ studies is RHEED. The quantitative analysis on island size and distributions, however, is mainly performed by LEED because of the more complicated geometry of RHEED and the influence of inelastic scattering. To overcome these difficulties, a novel RHEED instrument with extremely high angular resolution (0.1 mrad) and energy resolution of about 2 eV is developed. For the first time the SPA-RHEED instrument points out the important contribution of inelastic processes to the intensity distribution of the spot profiles. Electronic losses due to plasmons and band transitions are clearly detected and quantitatively described by the dipole scattering theory. A meaningful evaluation of the profiles with respect to defects is only possible with a good energy resolution. For well prepared Si(ll1) with various island sizes in different azimuths the elastic and inelastic scattering is investigated throughout several Brillouin zones. Mean terrace sizes between 50 and 500 Å are detected. Characteristic structures are correlated with the scattering vectors and are separated into surface and bulk related phenomena because of the refraction effect. The extremely high angular resolution of SPA-RHEED enables determination of island size up into the micrometer range and lattice constants with an accuracy <0.1%. Surface damage is reduced due to the extremely low current. Therefore the features of the SPA-RHEED instrument offer new applications for RHEED.

11:20 am SS5-ThuM10 Experimental and Numerical Investigation of Ignition Conditions of H2/O2 Mixtures on Pt, M. Rinnemo, O. Deutschmann, P. Ahlström, F. Behrendt, and B. Kasemo, Department of Applied Physics, Chalmers University of Technology and University of Göteborg, S-41296 Göteborg, Sweden, 2Universität Stuttgart, Institut für Technische Verbrennung, Pfaffenwaldring 12, D-70569 Stuttgart, Germany.

Catalytic ignition is the sudden transition in a catalytic reaction from kinetic control to mass transport control. Its description requires a detailed knowledge of both the elementary reactions steps on the surface as well as of the transport processes between surface and gas phase.

In the experiment mixtures of hydrogen and oxygen, diluted with nitrogen and flowing slowly at atmospheric pressure, were ignited by a resistively heated platinum wire. Temperature vs. time traces were recorded for a wide range of compositions. Such data have been used to verify the Frank-Kamenetskii criterion for ignition, which states that at this point the derivatives of the dissipated chemical power and of the heat loss, respectively, become equal. In this work we have investigated further the ignition condition, and in addition the transient behaviour after ignition until a new equilibrium is established.

The experimental results are compared with detailed numerical simulations. For this purpose a code simulating laminar reactive flows above catalytically active surfaces is applied to the experimental conditions. The chemical reactions in the gas phase as well as on the surface are described using a mechanism consisting of elementary reaction steps. Experimental found ignition behaviours are compared with numerical results. Additionally, the evolution of surface coverages and gas phase gradients with time is discussed. The comparison elucidates the complex interaction between surface and gas phase kinetics.


11:40 am SS5-ThuM11 The Synthesis of Ammonia on Ru(0001), P. A. Taylor and E. Törnquist, Haldor Topsøe Research Laboratories, DK-2800 Lyngby, Denmark.

Ruthenium based catalytic systems have been subject to several studies as an alternative to the traditional iron ammonia catalyst [1]. In this work the synthesis of ammonia over Ru(0001) has been studied in a combined high pressure/ultrahigh vacuum (UHV) system. The Ru(0001) crystal was cleaned and characterized in UHV using conventional surface science techniques. Once clean, the crystal was placed into a high pressure cell (HPC) contained within the UHV chamber, where the design pressure of the HPC is 10 bar. A controlled leak from the HPC to a quadrupole mass spectrometer made it possible to monitor the gas composition during synthesis. The concentration of ammonia was measured as a function of crystal temperature and total pressure, both under static and flow conditions. In the temperature range of 573–673 K (at 9 different temperatures) the overall activation energy for ammonia synthesis was measured to be 128 kJ mol⁻¹ in a stoichiometric (N₂/3H₂) gas-mixture at 2 bar. After synthesis, the only detectable adsorbed species was nitrogen. The presence of low concentrations of water during ammonia synthesis had no measurable effect on the measured rates. Adsorbed sulfur, however, acts as a poison blocking approximately 6 active surface sites. The promotional effects of adsorbed potassium will also be reported. The results provide essential information needed to construct a micro-kinetic model for the synthesis of ammonia over ruthenium.


NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY/BIOMATERIAL Interfaces Room A209 – Session NSBI-ThM

Biology at the Nanoscale: II
Moderator: S. Lindsay, Arizona State University.

8:20 am NSBI-ThM1 Molecular Recognition Between DNA Base Pairs by AFM, B. D. Rainier, T. Boland, Department of Chemical Engineering and Center for Bioengineering, University of Washington, Seattle, WA 98195.

Small, planar aromatic molecules such as purines and pyrimidines are systems that exhibit spontaneous self-assembly on gold surfaces. The self-assembly processes have been investigated by STM, AFM, ESCA. Static SIMS, and TPD characterizations of the organic systems corroborate the observations. At equilibrium, ordered monolayers of molecules in an edge on conformation were observed for most systems. A lone nitrogen pair is chemisorbed to the surface, and molecules are interacting through mutual π-base stacking to yield lateral stability. To probe if these biological monolayers exhibit similar molecular recognition capabilities as in living organisms, the four DNA bases were self-assembled onto gold coated AFM tips and flat gold surfaces that were then allowed to interact. Only when complementary bases are assembled on tip and surface, the AFM detects additional forces that are absent for bare gold samples or assemblies of noncomplementary bases. This additional, attractive force is attributed to hydrogen bonding, and can be used as a qualitative indicator of molecular recognition. Force curves and recognition AFM images will be shown. Quantitative statistical analysis of the measured forces will be presented. Finally, a strategy for patterning surfaces with DNA bases and subsequently recognizing the pattern and its relevance to DNA sequencing and biological sensors will be discussed.


We have used an Atomic Force Microscope (AFM) to measure directly the binding interaction associated with Watson-Crick base pairing between single strands of DNA that have been immobilized on the surfaces of the AFM probe and substrate. The binding interaction is measured by first bringing the two surfaces together, allowing the molecules to interact, and then separating them. The measurements are conducted in solution to maintain bioactivity of the molecules and reduce nonspecific surface forces. Two different experiments are performed: a direct assay in which oligonucleotides are immobilized on opposing surfaces and a sandwich assay in which a homopolymer nucleotide is placed between opposing surfaces that are covered with its complement. The first experiment revealed strong adhesive forces (∼1.6 N/m) as the result of the Watson-Crick base pairs bonding together.
only when the base sequences of the oligonucleotides are complementary. The magnitude and distribution of the rupture forces are consistent with single molecule interactions. In fact, a direct correlation is observed between force and the number of base pairs in the double-helix. The addition of the homopolymer did not change the magnitude of the adhesive force but increased the distance (up to 400 nm) between the surfaces at which the bond rupture occurs. As the surfaces are separated an exponentially increasing force is required to elongate the molecule resulting in the measure of the molecule’s elasticity. These results demonstrate that the AFM can measure intra- as well as intra-molecular forces in complex macromolecular systems.  

9:00 am NSBI-ThM3 Scanning Probe Microscopic Visualization of Electrostatically Immobilized Intercalating Drug-Nucleic Acid Complexes, L. A. Bottomley, J. E. Coury, G. Gardner, E. A. Handley and L. D. Williams, School of Chemistry & Biochemistry, Georgia Institute of Technology, Atlanta, GA 30332-0400 USA. DNA intercalators are small molecules which insert between the base pairs of the DNA double helix. Intercalation into DNA necessarily results in radical changes in DNA conformation, separating base pairs along the helical axis. Thus, intercalators are of great importance as probes of nucleic acid structure and as interferents or inhibitors of DNA replication, transcription and/or topoisomerase activities. Relationships between structures of intercalators and conformational changes of intercalated DNA complexes are subtle and unresolved. Intercalating drug molecules have been synthesized incorporating markers easily distinguishable by STM and AFM. Specifically designed psoralen and methidium bromide derivatives have been intercalated into DNA. Scanning tunneling and atomic force micrographs were obtained following electrostatic immobilization of the complexes onto chemically modified gold or mica surfaces. To facilitate recognition of the point of intercalation, the psoralen and methidium derivatives were covalently linked to biotin and then exposed to streptavidin-coated colloidal gold beads. The effects of intercalation on the tertiary structure of DNA will be presented. Comparisons will be made between the structures determined by AFM and STM to those determined crystallographically. INVITED  

9:40 am NSBI-ThM5 Sequence Effects in the Images and Bonding of Single Stranded DNA on Au(111) Observed by Electrochemical STM, Y. Lyubchanska, Dima Rekesh, T. W. Jing and S. M. Lindsey, Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85287. STM images of the adsorption process of the complementary nucleic acid oligomers 5'-CCCCCTTTTCCTCCCTTT and 5'-AAAAGGGGAAAAAGGGGUG on Au(111) show that it is strongly dependent on rupture force and the number of base pairs without potential control, even on electrodes that are slightly negative of the potential for zero charge. The adsorption process for the polypurine is similar to what we have found for random sequence DNA, requiring a positive charge on the electrode and co-adsorption of a small anion. The images of the spontaneously adsorbed polyamidine and the electrochemically adsorbed polyamidine are very different. At low potential, the polyamidines are electrochemically adsorbed, their STM images are similar to those of the polyamidines adsorbed in the same conditions. The images of both polymers are fitted by a base-stacked helix with a pitch of 3 to 4 nm. In contrast, the polyamidines adsorbed near the potential for zero charge give images that are fitted by an elongated helix with a pitch of 6 to 9 nm. This interpretation is confirmed by images which show a single molecule transforming from the elongated phase to the compacted phase. The transformation is followed by an azimuthal rotation of the molecule so that its alignment on the substrate is identical to that of its neighbors. These transitions may be a consequence of the changes in screening of the phosphate backbone as the effective ionic strength at the electrode surface is changed by altering the electrode potential.  

This work was supported by grants 1R21 HG00818-01A1 from the National Institutes of Health and N00014-90-J-1455 from the Office of Naval Research.  

10:00 am NSBI-ThM6 Imaging Polytenic Chromosomes with the Atomic Force Microscope, Daniel M. Jondle, Linda Ambrosio, James Vesenka & Eric Henderson, Department of Zoology and Genetics, Iowa State University, Ames, IA 50011. Polytenic chromosomes from the salivary glands of Drosophila melanogaster have been examined under a combined inverted fluorescence/atomic force microscope (AFM). This study revealed a correlation between the banding pattern apparent by fluorescence microscopy and topographic features resolved by the atomic force microscope (Fig. 1). Since the bands are continuously observed between the number of base pairs in the double-helix, the addition of the homopolymer did not change the magnitude of the adhesive force but increased the distance (up to 400 nm) between the surfaces at which the bond rupture occurs. As the surfaces are separated an exponentially increasing force is required to elongate the molecule resulting in the measure of the molecule’s elasticity. These results demonstrate that the AFM can measure intra- as well as intra-molecular forces in complex macromolecular systems.  

10:20 am NSBI-ThM7 The Topology of Supercoiled DNA by SFM Imaging, Bruno Samori, Carmelo Nigro, Innocenzo Muzzalupo, Giampaolo Zuccheri, Carla Quagliariello, University of Calabria, Arcavacata di Rende, 87030 Italy. Agarose gel electrophoresis provides a means for separating single topoisomers from a population of supercoiled pBR322 DNA molecules. The DNA electroluted from a single electrophoretic band is constituted of molecules with the same linking defect (ΔLk). Molecules with relatively high and low linking deficits are obtained from electrophoresis of native supercoiled and topoisomerase relaxed pBR322 molecules.  

We image single topoisomers with known ΔLk. The analysis of the topologically equivalent molecules extracted from the gel provides insight on the equilibrium in which supercoiled DNA is involved. ΔLk is the purely topological parameter commonly used to describe supercoiling. SFM makes it possible to characterize the supercoiling of a single molecule of the sample by a more complete parameter which describes each conformation possible, the writing number (Wr). We already reported the calculation of Wr from a SFM topography. On this basis we can determine the portion of ΔLk between the changes in the supercoiling state (Wr) and the change in the twist of the DNA strand (Tw).  

The procedure of DNA deposition on mica has been tailored in order to consider the effects of both phase transition (B to A) and high salt concentration on the original supercoiling of the DNA under investigation.  

10:40 am NSBI-ThM8 Scanning Probe Microscopy Studies of Macromolecular Interactions, S. J. B. Tendler, C. J. Roberts, P. M. Williams, M. C. Davies and D. E. Jackson, Laboratory of Biophysics and Surface Analysis, Department of Pharmaceutical Sciences, The University of Nottingham, Nottingham, NG7 2RD, UK. The scanning probe microscope (SPM) offers the ability to obtain high resolution structural information on a range of biological molecules including proteins, carbohydrates and nucleic acids. In order to obtain this data, a number of sample preparation methods have been developed, including coating the sample with a metallic layer, the use of immobilizing self-assembled monolayers and streptavidin-biotin binding systems.  

We have been utilizing the above techniques in order to investigate biomolecular interactions with the scanning tunneling microscope (STM) and the atomic force microscope (AFM). The process of self-association of short synthetic peptides to produce protein-like macromolecules has been studied in detail. STM and AFM images of protein self-assembly have been obtained at various stages of assembly, and data has been correlated with that obtained using electron microscopy. The SPM allows the study of this self-assembly process and provides information on structural features of the molecular systems that are not normally observed using more conventional microscopy techniques.  

11:00 am NSBI-ThM9 DNA Surface Attachment Schemes for Scanning Probe Microscopy, Lisa A. Wenzler and Thomas P. Bostee, Jr., Department of Chemistry, University of Utah, Salt Lake City, UT 84112. Using the thiol-gold adsorption system, we are developing reproducible methods for depositing, immobilizing and imaging deoxynucleic acid (DNA) on a supporting gold substrate for scanning tunneling microscopy (STM) and atomic force microscopy (AFM). These methods involve chemical modifications which can be made to a DNA oligonucleotide that will cause the DNA to adsorb to a suitable
surface. In addition, chemical schemes involving the modification of the surface as opposed to the DNA will be presented. This involves the use of self-assembled monolayers that contain groups at the outer interface which are known to interact with DNA. Additional sensitive methods such as X-ray Photoelectron Spectroscopy (XPS) and reflection-FITR will be presented for independent structural information, verification and quantification of the amount of DNA adsorbed. The strength of this project is the correlation between STM/AFM results and other spatially averaged traditional surface analytical techniques.

11:20 am NBS-ThM10 Studying the Dynamics of Polymer Surface Degradation by Scanning Force Microscopy, M. K. M. C. Davies, A. Domb, C. J. Roberts, A. Shard, S. J. B. Tendler, and P. M. Williams, Laboratory of Biophysics and Surface Analysis, Department of Pharmaceutical Sciences, The University of Nottingham, Nottingham, NG7 2RD, U.K. The Hebrew University of Jerusalem, School of Pharmacy, Jerusalem, Israel 91120.

The controlled release of drug molecules from surface eroding biodegradable polymer devices is an area of active research which promises many clinical and economic opportunities. Drug release from these devices occurs only as a result of polymer chain scission at the polymer-amphiphile interface. In the design of these novel devices, it is vital to understand the dynamics of polymer degradation at the interface with the polymer. The use of these dynamics has advanced by the development of in situ scanning force microscopy (SFM) which enables the polymer surface degradation to be visualized within an aqueous environment. 

For the semi-crystalline polyanhydride, poly(sebacic acid) (PSA), we have visualized the preferential loss of amorphous material resulting in the exposure of crystalline fibres and spherulites at the polymer-amphiphile interface and the pH dependence of the kinetics of surface morphological changes has been recorded. Phase separations at the surface of incompatible polymer blends composed of PSA and poly(lactic acid) (PLA) have been imaged and the differential rates of degradation of these two polymers has been visualized by SFM. These differential degradation rates result in the exposure of slowly degrading PLA regions at the surface. The successful implementation of in situ SFM in the study of surface degradation dynamics of biodegradable polymers has generated a new powerful approach to advance our understanding of erosion processes which can assist the rational design of novel degradable biomedical materials and devices.

11:40 am NBS-ThM11 In-situ Observation of the Protonation of Cytochrome c on Au(111) by Electrochemical STM, D. Lampner, T. W. Jing, J. Pan, and S. M. Lindsay, Department of Physics and Astronomy, Arizona State University, Tempe, AZ 85287.

We have studied the adsorption of cytochrome molecules onto an Au(111) 22X3 surface in NaClO4 (which does not adsorb to gold). Spontaneous adsorption on an uncontrolled electrode only occurs when the rest potential is slightly positive of the potential for zero charge. At least two phases are commonly seen, coexisting in adjacent domains. One corresponds to the cytochrome stacked on edge (edge-on) phase and the order to the cytochrome lying flat (flat-on) phase. When the substrate potential is controlled and raised to larger positive values, the edge-on phase is largely transformed into the flat phase. Furthermore, systematic variations in brightness across the unit cell that were present at low potential disappear at the higher potentials. Therefore the electrolyte is unbuffered, these effects almost reflect changes in protonation of the cytochromes at the N3 position. Chrono-coulometry measurements indicate that charge regulation may be occurring. We confirm that the changes in the form and contrast of the images are owing to protonation by examining the same area of a 22X3 surface (at molecular resolution) as the pH is lowered through addition of HClO4. We find that images characteristic of low electrode potentials in neutral, unbuffered electrolyte are found at low pH, while images characteristic of positive electrode potentials in neutral, unbuffered electrolyte are observed at higher pH. These results indicate that the protonation of a single molecule can be followed by contrast changes in the STM image.

This work was supported by grants 1R21 HG00818-01A1 from the National Institutes of Health and N00014-90-J-1455 from the Office of Naval Research.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room A207 – Session NS2-ThM

Proximal Probe Based Fabrication
Moderator: H. G. Craighead, Cornell University.


Measurement of electrical and mechanical properties of nano-scale junctions and wires is of extreme relevance in the area of integrated nanoelectronics and related technology. It has been shown recently [1] that it is possible to fabricate small nanowires using a scanning tunneling microscope (STM). Furthermore, atomic scale processes underlying the formation of intermetallic nano-contacts have been revealed via molecular dynamics (MD) simulations [2]. In this work, we present results for two regimes of nanowires produced by STM. For short gold wires (50 Å) we show clear evidence at room temperature of conductance quantization, with additional fine structure correlating with the order-disorder states of layers of atoms in the wire, predicted by MD simulations [2]. The resistance of long wires, as long as 400 Å, demonstrates the transition to a localization regime. Current versus voltage characteristics indicates gradual loss of metallic character as the wire narrows.


8:40 am NS2-ThM2 Quantized Conductance in an Atom-Sized Point Contact, F. Besenbacher, L. Olesen, E. Laegsgaard, and I. Stensgaard, Center for Atomic-Scale Materials Physics and Institute of Physics and Astronomy, Aarhus University, DK 8000 Aarhus C, Denmark.

Point-contact experiments are carried out at room temperature on single-crystal Cu, Ni, and Pt surfaces with an STM. The preamplifier is modified such that both the current and the voltage in the tip-surface junction are measured simultaneously during tip indentation and retreat from the surface. During the indentation, the tip suddenly snaps into point contact with the surface, causing a jump in the conductance. A point-contact neck is formed, and when the tip is withdrawn, the contact neck stretches. During this stretching, it is observed that for all the three metals, the conductance is quantized in units of one or two times $2e^2/h$ (77.5 $\mu$S) even at room temperature. A typical length per quantum step is $\sim 1$ Å. Exactly which quanta of $2e^2/h$ are observed differ from one indentation to the other. Recently, we have [1] combined these experiments with atomistic simulations of the contact and model calculations of the conductance based on a Landauer-Büttiker type theory. We show that the jumps in the conductance is a direct consequence of the transverse electron motion in the contact consisting of one to ten atoms only rather than of a quantization of the cross-sectional area as the contact is stretched. Experiments are in progress to use these thin wires to study stick slip motion on clean and chemically modified surfaces.


9:00 am NS2-ThM3 Nanoscale Mechanical and Chemical Processing of Surfaces: Molecular Dynamics Simulations, 1S. B. Sinner,2 Richard J. Colton, Carter T. White and Donald W. Brenner,3 US Naval Research Laboratory, Surface Science Branch, Code 6170, Washington, DC 20375-5342, Atomic force and related microscopies are powerful techniques, not just for imaging, but also for the engineering and processing of matter on the nanoscale. Computer simulations can provide important insights into the mechanisms at work in these processes. We have been using molecular dynamics simulations to study the interaction of nanometer-scale diamond tips and asperities with surfaces. Specifically, we are interested in atomic-scale indentation and scratching of diamond and metal surfaces with diamond tips. Such simulations provide in-
formation on the stability of the tip compared to the surface as a function of the size of the tip and the nature of the surface. In addition, we have modeled the selective alteration of a diamond surface by using a chemically reactive tip to abstract a surface atom. This creates an active site on the surface where controlled chemistry can take place. We propose a novel tip design to perform the abstraction without inadvertently damaging the surface or tip.

1 Supported by ONR.
2 NRC Postdoctoral Associate.
3 Address after August 1, 1994: Dept. of Materials Science and Engineering, North Carolina State, Raleigh, NC.

9:20 am NS2-ThM4 Electrical Measurements on STM Patterned Silicon MOSFETs, Ty Mayfield and T. K. Higman, Department of Electrical Engineering, University of Minnesota, Minneapolis, MN 55455.

Periodic surface potential gratings have been created in the active area of a silicon MOSFET with a scanning tunneling microscope (STM). Gratings running both parallel and perpendicular to the direction of electron flow in the channel were produced. Electrical measurements were made to determine the effect of the potential grating on MOSFET transport. The STM writing was accomplished through a selective field enhanced oxidation process on the bare silicon channel. The devices were then cleaned with a modified RCA process and subsequently gate oxidized. Atomic force microscope images confirm that the gratings translate through the oxide and gate oxide steps to provide a modulation of the gate oxide thickness. Because the thickness modulation can be made as large as 100 Å, macroscopic threshold voltage shifts exist between areas that are STM modified and those that are unmodified. A device with a grating parallel to the electron flow has a continuous conductive path from source to drain at the unmodified threshold voltage. A perpendicular grating forms pools of electrons which are electrically disconnected from the source and drain. The contrast in these electrical properties can be measured at or slightly above the unmodified threshold voltage. Data will be presented which show these effects.

9:40 am NS2-ThM5 Nanolithographic Patternning of Metal Films with the STM, C. Van Haezendonck, L. Stockman, G. Neutelens, C. Struyk, Y. Bruynseraede, Laboratorium voor Vaste-Statistische en Magnetisme, Katholieke Universiteit Leuven, B-3001 Leuven (Belgium).

We have developed a new and reliable lithographic method to pattern this evaporated gold films by locally exposing a Langmuir-Blodgett layer of an electron beam resist with the STM. The resist layer consists of only 4 monolayers of omega-tricosenoic acid. For the exposure the STM operates at 1 V with 3 nA current. The resulting voltage difference of about 10 V between the electrochemically etched Pt-Ir tip and the gold layer on top of which the very thin resist layer has been deposited. The omega-tricosenoic acid acts as a negative resist, where, after development in ethanol, the unexposed areas can be removed via argon ion milling.

We have written several fine-line structures with a linewidth down to 15 nm. The lines interconnect larger predefined contact pads, which can be used for ultrasonic wire bonding. We will present the results of our detailed study of the dependence of the fine-line quality on the exposure parameters (tunneling voltage and current, exposure dose, Pt-Ir tip quality, ...). Low-temperature magnetoresistance measurements confirm the electrical quality of the narrow gold lines. While the potentiality to use the STM lithography for technological applications is still unclear, our lithographic patterning technique certainly provides an inexpensive and flexible tool for the preparation of mesoscopic samples. As a nice application of our technique, gold contacts have been attached to small bundles of diameters of about 50 nm from carbon nanotubes, enabling to measure their resistance as a function of temperature and magnetic field.


Self-assembled monolayers are of great interest as resist materials for STM-based lithography. We have investigated the STM interaction with phenethyl-phenacat (PEDA) on Si. The molecule consists of a silane group attached to a ligating amine group. PEDA differs from Langmuir-Blodgett film forming molecules in that the silane group chemisorbs to the native oxide of Si forming a densely packed film. Low energy electron exposure exhibits the ligating action of the PEDA. A Nl film will then selectively remove this film in the unexposed regions. STM lithography on PEDA was performed in vacuum with currents between 3 pA and 1 nA, tip-sample voltages between 8 and 100 V, and tip speed of 0.2-2 μm/sec. We have examined the exposure threshold bias, dose and tip-sample bias behavior, and IV characteristics of the tip-resist Si system. We find a threshold voltage between 8 and 10 V. This threshold depends on the condition of the tip. The changes brought about by STM exposure are visible in subsequent STM imaging under below-threshold bias conditions. We have observed that the width of exposed lines (in nm) in the latent resist image is on the order of the bias voltage (in V). The tip-sample I-V curves showed that, for negative tip bias, tip-sample conductivity is sensitive to the exposure history of the PEDA. The I-V curve in both directions are in good agreement with a field emission model of tunnelling through a barrier.

10:40 am NS2-ThM8 Scanning Probe Lithography of Novel Langmuir-Schaeffer Films: Electrochemical Applications, U. Demir, K. K. Balasubramanian, V. Cammarata, and C. Shannon, Department of Chemistry, Auburn University, Auburn, AL 36849 USA.

Langmuir-Schaeffer films composed of novel rigid rod molecules have been studied as potential resist materials for use in SPM lithography. These materials are ideally suited for electrochemical applications for two reasons: first, the blocking ability of the films can be easily controlled by the number of layers transferred to the substrate; second, the fidelity of the tip induced features is high due to the mechanical properties of the film. We have used the tip of a STM to define features with lateral dimensions ranging from 1 nm to 3 μm. The depth and overall morphology of the features depend on the lithography conditions. We have studied the electrochemical deposition of Cu into the lithographically defined domains. We find that for deposits consisting of a single LS monolayer, deposition of Cu occurs over the entire surface of the electrode; however, for two or more LS layers, deposition occurs selectively at the lithographically defined features. Furthermore, we find that material deposits first at the perimeter of the holes followed by deposition into the hole itself. In addition it appears that larger holes fill before smaller ones. Other electrochemical applications of these systems will be briefly discussed.

11:00 am NS2-ThM9 Cyanide Etching of n-Alkane-thiol-Modified Au(111) Surfaces Studied by Electrochemical Scanning Tunneling Microscopy, Y. Q. Li, O. Chalilappakul, R. M. Crooks, Department of Chemistry, Texas A&M University, College Station, TX 77843-3255.

A systematic in-situ electrochemical scanning tunneling microscopy (ECSTM) study of the electrochemical dissolution of Au(111) in dilute cyanide solutions has been carried out. Atomic resolution and large scale images showing the Au(111) dissolution process were obtained. The images indicate a voltammetric potential difference of about 10 V between the electrochemically etched Pt-Ir tip and the gold layer on top of which the very thin resist layer has been deposited. The omega-tricosenoic acid acts as a negative resist, where, after development in ethanol, the unexposed areas can be removed via argon ion milling.

We have written several fine-line structures with a linewidth down to 15 nm. The lines interconnect larger predefined contact pads, which can be used for ultrasonic wire bonding. We will present the results of our detailed study of the dependence of the fine-line quality on the exposure parameters (tunneling voltage and current, exposure dose, Pt-Ir tip quality, ...). Low-temperature magnetoresistance measurements confirm the electrical quality of the narrow gold lines. While the potentiality to use the STM lithography for technological applications is still unclear, our lithographic patterning technique certainly provides an inexpensive and flexible tool for the preparation of mesoscopic samples. As a nice application of our technique, gold contacts have been attached to small bundles of diameters of about 50 nm from carbon nanotubes, enabling to measure their resistance as a function of temperature and magnetic field.


Field evaporation and current induced point heating using scanning probe microscope (SPM) for fabrication of nanometer structure on an insulator and a magnetic material are presented. We have studied them using an atomic force microscope (AFM) and a scanning tunneling microscope (STM). In AFM experiment, applying the pulsed
Self-assembled monolayers (SAMs) of thiolsates on gold open exciting new possibilities of engineering smooth surfaces with their chemical properties fine-tuned at the molecular level. This leads to new avenues of control of physico-chemical properties of surfaces. Wettability studies of mixed monolayers containing hydrophobic (CH₃) and hydrophilic (OH) terminal groups are discussed.

Covalent hexadecane for HOC(H₂)ₙSH(CH₃)(CH₂)ₙSH (n), and for HO(CH₃)ₙSH(CH₂(CH₃))ₙSH (*), mixed alkanethiolate monolayers on gold, as a function of the surface OH-concentration.

INVITED

9:00 am AS-Thm3 Probing the Phase Transition in Alkyl Thiol Assemblies on Gold Surfaces, F. Bensebaa, "T. Ellis," A. Badia" and B. Lennox, ² ¹Department of Chemie, Univ. de Montréal, Montréal, H3C 3J7, ²Dept. of Chemie, Univ. de McGill, Montréal, H3A 2K6.

In spite of the numerous experimental and theoretical investigations on self-assembled monolayers (SAM) of n-alkyl thiol, CH₃(CH₂)ₙSH, on gold surfaces, there is some uncertainty about the nature of the phase transition. Whether the disordering, believed to be the origin of the phase transition, is due to gauche defects or to random chain tilt is the main focus of our work. Thiolates, with chain length varying from n = 10 to n = 22, were adsorbed on evaporated gold films and investigated by infrared reflection absorption spectroscopy (IRAS), with and without polarization modulation, in the temperature range 140–150 K. The spectral deconvolution was performed with commercial software and the temperature dependence of the linewidth, peak frequency and integrated intensity were extracted. We will discuss our results within existing models. Relevance of IR studies on bulk n-alkanes is also discussed.


In order to determine the molecular orientation of PMDA on Pt(111) NEXAFS spectra were measured at the C1s and O1s absorption edge. For the multilayer the absence of angular variation in the resonances indicates that the PMDA molecules are oriented randomly. For the monolayer the observed strong angular variation of the σ*-resonance intensities reveals an orientation of the PMDA molecules with their ring-plane parallel to the Pt surface. Further support for this planar arrangement is provided by the substantial broadening and shift of the σ*-resonances indicating a strong interaction between the monolayer and the substrate. If all molecules in the well oriented monolayer were adsorbed in a planar arrangement, the NEXAFS spectra taken at normal incidence should show no intensity in the energy range of these resonances. In our experiments, however, a finite transition intensity of about 20% of the edge jump is observed. A careful analysis of our NEXAFS data suggests the presence of excitations into empty hybrid substrate-molecule orbitals for photon energies above the XPS-binning energy. Such transitions are expected for strongly chemisorbed molecules and have been discussed previously for benzene on Ag(111) [1]. This explanation is supported by the observation of a considerably strongly angular variation for an epitaxially grown triple layer.


8:20 am AS-Thm1 Surface Engineering Using Mixed Alkanethiolate Monolayers on Gold, Abraham Ulman, Department of Chemistry, Polytechnic University, Brooklyn, NY 11201.

Voltage to the gold thin-film covered AFM tip has made nanometer-sized gold dots of around 10 nm in diameter on a SiO₂/Si as an insulator/conducting substrate in the case of negative tip bias. On the other hand, positive tip bias has made small balls to evaporate and remove the silicon dioxide. The former is due to field evaporation of gold atoms on the insulator. This is because the bias means that negative gold ions are contributed in field evaporation and the threshold field is about ~1.6 V/Å, which agree well with theoretical results. The latter is due to current induced heating of surface. The silicon appears in bottom of the half after the voltage application. This means that the current heating makes temperature in the local area rises up to higher than 1000 C. Furthermore, we applied the current heating to make nanometer-sized magnetic domain structures in magneto-optical material by STM. Using a current imaging tunneling spectroscopy technique, we made magnetic domains with around 100 nm in diameter were made in PtCo multilayer at the voltage of less than 2V for 1 ms. It is considered that the temperature in local area rises up to over Curie point of about 400 C.

These results indicate that SPM has a potential to achieve an ultrahigh density recording and to fabricate nanometer-sized electron device patterns.

* Supported by the National Science Council of R.O.C. under grant No. NSC83-0208-M001-038.

APPLIED SURFACE SCIENCE
Room A101 – Session AS–THM

Self-Assembled Monolayers


We report the results of an ultrahigh vacuum scanning tunneling microscope (STM) study of n-alkanethiol [CH₃(CH₂)ₙSH]ₙ(Cₖ) monolayers self-assembled on Au(111) single-crystal surfaces. The STM images show that the saturation coverage self-assembled monolayer (SAM) unit cell measures 0.86 × 1.0 nm and contains four thiolate molecules (see graphic). The observed unit cell is consistent with results from recent x-ray and helium diffraction studies. This unit cell corresponds to a (4 × 2) superlattice of a basic (v3 × v3)R30° dense-packed monolayer. The domain size ranges from 5.0 to 15 nm with domains separated by molecular-width rotational boundaries, anti-phase
boundaries and stacking faults. SAMs composed of longer chain thiols (C8 and C10) were stable in vacuum for over 40 days. Short-chain homologues (C4 and C6) exhibited a 2-D liquid phase at room temperature. Facile mass transport of surface gold atoms was observed in the presence of the liquid phase using time-lapse STM movies. Nucleation and growth of ordered domains having a unit cell of p×v×3 (p=8-10) were observed for the short-chain liquid-phase SAM.


We present a study by XPS of the chemical and physical interactions of Cr with the COOH groups of a self-assembled monolayer (SAM) of HSi(CH3)3-COOH on gold. For each overlayer thickness, a new SAM was exposed to the evaporative deposition of Cr at ≤1×10⁻⁸ torr and then sublimed immediately in-situ to a surface analysis chamber held at ≤5×10⁻¹⁰ torr. Metallic Cr can be produced in this way as evidenced by a Cr 2p 3/2-1/2 splitting of 9.2 eV for 10 Å Cr films on Au. The C ls and O ls binding energies and intensities for the initial COOH-terminated SAM are consistent with the presence of the O=C-CH₃ end group at the surface. For Cr overlayers of 0.4-10 Å average thickness, we identify Cr-COOH reactions primarily by changes in the O ls level, and by comparison with the O ls spectra for Cr/CH₃ and Cr/Au films in UHV and after exposure to oxygen. For 0.4 Å Cr-COOH, the O ls peak at 532.7 eV is narrowed and located between the original C-O (533.9 eV) and C=O (532.5 eV) components, indicating a Cr/COOH reaction. In comparison, an oxidized 30 Å Cr film on Au yields large peak at 530.3 eV consistent with CrO₂, as well as a smaller peak at 532.1 eV that we attribute to surface-bound oxygen species. For 0.7-10 Å Cr/COOH, a growing second O ls component is found at 531.1-530.6 eV that may be attributed to an adsorbed oxygen species on the Cr surface (although it is 3 times greater in intensity for Cr/CH₃) or to a second, more highly oxidized form of Cr/COOH bonding.

*This work was supported by the U.S. Department of Energy under Contract DE-AC02-83CH10093.

10:20 am  AS-Thm7  Unexpected Packing of Fluorinated n-Alkane Thiols on Au(111): An Atomic Force Microscopy Study, G. Lin,¹  C. E. D. Chidsey,² D. F. Ogletree and M. Salmeron, Material Science Division, Lawrence Berkeley Laboratory and Department of Chemistry,¹ University of California, Berkeley, CA 94720; ²Department of Chemistry, Stanford University, Stanford, CA 94305.

Using atomic force microscopy (AFM), we have conducted a comprehensive study of the structure of CF₃(CH₂)₃(CH₂)₉SH (n=11, 7 and 5) on a Au(111) surface. Surprisingly, although the nearest neighbor fluorinated alkane thiol distance is very close to the lattice constant of a commensurate p(2×2) structure, the close-packed rows of molecules are rotated -30° with respect to the underlying gold lattice. This packing is incommensurate or at most only close to a high-order commensurate p(3×3) structure. Our finding is consistent with the observation of an independent x-ray diffraction experiment. The relative orientation of the organic monolayer and the Au(111) substrate has been determined unambiguously because AFM tips can reversibly displace the thiols molecules under high loads. In addition, the issues of defects and domain boundaries within these fluorinated monolayers will also be discussed.

11:00 am  AS-Thm9  Quantitative Analysis of Monolayer Molecular Films Using 'Molecule Corrals' and STM, David L. Patrick, Victor J. Cee, and Thomas P. Beebe, Jr., Department of Chemistry, University of Utah, Salt Lake City, UT 84112.

Nanometer-diameter etch pits on the Au surface plane of highly oriented pyrolytic graphite (HOPG) are used as 'molecule corrals' to study self-assembled monolayer films of organic molecules. The corrals are produced during high temperature oxidation by a method that allows precise control over their size, and are uniformly circular and one graphite layer (3.4 Å) deep. Molecular self-assembly along the bottom of pits occurs semi-independently from that on the surrounding terrace, allowing a statistical approach to the analysis of information in STM images. Rates of molecular self-assembly, active nucleation sites (step edge vs. open terrace sites), as well as the relative configurational free energies of different monolayer structures can all be determined as a function of corral size. Variable-temperature STM experiments allow measurement of nucleation activation energies and other film characteristics dependent on thermal conditions. We also present a general method for the extraction of orientational interaction energies between nearby two-dimensional crystalline domains of molecules directly from STM images of self-assembled films in etch pits.

11:20 am  AS-Thm10  In Situ AFM/STM Study of Nucleation, Growth and Electron Transfer Reactions of Purines at the Graphite-Water Interface, N. J. Tao and Z. Shi, Department of Physics, Florida International University, University Park Campus, Miami, FL 33199.

We have studied xanthine and guanine at the graphite-water interface with AFM and STM. The molecules were found to condense spontaneously into monolayer films at the interface. The nucleation and growth processes were studied by AFM in real-time as a function of the surface potential. The molecular packing structures were determined from the molecular resolution AFM images. Although the STM images of the molecules were obtained with submolecular resolution, they are not straightforward to interpret because the mixed states of the molecules and the underlying graphite were imaged. At low potentials the monolayers dissolved into the bulk solution, while at high potentials oxidations took place. The kinetics of the oxidations was studied in real-time with AFM. The reactions were observed to start preferentially from defect sites, and the reaction rates were determined from the images. Both AFM and STM images of the reaction product of xanthine were obtained with molecular resolution which allowed us to extract the molecular packing structure of the product molecules. However the oxidation product of guanine dissolved into the bulk solution which made it difficult to examine with AFM/STM. From the total amount of charge transfer and the number of reacted molecules, the numbers of electrons per molecule involved in the reactions were directly determined to be two for guanine and four for xanthine.

*This work was supported by Research Corporation and the Petroleum Research Fund of the American Chemical Society.

11:40 am  AS-Thm11  Multi-Step Assembly of Polymeric Monolayers, D. W. Grainger, G. Mao, and F. Sun, Colorado State University and D. G. Castrner, Univ. of Washington, Seattle, WA.
Self-assembly of organic silanes provides a promising method of preparing materials with well-defined surface chemistry for solid lubricants, photoresists, biosensors, biocomponents, protective coatings, etc. This study examines a three-step process that combines the benefits of monomeric and polymeric self-assembly. First 7-Octyltri-chlorosilane (OTS) is self-assembled onto a Si wafer. Then siloxane polymers were coupled to the bound OTS olefins by hydrosilylation of Si-H groups or photo-initiated side chain thiol radicals. Finally, the attached siloxane polymers were derivatized with ethylene glycol allyl ether oligomers (PEG) or a perfluoroalkyl allyl ether (FC). An alternative two-step method of directly coupling a PEG or FC derivatized siloxane polymer to the OTS monolayer was also examined. XPS, contact angle, ellipsometry, and FTR were used to characterize the film structure at each step of the processes and to determine the efficiency of each assembly step. Both processes produced monolayers that were surface enriched in either PEG or FC groups. The attachment of the PEG oligomers was more facile than the attachment of the FC chains. The amount of PEG attached depended on the degree of siloxane polymer attachment to the OTS surface and the molecular weight of the PEG oligomer. The three-step method produced films with higher PEG concentrations than the two-step method. The results from these experiments will also be compared to our previous results for multifunctional polymeric monolayers on Au.

PLASMA SCIENCE

Room A109 – Session PS-ThM

Charge Free Processing

Moderator: K. L. Maxwell, SEMATECH.

8:20 am PS-ThM1 Overview of Charge-Free Processing—Mechanisms and Applications, H. H. Sawin, Massachusetts Institute of Technology, Cambridge, MA 02139.

Charge-free processing of microelectronics is currently being considered to augment conventional plasma processes in order to eliminate damage of the devices being processed and skewing of the etching directionality caused by the deflection of ion bombardment. Many experimental means, e.g. neutral beams and atom sources, are under investigation to replace reactive ion etches and wet cleans. The first success of neutral processes is likely to be realized in cleaning applications since the rate of directional neutral etching processes is typically an order of magnitude slower than that of plasma processes. The fundamental barriers associated with pumping and surface mechanisms encountered in designing directional neutral beam systems with etching rates comparable to plasmas will be discussed. The rate of etching and pumping dictates a minimum process pressure of order 0.1 mTorr. However, at these pressures neutral beam processes with surface etching probabilities of order of 1 could have etching rates comparable to plasma processes.

The cleaning of surfaces represents a different set of challenges as less material must be removed, but that processes which are non-selective must be developed to clean the damaged/contaminated surfaces left by reactive ion etching. All cleaning products must be volatile at reasonable temperatures. The roughening of surfaces by dry cleaning and selectivities of these processes will be discussed. It is likely that multi-step dry processes will be necessary for cleaning. Proposed processes involving sacrificial oxide layers will be discussed.

9:00 am PS-ThM3 Charge Measurements and Damage Effects in the Reaction Chamber of a Remote Plasma Etcher, D. J. Bonser, P. K. Aum, T. Ta, SEMATECH, Austin, TX 78741, M. G. Blain, T. L. Meisenheimer, Sandia National Laboratories, Albuquerque, NM 87185, W. M. Holter, ASTex, Inc., Woburn, MA 01801.

Remote plasma etching is thought to produce a charge free environment in which neutral species are efficiently reacted. We have measured the charge present in an etching chamber that is approximately one meter removed from a microwave plasma discharge. In contrast to the notion that etching is accomplished in a charge free environment, both electronically excited and ionic species were detected near the wafer surface. We present results of high sensitivity, direct current measurements using both single and double probes as well as a.c. measurements in a remote etching chamber for various inert and process plasma conditions. Charge measurements are compared with damage induced in gate oxide integrity test structures. The species responsible for the charge are investigated by Fourier transform infrared spectroscopy, optical emission spectroscopy, and quadrupole mass spectrometry.


Remote plasma etching is growing interest as an alternative to conventional close-coupled plasma technology in an effort to reduce wafer charge damage. Despite the potential of remote plasma etching, little is known about the chemistry of the processes used in these reactors. We report the gas phase species in an etch chamber that is approximately one meter removed from a microwave discharge region for three different gas mixtures; N₂, CF₄/O₂/N₂, and CF₄/O₂/N₂/C₂H₆. Fourier transform infrared spectroscopy, optical emission spectroscopy, and quadrupole mass spectrometry were used to detect etch reagents and products while etching Si, N₂, and polycrystalline silicon. Sensitive charge measurement techniques were employed to measure charge in the etch chamber. All gas mixtures produced highly dissociated reagents as measured in the etch chamber. In contrast to the notion that etching is accomplished in a charge free environment, both electronically excited and ionic species were detected near the wafer surface. In situ gas measurements are directly compared to measured etch rates and the species responsible for etching are inferred.


High stripping rate is required in downstream resist stripping, even though it has the advantage of less processing damage to devices in charging and metallic or alkaline contamination. Metallic shielding plate between plasma and wafer is required to prevent charged particles from reaching the wafer surface. Stainless steel or heavy metals cause metallic contamination. So, aluminum is used as a shielding plate. Aluminum surface, however, causes recombination of radical species and reduces the stripping rate. We studied the transportation of O and OH radicals using ESR (Electron Spin Resonance) with different wall surface conditions in O₂/H₂O downstream. Radical recombination was enhanced on aluminum surface, especially after long time O₂ plasma exposure. On the contrary, recombination was significantly small on quartz surface. Thus, high stripping rate was obtained with quartz covering of aluminum shielding plate. Moreover quartz covering prevented the surface oxidation of aluminum, and high stripping rate was maintained for long time in stripping processes, and striping equipments. Temperature control of aluminum surface is also important. More radicals recombined with higher wall temperature. We believe that build up of water molecules on the surfaces prevented sticking and recombination of radicals at low temperature. We also discuss the effects of residual charged particles or radiation on the wafer surface.

10:00 am PS-ThM6 UV-Stimulated Dry Stripping of Silicon Nitride Films, David C. Gray, Jeffrey W. Butterbaugh, C. Fred Hitt, FSI International, Chaska, MN 55318.

UV light-stimulated processes have been developed for rapidly stripping films of silicon nitride, silicon oxynitride, silicon, and composite films thick enough to be photographed in a dry reagent environment, free of plasma or plasma etchants. These processes are carried out in vacuum reactor which allows simultaneous exposure of a substrate wafer to a polyatomic halogen gas and UV radiation. Silicon nitride stripping rates in excess of 1000 Å/min have been demonstrated for UV-stimulated fluorine-based processes, while maintaining the bulk wafer temperature below 250°C. It has been shown that the mechanism for UV-stimulated silicon nitride etching requires direct photon exposure of the etching surface. Selectivities between silicon nitride, silicon oxide, and silicon films are controlled through UV lamp exposure, substrate temperature, and addition of oxygen, nitrogen, and various halogen-containing gases. Selectivities for silicon nitride to silicon oxide etching of greater than 30 can be achieved for the stripping of silicon nitride LOCOS masks layers in the presence of field oxide and pad oxide layers. These processes address many of the limitations of plasma-downstream etch tools for dry silicon nitride stripping, including complete elimination of charged particles and sputtered contaminants associated with plasma etchants.
11:00 am PS-ThM9 Hyperthermal Neutral Beam Etching, K. P. Glapin and T. A. Moore, Division of Chemistry and Chemical Engineering, and Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California 91125.

Fast neutral beams of reactive species have been suggested as an inherently better means for the anisotropic etching of semiconductors due to minimalization of bombardment-induced damage and complete elimination of charge damage. However, difficulties associated with the generation of high fluxes of neutral reactive species with hyperthermal (2 eV) kinetic energy have hindered progress towards assessing their potential for etching. We report here on the generation of beams of neutral fluorine atoms with translational energies between 2-10 eV and their use in the anisotropic etching of silicon. The fluorine atoms are produced by laser-induced detonation of SF\textsubscript{6} in a nozzle, then collimated and brought onto the etched wafer. For room temperature Si, we measured 7:1 anisotropy ratios and 30:1 selectivity to SiO\textsubscript{2} while the photoresist mask underwent no erosion whatsoever.

The ability to control the translational energy of the incident beam in the hyperthermal range (2-10 eV) facilitated the investigation of the dynamics of fluoride atom scattering from the fluorinated Si surface during steady-state etching. A rotatable quadrupole mass spectrometer was used in a Time-Of-Flight mode of operation to angularly resolve the flux and energy distribution of fluoride atoms after the collision with the etched surface. A low (3.2 eV ± 0.4 eV) and a high (6.4 eV ± 1 eV) incident translational energy have been employed. It is found that, for both energies, the flux-weighted angular distribution of inelastically scattered fluoride atoms does not follow a cosine law but it rather peaks near the specular direction. More remarkably, for wide incident angles, the unreacted fluoride atoms scattering in the specular direction may retain up to 60% of the incident energy. This wide-angle scattering mechanism dramatically affects profile evolution and explains phenomena such as microetching and reverse microloading.


The anisotropic etching of electronic materials presently relies upon charged particle bombardment to produce damage sites that react with suitable adsorbed chemical reactants. This technique of reactive ion etching has gained widespread acceptance for the mass production of large-scale integrated circuits and is routinely used in the manufacture of memory and microprocessor chip having feature sizes down to 0.5 μm. The desire to put more functionality onto a chip requires that feature sizes smaller than 0.5 μm be manufactured with high reliability and yield. This drive for smaller feature size requires that process induced damage be minimized in order to maximize production yield. The use of fast neutral beams as the anisotropic reactant eliminates essentially all of damage mechanisms associated with ions. We have performed some preliminary atomic oxygen etching studies that show striking differences between ion and neutral beam processes. Feature size of 0.3 μm has been etched in photoresist polymers using high kinetic energy atomic oxygen with an etch rate of \approx 1800 Å/minute at an incident O-atom flux of 10\textsuperscript{17} AO/s/cm\textsuperscript{2}. Our results in etching photoresists and other polymers shows that the reaction of the fast O atoms is direct. At a translational energy of 5 eV, roughly 10% of the incident flux reacts to form the volatile products water, carbon monoxide, and nitrogen oxides. Reaction rates for various polymers are independent of the polymer structure as the polymer only contains carbon, hydrogen, oxygen, and nitrogen. The reaction rate depends strongly on the incident energy of the O atoms, and thermal O atoms react at a negligible rate (approximately 10\textsuperscript{-4} that of 5 eV atoms). This direct reaction of fast O atoms with the polymer is unprecedented and is the key to fast neutral anisotropic etching. Extension of these concepts to anisotropic etching of silicon, GaAs, and other materials is presently being investigated and will be discussed.

ELECTRONIC MATERIALS/SURFACE SCIENCE

Room A102 – Session EMSS-ThM

Semiconductor Surface Reactions III

Moderator: J. R. Engstrom, Cornell University.

8:30 am EMSS-ThM1 Sputtering of GaAs(110) Studied with Scanning Tunneling Microscopy, R. J. Pechman, X.-S. Wang, and J. H. Weaver, University of Minnesota, Department of Materials Science and Chemical Engineering, Minneapolis, MN 55455.

Ion bombardment is a common surface processing technique that is important in sputtering and ion-assisted etching applications. Scanning tunneling microscopy (STM) provides a atomic-scale view of the result of such bombardment. We have used STM to investigate material removal from GaAs(110) via Ar\textsuperscript{+} and Xe\textsuperscript{+} bombardment, spanning the range from initial impact on a pristine cleaved surface through multilayer roughening. Low energy ions (300-3000 eV) create defects that are largely confined to one or two unit cells. Diffusion of the vacancies created in this way produces two-dimensional single-layer-deep pits in a manner analogous to island formation during growth. Temperature-dependent studies of vacancy island nucleation yields the activation energy for diffusion. Continued sputtering results in temperature-dependent roughening that relates to interlayer diffusion. Finally, sputter yields have been determined as a function of ion energy and incident direction.

8:40 am EMSS-ThM2 Atomically-Resolved Decomposition of Phosphine and Formation of P-terminated Layers on Si(001), R. J. Hamers and Y. Wang, Dept. of Chemistry, University of Wisconsin, Madison, WI 53706.

We have investigated the thermal decomposition of phosphine and the formation of phosphorus-terminated layers on Si(001) using scanning tunneling microscopy (STM) and tunneling spectroscopy. While at room temperature PH3 adsorbs as molecularly, as elevated temperatures PH3 decomposes to P and H atoms. The phosphorus atoms are readily incorporated into the outermost layer of silicon, ejecting silicon atoms onto the terrace and allowing the P atoms to mix with Si in the outermost dimerized layer. The resulting surface is an alloy of Si-Si, Si-P, and P-P dimers. Surprisingly, a statistical analysis of the images reveals that Si-P heterodimers are found more often than expected on the basis of random mixing, despite the fact that Si-P heterodimers
have a “dangling bond.” At higher coverages where P-P dimers predominate the surface reveals strain-induced defects. The role of lattice strain and Si-P chemistry in determining the chemistry and morphology of the surface will be discussed.

This work is supported in part by the U.S. Office of Naval Research and by the National Science Foundation.

9:00 am  EMSS-ThM3  Semiconductor Etching with Halogens: Scanning Tunneling Microscopy Investigations, J. H. Weaver, Department of Materials Science and Chemical Engineering, University of Minnesota, Minneapolis, MN 55455 USA.

This talk will review recent progress made in understanding surface etching of Si(100)-2×1 and GaAs(110), particularly as offered through in situ scanning tunneling microscopy investigations. These studies have involved exposure of the surface to controlled fluxes and fluences of Br2 and Cl2 as a function of temperature. The results show that ordered removal of surface layers can be achieved under suitable conditions. They make it possible to compare phenomena related to material removal (etching) to those associated with overlayer formation (growth) and offer insights into surface chemical pathways and the dynamics of etching.

INVITED

9:40 am  EMSS-ThM5  Analysis of Nanometer-Scale Surface Roughness with Reflection Inelastic Electron Scattering from Silicon Surfaces, H. N. Fraser, H. A. Atwater, S. S. Wong, and C. C. Ahn, California Institute of Technology, Pasadena, CA 91125.

In this paper, we demonstrate how inelastic electron scattering measurements can be used to complement reflection high energy electron diffraction (RHEED) to obtain information about nanometer-scale surface roughness. Reflection electron energy loss spectroscopy (REELS) at 25 keV has been employed to investigate nanometer-scale roughness via low-loss (0-80 eV) inelastic electron scattering from silicon (001) surfaces. Surfaces with roughness length scales from the atomic scale to microns were produced by microfabrication and epilaxial growth. Roughness was quantified from scanning electron and atomic force microscopy images. REELS measurements at the loss region, which are dominated by surface and bulk plasmon scattering, were performed as a function of incidence angle and surface roughness was correlated with the extent of multiple inelastic scattering. While the inelastic scattering spectra are complicated functions of roughness and incidence angle, several conclusions can be made about roughness based on trends apparent in the relative bulk and surface plasmon intensities. First, very smooth surfaces with only atomic-scale roughness exhibit considerable multiple plasmon scattering and significant surface plasmon contributions at typical RHEED incidence angles. Second, as-polished silicon surfaces, and surfaces intentionally roughened by microfabrication exhibit reduced multiple inelastic scattering as compared with very smooth surfaces. A method for fitting experimental inelastic intensity distributions, and correlations with roughness, will be discussed.

10:00 am  EMSS-ThM6  Sub-surface Diffusion of Ni on Si(100) and (111), M. Y. Lee and P. A. Bennett, Box 871504, Physics Dept., Arizona State University, Tempe, AZ 85287-1504.

We directly compare the rates of surface vs. bulk diffusion for Ni on/in Si by depositing a laterally confined dot of Ni on one side of a double-polished and UHV cleaned Si wafer and measuring the lateral Auger profile on the reverse side following Ni deposition. Ni reaches the far side of the wafer at temperatures as low as 500°C via bulk diffusion with no measurable contribution from surface paths. This is attributed to the relatively high product of solubility and diffusivity for Ni in bulk Si. In related experiments with a uniform lateral dose of Ni, we find that surface phases (a×1 ring-cluster structure and an ordered 1×9 structure) disappear by solution of Ni into bulk silicon. For low initial coverages, the 1×7 structure can even be recovered. Virtually all the metal so dissolved can be restored to the surface (both top and bottom) by quench cooling, driven by the vertical temperature gradient. The role of these diffusion and segregation phenomena in silicide reactions is described.

10:20 am  EMSS-Thm7  Chemical Vapor Deposition of TSi3 using SiH3 and TiCl4, M. A. Mendicino, R. P. Southwell, and E. G. Ostlund, Department of Chemical Engineering, University of Illinois, Urbana, IL 61801.

TSi3 has been the object of considerable study because of its low resistivity among the transition metal silicides and its compatibility with existing ULSI technology. However, CVD of TSi3 from gas phase SiH4 and TiCl4 is accompanied by a competing reaction which consumes intolerable amounts of the Si substrate. The reaction mechanism for TSi3 growth is poorly understood, and some disagreement even exists about the reaction stoichiometry. The combined CVD/UVH approach we have developed fills many gaps in the current understanding of TSi3 CVD. Our approach is unique in that it attempts to produce optimal processing conditions from surface reactivity measurements under well-characterized ultrahigh vacuum conditions. These predictions are then tested directly in a CVD chamber, connected to the ultrahigh vacuum system, that operates at normal processing temperatures and pressures. Special effort centers on understanding and controlling the first stages of TSi3 film growth on the silicon surface. In addition to tackling these problems in the particular case of TSi3 growth, we are developing a framework of chemical reasoning that will be widely applicable in deposition technology.


Tetraethoxysilane (TEOS) is used in many microelectronics processes for the deposition of conformal SiO2 films. Good conformality is achieved only if the surface chemistry is controlled by the appropriate rate-limiting step during deposition. Therefore, to provide process reliability and improved methods for TEOS-based SiO2 CVD, it is essential to understand the rate-limiting step and its dependence on process conditions.

To improve the molecular-level understanding of both the overall rate-determining step for SiO2 deposition and the transition state for initial chemisorption, we have used FTIR and isotopically labeled species to measure the intrinsic kinetics of the TEOS reaction on SiO2. Importantly, we have performed these measurements in a cold-wall reaction reactor under carefully controlled conditions that pertain to TEOS-based CVD processes (10 to 100 mTorr TEOS at 765 to 1200 K).

We have determined that the Ea for the initial chemisorption step is only 6 kcal/mol between 900 and 1200 K. Nevertheless, the initial chemisorption probability is 3×10-7 and decreases to lower values as the reaction proceeds. In addition, there is a continuous build-up of ethoxy groups on the surface. This suggests that the rate of ethoxy decomposition to SiO2 and silanols may be insufficient to keep up with the rate of TEOS chemisorption under steady-state CVD conditions. If the ethoxy build-up is responsible for the observed decrease in the TEOS chemisorption probability, this may indicate that ethoxy decomposition is the overall rate-limiting step in SiO2 CVD. Our results also show that silanols are consumed by TEOS surface reactions at CVD temperatures and yet their steady-state concentration remains constant as a result of ethoxy decomposition. Although surface silanols are consumed during chemisorption, TEOS reactions with Si-OH and Si-O with equal rates at 765K. This suggests that proton transfer is not the rate-limiting step in the chemisorption transition state.

This work was performed at Sandia National Laboratories under DOE contract DE-AC04-76DP00789.

11:00 am  EMSS-ThM9  Investigation of the Chemical Mechanisms of Ta/Ta-oxide Halogen Etch Selectivity, L. A. Delouise, Xerox Corporation, 800 Phillips Road, 114-41D, Webster, NY 14580.

XPS and LEED techniques are used to investigate the interaction of a Cs2+ supersonic molecular beam with a Ta(110) surface as a function of halogen concentration. The starting surface is an ordered submonolayer oxide with ~1/3 monolayer coverage. Surface oxygen originates from a bulk contamination. Low and high 0 is binding energy components are found and associated with Ta-+ and TaO2+ oxidation states, respectively. The oxide states are unequivocally populated and studies show they also exhibit an inequivalent reactivity toward chlorine. Three temperature regimes were distinguished in terms of the reactivity of the surface towards chlorine. At room temperature chlorine readily adsorbs on the Ta(110)-suboxide surface causing further oxidization of Ta sites. Between room temperature and 300°C a chemical state selective etching reaction involving only oxygen bonded in Ta2+ sites occurs. LEED studies show the initial Ta-oxide overlaver is slightly deordered along the (100) direction and that etching preferentially depletes oxygen bonded in Ta2+ disordered domains. Between 300°C and 650°C surface states are passivated by Cl overlaver domains of p(4×1) orientation in which a slight disordering along the (110) site stacking direction is observed. Above 650°C chlorine is thermally depleted and surface segregation of bulk oxygen contamination occurs. Oxygen segregation occurs at 400°C in the absence of Cl. At room
It is known that the reaction of gaseous ammonia with the (110) surfaces of GaAs and InP produces non-dissociative chemisorption, but important details concerning the adsorption geometry and local electronic structure, as well as the extent of surface etching, are not well understood. These issues are relevant to such complex processes as the Atomic Layer Epitaxy of nitride compounds (e.g., GaN), and to continuing efforts to grow suitable insulating nitride layers for compound semiconductor device applications.

We have examined the adsorption of submonolayer amounts of ammonia on the (110) surface of p-type GaAs with scanning tunneling microscopy (STM) and spectroscopy (STS), and find two chemisorption-related features whose structure is attributed to the perturbations in substrate surface atoms rather than to molecular orbitals of the adsorbate.

The first structure appears as two symmetrically raised in-chain arsenic atoms whose mutual gallium neighbor is either depressed or enhanced depending on the nature of the probe tip. This structure is accompanied by asymmetric screening similar to that expected for a tilted dipole above a dielectric surface. Simple chemical arguments, symmetry, and the dipole moment of NH₃ suggest that these structures reflect ammonia bonded to gallium atoms. The second structure appears as an arsenic vacancy along with ammonia adsorption at one or both of the adjoining gallium sites. Since these defects occur with greater frequency than the native vacancies on clean GaAs(110), they imply that surface etching of arsenic atoms can occur along with adsorption.

Preliminary spectroscopic measurements indicate that ammonia adsorption is also associated with a shift of the surface Fermi-level from below the valence band edge (on degenerate material) into the band gap, and the relationship of this observation to chemisorption-induced charge transfer and surface states will be addressed.

Both of the layered semiconductors, SnₓSᵧ, and SnₓSeᵧ, are possible materials for photovoltaic solar energy conversion. In window-absorber structures SnₓSᵧ (bandgap: 1.1eV) could be used as the absorber whereas SnₓSeᵧ (bandgap: 2.2eV) is promising for use as a window material. The growth of heterojunctions of these materials by straightforward MBE techniques is therefore quite attractive. In this paper we describe the epitaxial deposition of SnₓSᵧ and SnₓSeᵧ on several other layered semiconducting substrates. RHEED and LEED were used to demonstrate ordered layer growth of these materials, and XPS/UPS studies were used to confirm stoichiometry, sharpness of the interfaces formed, and band edge positions at the interface. STM investigations of these materials suggest that, although the electron diffraction techniques indicate layer-by-layer growth, the actual surface layers formed vary considerably in their long range order, and in the mechanism of film growth.
inhibited and evidence for formation of 3D islands in the early stage of growth followed by transition to an amorphous phase was observed. However, AFM results of films of 200 Å thick showed that the surface of films deposited at 300 °C and below is smooth and featureless. On the other hand, the surface of films deposited at 350 °C and above is characterized by islands of typical height and diameter equal to 2.5 and 100 nm, respectively, and the density of these features increases with temperature. The effects of substrate temperature, contamination, and surface damage on the growth mechanisms are discussed.

9:40 am  TFVM-ThM5  Formation of Thin C-N Films by Ion Beam Deposition, K. J. Boyd, D. Marton, S. S. Todorov, A. H. Al-Bayati, J. Kulik, and J. W. Rabalais, Department of Chemistry, University of Houston, Houston, Texas 77204-5641.

Thin carbon-nitrogen films have been deposited by direct impingement of 5-50eV C" and N" or N' ions on surfaces, using the dual-source ion beam deposition system at the University of Houston. The influences of ion energy, N and C arrival rates, and substrate have been studied. The deposited films are essentially amorphous, with some graphitic regions on a scale of 1-20 nm. The films are analysed by in situ Auger electron spectroscopy (AES) and excite X-ray photoelectron spectroscopy (XPS). Both AES and XPS show two distinct types of C-N bonding: one assigned to a graphite-like local structure (C-N bonding), with stoichiometry varying from CN to C2N, and one attributed to a C-N-like local structure (C-N bonding), with stoichiometry very near C2N. Overall nitrogen fractions of up to 0.47 are obtained. No phase evolution with increasing ion fluence is observed, although the stoichiometry of the graphite-like phase does increase with ion fluence up to an energy dependent steady-state value. Initial growth proceeds through a mixed nitride-carbonitride layer. Film growth can be described by a subplantation model, with little or no interconversion between the two phases. The merits of IBD as a model for other film growth techniques utilizing energetic species are discussed, and implications of this work for C-N film growth by other methods are mentioned.

10:00 am  TFVM-ThM6  Energetic Condensation Using Filtered Arc Evaporation, P. J. Martin, A. Bendavid, T. J. Kinder, X. Wang, CSIRO Division of Applied Physics, Lindfield, NSW 2070, Australia.

The cathodic arc is an intense source of low energy particles which may be separated from the associated microdroplets of cathode material by a simple magnetic plasma duct. The resulting filtered energetic particle beam may then be deposited onto a substrate to produce smooth, dense films. Oxide and nitride films are formed by condensation of metal ions in the presence of reactive gases. High quality oxide films are readily deposited at ambient temperature with good optical properties and when deposited at substrate temperatures, have significant optical anisotropy and selectivity. Such coatings have practical applications in thermal control of solar radiation. Hard, smooth and adherent coatings of TiN are deposited onto heated substrates. The stress and hardness of the deposited layers is determined by the degree of negative bias applied to the substrate which in turn controls the energy of the depositing ions. The properties of the depositing films can be influenced by the current bombardment with low energy (0.5-1 keV) inert or reactive ions. Ion assisted arc deposition (IAAD) enables the deposition of hard materials such as amorphous diamond-like carbon, carbon nitride and TiN onto unheated substrates. TiN may be synthesized by nitrogen bombardment of condensing Ti on Ar or Xe ion bombardment of Ti in a nitrogen background. IAAD provides a controllable method of modifying the stress in the growing films, to a C-N-like local structure and the requirement of raising the substrate temperature significantly. TiN films prepared by IAAD show a strong (220) orientation and have hardnesses up to 3700 Hv. Films deposited onto Si show a stress reduction from around 9 GPa to 5 GPa by IAAD.


Ionized Sputtered Deposition (MSD) has been developed as a means of depositing metal and compound films from highly ionized metal discharges. The technique uses post-ionization of the metal ions sputtered from a conventional magnetron cathode in an RF-Inductively-coupled (RFI) discharge situated in the region between the cathode and the sample. Metal ions are then accelerated across a sample sheath by means of a DC potential on the sample. The primary advantages of this deposition technique are the high level of control of the depositing metal kinetic energy, the controlled directionality of the deposit, and the ability to drive various surface reactions (e.g. Ti2N) at low temperature. The technique has been used successfully for microelectronic applications to deposit diffusion barriers (liners) as well as fully filled trenches and vias of moderate aspect ratio at sub-6.5 micron dimensions. The deposited films have been examined using plain view and cross-sectional TEM, X-ray diffraction and RBS and compared to films deposited with Collimated Magnetron Sputter Deposition (CMSD) and conventional sputtering. The IMSD AlCu films are more highly oriented than the CMSD films, with virtually no Ar contamination. The stress was slightly tensile for the IMSD films and weakly compressive for CMSD films. Ti-nitride films were also measured.

INVITED

11:20 am  TFVM-ThM10  Further Perspectives on Stresses in Sputter-Deposited Thin Films, D. W. Hoffman, Advanced Modular Power Systems, 4667 Freedom Dr., Ann Arbor, MI 48108.

In a recent review the author gave a perspective on the principal findings of John Thornton and himself in their survey of stresses and properties of magnetron sputtered thin films. The present talk picks up where the previous review left off by describing some of the more novel and significant spin-offs that evolved from that central body of work. Among the topics of interest, special attention is given to a novel device for in-situ measurement of film stresses, which appears to solve the problems of previous in-situ stress instruments and offers the possibility of a work-a-day film stress monitor that could be employed in much the same way as, and perhaps in tandem with, a quartz crystal thickness monitor.

INVITED

ELECTRONIC MATERIALS
Room A108 - Session EM-ThM

Optical Diagnostics for Materials Processing
Moderator: P. Herman, Columbia University.


Spectroscopic ellipsometry's (SE) strength as an in-situ semiconductor crystal growth process diagnostic lies in the fact that the material's dielectric properties (pseudodielectric functions) are dependent upon temperature and alloy composition. These dependencies can be exploited to monitor and control epitaxial growth in real-time. This paper reviews several aspects of growth and control of II-VI semiconductor grown by molecular beam epitaxy (MBE) and gas source MBE (GSMBE) using SE.

Chamber and manipulator design considerations for implementing SE on an MBE and GSMBE with substrate rotation will first be discussed. A commercially available "ellipsometer-ready" MBE system will be discussed.

It will then be shown that an MBE process can be established without conventional analysis tools such as RHEED. Measurement and control of substrate temperature by SE will be shown. This is possible by using the temperature-dependent pseudodielectric functions for GaAs and AlGaAs which we have measured. Calibration of MBE growth will be discussed including in-situ measurement of oxide desorption, V/III flux ratio, growth rate and alloy composition measurements. Tracking the growth of the epitaxial layer provides information on alloy composition and surface roughness. Algorithms for deconvoluting these effects from the data will be discussed.

Finally, examples of structures used in optoelectronic devices will be presented including quantum well thickness control and microcavity laser device growth.

INVITED

9:00 am  EM-ThM2  Real-Time Monitoring of Resonant Tunneling Diode Growth Using Spectroscopic Ellipsometry, F. G. Celli, Y.-C. Kuo, W. M. Duncan, T. S. Boise and A. J. Katz, Corporate Research & Development, M/S 147, Texas Instruments, P.O. Box 655916, Dallas, TX 75265.

Sensor-based growth control appears to be necessary to insure manufacturability of quantum devices, such as resonant-tunneling diodes (RTDs), due to the inherent sensitivity of the devices to monolayer
changes in layer thickness. We are applying spectroscopic ellipsometry (SE) to the MBE growth of AlGaAs/GaAs, with the eventual goal of SE-based control. SE provides a versatile and sensitive method to monitor wafer-state properties, such as layer thickness and composition, in real-time.

We acquired SE data from rotating wafers during MBE growth of single and multiple RTDs. The active regions consisted of 50 Å wells, including a 20 Å thick InGaAs barrier layer. Layer thicknesses were derived from real-time data fits to the SE spectra using growth-temperature optical constants (determined in the same MBE/SE system). Effusion cell flash and surface roughness data were obtained independently by reflection mass spectrometry and laser light scattering, respectively. Correlations between electrical I-V curves of the processed devices and the in situ sensor data will be discussed. We have found that the effects of multiple layers (AlAs or InAs) relaxation on the I-V curves depended on the (tensile or compressive) nature of the strain, and can be distinguished with in situ monitoring. Prospects for SE-based real-time control of RTD growth will be addressed.


SiGe, heterostructures and multi-quantum wells are being studied for applications as heterojunction bipolar transistors (HBT) and far-IR detectors. For the growth of these structures to a tight specification, in-situ optical monitoring and control is required. This paper reports the first results of RTSE obtained during Si1-xGex/Si vapour-phase epitaxial growth. Spectra were obtained using a SOPRA RTSE system which uses an optical multi-channel analyser to provide typically ten Ψ, cosΔ spectra at 256 energies over the range 1.5-4.7 eV in 1 sec.

RTSE has been used to study all stages of HBT growth: surface oxide desorption at ~900°C, B-doped Si0.05Ge0.95 (p-base) growth at 610°C, As-doped Si (n-emitter) growth at 700°C. Spectra have been obtained as a function of temperature for the substrate initially covered by 10 Å oxide and after desorption, which was clearly observed at ~850°C.

The Ψ spectra of Si0.05Ge0.95 and Si at 610°C are dominated by E1 peaks near 2.95 eV and 3.1 eV, respectively. In this region the alloy layers become optically thicker after ~300 Å deposition. Below this thickness the observed spectra are a convolution of layer and substrate spectra, and the peak shifts to higher energies and to energies below 2.9 eV before approaching the bulk alloy value. Multilayer modelling shows this behaviour to be consistent with growth of a uniform alloy layer. Si-rich regions with thicknesses of a few monolayers have been detected at hetero-interfaces and in alloy layers grown with growth interruptions. Beyond these initial regions growth rates are uniform for undoped and B-doped Si0.05Ge0.95 and for undoped Si, but for As-doped Si decrease with time due to As surface accumulation.

9:40 am EM-ThM5 Real Time Investigation of Nucleation and Growth of Si on SiO2, Using Silane and Disilane in a Rapid Thermal Processing System, Y. Z. Hu, D. Diehl, C. Y. Zhao, Q. Liu and E. A. Irene, Department of Chemistry, University of North Carolina, Chapel Hill, NC 27599, USA; and C. Christensen and D. M. Tatum, Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC 27695.

Systematic studies of the nucleation and film growth of Si on amorphous SiO2 covered Si using rapid thermal chemical vapor deposition from SiH4 and SiH4 were performed at temperatures between 600 and 850°C and reactant gas (5% in He) pressures between 20 and 500 mTorr. Quantitative assessment of the nucleation parameters (incubation time, mean nuclei density, growth rate and coalescence) have been determined by a numerical analysis of in situ real time single wavelength ellipsometry measurements. Optical properties of polysilicon films have been investigated using in situ spectroscopic ellipsometry in the 2.5-5.0 eV photon energy range. A Bruggeman effective medium approximation for the ellipsometric data reveals that the rough surface overlayers and microstructure of the deposited films depend significantly on the substrate temperature, reactant gases and their partial pressures. Atomic Force Microscopy was used ex-situ to observe the initial stage of nucleation and the roughness of the films. The structure of the deposited Si films were characterized by cross-sectional transmission electron microscopy.


Processing of III-V materials by high density plasmas has been extensively studied in the electron cyclotron resonance (ECR) tool, where a CH2/He/Ar gas mixture has been shown to etch GaAs, AlGaAs, InP, AlInAs, and InGaAs. Accurate endpoint detection, overall process monitoring and control of the etching of multilayer stacks is essential to reproducible fabrication of devices. Spectroscopic ellipsometry is a nondestructive optical technique that can determine thicknesses and compositions in multilayer stacks of III-V materials. Recent advances in ellipsometric data acquisition and processing enable us to accurately determine the etching rates of 44 wavelengths simultaneously in real time. A rapid analysis algorithm extracts the top layer film thickness and allows process monitoring.

In-situ ellipsometry was employed to monitor the ECR etching of diamond-like carbon, AlGaAs/GaAs structures, and bulk GaAs in real time. Since the film thickness in real time is determined, a wide range of etch parameters can be investigated without changing samples. This allows rapid optimization of the etch rate, and provides endpoint detection to within a nanometer, in the face of a time dependent etch rate.

10:20 am EM-ThM7 Bulk Ordering and Optical Anisotropy of In0.5Ga0.5As/InP, E. As, D. E. Aspnes, K. Hingerl, I. Kamiya, L. T. Florez, B. Poland, J. S. Majewski, S. Mahajan, and J. P. Harbison, Red Bank, N.J. Physics Dept., NCSU, Raleigh, NC; Materials Science Dept., CMU, Pittsburgh, PA.

We report the first real-time observation of atomic ordering during epitaxial growth, specifically of In0.5Ga0.5As lattice-matched to InP. Reflectance-difference (RD) spectra of thick layers of In0.5Ga0.5As grown at 335 °C show extra derivative-like structure in the vicinity of the E1, E1 + Δ1 transitions near 2.5 eV relative to RD spectra for In0.5Ga0.5As grown at 450°C. For the lower-temperature material, both RHEED patterns during growth and TEM micrographs after growth exhibit threefold periodicity, consistent with results recently reported for AlInAs by Gomyo et al. (Phys. Rev. Lett. 72, 673 (1994)). If the ordered phase is grown on the disordered phase or vice versa, the 2.5 eV RD signal exhibits interference oscillations with increasing thickness, thereby unequivocally relating the extra structure to bulk anisotropy. Post-growth ellipsometric spectra of ordered material show that this extra structure is due primarily to an increased broadening of the dielectric response for polarization along (110). This is consistent with a simple model where the increased broadening is attributed to fluctuations in the E1 and E1 + Δ1 absorption edges that result from intraband coupling (zone folding) driven by the superperiodicity. These fluctuations occur whether or not the ordering is complete. The results show how, and under what conditions, ordering in the bulk can be assessed by optical measurements during growth.

*Work supported by the Office of Naval Research.

10:40 am EM-ThM8 Monitoring of Deposition and Dry Etching of Si/SiGe Multiple Stacks, B. Tillack, R. Ritter, H. H. Richter, A. Wolff, G. Morgenstern, C. Egg*; Institut für Halbleiterphysik der Technischen Universität Berlin, Germany.

Reflection supported interferometry (PYRITTE) has been used for the in situ observation of deposition of Si1-xGex/Si multiple stacks by Rapid Thermal Chemical Vapor Deposition (RTCVD). The thickness and optical parameters of each of the thin films of the stack have been evaluated during the deposition process. During patterning of the so prepared Si1-xGex/Si stacks by reactive ion etching (RIE), the nonselectivity of the etching process has been studied by optical emission spectroscopy (OES). The monitoring of the 261.1 nm Ge line provides a sensitive and accurate indicator which allows an etch stop within any of the thin films of the stack.

TEM has been used to analyse the structure and thickness of the Si1-xGex/Si multiple stacks after deposition. The composition of the surface layer formed as a result of RIE was examined using X-ray photoelectron spectroscopy (XPS).

The results demonstrate for the first time an opportunity for in situ investigation and control of deposition and patterning of Si1-xGex/Si multiple stacks.
11:00 am EM-ThM9 Observation of Etching Reaction Using Second Harmonic Generation, S. Haraichi, F. Sasaki, S. Kobayashi, M. Komuro, T. Tani, Electrotechnical Laboratory, Tsukuba-shi, Ibaraki 305, JAPAN.

The understanding of atomic scale mechanism in dry etching process is very important for the next microfabrication technology. We have investigated the basic characteristics of surface second harmonic generation (SHG) in the Si etching to pursue the possibility of time-resolved observation of etching reaction. A Nd:YAG laser at 1.064 μm was used as the fundamental incident light which was focused on the sample at 45° incident angle. The surface SHG output was detected by a photo-multiplier and a gated integrator. Figure shows SHG in the steady state for Si/XeF₂ system. The suffix of SHG indicates the combination of input-output polarization and the p- and the s-polarization mainly reflect dangling-bond and back-bond dipoles respectively. Both SHG + and SHG - signals quickly decay with the etching because of the formation of SiF₄ layer and approach to the values independent of XeF₂ pressure. Fluorination atom probably does not attack only dangling-bonds but also back-bonds in the time duration observed here and the thickness of SiF₄ layer is independent of XeF₂ flux in the steady-state of the etching.

Epitaxial growth of III-V semiconductor materials is probed in situ in a molecular beam epitaxy reactor by single photon ionization of the gaseous fluxes using vacuum ultraviolet (VUV) laser radiation. The ninth harmonic of the Nd:YAG laser is used to ionize the species of interest. This radiation is produced by frequency tripling the Nd:YAG laser output to 355 nm and then to 118 nm in a Xe/Ar mixture. Coupled with time-of-flight mass spectrometry, this radiation selectively probes the gaseous fluxes of Ga₂, As₂, and As₃ during molecular beam epitaxy of III-V materials. The essential aspects of the method and details of calibration procedures to obtain relative flows are described. Cracking of the arsenic species does not occur in the laser/mass spectrometer, making relative species concentration measurements very reliable. Rapid data acquisition provides real time measurements of the incident and scattered fluxes and of materials desorbed during growth. Recent work to correlate the spectrum with reflection high energy electron diffraction (RHEED) oscillations during GaAs epilayer growth will be discussed.

2Staff Members, Quantum Physics Division, National Institute of Standards and Technology.

MANUFACTURING SCIENCE AND TECHNOLOGY
Room A110 – Session MS-ThM

Micro-Contamination and Defects
Moderator: A. C. Diebold, SEMATECH.

MS-ThM1 ABSTRACT NOT AVAILABLE

9:00 am MS-ThM3 An in situ XPS Study of Metal Surface Recontamination and Hollow Cathode Plasma Cleaning, H. Li, A. Belkand, Z. Orban, BOC Group Technical Center, Murray Hill, NJ 07974, and F. Jensen, Airco Coating Technology, Concord, CA 94524.

Adhesion of coatings depends on the substrate surface condition prior to the coating deposition. Pre-cleaning of metallic substrates has been known to improve the coating adhesion; however, little has been published on the quantitative effect of in situ plasma cleaning and metal surface recontamination upon exposure to the air.

Using in situ XPS, we have studied the recontamination of previously plasma cleaned metal surfaces by exposure to controlled amounts of air. It was found that the initial formation of a thin metal oxide film was followed by the continuing growth of hydrocarbon film as well as hydroxides and moisture from the air. The main source of hydrocarbon is believed to be due to the backstreaming of vacuum pumps. We have evaluated the suitability of a reactive hollow cathode plasma for the removal of the surface contamination. The quantitative reduction of hydrocarbon, moisture, hydroxides and metal oxide films is discussed. A linear plasma source, based on the hollow cathode principle, used for in-line cleaning of metal coil is shown.


Wet cleaning processes are ubiquitous in silicon integrated circuit manufacturing. Due to the large consumables cost and potential surface roughening effects there is great interest in evaluation of new and dilute chemical cleans. Thus, it is desirable to use well defined contaminant test wafers so as to enable quantitative comparison of cleaning processes prior to costly evaluations on product wafers. In this study, particle (Si₃N₄ and SiO₂), metal (Fe, Cu, Ni), and hydrocarbon (hexamethyl-di-silazane) standards were prepared and analyzed for use as test wafers for wet cleaning process development. These standard wafers are representative of typical line contamination and at concentrations which are high enough to be a "worst case" situation. They were designed such that high cleaning efficiency is obtained with a "good" clean, while for example, water rinsing alone is ineffective for removal. The test wafer preparation and analysis is reproducible cost effective and readily accessible. Using a design of experiments package (BestDesign) which minimizes the number of runs and simultaneously
optimizes multiple responses, it is possible to use this approach for process optimization on manufacturing tools. Requirements for successful implementation are high measurement accuracy and details of the test wafer preparation/analysis will be described.

9:40 am   MS-ThM5 Rapid Yield Learning, D. M. H. Walker, Dept. of Computer Science, Texas A&M University, College Station, TX 77843-3112.

Yield is a dominant economic consideration in semiconductor manufacturing. The yield ramp must occur quickly to maximize profits. Rapid yield learning is the set of activities directed at increasing the slope of the yield ramp and maintaining high yields once they are achieved.

The basic yield learning cycle is: process a wafer, measure the result, diagnose problems when they occur, and take corrective action. Measurements can be either direct (e.g. optical inspection of a test structure), or indirect (e.g. electrical test of a product chip). Direct measurements can accurately diagnose problems, but are high in cost and low in throughput. They are most useful early in process development, but later become a bottleneck. Indirect measurements are relatively cheap and fast, and are ideal for use during the yield ramp, but require models relating process disturbances to test failures. This has limited their use in the past.

We have developed models relating process disturbances to failures, simulators that implement these models and methods for using them in rapid yield learning. The models must traverse the domains from equipment to process to device to circuit to gate to test. We have used existing and new technology CAD and EDA tools to develop compact, computationally-efficient models that span the domains, providing the defect to failure relationship.

These models have been successfully applied at several companies for diagnosis of parametric and catastrophic defects. Some of these techniques have been commercialized in the pdfab simulator from PDF Solutions, Inc. Two challenges have been rapid model characterization and the cultural problems in the use of simulation models in semiconductor manufacturing and test.

INVITED


Photore sist has been employed in all aspects of wafer processing, such as etching, deposition, and ion implantation etc., to protect a surface from damage caused by highly reactive, or heavily damaging chemical environments to which the wafer is exposed. During the processing, the photore sist mask is subjected to physical and chemical erosion, high temperatures, impregnation with assorted chemical species, surface coating with all sorts of contaminant films and polymers. Considering all of the possible individual processes, and the variety of different chemistries they involve, any manufacturing tool purposed to be a stripper of photore sist faces a severe test of flexibility. Historically, photore sist dry stripping equipment has concentrated on the simple chemistry of oxygen plasma, which quite effectively handles the virgin organic resist material. However, this approach ignores completely the complexity of the chemistry associated with the residue problems. It has been the practice of the industry to leave that problem up to the device manufacturer, to develop the necessary complex wet chemical process steps. We have developed a new process methodology, that combines microwave downstream free-radical chemistry and low-bias ion chemistry in one reactor, and in appropriate processing-step sequences to remove bulk photore sist, leaving behind most residuals converted to a water-soluble state. This suggests that DI Rinser-Dryers could replace most of acid and solvent wet stations. Processes have been developed for all significant masking steps. Questions of substrate loss and damage, undercutting of multiple layers, gate-oxide damage, are all being carefully considered. In this paper, we discuss about the reactor concept, and present examples of process results and its advantages.


Silicon dioxide etching and pre-gate oxide Si surface cleaning with vapor phase processing has been studied in a high vacuum, integrated cluster system. The system contains cleaning/etching and rapid thermal oxidation chambers. The integration enables us to process (pre-gate cleaning and oxide growth) an entire cassette without interruption. The SiO₂ etch rate fluctuates from wafer to wafer. The standard deviation is less than 10% for a 25 wafer lot etched at a rate of 70 Å/min. The on-wafer uniformity is controlled by the IR lamp configuration with higher non-uniformity at higher temperature. The non-uniformity of etching across a 5 inch wafer is less than 1% with lamps turned off (i.e. room temperature). Both the non-uniformity and the etch rate fluctuation from wafer to wafer can be compensated by an over-etch process since AFM measurement reveals no added roughness to the Si surface. Particles (>0.5 μm) generation during the etch process is in the range of 20 per wafer after about 100 Å oxide has been etched off. However, the particles counts can be reduced if the etching is carried out at a higher temperature. We are currently engaged in electrical device characterization studies.

11:00 am   MS-ThM9 Detection and Analysis of Ultra-Small Particles on 8" Unpatterned Si Wafers, C. R. Brundle, C. R. Brundle and Associates, San Jose, CA 95125, and Y. Uritsky, Applied Materials, Santa Clara, CA 95054.

As the line-widths of ICs get smaller, the allowed size of "adder" particles deposited on Si wafers by the manufacturing tool/process also decreases. Currently it is 0.16 μm as detected by laser scanners which allow process engineers to determine the number of "adder" particles on an unpatterned test wafer and establish whether the process/tool is out of spec.

The laser scanner-generated particle map then becomes the reference co-ordinate system for our subsequent attempts to identify the nature of the particles and, therefore, their origin. Particle identification is usually performed using SEM/EDX (morphology information and elemental composition). Problems with this approach increase as particle size decreases, especially since some "particles" turn out to be thin film and organic/polymeric like. This paper reviews these problems and describes approaches for improved characterization capability. The problems are: the difficulty of relocating very small particles in an SEM on 8" unpatterned wafers (a co-ordinate transfer accuracy and an SEM contrast problem); the inability of EDX to effectively separate particle signal from substrate signal; and the fact that morphology and elemental composition are not always sufficient for a positive ID. The improved approaches described are: improved 8" wafer reference origin description and co-ordinate transform algorithms; improved EDX capability; and evaluation of Auger, SIMS, and FIB as "add-ons" to an SEM/EDX system.

11:20 am   MS-ThM10 The Nature of Copper Precipitation From Dilute HF Solutions on to Si Surfaces, T. S. Sriman, R. Sampson, J. Shyu, W. C. Harris, D. Liu and S. Bill, Digital Equipment Corporation, 77 Reed Road, Hudson, MA 01749.

It is well known that the presence of Cu and other transition metals on the Si surface prior to oxidation can lead to increased junction leakage and possible loss of gate-oxide integrity. This contribution examines the effect of Cu contamination of the HF used in HF-last cleaning processes prior to gate oxidation in deep sub-micron Si CMOS processes. We examined P-type (20-40 Ω-cm) Si [100] wafers exposed to a dilute HF solution which was quantitatively contaminated with Cu. Two concentrations of Cu, 1 ppm and 0.1 ppm were used in this study. The surface of the wafers was examined using TXRF and AFM. It was found that the Cu distribution on the wafer surfaces was non-uniform on both large and small scales. AFM imaging showed that the copper deposited in the form of small circular precipitates on the surface. Some areas of the sample exposed to the higher Cu concentration solution showed the aggregation of smaller precipitates in large platelets. Subsequent Cu deposition on these platelets appears to occur in a layer-by-layer fashion. The surfaces of these platelets were smooth (~0.1 nm RMS). No aggregation of platelets was seen in the sample exposed to 0.1 ppm Cu in HF. Diffraction analysis of planar samples from these wafers in the TEM revealed that the precipitates were composed of metallic copper.

VACUUM METALLURGY
Room A106 – Session VM-ThM

Pulsed Laser and Pulsed Ion Technology for Film Deposition and Surface Modification
Moderator: B. Sartwell, Naval Research Laboratory.

8:20 am VM-ThM1 Dual Laser Ablation of Particulate Free Optical Films, S. Witanachchi, K. Ahmed, P. Saktiviel and P. Mukherjee, Department of Physics, University of South Florida, Tampa, FL 33620.

The eximer laser ablation of a poorly absorbing ceramic target has been shown to produce high density of particulates that render the films grown by this method inappropriate for optical applications. We report here the results of a dual laser process, where a pulsed CO2 laser and an excimer laser were spatially overlapped on a Er doped YO2 target, that produced defect free films that are suitable for waveguide laser fabrication. The delay between the two lasers was seen to significantly affect the particulate density in the deposited films. The particulate ejection was observed to be minimum when, temporarily, the excimer pulse arrives at the target during the rising time of the CO2 pulse. The optical emission of the particulate was studied for different delays between the lasers, and indicate a significant enhancement on the plume excitation. The role of the two lasers and the results leading to the optimization of this process will be discussed.

8:40 am VM-ThM2 Nanostuctured Films and Particles Produced by Femtosecond Pulsed-Laser Ablation, S. L. Rohde, A. Latiesf, B. Robertson, T. Volles, D. Doerr, and D. R. Alexander, University of Nebraska-Lincoln, Lincoln, NE 68588.

Investigations of this film formation and particle generation have been carried out using ultrafast femtosecond laser ablation of pure metals onto a variety of substrates. Both particles and thin films of Au and other metals have been deposited using femtosecond laser producing 20 μs pulses with a pulse-width of 100 femtoseconds, focused into a 5 μm spot-size. Particles produced and collected on carbon grids were examined using TEM and shown to have particles sizes down to a few nm in diameter; similar grain sizes were observed for very thin films of Au as well. Comparisons are presented for particles deposited in air, low vacuum, and moderate vacuum environments. Other parameters investigated include location of the substrates relative to the incident laser, target-to-substrate distance, and various laser pulse parameters. Comparisons are drawn between this work and those found in the literature for pulse durations of several nanoseconds and longer. These initial results indicate that use of femtosecond pulsed-lasers may provide a means of producing ultrafine particles and nanostructured thin films with unique properties.

9:00 am VM-ThM3 Large-Area Pulser Laser Deposition: Techniques and Applications, J. A. Greer and M. D. Tabat, Raytheon Company-Research Division, Lexington, MA 02173.

Due to its unique ability to quickly produce high quality films of complex chemical compounds, Pulsed-Laser Deposition (PLD) is currently being used to grow an estimated 200 different materials in laboratories world-wide. Most of the effort involving PLD has been geared toward obtaining high-quality films for electronic and optical applications. Also, significant research has been conducted on obtaining an understanding of the laser-target interaction and the laser-induced plume dynamics. However, relatively little work has been conducted on scaling-up this process to substrate sizes compatible with most device and process line requirements. Clearly, techniques for scaling the process will be needed in order for PLD to become more than a unique laboratory tool. This talk will focus on several large area techniques which have been used to scale the PLD process to substrate sizes from 50 mm to 150 mm in diameter. The uniformity of a variety of film properties produced by PLD using these large-area PLD techniques will be presented, and the advantages and disadvantages of each approach will be discussed. A variety of properties and applications of large-area PLD films will be presented throughout the talk including substrates such as high temperature superconductors, thin film photovoltaics, and optical AR coatings. Finally, potential techniques and future equipment requirements to scale the process to substrates sizes of at least 200 mm in diameter will be examined.

9:40 am VM-ThM5 Laser Induced Fluorescence Studies of Atomic and Molecular Species in Laser Generated Plumes, Terry L. Thiemo, Dept. of Chemistry, USAF Academy, CO 80840, and Paul J. Wolf, J. J. Seiler Research Laboratory, USAF Academy, CO 80840.

Thin film growth by laser ablation deposition depends on both the nature and the energy of the material in the plume. The state of the material can be modified through interactions with a reactive ambient gas. In the case of oxide and high Tc superconducting film formation in the presence of O2 which is required for correct film oxidation. In addition, an understanding of the interaction of the plume species with a background gas and the substrate is needed to delineate the relative importance of gas phase or surface reactions in stoichiometric thin film formation.

As an initial measure of the relative importance of the film growth mechanisms mentioned above, we tracked ground state Ge and GeO using laser induced fluorescence in a plume generated by ablating a GeO target at various pressures of O2, Ar and N2. Time of flight (TOF) profiles and the intensity behavior were mapped in time resolved experiments. The Ge TOF profiles in O2 revealed a bi-modal behavior suggesting plume-ambient interactions which affected the Ge distributions. This behavior was absent in both the Ge distributions with other ambient gases and the GeO distributions. The most probable velocity was determined by fitting the TOF distributions to an isentropic expansion expression. Typical values ranged from 6 x 10^6 cm/s to 1 x 10^7 cm/s for Ge in vacuum and 100 mTorr of O2, respectively. The population of the ground state species decreased with increasing pressures for all ambient gases which indicated that gas phase chemical reactions were not kinetically important. These results will be detailed and subsequently correlated to mechanisms for thin film growth.

10:00 am VM-ThM6 GeO2 Films Prepared by Pulsed Laser Deposition, B. M. Patterson, P. J. Wolf, F. J. Seiler Research Laboratory, USAF Academy, CO 80840; M. Scott, Dept. of Physics, USAF Academy, CO 80840; T. Christensen, University of Colorado, Colorado Springs, CO 80918.

In recent years pulsed laser deposition (PLD) has been used to prepare thin films of several materials having applications in integrated optical systems. Our group, in particular, has used the PLD technique to fabricate thin films of amorphous germanium dioxide (GeO2), as a preliminary step towards preparing GeO2-doped silica films for optical waveguiding and nonlinear optical applications. Our goal in this present work is to assess the suitability of PLD for preparing the flat and uniform films required for waveguiding.

The GeO2 films were prepared on heated quartz substrates by ablating a rotating GeO2 target in the presence of an oxygen plasma. In order to improve the thickness uniformity of the films, the target was inclined with respect to its rotation axis, thereby spanning the ablation plume over the substrate. These GeO2 films were characterized to determine the optical properties and thickness uniformity. The null results of these measurements confirmed that the films were amorphous.


There is an increasing interest in the use of ion-beams as broad-area sources for modifying surfaces using rapid thermal excursions. Recent developments in pulsed power technology have enabled the production of high average power (5-250 kW), repetitively pulsed (100 ns pulse duration) ion beams with beam diameters of 10-20 cm and with energies up to 2 MeV. The ion beams efficiently deposit energy in a thin surface layer (top 2-20 micrometers) of the surface of materials. Deposition of small energy densities of less than 100 J/cm^2 is typically sufficient to melt the surface of metals and ceramics. Following melting, the surface layer rapidly cools at rates of approximately 10^{-10}-10^8 K/sec. These cooling rates are sufficient to produce amorphous, nanocrystalline, and/or supersaturated solid solutions in the treated layer. The resultant unique phases can, in turn, enhance the hardness and wear resistance of tool steels, the corrosion resistance
of engineering alloys, and the density of ceramic surfaces. This talk will review the effects of using ion beam surface treatment to enhance the properties of ferrous, nonferrous, and ceramic materials.

Invited

*This work was performed at Sandia National Laboratories, supported by the US Department of Energy under contract #DE-AC04-94AL85000.


In Plasma Source Ion Implantation (PSII), a target to be implanted is immersed in a weakly ionized plasma and pulsed to a high negative voltage. Ions in the plasma are accelerated toward the target and implanted in its surface, thereby modifying the properties of the surface. A large-scale PSII experiment is operating at Los Alamos, in which implantation of targets with surface areas of several m² is performed in an 8 m³ vacuum chamber using a 100 kV modulator supplying 20 µs pulses at 2 kHz, with a peak current of 50 A. Our goal is to demonstrate that PSII can be scaled up to an industrially useful process. In this talk I will describe the operational problems encountered and the results obtained in the implantation of carbon from methane, ethane, and acetylene plasmas, nitrogen implantation of large chrome-plated industrial parts, and nitrogen implantation of tool steels at elevated temperature.

*Work supported by U.S. Department of Energy.

11:20 am VM-ThM10 Film Deposition and Surface Modification Using Intense Pulsed Ion Beams, C. A. Meli, K. S. Grabowski, D. D. Hinshelwood, S. J. Stephanakis, Naval Research Laboratory, Washington, DC 20375; D. J. Rej, W. J. Wagenaar, Los Alamos National Laboratory, Los Alamos, NM 87545; Michael O. Thompson, Cornell University, Ithaca, NY 14853.

High-power pulsed ion beams have been used to ablate material for ultra-high-rate film deposition, and to modify the surface properties of alloys. Pulsed ion beams were provided by the Anaconda facility at LANL (~500 keV H, C, and O ions, ~1:1:1 ratio, ~400 ns duration. E ~ 30 J/cm²) and the Gamble II facility at NRL (~1.2 MeV H and C ions, ~9:1 ratio, ~100 ns duration, E ≤ 100 J/cm²).

Instantaneous film deposition rates produced by ablation reached up to ~0.1 cm/s, at the maximum available fluences. Films of pure Al, pure Ta, and a nickel-zinc ferrite compound were deposited on glass and single crystal substrates, in some cases heated. The film deposition process was studied with framing photography, a bolometer, and other diagnostics to gain an understanding of the ablation, transport, and deposition steps. These diagnostics provided guidelines for the production of desirable films. Smooth films, and oriented ferrite films have now been produced. Beams with a lower fluence (~5 J/cm²) were used to investigate rapid thermal processing of metal surfaces for the enhancement of corrosion resistance. Bare steel surfaces were treated to smooth surface features and dissolve precipitates, and thin metallic films on aluminum were treated to produce supersaturated surface alloys. Recent results in these two areas of effort will be presented.


Conventional DC magnetron sputtering of nonconducting oxide films via reactive sputtering is extremely difficult. Without good separation of the oxygen from the target, the surface of the target rapidly becomes covered with an oxide film or "poisoned" when there is a high enough partial pressure of the reactive gas to form the desired film composition on the substrate. Breakdown of the oxide film on the target surface leads to arcing, which can damage the power supply and which can also eject droplets into the growing film. Depending on the application, these droplets can degrade performance of the oxide film. Recent advances in power supply technology with the introduction of pulsed DC power has overcome many of the problems in using DC power for the reactive sputtering of oxide films. The DC power is switched rapidly, at a rate up to 20 kHz, between the normal negative target potential and a positive potential, and during the positive pulse any charging on the target surface is discharged, which prevents the formation of an arc. It is now possible to reactively DC sputter such previously difficult materials as aluminum oxide with pulsed power. Details of these pulsing sources, the reactive sputtering process, and the properties of the resulting films will be discussed.
Thursday Afternoon, October 27, 1994

SURFACE SCIENCE
Room A205 – Session SS1-THA

Oxidation and Adsorption
Moderator: J. W. Rogers, University of Washington.

2:00 pm SS1-THA1 Molecular Beam Homoeptaxial Growth and Surface Characterization of MgO(001), T. T. Tran, T. A. Hideman, and S. A. Chambers, Pacific Northwest Laboratory*, Richland, WA 99352.

Homoeptaxial growth of thin MgO(001) films was accomplished by evaporating magnesium in a high-flux, low-energy beam of oxygen ions produced by an electron cyclotron resonance plasma source. Growth at different substrate temperatures in the 450–750°C range were attempted with improved film topography, as determined by ex situ atomic force microscopy (AFM), obtained at the most elevated temperature. During growth, film composition was monitored by Auger electron spectroscopy and long-range crystallographic order was determined by reflection high energy electron diffraction. Post-growth analysis include low-energy electron diffraction and x-ray photoelectron diffraction data, providing long- and short-range crystallographic order information, respectively. Auger Mg KLL/O KLL intensity ratios obtained during growth were calibrated against Mg 2p/0 1s photomission intensity ratios which had been previously normalized with data from a cleaved MgO(001) reference surface. Reproducible stoichiometry in the MgO films was obtained. Excellent short- and long-range crystallographic order in the films grown at 750°C, as observed with diffraction probes, along with AFM data suggests that a step-flow growth mechanism is operative in this temperature range.

*Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RL01830.

2:20 pm SS1-THA2 Step Fluctuation Kinetics and the Formation of (n × 1)-O/Ag(110), W. W. Pal, M. R. Peng, N. C. Bartol, and J. E. Reut-Robey, University of Maryland, College Park, MD 20742.

We will present strong experimental evidence that (n × 1)-O/Ag(110) adopts an "add-edrow" structure similar to (2 × 1)-O/Cu(110). Despite the structural similarities between these two systems, we show that the formation mechanism for O/Ag(110) is different from that of O/Cu(110) because of differences in mass transfer from step edges.

STM images of the oxygen chains indicate that the O atoms are situated in the short 2-fold bridge sites with respect to the original Ag(110) substrate. The apparent contradiction to oxygen's observed preference for long 2-fold bridge sites is resolved by incorporating extra Ag adatoms to form a Ag-O-Ag chain-like structure. Additional evidence for this added row structure is provided by the 100 Å-scale displacements of 'isolated' steps upon oxygen adsorption, revealing that Ag atoms detach from the step edges to create the added rows.

With infrared vibrational spectroscopy and LEED, we observe the atomic oxygen stretching frequency shifts continuously from 310 cm⁻¹ to 330 cm⁻¹ as the (7 × 1) phase is compressed into the (3 × 1) phase. This shift is consistent with a first principles calculation based on an optimized add-edrow structure.

Details of the mass transport are obtained through analysis of step fluctuations on the clean surface at room temperature. From the autocorrelation in the position of the step edge, thermal step fluctuations appear dominated by adatom exchange processes at the step edge. The mean time between attachment/detachment is of the order of 100 ms. This rapid exchange indicates the presence of mobile silver adatoms on the terraces and that the rate of overlayer formation is not limited by step detachment kinetics. We suggest that the formation of Ag-O-Ag nuclei on terraces is the rate limiting step, in contrast to the stepatom supplied limited process for O/Cu(110).


2:40 pm SS1-THA3 Oxidation Behavior of the (100) and (110) FeAl Surfaces, H. Graupner, L. Hammer, and K. Müller, Univ. Erlbg.-Nbg., Erlangen, Germany, and D. M. Zehner, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6057 USA.

The initial stages of oxidation of the (100) and (110) surfaces of FeAl have been examined with XPS, AES, and LEED. Exposure to oxygen at sample temperatures up to 500°C leads to the formation of an amorphous Al2O3 film on both surfaces. At higher temperatures, oxide film phases are observed, where six x 6 structure is formed on the (100) surface at an oxidation temperature of 700°C, which is probably caused by an oxide film with a structure similar to γ-Al2O3.

The thickness of this film has been determined to be 4 ± 1 Å, which corresponds to two Al-O bilayers in γ-Al2O3. Apart from the chemically shifted Al 2p line associated with the oxide film, a second component is observed in the XPS spectra, which can be attributed to Al atoms in the substrate-oxide interface. Oxidation at 850°C leads to the development of a streaky (2 x 1) diffraction pattern similar to that reported for the NiAl(100) surface. For the (110) surface, the initial rate of oxygen uptake is significantly higher compared with that of the (110) surface at all temperatures. The composition and thickness of the oxide film on the (110) surface is dependent on the initial oxidation rate. Low rates, as in normal ultrahigh vacuum experiments, lead to the formation of thin Al2O3 films (~ 6 Å), while at higher rates thicker films are grown (8–15 Å) which contain iron oxide as well. Exposure to oxygen at a sample temperature of 850°C leads to the formation of a well-ordered oxide overlayer of 6 ± 1 Å thickness and a large, nearly rectangular unit cell. The structure of this film is probably related to δ-Al2O3. A film with identical unit cell parameters is present on NiAl(110). All observed oxide films are stable up to temperatures of 900°C.

This work is supported by *Univ. of Erlbg.-Nbg., FRG and DAAD, and **U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

3:00 pm SS1-THA4 Electron Stimulated Oxidation of Metals at Low Temperature: Ni(111) at 120 K, Wat Li, M. J. Stillman and S. J. Silbern, The James Franck Institute and Department of Chemistry, The University of Chicago, 5640 S. Ellis Ave., Chicago, IL 60637.

Oxygen adsorption and oxide growth on Ni(111) has been investigated at 120 K. We have found that electrons stimulate nickel oxide growth at all energies examined, spanning the range from 5 eV to 2 keV. These electron stimulated effects occur under conditions which would otherwise lead to oxygen chemisorption in the absence of electron irradiation. Moreover, we find that electron-induced oxidation rate can be achieved by two different procedures: one with the electron beam and oxygen exposure occurring simultaneously; the other with the electron beam and oxygen exposure alternating during the course of oxidation. These results differ from previous work, which concluded that appreciable oxide formation would occur upon exposure of low temperature Ni(111) to molecular oxygen—a result which we now attribute to synergistic effects involving electrons. We propose a model for the observed effect in which electrons create oxygen nucleation centers on the Ni(111) surface in the presence of chemisorbed oxygen. This model allows us to quantitatively account for the data, including extraction of the relevant cross sections. These findings are of fundamental importance in the areas of metallic oxidation and corrosion, and may find application in electron beam lithography.


The nature of alkali metal/oxide surface interactions is important for predicting the stability and surface electronic properties of insulators in thermionic systems as well as understanding the chemical activity of promoters in catalytic systems. The chemical nature of the Cs-sapphire (α-Al2O3) interaction along with the growth of ultrathin Cs films has been studied using electron spectroscopy (XPS/AES),
** electron diffraction (LEED) and mass spectrometry (RMS/TDMS). Reflection mass spectrometry (RMS) demonstrates facile adsorption of Cs, with an initial sticking coefficient of 0.9, on the unreconstructed (001) surface at 300 K. This initial, energetic interaction is evident in the thermal desorption of Cs at temperatures in excess of 1000 K. Core level spectroscopic data show no formal oxidation of Cs despite this initial high affinity between the adsorbate and the surface. A critical coverage of Cs is reached at $1.5 \times 10^{16}$ atoms cm$^{-2}$ after which the sticking coefficient decays exponentially to a value less than 0.1.

**LEED results show only increased background intensities indicative of limited long-range order in the film. Comparison of TDMS and XPS/AES data indicate that cluster formation occurs with increased Cs exposure. The absence of an oxidized Cs signal coupled with cluster formation suggests selective interaction of Cs with coordinatively unsaturated O sites on the surface. Selective Cs-O interaction is further supported by comparison of the sticking coefficient versus exposure data for the ($3\sqrt{3} \times 3\sqrt{3}$)R30$^\circ$ and the ($3\sqrt{3} \times \sqrt{3}$)R $\approx 9^\circ$ reconstructions of the (001) surface. These surfaces show a decrease in sticking coefficient consistent with their reported oxygen deficient structure. The impact of adsorbed C on the Cs/substrate interaction will also be discussed.

* Funded by Ballistic Missile Defense Organization and supported by the U.S. Department of Energy under Contract No. DE-AC04-94AL85000.


We have studied the adsorption of oxygen and formate on a Cu(100) surface using synchrotron-based IR spectroscopy, from 100 to 2000 cm$^{-1}$. Oxygen adsorption induces a frequency-dependent broadband reflectance change. Our measurements provide the first independent test of a model based on conduction electron scattering that incorporates non-local electrodynamics. Excellent agreement is found for low O coverage. Some discrepancies with theory remain, in the high-coverage behavior and the value of the rolloff frequency.

Exposure of the O-adsorbed surface to formic acid removes the oxygen and leaves a formate layer. The broadband reflectance returns to its clean-surface value. Two low-frequency vibrational modes are observed, at 360 and 380 cm$^{-1}$, as shown in the figure. Previous EELS investigations could not resolve these modes. The observation of two modes presumably due to Cu-O vibrations suggests that more than one adsorption site may be occupied.

Supported by NSF Grant Nos. DMR-885166 and DMR-9201077 and NSF/CNRS Int'l. Collaborative Award No. INT-9016771.

4:00 pm **SS1-ThA7 Atomically Resolved STM Study of the Interaction of Boron with Si(001), Yajun Wang and Robert J. Hamers, Department of Chemistry, University of Wisconsin, Madison, WI 53706, USA.

The interaction of boron with Si(001) is very important for semiconductor processing. STM, tunneling spectroscopy and surface photovoltaic measurement have been used to study the geometric and electronic structures of boron-induced reconstructions produced by thermal decomposition of diborane on Si(001). At T $\geq 675$ K, boron atoms organize into ordered structures on the surface. STM images reveal a number of boron-induced reconstructions which arise from the arrangements of three structural subunits. Boron atoms bond at

** substituional sites and form a delta-doped layer. These boron-induced structures are chemically inactive, but electrically active. Over 1075K, most boron atoms diffuse into bulk and it also produces surface facetting. This is the first atomic-scale study of boron-induced reconstructions on Si(001) and it will provide a deeper understanding of semiconductor doping. The formation process of new structures and their effects on further silicon films are still a topic of discussion. This work is supported by the U.S. Office of Naval Research.

4:20 pm **SS1-ThA8 The Structure and Stability of Methyl Thiolate on Ni(111), D. R. Huntsley, D. R. Mullins, S. H. Overbury, Oak Ridge National Laboratory, Oak Ridge, TN 37871-6201 and H. Yang, North Carolina State University, Raleigh, NC 27695-8204.

The production of methane from the reaction of either methanethiol or dimethylsulfide on the Ni(111) surface involves at least two intermediate species at high coverage which are readily discernable by high resolution XPS. One of these intermediates is formed upon adsorption at temperatures less than 100 K and is identified as methyl thiolate, based on the HREEL spectra. A second intermediate is formed upon heating the surface, and is stable between 200 and 275 K. HREEL spectra measured both on and off specular scattering conditions as a function of both annealing time and temperature exhibit no new modes due to the activated formation of this species, indicating that it is also a methyl thiolate fragment. However, changes in the relative intensities of the methyl deformation and stretching modes suggest a difference in orientation of the methyl group. The thiolate formed at low temperature is proposed to reside in a low coordination site such as a two-fold bridge site and have the C-S bond tilted toward the surface. The thiolate formed at higher temperature occupies a three-fold hollow site and has the C-S bond nearly perpendicular to the surface. Recent calculations using the many electron embedding theory support this conclusion, indicating nearly equal adsorption energies for these two configurations.

Research Sponsored by Division of Chemical Sciences, Office of Basic Energy Sciences, U.S. Department of Energy.


The decomposition mechanisms for precursors on surfaces are important in determining the efficiency of CVD processes. As a model system, the decomposition of P(CH$_3$)$_3$ and PH$_3$ on Ru(0001) has been studied by soft X-ray photoelectron spectroscopy using synchrotron radiation. By mean of the chemical shifts in the P(2p) core-levels, we are able to identify stable phosphorus containing surface reaction intermediates and elucidate the overall reaction mechanism. P(CH$_3$)$_3$ undergoes a step-wise dehydrogenation, i.e., P(CH$_3$)$_3$ $\rightarrow$ P(CH$_3$)$_2$ $\rightarrow$ P(CH$_3$) $\rightarrow$ P, which is complete at $\sim$ 450K. In contrast, PH$_3$ decomposes involving a single stable intermediate of undetermined stoichiometry, PH$_3$ $\rightarrow$ PH$_2$ $\rightarrow$ P, and is complete at $\sim$ 190K. The subsequent conversion of chemisorbed phosphorus to ruthenium phosphide by annealing is complete at $\sim$ 700K. Molecular adsorption and decomposition energetics are estimated using the bond order conservation Morse potential (BOCMP) method, and are found to agree semi-quantitatively with the experimental data. These mechanistic and energetic insights are useful in designing future phosphorus-containing molecular precursors for low-temperature, selective deposition of phosphorus in materials growth technologies.
SURFACE SCIENCE
Room A201 - Session SS2-ThA

Surface and Adsorbate Structure
Moderator: P. Avouris, IBM T. J. Watson Research Center.

2:00 pm SS2-ThA1 Br/Si(211)x2 x1 Structure Investigated by X-Ray Standing Waves, V. E etlaniemi, E. G. Michel, and G. M materlik, HASYLAB at DESY, 22603 Hamburg, Germany.

The interaction of halogens with silicon surfaces has been a subject of interest in recent years since it represents the first step of the etching process. We have studied the system Br/Si(211)x2 x1 by using the x-ray standing wave (XSW) technique, which allows a precise determination of the adsorption geometry of an interface. A clean, non-faceted Si(211)x2 x1 surface was prepared by annealing in vacuum a chemically etched crystal. The ideal Si(211) surface is formed by [111] terraces and [100] steps, and thus it is an ideal model surface to investigate the interaction between both types of sites. After Br adsorption, the surface reconstruction reverts to 1 x1 (as is typical of several other silicon surfaces). The adsorption sites of Br were investigated as a function of Br coverage and annealing temperature to characterize the competition between step and terrace sites at the surface. Three different substrate reflections ([222], [111], and [220]) were employed to locate Br atoms independently of the silicon surface geometry. Our results indicate that both atop (on terraces) and step sites (with Br atoms along empty dangling bond directions) coexist at the surface at room temperature. After annealing, the step sites are relaxed in a such a way that the Br-Br interatomic distance is maximized. The change in bond angles estimated from our analysis fits well within the limits fixed by current models for the elasticity of silicon bonds. Annealing to higher temperatures favors the occupation of terrace sites only.

2:20 pm SS2-ThA2 The Structural Characterization of Ga on Si(112), J. E. Yater*, A. Shih, Y. Iderza, Naval Research Laboratory, Washington, DC 20375.

The structure of gallium overlayers on the Si(112) surface has been investigated using the technique of angle-resolved Auger electron spectroscopy (ARAES). This system is of scientific interest because previous studies suggest that the Ga atoms form well-ordered chains along the stepped Si(112) surface. The fabrication of quantum well wire arrays has much significance in solid state device applications, as does the growth of III-V materials on silicon substrates. However, no studies to date have determined the exact gallium atomic site locations relative to the Si(112) substrate. In this study, a single-resolved AES data is used to distinguish between a host of possible overlayer/lattice structures based on a comparison with single scattering cluster (SSC) calculations. These calculations show that low energy AES data is very surface sensitive and therefore appropriate for overlayer structural studies. A well-ordered gallium overlayer is grown on a clean (1 x 2) reconstructed Si(112) surface such that a (1 x 1) LEED pattern is observed. The angular dependence of the Auger electron intensity is measured for two low energy gallium peaks along the [110] and [111] directions. The theta scans are taken in 5° incremental steps, and modulations are observed in the intensity patterns which correspond to scattering minima and maxima along the forward direction of the overlayer structure and reflect the symmetry of the overlayer structure along the scan direction. These measurements are the first ARAES data from such chain-like overlayer structures. A comparison with cluster calculations can be used to determine the gallium atomic site locations and the resulting Si(112) surface structure.

*NRC Postdoctoral Fellow.

3:00 pm SS2-ThA3 Transition Metal Induced Ring-Cluster Structures on Si(111) Studied by STM, S. A. Purik, M. T. Lee and P. A. Bennet, Physics Dept., ASU, Tempe, AZ 85287-1504.

We characterize a "ring-cluster" (RC) structure produced by high temperature annealing of transition metals (TM) on Si(111). Originally discovered for Co, this structure consists of a single metal atom on a substitutional site in the top layer of Si(111) plus an overlying ring of 6 Si adatoms with an unusual bridge-bonded topology. The same structure occurs for all near noble metals (Co, Ni, Pt, Pd, Ir) but not for earlier TM (Fe, Ti and Ta) or noble metals (Cu). The RCs first form at 700°C and at higher temperatures will phase separate from clean Si(111)-7 x 7 into large triangular patches containing a 1 x1 lattice gas of RCs plus small 2 x 2 patches of Si adatoms on T4 sites. The metal coverage in the 1 x 1 phase ranges from a minimum of 0.02 ML to a maximum of 0.14 ML at which point the RCs are close-packed and can order into a v/7 superlattice. In the case of Ni, a second ring-like structure forms simultaneously with the RCs and orders into a /19 superlattice at a minimum coverage of 0.16 ML. These rings are centered on T4 sites of Si(111). A unit cell structure with 3 sub-surface Ni atoms is presented.


3:20 pm SS2-ThA4 Surface Geometry of S-Passivated InP(100)-(1 x 1), O. L. Warren, G. W. Anderson, M. C. Hauf, and P. R. Nortson, University of Western Ontario, London, Ontario, Canada.

Surface cleaning/passivation is a key step in semiconductor manufacturing processes. In the case of InP(100), the surface is passivated effectively by S after dipping in an aqueous solution of (NH4)2S at 65°C. Upon transfer to UHV, the surface exhibits a coherent (1 x 1) LEED pattern without further treatment. In order to ascertain the role of S in surface passivation, we have determined the geometric structure of S-passivated InP(100)-(1 x 1) by dynamical LEED analysis. Sulfur atoms occupy the expected surface bridge sites which continue the zincblende structure of the substrate, the bond only to In atoms in the next layer. Other structural models, such as S bonded to sp-hybridized In, can be clearly ruled out. Investigation of random vacancies in the S layer favors nearly a full monolayer coverage. The distance between the S layer and the In plane directly beneath is 1.45 Å, and all interlayer spacings of the substrate remain at the bulk value of 1.47 Å; therefore, the In-S bond angle is nearly exactly tetrahedral. The quality of fit between experiment and theory is good, as indicated by a minimum Pendry r factor of 0.26. The bonding configuration determined in the analysis is consistent with the idea that S passivates the surface by saturating the dangling bonds. These results are in general agreement with those obtained previously by XANES, although the details differ on a quantitative level.

3:20 pm SS2-ThA5 Structural Determination of Methyl Halides on GaAs(110): Analysis by NEXAFS and TOF Measurements of Photodissociation, P. J. Lazsky, P. H. Lu, Q. Y. Yang, and R. M. Ogston, Jr., Columbia Radiation Laboratory, Columbia University, NY, NY 10027.

The structure of methyl halides physisorbed on GaAs(110) has been investigated by NEXAFS and correlated with angular resolved time of flight (TOF) measurements of methyl radicals ejected from the photodissociation of the adsorbed molecules. The photo-induced dissociation is due to electron attachment to the adsorbate molecules from electron-hole pairs created in the GaAs substrate. The results from both experiments show that the methyl halide molecules in the first monolayer are oriented so as to tilt away from the surface normal in the [010] direction. These results indicate that the dissociation process takes place on a time scale faster than reorientation of the CHX+ ion intermediate and demonstrates clearly the ability to use angle-resolved TOF measurements of fragments generated from photo-induced dissociation by low energy electron attachment as a probe of the structure of adsorbed molecular layer. In addition, the results show that the molecules in the second layer are oriented differently from...
those in the first and have also been investigated with NEXAFS and TOF. The correlation of results from NEXAFS and TOF measurements, along with insights gained from the modeling of temperature programmed desorption (TPD) data has elucidated the structure of methyl halides on the GaAs(110) surface. This structural information provides insight into the interactions of small, dipolar molecules with the corrugated GaAs(110) surface. Preliminary computations indicate that the molecular tilt is in agreement with electrostatic interactions between the adsorbate molecule and the surface fields.

3:40 pm SS2-ThA8 Structure of Sulfided Ultra-Thin Ni Films on W(001), S. H. Overbury, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6201.

Low energy alkali ion scattering, LEED and AES have been used to determine the interaction and structures of co-adsorbed S and Ni on the W(001) surface. This work follows previous research to characterize the structures obtained for Ni/W(001) and of S/W(001), and provides structural details on a model system study of sulfidation of ultra-thin metal films. Ni alloys with W(001) to form a single mixed layer at sub-monolayer coverages, but at 1 ML it remains at the surface and forms a relatively pure, pseudomorphic overlayer. Pseudomorphic growth continues into the second layer. It is found for annealing temperatures of around 900 K that S adsorbs as an overlayer on Ni films of thicknesses of 1 ML to 2 ML without inducing separation of Ni into islands of Ni or of Ni sulfide. A c(2x2) pattern is observed for a ML coverage on W(001) and on the Ni thin films. Ion scattering demonstrates that this pattern is due to ordering of S atoms adsorbed on four-fold sites on the W or pseudomorphic Ni. This structure can also be obtained by reversing the order of deposition, due to thermally induced phase exchange of Ni and S atoms. Surface bonding distances can be determined from the location of shadowing edges observed in angular scans of Ni and W single scattering. A ML of Ni allows S adatom to be slightly closer to underlyng second layer W atoms than on Ni-free W(001). Sorbed S causes outward relaxation of the Ni layer. As S coverage increases above 0.5 ML, the 1/3, was beam of the c(2x2) pattern is replaced by diffuse features. LEIS indicates that S continues to fill four-fold Ni hollow sites.

Research sponsored by Division of Chemical Sciences, Office of Basic Energy Sciences, U.S. Department of Energy.

4:20 pm SS2-ThA9 Second Harmonic Generation from Alkali Metal Overlayers on Al(111) Surface, Jang Wang, Z. C. Ying, University of Pennsylvania, Philadelphia, PA 19104; E. W. Plummer, University of Tennessee, Knoxville, TN 37996.

In order to fully explore the potentials of the optical second harmonic generation (SHG) as a useful surface probe, it is necessary to understand the microscopic mechanism of the surface nonlinear optical process. Surface SHG from metal surfaces is paid special attention since this system is theoretically tractable by first-principle calculations. Using a mode-locked Nd:YAG laser at 1064 nm (hω = 1.17 eV) and 532 nm (hω = 2.34 eV), we were able to measure the SHG from clean Al(111) surface as well as from alkali metals (Na, K, Rb) adsorbed on Al(111) surface as functions of azimuthal angle, coverage and temperature. The SHG from clean Al(111) and (Na, K, Rb)/Al(111) exhibits both isotropic and anisotropic responses with respect to the rotation of the crystal azimuthal angle. For isotropic components, the enhancement of SHG from alkali metal overlayers adsorbed on Al(111) over that of the clean Al(111) surface agrees well with the recent time dependent local density calculations (TDLDA). This agreement confirms that the induced second-order current normal to the surface is the dominating component in SHG from metal surfaces and alkali metal overlayers on metal surfaces. Yet, the strong anisotropic SHG from our experiment suggests that there is still a need to extend our current physical picture as embedded in the TDLDA calculations beyond the free-electron models, even for the simplest metal surfaces.


4:40 pm SS2-ThA9 Depolarization and Phase Behavior in a Model for Alkali Adsorption on Simple Metals,1 D. R. Roels and Daniel Fromowitz, Physics Dept., Haverford College, Haverford, PA 19041 USA.

Alkali adsorption on metallic surfaces results in complex phase behavior due to the interplay between the charge-transfer-based bonding behavior and adatom interactions. Observed phenomena1 include phase transitions involving depolarization,2 coverage-driven changes in binding site and substrate reconstruction. We have developed a model for these systems based on dipole-dipole adatom interactions with momentum strengths determined (self-consistently) by the competition between bonding-induced momentum formation and depolarization based on adatom proximity. The model also incorporates multiple binding sites. We report simulation results obtained from this model including the nonlinear non-ideal variation (with coverage) characteristic of these systems; low-coverage ordered phases; and the novel coverage-driven depolarization transitions proposed in ref. 2. Some parameter variation is also presented in order to point toward consistency with experimental systems.

1 Supported by NSF grant DMR-9105804.

5:00 pm SS2-ThA10 The Structures of CO and O on the Rh(110) Surface as Determined by Tensor LEED, J. D. Batteas, A. Barbieri, E. K. Starkey, M. A. Van Hove, and G. A. Somorjai, Materials Sciences Division, Center for Advanced Materials, Lawrence Berkeley Laboratory, and Department of Chemistry, University of California, Berkeley, CA 94720.

The structure and bonding of atoms and molecules on defect sites such as steps and kinks are of particular interest since these are proposed active sites of catalysis. CO adsorbed on the Rh(110) is a densely stepped surface with rows of (111) microfacets. Both CO and O form p2mg (2x1) surface phases at 1 monolayer coverage on the Rh(110) (1x1) surface. The detailed structures of these phases have been determined by an automated tensor LEED intensity analysis.

In the Rh(110)-p2mg(2x1)-2C0 structure, the clean surface relaxations are removed with the top interlayer spacing expanded by 0.15 Å with respect to the clean surface. The CO molecules are bonded near the short bridge sites with Rh-C bond lengths of 1.97 Å, a C-O bond length of 1.13 Å, and the molecular axis tilted by ~24° from the surface normal. In the Rh(110)-p2mg(2x1)-2C0 structure, a cluster-like bonding for the O is found. The clean surface relaxations are removed and the oxygen atoms are bonded asymmetrically in the fold hollow-site. Two Rh-O bonds are formed with the top metal layer with bond lengths of 1.86 Å and a third Rh-O bond is formed with the second metal layer of 2.07 Å. The second metal layer Rh atoms
are found to shift away from their clean surface positions, towards the oxygen positions, introducing a zig-zag lateral "splitting" of the layer by ~0.10 Å.

**NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY/ BIOMATERIAL INTERFACES**

Room A209 - Session NSBI-ThA

**Micro- Instrumentation and Sensors**

**Moderator:** M. H. Hecht, Jet Propulsion Laboratory.

2:00 pm NSBI-ThA1 Micromachined Neural Interface Technology, Gregory T. A. Kovacs, Stanford University.

Direct interfaces between living neural tissue and electronic circuits have long been desired, both as research and rehabilitation tools. Recent developments in the micromachining of silicon and other materials has led to the ability to fabricate arrays of microelectrodes that offer great promise in this arena. Currently, this technology is being used to fabricate three types of neural interface: penetrating cortical probes, regeneration electrode arrays for peripheral and selected cranial nerve applications, and "electric Petri dishes" for in vitro preparations. Work at Stanford's Center for Integrated Systems in each of these areas will be reviewed. Issues of microfabrication and the potential integration of signal processing circuitry will be discussed, as well as future research directions.

**INVITED**


We have fabricated nanometer-sized interdigitated electrode patterns utilizing electron beam lithography and lift-off techniques. The silicon dioxide surface was then patterned treated with APT for adhesion and promoted selective adhesion of biomolecules in between the electrodes. The aim of the work was to find out whether the dimensions (i.e., the electrode separations) of the pattern would affect the admittance signal of the biomolecules in between the electrodes. The biomolecule we chose to study was horseradish peroxidase. Since the admittance signal scales with the a geometrical factor A/d, where A is the electrode area and d is the separation, we chose to keep A/ d constant when changing the electrode separation in order to eliminate this trivial effect on the admittance signal. Our findings suggest that a much more sensitive "biosensor" is possible to fabricate with nanometer technology than present "biosensors" having dimensions on the sub-micron range. We also found that the biosensor in the nanometer regime—the nanosensor—actually is not only more sensitive but also more biospecific than a larger micro-sensor would be.

3:00 pm NSBI-ThA4 Interfacial Aspects of Acoustic Plate Mode (APM) Biosensor Response, J. Renken, R. Dahini, F. Jossé*, M. Grunze, Universität Heidelberg, Lehrstuhl für Angewandte Physikalische Chemie, Im Neuenheimer Feld 253, 69120 Heidelberg, Germany, *Department of Electrical and Computer Engineering, Marquette University, Milwaukee, WI 53233, USA.

Recently, preliminary results on a mass-sensitive 150-MHz APM biosensor on Zn-LiNbO$_3$ have been reported. It has been shown that the adsorption of non-labeled antigens can be monitored in real-time at the nanogram scale. However, interfacial effects of the antibody film have not yet been taken into account. Therefore, films of different chemical structure were used for the coupling of antibodies to the crystal surface. An aminosilane, a dextran, and a new polyether-polylurea based immunosorbent were prepared and their antigen binding capacities determined by a modified ELISA test. The results show that the antigen binding capacities for the dextran and the polyehter-polylurea films are enhanced by a factor of 4.5 and 2.7, respectively, compared to the amino silane film. Sensor experiments, however, reveal that an increased antigen binding capacity does not necessarily result in a higher dynamic range of the acoustic device. No significant enhancement is observed neither for the dextran film nor for the polyehter-polylurea film. This deviation is discussed in terms of the viscoelastic properties of the different films.


Many solid state gas sensors use the electrical conductance change of a semiconducting oxide as the sensing principle. These devices, usually fabricated as discrete single sensors, typically use elevated temperatures in the range 200°C-300°C to effect the surface reactions required for sensing. A key drawback of this class of sensors is that many different reducing gases will cause a conductance change. Partial selectivity is attempted through the use of catalytic additives to the semiconducting oxide. There is now considerable interest in the application of thin film technology to produce arrays of sensors with different additives to create a selective sensing instrument on a chip. Here we describe an approach which takes advantage of the fast thermal response of micro-machined "hotplates," fabricated on a silicon wafer using CMOS technology. Micro-hotplates have a wide range of temperature control, from 20°C-500°C or 1000°C, depending on design materials, and a fast thermal response of about 1 ms. They may be fabricated in arrays with electrical circuitry on the same chip. We demonstrate operating modes in which ramped sequences of temperature pulses are applied to a micro-hotplate, while monitoring the conductance of a SnO$_2$ film grown over contact pads on the micro-hotplate. The kinetics for adsorption, desorption, and reaction on the sensing surface are thereby controlled by the sensing instrument. A smart sensor would use on-chip pattern recognition techniques to analyze incoming data, and adjust the temperature-pulse sequence to refine composition analysis. Response differences between the vapors of ethanol, methanol, and other gases are demonstrated.

*Dept. of Materials and Nuclear Engineering, Univ. of Maryland at College Park.

3:40 pm NSBI-ThA6 New Techniques of Thermal Imaging Using the Atomic Force Microscope, M. Chandrachood, O. Nakabeppu, Y. Wu, J. Lai, and A. Majumdar, Department of Mechanical Engineering, University of California, Santa Barbara, CA 93106.

Trends in VLSI technology suggest that when micromachined devices are fabricated in the deep silicon range, power dissipation will be a critical factor in chip design and operation. With the goal of developing a temperature measuring technique in the nanometer range to understand the thermal behavior of sub-micron devices, this paper presents two new techniques of thermal imaging using the atomic force microscope (AFM). The first uses the mismatch in thermal expansion coefficients of a metal, such as aluminum or gold, and silicon nitride. When an aluminum film is deposited on a regular silicon nitride AFM cantilever, a temperature change results in cantilever bending. By sensing the temperature-induced deflections while scanning the probe, the temperature of a surface can be measured. An AC heating method is used to separate deflections due to topography and temperature. The second technique uses a microfabricated thin film thermocouple on a regular silicon nitride AFM probe. By scanning the thin-film thermocouple tip on the surface, the surface temperature can be mapped. For both techniques, experiments done in air and in vacuum suggest that due to the small tip-sample contact area, heat conduction through air dominates over that through the tip-sample point contact and could be the limiting factor in the spatial resolution. Thermal images of sub-micron metal lines and semiconductor devices will be presented.

4:00 pm NSBI-ThA7 Initial Tests of a Micromachined SEM, D. A. Crewe, A. D. Feinerman, University of Illinois at Chicago, Chicago, IL 60680.

A Micromachined Scanning Electron Microscope (MSEM) consisting of a square-cm sized silicon die aligned and separated by optical fibers has been built and is in the initial stages of experimental testing. Each die contains a set of four orthogonal anisotropically etched v-grooves and a small (5-200 μm) aperture. Short (5 μm) lengths of precision 300 μm diameter Pyrex fibers are placed in the grooves and a die with a matching v-groove pattern is placed on top of the fibers. The two die are then anodically bonded together. In its final form the MSEM will consist of 6 silicon die aligned, stacked, and bonded from one anode in this manner. One die will contain a field emission electron source, 4 die function as electrostatic electrodes for extraction, focusing, and deflection of the beam, and a final die for detection of secondary electrons. The entire structure measures less than one cubic cm. Arrays of MSMES can also be fabricated for applications in high throughput e-beam lithography.

Our goal is to provide a small, inexpensive low voltage SEM that performs as well as commercially available instruments. Because of
the advantages inherent in size reduction and batch processing the cost of the MSEM is dramatically lower than that of conventional machines. The MSEM operating at 2.5 kV with a working distance of 0.5 mm has a theoretical peak-to-peak voltage of 5.5 kV. In its as-fabricated form the MSEM being tested utilizes a micromachined 3-electrode electrostatic lens, a commercial ZrO2 TFE electron source, and conventionally machined electrostatic parallel plate deflectors. Initial line scan experimental data and early images will be presented along with results of resolution experiments.


4:20 pm NSB-I-ThA8 Localized Photodiode for Near-Field Photodetection Optical Microscopy, R. C. Davis, C. C. Williams, Dept. of Physics, University of Utah, SLC, UT 84112.

In Near-Field Scanning Optical Microscopy (NSOM) an optical fiber with a small aperture at the end is the light collector. In contrast Near-Field Photodetection Optical Microscopy (NPOM) employs a small photodetector placed directly in the near-field as the light collector. We have fabricated a Schottky photodiode localized to the end of a silicon tip for use as the photodetector in NPOM.

A silicon tip has been cleaved to create a sharp tip. We have developed a novel approach for constructing a very small device at the end of a silicon tip without the use of lithography. Using this approach we have fabricated a small Al-Si Schottky diode. The diode contact area is approximately 1 micron square with a sub-micrometer tip curvature. We have electrically characterized the diode by I-V and have measured the optical sensitivity as a function of position on the diode using a micrometer size focused laser spot.


Nano-meter resolution atomic force microscopy (AFM) has led to the development of special-purpose, high-aspect-ratio probes. The probe radius and aspect ratio are the primary determinants of the confluence of the acquired AFM image to the true profile of the sample. Unfortunately, batch fabricated silicon probe geometry (radius, symmetry, roughness and aspect ratio) varies from sensor to sensor. Controlled geometry AFM probes have been developed. High aspect ratio AFM probes of ≈100 nm shaft diameter and microns in length are created by electron-beam induced growth. Focused ion beams have also been used to sharpen conventional probes. This paper extends the application of these techniques to the creation of probes for resonance-mode AFM, and characterizes the performance of the resulting probes.

Combining the advantages of high aspect ratio probes with the reduced lateral and contact forces of resonance-mode AFM provides an improved ability to measure different samples. This has been demonstrated with recently developed tip characterization structures. The advantages of regular tip shape for crystal grain metrology have also been shown. The lower lateral forces of resonance mode scanning significantly reduce artifacts associated with tip flexing that are often observed during contact mode imaging with e-beam grown microtips. A discussion of the probe-forming techniques, and an experimental evaluation of the imaging capabilities will be presented.

2:00 pm NSB-I-ThA8 Observation of Superlubricity by Using Scanning Tunneling Microscope Method, Motohisa Hirano, NTT Interdisciplinary Res. Labs., Musashino, Tokyo 180, Japan; Kazumasa Shinojo, ATR Optical and Radio Communications Res. Labs.; Reizo Kaneko, NTT Interdisciplinary Research Labs.; Yoshitada Murata, The University of Tokyo.

This study aims to observe the state of vanishing friction, called superlubricity, by examining sliding between atomically clean surfaces. Theoretical study prior to experiments found that the case occurs in which frictional forces exactly vanish even in realistic systems such as metals when incommensurately contacting crystal surfaces slide against each other [1]. The measured frictional forces between mica surfaces under very dry atmosphere decrease when the contacting surfaces approach being incommensurate [2]. In this experiment, silicon tips with atomic force microscopy, 0.01 εm, are one-dimensionally scanned against a W tip in UHV by using STM method, and the W tip-deflection caused by the friction appearing between the scanned Si(001) and the W(110) on the polycrystalline W tip is measured. This STM method achieves the sliding with the elastic contact of the surfaces as in the attractive mode of AFM operation. The commensurate contact is obtained by aligning the lattice orientation of the contacting surfaces in such a way that [111] of W(110) corresponds to [100] of Si(001) since the inter-atomic distances are almost identical to each other along these directions. The lattice orientation of W(110) is determined by field emission microscopy and that of Si(001) by low-energy electron diffraction. Typical scanning amplitude is 100 nm and the scanning rate is 0.5 Hertz. We have observed friction of magnitude 10⁻² N, which is comparable to the calculated value, when the contact is commensurate. However, when the contact is incommensurate, friction is not observed in this measurement which can resolve the frictional forces of 10⁻⁴ N. It is thus concluded that the observed frictional anisotropy stemming from the differences in the commensurability of the contacting surfaces implies the existence of superlubricity.


2:20 pm NSB-I-ThA8 Influence of Water Vapor on Nanotribology Studied by Friction Force Microscopy, M. Binnig, Centre Suisse d'Electronique et de Microtechnique (CSEM), CH-2007 Neuchatel, Switzerland; C. M. Mute, IBM Research Division, Almaden Research Center, San Jose, California 95120 USA.

It has long been known that presence of water vapor can dramatically influence the friction and wear properties of materials. In order to simulate the nanotribological effects of adsorbed water molecules and the capillary condensation of water around contactingasperities, we have studied the friction and adhesive forces acting on the tip of an atomic force microscope in contact with a flat surface as function of humidity. On a hydrophilic surface such as silicon oxide, water films grow to tens of nanometers in thickness at high relative humidity, and strong capillary formation occurs around the tip. In contrast, on hydrophobic surfaces such as amorphous carbon films or lubricated silicon oxide, only a few layers of water molecules condense, and the water capillary formation around the tip is greatly suppressed. Friction

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY

Room A207 – NS2-ThA

Nanomechanics and Nanotribology: II


5:00 pm NSB-I-ThA10 Tunnel Sensors for High Spatial Resolution and Sensitivity Force Sensors, R. C. White and J. C. Jiang, Columbia University, New York, NY 10027 USA.

A microwave frequency transducer for implementation in array force sensors has been fabricated and evaluated for performance and reliability. The mechanism of operation is based on cold field emission from a Si emitter which can be fabricated individually or in field emitter arrays. The performance of the vacuum diode is dependent on a nanometer scale radius of curvature at the emitter surface for field enhancement and tunneling current which is exponentially dependent on distance between emitter and anode. Initial devices exhibited stable emission current of greater than 1 mA at bias voltages less than 10V. This result, although stable and reliable for sensor applications, was inconsistent with the physics of cold field emission for these structures, described by the Fowler-Nordheim model. Investigation of individual tip performance has been carried out using a "STM-like" setup where radius of curvature of the anode could be controlled, as well as anode-cathode spacing, and vacuum environment. Results indicate an unstable operating region for the devices, which can result in breakdown of the emitting material if the bias voltage is increased. Such breakdown results in large emission currents, and physical damage to the nanometer scale emitter. Surface modification of the "as-prepared" Si emitters has been performed which allows for better control of the device near the unstable point. This modification is the simple addition of an ultra-thin (<100 Å) metal overlayer. Modeling indicates that the modification creates a resonant tunnel enhancement at the emitter surface.

2:00 pm NSB-I-ThA8 Observation of Superlubricity by Using Scanning Tunneling Microscope Method, Motohisa Hirano, NTT Interdisciplinary Res. Labs., Musashino, Tokyo 180, Japan; Kazumasa Shinojo, ATR Optical and Radio Communications Res. Labs.; Reizo Kaneko, NTT Interdisciplinary Research Labs.; Yoshitada Murata, The University of Tokyo.

This study aims to observe the state of vanishing friction, called superlubricity, by examining sliding between atomically clean surfaces. Theoretical study prior to experiments found that the case occurs in which frictional forces exactly vanish even in realistic systems such as metals when incommensurately contacting crystal surfaces slide against each other [1]. The measured frictional forces between mica surfaces under very dry atmosphere decrease when the contacting surfaces approach being incommensurate [2]. In this experiment, silicon tips with atomic force microscopy 0.01 εm, are one-dimensionally scanned against a W tip in UHV by using STM method, and the W tip-deflection caused by the friction appearing between the scanned Si(001) and the W(110) on the polycrystalline W tip is measured. This STM method achieves the sliding with the elastic contact of the surfaces as in the attractive mode of AFM operation. The commensurate contact is obtained by aligning the lattice orientation of the contacting surfaces in such a way that [111] of W(110) corresponds to [100] of Si(001) since the inter-atomic distances are almost identical to each other along these directions. The lattice orientation of W(110) is determined by field emission microscopy and that of Si(001) by low-energy electron diffraction. Typical scanning amplitude is 100 nm and the scanning rate is 0.5 Hertz. We have observed friction of magnitude 10⁻² N, which is comparable to the calculated value, when the contact is commensurate. However, when the contact is incommensurate, friction is not observed in this measurement which can resolve the frictional forces of 10⁻⁴ N. It is thus concluded that the observed frictional anisotropy stemming from the differences in the commensurability of the contacting surfaces implies the existence of superlubricity.


2:20 pm NSB-I-ThA8 Influence of Water Vapor on Nanotribology Studied by Friction Force Microscopy, M. Binnig, Centre Suisse d'Electronique et de Microtechnique (CSEM), CH-2007 Neuchatel, Switzerland; C. M. Mute, IBM Research Division, Almaden Research Center, San Jose, California 95120 USA.

It has long been known that presence of water vapor can dramatically influence the friction and wear properties of materials. In order to simulate the nanotribological effects of adsorbed water molecules and the capillary condensation of water around contacting asperities, we have studied the friction and adhesive forces acting on the tip of an atomic force microscope in contact with a flat surface as function of humidity. On a hydrophilic surface such as silicon oxide, water films grow to tens of nanometers in thickness at high relative humidity, and strong capillary formation occurs around the tip. In contrast, on hydrophobic surfaces such as amorphous carbon films or lubricated silicon oxide, only a few layers of water molecules condense, and the water capillary formation around the tip is greatly suppressed. Friction

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and adhesive forces during sliding of the tip across the sample surfaces decrease substantially with increasing relative humidity, implying that the surfaces are lubricated by the condensed water.


In a number of mechanical wear situations, a surface experiences simultaneous tribological loading and chemical exposure; the combination can greatly increase wear rates. We have modified our air AFM to permit measured variations in relative humidity during scanning. The relative humidity can be varied from less than 10% up to 95% in 10°C steps and the procedure performed using Si₃N₄ tips with nominal radii of 40 nm, normal loads (Fz) from 1 to 30 nN, and tip velocities from 1 to 200 μm/s. The surface of interest is cleaned single crystal NaNO₃, a hygroscopic salt with rhombohedral calcite structure. Initial scans (dry) exhibit numerous long, sharp 6 Å steps, corresponding to two unit cells, exhibiting high stability. In high humidity, infrequent scans at low Fz show clear uniform step dissolutions at typical velocities of 100 nm/s. With frequent scanning at Fz = 10 nN at high humidity, damaged mechanically stimulated corrosive attack is observed at the steps, producing 3 Å staircases, oriented groves, and possible formation of carbonate from reaction with CO₂ in the air. We present the influence of Fz, tip velocity, and relative humidity on the rates of corrosive wear and possible mechanochemistry.

3:00 pm NS2-ThA5 Nanotribology in Electrochemically Controlled Environment, M. Bingelli, J. Burger and R. Christoph, Centre Suisse d'Électronique et de Microtechnique S.A., 2007 Neuchâtel, Switzerland.

In order to achieve information about nano-mechanical properties of materials and to complement classical tribological research with nanometer scale investigations, friction force microscopy has become a well established tool over the last few years. Simulation of a single asperity contact of a real tribosystem using FFM tip has proven to be a promising approach towards a more detailed understanding of tribological processes. As the surface conditions of two sliding materials determine their friction behavior, rigorous control of these conditions is crucial. Therefore, a standalone bidirectional force microscope, the atomic scale tribometer, has been combined with an electrochemical environment, which enables to control the cleanliness and thermodynamics of the sample (and) tip surface. The standalone design allows for scanning the probe (not the sample) and therefore no restrictions to the electrolyte cell design and weight apply. Sliding experiments carried out on graphite in 0.1 M NaClO₄ electrolyte evidenced the potential of this method. Lateral forces on the tip during the scanning process (conventionally: friction) show a clear dependence upon the applied electrochemical potential and therefore the chemical state of the surface. Friction force microscopy in electrochemical environment is thus the first tool capable of detecting local electric double-layer properties, thereby contributing analytical information for the modelling of this study significantly disputed subject.

3:20 pm NS2-ThA6 Friction and Adhesion Properties of Hard Carbon Surfaces Measured by Atomic Force Microscopy, Scott S. Perry and Seher A. Somorjai, Lawrence Berkeley Laboratory and Department of Chemistry, University of California, Berkeley, CA 94720, C. Mathew Mote, IBM Almaden Research Center, 650 Harry Road, San Jose, CA 95120.

The friction and adhesion properties of amorphous carbon films have been studied on the nanometer scale by atomic force microscopy (AFM). Sputtered hard carbon films are used as contact mechanics applications including computer disk drives. Interaction forces between a sharp tungsten tip and the surface of the carbon films have been measured as a function of chemical composition and structure. Normalization of adhesive forces at different surfaces has been accomplished by estimating radii of curvature through electroscopic measurements. Variations of hydrogen content within the films, surface oxidation and the hardness of lubricant films all influence the tip-surface adhesion. Friction forces between a sliding tungsten tip and carbon surface have been measured simultaneously to load forces allowing the calculation of coefficients of friction. Friction forces are seen to vary with both the chemical and topographic structure of the films. The relevance of atomic force measurements to industrial tribological applications will be discussed.

4:00 pm NS2-ThA8 Nanotribological Investigations of Epitaxial C₅₀ Films on Ge(001) by Lateral Force Microscopy and Force Spectroscopy, W. Allers, U. D. Schwarz, G. Gensterblum* and R. Wiesendanger, University of Hamburg, D-20355 Hamburg, Germany; *Facultés Universitaires Notre-Dame de la Paix, B-5000 Namur, Belgium.

C₅₀ films. We have applied lateral force microscopy and force spectroscopy to study the variation of the local frictional coefficient on submonolayer C₅₀ films epitaxially grown on Ge(001) substrates under ultra-high vacuum conditions. The local frictional coefficient derived for the C₅₀ islands was found to be significantly smaller than on the layer of Ge(001) substrate (see figure). This demonstrates that well-ordered C₅₀ films can indeed lower the frictional force even compared with a layered substrate. On the other hand, graphite was found to exhibit significantly better lubricating properties compared with the C₅₀ films.

4:20 pm NS2-ThA9 UHV Boundary Lubrication of the Cu(111)/Cu(111) Interface by Submonolayer Coverages of Trifluoroethanol, Christopher F. McFadden, University of Illinois at Urbana-Champaign, Urbana, IL 61801; Andrew J. Gellman, Department of Chemical Engineering, Carnegie Mellon University, Pittsburgh, PA 15213.

The ability of adsorbed films of molecular thickness to lubricate solid-solid interfaces is of critical importance to many areas of technology and engineering. An ultrahigh vacuum (UHV) tribometer has been used to study the friction behavior of Cu(111) surfaces separated by adsorbed films of 2,2,2-trifluoroethanol ranging from ~0.05 monolayers (ML) to ~10 ML thickness. The instrument is designed such that both samples may be prepared and characterized under UHV conditions and then brought into contact and sheared relative to one another without exposure to atmosphere. Shear and normal forces are measured independently and simultaneously during sliding. The friction behavior of clean Cu(111) surfaces is characterized by high, erratic friction forces with static friction coefficients in the range 3–8. The friction coefficient is reduced to approximately 0.87 by as little as 0.05ML of trifluoroethanol and remains constant at coverages up to 1ML. This represents the first demonstration of boundary lubrication of metals by submonolayer films and challenges the classical view that a close-packed monolayer film is prerequisite for effective boundary lubrication.
APPLIED SURFACE SCIENCE
Room A101 – Session AS-ThA

Polymer/Organic Surfaces
Moderator: A. B. Ulman, Eastman Kodak Company.

2:00 pm  AS-ThA1 Polymer Surface Damage by X-ray Photoelectron Spectroscopy, W. F. Steckle, P. E. Sobol, H. Iwai, Physical Electronics Laboratories, 6509 Flying Cloud Drive, Eden Prairie, MN 55344.

X-ray photoelectron spectroscopy is often regarded as not damaging to surface chemistry. Polymer surfaces can show degradation during analysis, especially with non-monochromatic x-ray sources because of thermal effects, exposure to Bremsstrahlung radiation, or interaction with low energy electrons. With the common use of modern monochromatic XPS, damage to a polymer surface during analysis is minimized or eliminated, except in the cases of many halogenated polymers. While some damage to polymer surfaces can still occur, the resolution and sensitivity of modern spectrometers makes it possible to more closely examine the effect of monochromatic radiation damage to a polymer surface. Using high by focused x-ray sources and focused electron optics for analyzers, changes in surface chemistry can be especially important for polymers as well as other materials. Chlorinated and fluorinated polymers have been studied using different analytical conditions. Experiments were conducted by varying the x-ray flux density by using focused and broad beam monochromatic radiation combined with analysis from different size areas. From these experiments a methodology for examining polymer degradation will be discussed by contrasting the different analysis conditions.


We demonstrate that high-resolution electron energy loss spectroscopy (HREELS) can be used to elucidate the surface vibrational structure of both <30 Å thick and thick (>1 micron) polymers. The effects that charge neutralization may have on vibrational selection rules will be addressed. Experimental conditions necessary to achieve a unique vibrational identification of the polymer surface will be discussed. Examples include end-group terminations in polyethylene and polycarbonate, surface reorganization of end-functionalized polyesters, surface chemical reactivity of oligomeric vinylidene chloride, and plasma/corona modification of polyethylene terephthalate.


In medical grade silicone (poly(dimethyl siloxane)) (PDMS) elastomers, silica filler is added to improve mechanical properties. This study asks a question important to the performance of silicone medical devices: can the filler be observed at the surface? Silicone elastomer surfaces were studied by ESCA, SSIMS, SEM, and AFM. Specimens were challenged by abrasion, enzymatic, and peroxide treatments.

Standard materials were analyzed to establish that SiO2 and PDMS could be distinguished. A complex curve envelope associated with adventitious carbon contamination was present in the ESCA C1s spectrum for all silica standards. Although the high resolution Si 2p envelope will fit with one peak, with a binding energy (BE) consistent with SiO2 (104.0 eV), a low BE shoulder was present in all spectra. This may be indicative of sample charging or a low level of a (CH3)2SiO (102.4 eV) contaminant.

The elemental composition for “silica-free” silicone was near the expected theoretical values for PDMS. The BE’s were consistent with C-1s (285.0 eV), (CH3)Si-O (532.6 eV) and Si-O (102.4 eV). SIMS spectra for an SiO2 standard were consistent with the negative ion spectra for silica with peaks at 60 m/z and 76 m/z. Although there appears to be a small 76 m/z feature in the negative ion mass spectra for some of the silicone materials, there is no enhancement of the 60 m/z mass fragment one would expect if silica were present. The SIMS negative ion mass fragments for biomedical silicone elastomers do not indicate silica is present in the top 15 Å of the samples. Negative ion peaks at 39, 60, 75 and 149 m/z, as well as, positive ion mass peaks at 73 and 147 m/z are consistent with PDMS. Thus, on all silicone biomedical elastomers, by ESCA or SIMS, only PDMS was found in the outermost 10–80 Å.

The possibility of silica coming to the surface upon hydration of the elastomer was also investigated. No silica was observed by ESCA of a frozen-hydrated specimen following a 24 hour soak in water.

3:00 pm AS-ThA4 High Resolution XPS and ToFSIMS Studies of Polyamidoamines, A. G. Shand, M. C. Davies, S. J. B. Tendler, G. Broomfield, A. J. Paul, L. Bartore*, and P. Perrut*, Department of Biophysics and Surface Analysis, Department of Pharmaceutical Sciences, The University of Nottingham, Nottingham, NG7 2RD, UK, SERC, Daresbury Laboratory, *CISMA Ltd., *Universita di Brescia.

The surface analysis of polymers containing amine groups is an area which has not received a great deal of attention. In this work we describe for the first time high resolution X-Ray Photoelectron Spectroscopy (XPS) and Time of Flight Secondary Ion Mass Spectroscopy (ToFSIMS) analysis of a novel series of polyamidoamines, polymers which are of interest to the biomedical community.

The Scienta ESCA300, which attains an instrumental resolution of well below 1 eV was employed for XPS analysis. Following the deconvolution of C1s, O1s, and N1s regions the component peaks within each spectra revealed detailed information concerning the chemical environments of each element. In addition, degradation of some of the polymers under X-Ray irradiation was investigated and valence band spectra recorded. Polymers with closely related compositions could be readily distinguished in the ToFSIMS spectra. High resolution ToFSIMS work allows accurate ion assignment and second ary ions are interpreted as being derived from intact polymer structural units, which implies the occurrence of specific fragmentation mechanisms. A good understanding of polyamidoamine surface chemistry has been achieved which will prove invaluable in future studies of similar materials.


Corona discharge treatment is widely used in the industrial processing of polymeric films, particularly to improve wetting and adhesive properties. Corona discharge treatment in air leads to the formation of a complex low-molecular-weight oxidized material (LMWOM) which has been only partially characterized by standard surface analysis methods. We utilize in these experiments laser desorption ionization mass spectrometry to analyze LMWOM which has been solven extracted from the surface of corona-treated polypropylene. Molecular weight distributions are determined as a function of total discharge energy and relative humidity of the ambient air. A comparison of mass spectrometric data with prior ESCA, IR, and contact angle measurements permit at least partial elucidation of the chemical composition of the LMWOM. These results indicate the general feasibility of using ion trap mass spectrometers for the analysis of complex organic surfaces.

3:40 pm AS-ThA6 Addition of a Single Chemical Functional Group to a Polymer Surface with a Mass-Separated Low Energy Ion Beam, P. Nowak and N. S. McHenry, Surface Science Western and Department of Chemistry, University of Western Ontario, London, On-
tario, Canada N6A 5B7; I. Bello and W. M. Lau, Department of Materials Engineering, University of Western Ontario, London, Ontario, Canada N6A 5B7.

A 10-electron laser ion beam system was used to deliver pure OH* and NH* to 15nm thick polystyrene films on silicon in ultrahigh vacuum for the study of polymer surface modifications yielding specific surface chemical functional groups. X-ray photoelectron spectroscopy showed that when the bombardment energy of OH* exceeded 10 eV or dose was higher than 1 x 10^12/cm^2, the introduction of a mixture of C-OH, C-O-O, and C-OOH together with a severe damage of the polymer rings, was observed. However, for bombardment at 10 eV with a dose of 1 x 10^12/cm^2, only C-OH (or COR) was found. Similarly, bombardment with NH* at 10 eV and a dose of 1 x 10^12/cm^2 induced nitrogen incorporation with a symmetrical N= peak, which suggests the absence of vastly different surface functionalities. The C 1s data on such surfaces indicated the major surface functionality being C-NH2 (or C-NH) with a minor component of C-(N=)

4:00 pm AS-THA7 Polyfluorother Lubricant Analysis by TOF-SIMS, P. Kasai, A. M. Spool, IBM Storage Systems Division, San Jose, CA 95193.

Perfluoro-poly(ether) (PFPE) produce rich and intense SIMS spectra. This study represents an attempt to further understand how to interpret these spectra. We compare and contrast TOF-SIMS analyses of dimethyl, kryptof, perfluoro-poly(dioxolane), and Z-formblins. For all of these compounds, the negative ion spectra have intense regular peak patterns that persist up to high masses approximating the mean MW for the polymer. In contrast, the positive ion spectra are much more complex, with less pronounced repeat patterns at high masses. The results suggest differences in ion formation/decay mechanisms between positive and negative ions. The dimethyl molecule, with its symmetric monomeric unit, produces two patterns in the negative ion spectrum that differ by the weight difference of the 2 end groups. The other compounds studied here have asymmetric monomers and produce spectra indicative of asymmetric cleavage. The negative ion fragmentation patterns of the Z-formblins can be modeled with a random distribution of the 2 monomers that comprise these polymers, and with symmetric fragmentation of the polymer. The addition of functionalized endgroups of these molecules has surprising effects on the TOF-SIMS spectra. The endgroups affect either ion formation probabilities or ion lifetimes.


Well ordered Langmuir-Blodgett monolayers of Poly(methyl methacrylate) have been studied with ToF-SIMS. The high mass range from 1000 to 3000 Da. is investigated in the present study. Samples of different thicknesses were prepared at two different surface pressures on a Langmuir-Blodgett trough. The surface coverage was confirmed by an ESCA study.

A Reflectance-Absorption FT-IR study of the samples provides information of the configuration of the polymers on a silver substrate. Previous work by Brinkhuis was compared to confirm the results.1

The ToF-SIMS results of L-B films of polymers with different tacticities have been analyzed following previous published approaches based on statistical chain breaking to form radical ions.2 Clearly, different ion formation patterns result from different structures (e.g., atactic, isotactic, syndiotactic) leading to the conclusion that polymer tacticity structures may lead to different ions. This will lead to a better understanding of the effects of polymer ion formation in ToF-SIMS.


A better understanding of polymer interfaces is being driven by the need to develop new materials for coating and adhesive applications.

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is emerging as a powerful analytical probe for unambiguously identifying surface species that can impact a material's performance. Here, we report on our efforts to elucidate the interfacial properties of poly(vinyl acetate-ethylene) copolymer (VA-E) based adhesives bonded to poly(vinyl chloride) (PVC) substrates. We find surface segregation of plasticizers, found within certain grades of PVC, contributes to the delamination of the VA-E adhesive from the PVC substrate. In addition, ToF-SIMS provides valuable information on the spatial distribution of components which contribute to adhesive failure.

5:00 pm AS-THA10 Morphological and Nanomachinery Investigations of Surface Modified Polytetrafluoroethylene, A. J. Howard, R. R. Rye, P. Tanguyong, and J. E. Houston, Sandia National Laboratories, Albuquerque, NM 87185.

Using both atomic and interfacial force microscopies (AFM and IFM), we have studied the surface morphology and nanomechanics properties of surface modified virgin poly(tetrafluoroethylene) (PTFE). The modified surfaces of PTFE were prepared by exposure to electron-beam irradiation, wet chemical etching in a sodium naphthalene solution, and a combination thereof. These surface treatments are used in our laboratory in combination with conventional integrated-circuit processing techniques to pattern adherent conductors directly on low dielectric constant PTFE substrates for high frequency device applications. AFM images (performed in tapping-mode since contact-mode AFM on this soft material yielded images dominated by tip-induced artifacts) show that stress dependent crazing resulting from chemical etching is necessary for strong metal adhesion to etched PTFE. IFM force versus displacement curves and creep experiments are used to probe the changes in mechanical properties of PTFE resulting from electron-beam irradiation. This information will be used to discuss why poor metal adhesion to irradiated and etched PTFE surfaces is observed. These nanomechanics results, as well as our AFM and SEM (or comparison) morphological data will be presented.

This work was performed at Sandia National Laboratories and supported by the U.S. Dept. of Energy under contract DE-AC04-94AL85000.

PLASMA SCIENCE

Room A109 Session PS1-THA

Plasma Surface Interactions
Moderator: M. Nakamura, Fujitsu Ltd., Japan.

2:00 pm PS1-THA1 Chemical Topography of Masked Poly-Si Films Etched in Cl2 and HBr-Containing, High-Density Plasmas, V. M. Donnelly, C. C. Cheng, K. V. Guinn, and J. P. Herman, AT&T Bell Laboratories, Murray Hill, NJ 07974.

The spatially-resolved adsorbate composition, or "chemical topography", of small features (polycrystalline Si masked with photore sist or SiO2 stripes) etched in Cl2, HBr-containing plasmas has been quantitatively determined by angle-resolved x-ray photoelectron spectroscopy (XPS), using shadowing of photoelectrons by adjacent features and geometric modeling. The plasma reactor consisted of a helical resonator source and a radio-frequency biased substrate stage. Samples with equal line and space width features (0.5-2 μm) were etched and then transferred under high-vacuum to the XPS chamber. Real-time, laser-induced thermal desorption showed that the Si-halide layer formed during etching was stable during the sample transfer period. For resist-masked samples etched in Cl2 plasmas, ~1 x 1014 Cl-atoms/cm2 cover vertical and horizontal surfaces of poly-Si and resist. Little or no C or O was found on the poly-Si, and a trace amount of Si was found on the side (but not top) of the resist. Analogous results were found with pure HBr plasmas, with slightly less Br covering all surfaces. Adding O2 to either Cl2 or HBr plasmas increases O coverage on the poly-Si sidewall, and leads to formation of a relatively thick SiO2 layer on the side of the resist. INVITED

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(Also at Columbia Univ., New York, NY.

2:40 pm PS1-THA3 Reactivity of Plasma Radicals with the Surface of Depositing Films, N. F. Dalleska and Ellen R. Fisher, Department of Chemistry, Colorado State University, Fort Collins, CO 80523.

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Understanding the interactions of molecules with surfaces is important for development of thin-film materials processing technologies, although many direct reactivity studies have been made during deposition. Direct measurements of radical reactivities are thus vital to acquiring knowledge of the complex chemistry of chemical vapor deposition (CVD) and plasma enhanced CVD processes. The presentation will center on a relatively new technique, IRIS (Imaging of Radicals Interacting with Surfaces), which combines spatially resolved laser-induced fluorescence (LIF) and molecular beam methods to measure the reactivity of radicals with the surface of depositing films. The technique also employs a state-of-the-art gated and intensified charge coupled device array detector. The radicals studied are observed in materials-processing systems and may be important to such processes as plasma deposition of Si, SiO2, Si3N4 and diamond thin films. Indeed, many proposed mechanisms indicate that small molecules such as SiH, SiO2, CH, CF, and CF2 play significant roles in these deposition processes. Previous results show major differences in radical reactivities, indicating that additional results for a large number of molecules are needed. Preliminary reactivity results on some of these radical species using a newly constructed IRIS apparatus at Colorado State University will be presented.

3:00 pm PS1-ThA4 Molecular Dynamics Simulation of Atomic Layer Etching of Si(100), Satish D. Athavale and Demetre J. Economou, Plasma Processing Laboratory, Department of Chemical Engineering, University of Houston, Houston, TX 77204-4792.

Atomic Layer Etching (ALET) is a technique capable of etching a crystalline solid with monolayer accuracy. ALET is a cyclic process consisting of: (1) adsorption of a monolayer of gas onto the solid surface, (2) evacuation of excess gas, (3) exposure of the surface to energetic particles (ions, electrons, photons) to induce reaction, and (4) pump out of the reaction products. These steps are repeated in a cyclic manner so that a monolayer of material is removed in each cycle. To achieve monolayer etching, it is necessary to limit reaction to the topmost layer. We have used molecular dynamics (MD) simulations to study the effect of Ar ion bombardment on the chemistry of a Si(100) surface covered with a monolayer of Cl atoms. In contrast to other studies, the surface exposed to ions only, without simultaneous neutral bombardment. MD results elucidate the effect of ion energy (<100 eV) and flux on the surface reaction, product distribution, and the extent of lattice damage and surface roughness. The goal is to determine the range of ion energy and flux to achieve ALET. Simulation results are used to guide an experimental ALET system.

3:20 pm PS1-ThA5 Generation of Ion Radicals by a Fragmentation Process on Surface, Y. Mitsuioka,1,2 S. Takahashi,1 H. Toyoda,1 S. Mukainakano,1 T. Hattori,2,3 H. Sugai,1 a) Nagoya University, Nagoya 464-01, Japan; b) Research Laboratories, Nippondenso Co., Ltd., Nissin-cho, Aichi-ken 470-01, Japan.

There has been a great need to understand ion-surface interactions in plasma-assisted deposition and etching. In this paper, we describe observation of radical generation by fragmentation, i.e., dissociative scattering on surfaces: a polyatomic ion breaks up on solid surface into smaller radical fragments (radicals). First of all, an ion species CH2+ (x = 2-4) is extracted through a mass filter from a methane plasma and irradiated on an aluminum target. The dependence of production rate of each fragment ion on the primary ion energy is measured, together with the branching ratio. In the case of CH2+ ion, two types of dissociation processes are clearly found, depending on the incident energy E. One is the dissociation into CH+ and CH2+ for E < 30 eV, and the other is the fragmentation into CH+ and C for E > 100 eV. Tentatively, the former is explained by dissociation of the molecule excited electronically with charge neutralization on the metal surface, and the latter by dissociation of the molecule excited vibrationally due to high translational energies. Further details will be presented, together with another experiment on fragmentation of fluorocarbon ion, a key radical CH2F+ (x = 1-3) in reactive ion etching.

3:40 pm PS1-ThA6 Aspect Ratio Dependent Etching of Polysilicon, T. J. Dalton and H. H. Sawin, Massachusetts Institute of Technology, Cambridge, MA 02139.

Aspect ratio dependent etching (ARDE) phenomena (e.g., RIE Lag) were measured for polysilicon etching in an HBr/Cl2 chemistry on an Applied Materials Precision 5000 plasma etcher using a CCD camera to measure etch rate in situ by spatially resolved optical emission interferometry. A test mask containing gratings of various dimensions was used. We were able to measure variations of etch rate with aspect ratio of only a few percent. Such measurements are very difficult to make by measuring film thicknesses from SEM photographs for a particular etch. ARDE was found to be a function of residence time (gas flow rate), pressure, magnetic field strength, temperature, feed gas composition, and the seasoning/history of the etcher. At high gas flow rates, no ARDE was seen; decreasing the flow rate resulted in RIE Lag (smaller features etched slower than larger features). Increasing pressure resulted in increased RIE Lag due to ion shadowing caused by the increase in off-normal ion bombardment. In addition, decreasing the pressure decreased ARDE. Decreasing the cathode temperature below 0°C resulted in inverse RIE Lag (see figure). Etch rate decreased more rapidly in large features/open areas due to unimpeded deposition. Narrow features experienced a slower decrease in etch rate due to the sticking of depositing species on the sidewalls.

4:00 pm PS1-ThA7 Real-Time Investigation of DC Bias Effects on Ultra Thin Silicon Oxide Growth in an Oxygen Plasma, M. Kitajima, T. Kamioka, and K. G. Nakamura, National Research Institute for Metals, 1-2-1 Sengen, Tsukuba, Ibaraki 305, Japan.

We have studied positive and negative bias effects on the plasma oxidation of silicon using real-time ellipsometry, to understand the oxidation kinetics in the ultra thin film region. The oxidation experiments were performed in UVH chamber having a background pressure of 10-6 Pa. A dry oxygen plasma was generated by a radio frequency (rf) discharge (frequency of 13.56 MHz, and power of 300W) with 2.0 Pa O2. The sample used was p-Si (111) wafer, and the distance from the rf coil to the sample was 1 m. Applied bias to the sample were −60V, −30V, −15V, +15V, +30V, +45V, and +60V. The oxide film growth was monitored with a time resolution of 1 s by using a PEM-type ellipsometer. We found that the plasma oxidation rate was strongly affected by the sample bias, and the bias effect depends on oxidation stage (oxide film thickness). For longer time oxidation (thicker oxide film), the oxidation rate increased as positive bias increases, but little effect was observed for negative bias. This shows that the dominant oxidizing species are negative ions. For initial oxidation stage immediately after starting the plasma ignition (thinner oxide film), the negative bias also enhanced the plasma oxidation rate; the oxidation rate increases as both the positive and negative biases increase with showing a minimum at +15V. This suggests that both positive and negative ions contribute in the initial oxidation process to the plasma oxidation of silicon surface. Results of XPS measurements will be also described.


Plasma processing produces damaged, micro-roughened semiconductor surfaces due to low-energy ion bombardment. We have quantified ion-induced point defect production experimentally on the Ge(001) surface in real time using in situ Reflection High Energy Electron Diffraction. We have developed Monte Carlo simulations of defect flow to major defect recombination in the bulk and on the surface, as well as to predict distribution profiles of defects percolating to the surface. Defect production statistics generated by our binary collision simulator, TRIMRC, coupled with our simulation of bulk defect diffusion, predict the number of ion-induced defects that reach the surface and suggest that TRIMRC may overestimate the depth
distribution of the defects. The experimental defect yield decreases as the substrate temperature increases from 175 to 475 K. Our Monte Carlo simulations of surface diffusion (applicable to any crystalline surface) support a defect annealing mechanism that involves surface recombination of defects generated within a single cascade (at low ion fluxes).

This work was performed at Sandia National Laboratories and supported by the U.S. Dept. of Energy under Contract DE-AC04-94AL85000.

*AWU-DOE Fellow, U. of Texas, dissertation work performed at SNL.

**Supported by NSF grant CHE 8920120.

4:40 pm  **PS1-THA9** Characterization of Ion Directionality in Plasma Enhanced Chemical Vapor Deposition of Silicon Dioxide, Jundong Li, Calvin Chang, James P. McVittie, and Krishna C. Saraswat, Center for Integrated Systems, Stanford University, CA 94305.

Plasma Enhanced Chemical Vapor Deposition has emerged in recent years as an important technique for the deposition of interlevel dielectric (ILD) and intermetal dielectric (IMD) materials in modern multilevel ULSI technology, especially when the device dimension decreases to sub-micron level. The main concern here is the filling of high-aspect-ratio structures. The step-coverage ability of a PECVD process is directly related to the spatial angular distributions of the incoming species (probably ions) but this has never been carefully studied. The ion angular distribution depends on the initial ion temperature, bias power, and the sheath transport kinetics, and is, however, very difficult to be experimentally characterized due to the complexity of real processes. In this work a profile simulator and an experimental overhang test structure have been used to study the contribution of directional fluxes in different PECVD systems. Detailed comparison between experimental results and simulation shows that for conventional PECVD processes (several Torr), both a directional (sticking coefficient of 1) component and an isotropic (probably thermal CVD) component are observed, whereas for low pressure ECR CVD system the directional component dominates. It is found that this directional component is consistent with the kinetics model of ion transport. In general, the ion directionality plays a major role in determining the conformity of PECVD processes and the characteristic shape of this directionality can be extracted from test structure results. This work will help to clarify the mechanism of ion induced deposition and makes it possible to provide a useful design tool to help optimize the PECVD processes.

5:00 pm  **PS1-THA10** An Auger and XPS Study of Ar⁺ Sputtering Yield and Angular Distribution of Al, P. C. Smith, R. B. Turkoi, J. P. Kelly, D. N. Rutze, University of Illinois, 103 S. Goodwin, Urbana, IL 61801.

Sputtering due to low energy ions occurs in many applications including plasma processing and magnetically confined plasma devices. A surface collector technique for the measurement of low energy sputtering has been investigated. A sample taken from an Al 1% Cu magnetron sputtering target has been exposed to 700 eV Ar⁺ beam. The sputtered material was collected on a p-doped silicon wafer that was subsequently analyzed with angularly resolved XPS and Auger surface analysis techniques to determine the density distribution of Al across the collector. This data was analyzed and compared to a reference wafer to determine the total sputtering yield and the angular distribution function of the sputtered material. In addition, the sputtered Al target was analyzed to determine the changes in its topology and to look for indications of resputtering. This experimental setup has also been modeled and compared to the vectorized fractal TRIM code for both reflection and sputtering. Results indicate the expected aluminum distribution in lower concentrations than predicted by computer models.

2:00 pm  **PS2-THA1** Target Area Design Basis and System Performance for the National Ignition Facility, Michael T. Tobin, V. Karpenko, K. Hagans, A. Anderson, Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, CA 94550, and R. Wavrik, and R. Garcia, Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185.

The Department of Energy is proposing to construct the National Ignition Facility (NIF) to achieve ignition and modest gain in the laboratory early in the next century. The NIF will consist of a 1.8 MJ, 0.35 μs laser with ~200 beams. This is a 50-fold increase in laser energy over the Nova laser at LLNL. Target experiments will be positioned and contained in a 5-m radius, 10-cm thick sphere. Diagnostics are positioned around the equator of the sphere to collect data. NIF is expected to make major contributions to the evolution of Inertial Fusion as an energy source, to defense sciences national security needs, and to basic science research. The specific requirements of each user community for the NIF Target Area is currently under evaluation and will be considered in the design process.

The NIF Target Area is designed to safely contain an annual yield of 385 MJ distributed among monthly ~20 MJ shots, weekly ~5 MJ shots, and ~100 kJ shot on a daily basis, a total of over 145 shots that produce significant D-T neutron yield. In addition, another 500–1000 non-yield shots will also be conducted. The environments created by this range of performance introduce unique design issues.

Solutions to these issues required development of new predictive capabilities and validation of existing capabilities in assessing the in-chamber material responses to multiple exposures of unconverted laser light, x-rays, debris, and neutrons, and the consequences to the overall system of those responses. Confidence in overall system performance was developed by evaluating the ability of the various target area systems, once designed to meet their individual design requirements, to interact safely, affordably, and with low risk to meet the required shot rate.

1:30 pm  **PS2-THA2** Target Fabrication For Inertial Confinement Fusion Moderator: H. G. Kim, University of Rochester.

2:40 pm  **PS2-THA3** Precision Shell Characterization Using Radial Averaging of X-ray Images, R. B. Stephens, General Atomics, P.O. Box 85608, San Diego, CA 92186-9784.

Measurement of shell dimensions from X-radiograph plates are normally limited by film graininess and camera resolution. We have taken advantage of the circular symmetry of microsphere X-ray images to substantially improve the resolution with which surfaces can be detected, and to detect interfaces between layers with only slight differences in X-ray absorption. This capability has been created with a desktop computer and slight modification to a freeware image processing program.

The digitized image of a shell is analyzed in three steps. 1) A radial averaging routine finds the image center to sub-pixel accuracy by maximizing the amplitude of the peaks in the 360° averaged radial intensity profile. 2) The 2nd derivative of the radial intensity profile is displayed as a rectangular plot of distance vs. radial angle. 3) The 2nd derivative of the 360° average of the radial intensity profile is plotted.

Peaks occur in the 2nd derivative plots at interfaces between materials of differing X-ray absorption strength. Therefore the rectangular plot shows a bright line indicating the radius of the shell surfaces and interfaces as a function of angle; fluctuations in radius ≤1 μm can be detected. The peak location can be located with a reproducibility of 0.25 μm off the graph of 2nd derivative of 360° averaged radial intensity profile.

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Such precision requires minimization of vibrations in the exposure and distortions during digitization.

3:00 pm **PS2-ThA4** High Yield Fabrication of Uniform, Large Diameter Foam Shells for Laser Fusion Targets Using Polymerization by Photo Initiation with UV Light, M. Takagi, Y. Kobayashi, T. Norimatsu, Y. Itawa and S. Nakai, ILE Osaka University, Yamadaoka, Suita, Osaka, 565, Japan.

In ignition experiments, one (1)-mm-diameter foam shells with 100-μm uniform walls will be used as liquid fuel sustainers for a laser with output power of several hundred kilo joules. The low density, uniform foam layer is saturated with liquid deuterium and tritium fuel to make a uniform hollow fuel layer and covered with a membrane to prevent the evaporation of the fuel.

In a previous work [1], we heated hollow water/oil/water emulsions to start polymerization. The temperature change, however, induced density mismatching and breaks of the hollow emulsions resulting very low yield for uniform foam shells. Recently we have developed a new technique to make such foam shells using polymerization of trimethylolpropane trimethacrylate (TMPT) initiated by UV light. The TMPT and polymerization initiator were dissolved in a mixture of diethyl phtalate and di-n-butyl phtalate (oil phase). The reaction was injected into a water bath using a dual orifice droplet generator to make a cylindrical oil column that broke into hollow water/oil/water emulsions with constant diameter and wall thickness. The emulsions were irradiated at room temperature using a high pressure UV lamp to gel the oil phase. After several rinse processes, the wet foam shells were dried in a critical point drying vessel using carbon dioxide as the drying fluid.

Since there is no temperature change in the process from formation of hollow emulsions through gelation of the oil phase, the yield for uniform hollow foam shells was greatly improved.

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3:20 pm **PS2-ThA5** The Historical Development of the Microencapsulation Technique Used to Fabricate ICF Capsules, U. Kado, H. Nakano, and H. Tsukahara, Kinki Univ., Dept. of Electrical Eng., Higashiosaka, Osaka 577, JAPAN.

The historical aspects regarding the development of the microencapsulation technique used to fabricate polymer capsules are presented. Initially, the production of paraffin capsules was attempted using this technique, which proved to be unsuccessful. However, polysulfone capsules were easily fabricated using microencapsulation at ambient temperatures. Recently, similar techniques have been used to fabricate foam capsules and polymer-layer-overcoated foam capsules. Foam capsules and their derivatives may make it possible to prepare cryogenic targets for ignition and ignition-scale experiments. The microencapsulation technique has been extended to fabricate polyvinyl alcohol (PVA) capsules overcoated with a polysulfone layer. These capsules retain fuel for a prolonged periods of time, and the polysulfone layer protects the hygroscopic PVA layer from degrading due to airborne water vapor. The microencapsulation technique produces capsules with diameters ranging from 50 to 1000 μm, PVA layer thicknesses of 6-10 μm, and 10-20-μm-thick polysulfone layers.

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4:00 pm **PS2-ThA7** Vapor Deposited Solid Hydrogen Crystals: Size, Structure, and Roughening, Gilbert Collins, Evan Mapoles, Wilt Unites and Tom Berneit, Lawrence Livermore National Laboratory, Livermore, CA 94550.

Grown slowly from the liquid phase, solid hydrogen crystals can easily be made very large, and at temperatures above 4 K have an HCP crystal structure. This tendency to form large crystals (several millimeters in extent) with flat crystal facets presents a problem when forming smooth, spherical hydrogen shells for ICF targets. We report the crystal size and crystal structure of solid hydrogen deposited from the gas phase from the triple point temperature down to 3 K. Hydrogen and deuterium crystals, deposited below half their respective triple point temperatures are microcrystalline with a mixed HCP + FCC crystal phase. This mixed phase transforms into an HCP phase continuously and irreversibly as the temperature increases through half the triple point temperature. During this crystal structure transformation the crystallite size increases.

As the deposition temperature approaches the triple point, single crystals of millimeter extent are easily formed. We report the roughening transition temperature from these crystals for two specific crystallographic orientations of the HCP lattice. Finally, above the roughening transition temperature, the equilibrium facet curvature is still larger than the curvature needed for NIF targets.

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Rayleigh-Taylor instabilities during implosions of inertially confined fusion (ICF) capsules effect capsule performance. During acceleration, surface imperfections grow and can, if large enough, lead to an asymmetric implosion or even shell breakup. For this reason, characterizing the topography of target capsules is extremely important. We have developed a profilometer based on an atomic force microscope combined with a precision rotary air bearing. Averaged 1D surface height power spectra obtained with this instrument are converted to 2D mode spectra, which are used as input to LASNEX simulations. We describe the design of the system and its performance in terms of runout and repeatability. We will also discuss the simulation of these measurements and the statistics in the averaging 1D power spectra. Finally, we will show the application of this measurement technique to capsules that have been laser-ablated, resulting in a well-defined surface topography. This special case provides an excellent test for the system, since the expected results are exactly calculable.

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4:40 pm **PS2-ThA9** Cryogenically Resealable Vacuum Seals,* N. R. Alexander, General Atomics, P.O. Box 85608, San Diego, CA 92196-8754.

General Atomics is developing a transfer cryostat that will permit gas cooled inertial confinement fusion (ICF) targets to be transferred cryogenically (T < 20 K) from a cryostat where they are filled to a cryostat where they will be layered and inserted into the target tank of the OMEGA Upgrade ICF facility at the University of Rochester's Laboratory for Laser Energetics. The transfer cryostat's bottom sections will be resealed inside of the fill cryostat and vacuum reestablished between the sections. Vacuum seals have been developed that allow resealing while at cryogenic temperatures. A number of designs and materials were investigated. The best design material combination had a median leak rate of 2 x 10^-12 mbar.l/sec for differential helium pressure of 100 torr and temperature 15 K. The median leak rate was computed from data gathered from a seal continuously kept at 15 K while being unsealed and resealed. During one test run, a seal was successfully resealed 11 times before the test was demonstrated.

The seal developed easily allows for the vacuum spaces of the transfer cryostat to be evacuated with a modest pump, once the bottom sections have been resealed.

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Recent interest in fabrication of laser fusion targets include a cryogenic target with a uniform solid or liquid fuel layer inside a spherical hollow capsule. We proposed plasma layering technique to redistribute a nonuniform solid fuel layer into uniform one with adds of glow discharge plasma initiated in the void of the capsule located in a strong microwave field (L). Higher surface temperature at the thicker solid fuel layer induces sublimation of fuel at the thicker area and conden- sation of the vapour at the thinner area. Through this sublimation and deposition cycle, the nonuniform solid fuel layer turns to be uniform after lapse of time.

The key points in this method are uniform heating of the solid layer by the plasma and uniform cooling of the capsule. Last year we es-
estimated the heating uniformity by the plasma under one dimensional electric field using data obtained by a simulation experiment at room temperature. The heat transfer by thermal radiation, however, was not included in the data analysis. This year we improved infrared imaging technique to measure the surface temperature. The heat transfer by thermal radiation inside the capsule was included in the estimation.

Resultant heating uniformity was worse than the previous estimate without radiation heat transfer, but still sufficient to obtain a seed layer with uniformity >99% in an abnormal glow discharge again.

Another interest of this method is a practical time scale to obtain a uniform solid hydrogen layer, which is currently supposed to depend on the cooling rate of the shell. We are going to demonstrate this technique using a quartz shell filled with deuterium gas cooled by liquid helium. The preliminary experimental results will be reported.


THIN FILM
Room A105 - Session TF-ThA

Thin Films for Energy Conversion and Efficiency/Active Films
Moderator: B. P. Hichwa, Optical Coating Laboratory, Inc.

2:00 pm TF-ThA1 Thin-Film Rechargeable Lithium Batteries, J. R. Rates, Solid State Division, Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, Tennessee 37831-6030.

Miniaturization of batteries has not kept pace with the reduction in size and power requirements of electronic devices. A lower limit on the dimensions of conventional batteries of such size in cells is dictated by bulk materials processing methods and by the battery container, which is typically a metal can. Recently, solid-state batteries that are less than 10 μm thick have been developed. These batteries, which can be fabricated in a variety of shapes and sizes on virtually any type of substrate, consist of a lithium metal anode and an inorganic insertion cathode that are separated by a lithium phosphorus oxynitride electrolyte. The cathode films, which include V₂O₅, Li₃MnO₄, and Li₃CoO₂, are deposited by reactive magnetron sputtering and evaporation. Processing conditions such as working gas composition and pressure, substrate temperature, and post deposition treatment have important effects on the physical and chemical properties of the cathode films and therefore have a major impact on the performance of thin-film batteries. Because they have high energies per unit of volume and mass and because they can be recharged thousands of times, thin-film lithium batteries have potentially many applications as small power supplies for electronic devices.

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This research was sponsored by the Division of Materials Sciences and Chemical Sciences, U.S. Department of Energy, under contract DE-AC05-84OR22400 with Martin Marietta Energy Systems, Inc.

2:40 pm TF-ThA3 Sputter Deposition of Cermet Fuel Electrodes for Solid Oxide Fuel Cells, T. Tsut and S. A. Barnett, Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208.

Electrodes for medium-temperature (600-800°C) solid oxide fuel cells (SOFC) should exhibit both low resistivity and low interfacial reaction resistance R, with the electrolyte. In order to achieve this, common fuel electrode materials, such as Ni-(YbO₂)-(ZrO₂)-(Ni₂O₃) cerments, must have both high porosity and small Ni particle size to achieve high three-phase contact area between Ni, YSZ, and the fuel gas. In this talk, the use of DC magnetron reactive sputtering of Ni-Zr-Y (68.8 wt% Ni, 26.6 wt% Zr, 4.52 wt% Y) targets in Ar₂O₂ mixes to deposit porous Ni-YSZ films is described. The films contained 30 vol% Ni, were deposited with 0.6 mTorr O₂ at a rate of ~3 μm/hr, and exhibited R of 0.005 Ohm. R values between Ni-YSZ films and YSZ electrolytes were measured in H₂/O₂ fuel gases by impedance spectroscopy. The spectra showed two arcs, attributed to electrochemical reactions and gas diffusion. Films sputtered at 20 mTorr total pressure gave the lowest R values, with SEM images showing cracks due to tensile stress at higher pressures and insufficient porosity at lower pressures. The initial YSZ substrate morphology also played an important role, with the lowest R values obtained for YSZ substrates roughened by polishing with 25 μm particles. The roughened surfaces presumably provided higher contact area and increased film porosity. The best R values of 0.15 Ohm/cm² at 750°C were much lower than bulk Ni-YSZ and low for high-power-density SOFC operation.


A novel two-stage, nontoxic selenium/arsenic free process consisting of sputtering deposition and homogenization of Cu-rich Cu-In-Ga precursor, first selenization, deposition of In-rich Cu-In-Ga precursor, and second selenization has been developed for the preparation of CuIn₃₋ₓGaₓSe₂ thin films. Morphology of precursors and CuInₓSe thin films was studied because of their effect on photovoltaic (PV) properties of solar cells. Very small sub-grain features were studied for the first time by atomic force microscopy (AFM) and quantitative data on rms roughness and 3D images were obtained. Morphology of precursors was found to change from initial very smooth layers with 200-300 Å size features and a rms surface roughness of <10 Å, to 3-5 Å size islands, coalescing grains with a fine sub-grain structure, and finally to compact, well-faceted, large-grain CuInₓSe₂ thin films with rms roughness in the range 950-1000 Å. Improvement of the morphology of completed CuInₓSe₂ thin films with the complete elimination of the sub-grain structure, substantial increase in the grain-size, more homogeneous distribution of the grain-size, and development of well-faceted grains was achieved by in situ homogenization of Cu-rich precursors prior to the first selenization and by employment of a maximum temperature of 550°C during the selenization so as to benefit from the fluxing action of copper selenide. Efficient PV solar cells were fabricated using 500 Å CdS conformal heterostructure layers even though rms roughness of completed CuInₓSe₂ thin films was ~140 Å.

3:20 pm TF-ThA5 Structure, Morphology, and Properties of CuInₓGa₃₋ₓSe₂ Epitaxial Layers on GaAs, G. Berry, D. Schroeder, L-Chung Yang, H. Z. Xiao, and A. Rockett, Coordinated Science Laboratory and Materials Research Laboratory, University of Illinois, 1101 W. Springfield Ave., Urbana, IL 61801.

Single crystal epitaxial layers of Cu(InGa)₃₋ₓSe₂ (CIGS) have been grown on (111) and (001) GaAs substrates by a hybrid sputtering and evaporation method. Results are presented showing the relationship of composition to film surface morphology, density and type of lattice defects, phases present, and optical/electronic properties. Films containing moderate amounts of Ga (10%) are shown to have structures and surface morphologies consistent with those of pure CuInSe₂ epitaxial layers. At higher Ga content (>20%) the surface becomes faceted and roughened and significantly increased bulk defect densities were observed. Second phases are also observed in the high-Ga content alloys. In-rich films having a composition CuInₓSe₂ have also been examined and found to include an ordered array of point defects. The In-rich films have increased energy gaps and show evidence of extended subgap absorption consistent with the presence of extended band tails. Interdiffusion between the layers and the GaAs substrates was higher when the Ga flux used to grow the epitaxial layer was lower. The use of these materials in photovoltaic applications is discussed and preliminary results of solar cell performance are described.


Recently several groups have shown that high efficiency multinary chalcophyrite thin film solar cells have a copper-poor surface layer which induces the formation of a buried homojunction. In the case of CuInSe₂ this Cu-poor phase is CuInₓSe₂ (CuOVC). In this composition we have characterized OVC films r.f. sputtered from a single target, on the other hand we have fabricated absorbers by sequential deposition of CuGaₓIn₁₋ₓSe₂ and CuInₓSe₂ by r.f. sputtering. We find that films grown at growth temperatures (Tg) below 400°C are homogeneous with a composition close to the target, no secondary phases are detected. These films have a
bandgap of 1.25 eV and a much stronger 112 orientation than CGIS thin films. For Ti above 400°C film composition in a ternary phase diagram approaches the line that joins CunSe₂ and InSe. X-ray diffraction shows the presence of InSe, and optical properties a bandgap of 1.0 eV with a high sub-bandgap absorption. We conclude that in the Cu-poor side of the phase diagram film formation takes place from the selenide binaries analogously to CGIs. Nevertheless, for high Ti, CuSe₂ and InSe do not react completely leaving unreacted InSe that decomposes in InSe(5) + Se(2). Absorbers grown by the sequential growth of high Tg Cu-rich CGIS and OVC films show a complete intermixing of the element, and an improved performance with respect to devices r.f. sputtered from a single target.

4:40 pm TF-Tha7 Attaining a Solar Energy Economy with Active Thin Film Structures, R. B. Golden, Electro-Optics Technology Center, Tufts University, Medford, MA 02155.
Quantitative arguments are presented which support the conclusion that the probability is high for the U.S. to attain a solar energy economy in the relatively near future. The basis of the arguments is the use of four active thin film structures: (i) electrophoretic "smart" windows for energy conservation; (ii) thin film photovoltaic cells for energy conversion; (iii) thin film batteries & fuel cells for portable energy storage and delivery. The paper focuses on the pivotal role of thin film deposition processes. Particular attention is paid to some of the deposition challenges presented by electrochromic smart windows and the thin film batteries.

4:40 pm TF-Tha9 Low Temperature, High Rate Deposition of Electrochromic Materials/Devices with MetaMode, Nada A. O'Brien, J. Gordon, H. Mathew, and Bryant P. Hichwa, Optical Coating Laboratory, Inc., 2789 Northpoint Parkway, Santa Rosa, California 95407-7397.
MetaMode reactive sputtering [1] is used to manufacture uniform multilayer anti-reflection coatings and transparent conductive films. Recently, we have deposited electrophoretic(EC) materials and devices with the MetaMode process. MetaMode offers the advantage of low temperature deposition and relatively high deposition rates compared to conventional reactive sputtering. Moreover, we have reproducibly fabricated complete monolithic EC devices at manufacturable rates with the MetaMode process. For example, a 25 cm² translucent device shows a photopic transmission change ≥ 50% and coloring/bleaching time ≤ 20 seconds. In this paper, a description of the MetaMode process and its capabilities will be given. In addition, we will compare the behavior of MetaMode-prepared WO₃/EC films with other more conventional EC deposition techniques. We will report on electrochromic properties, i.e., coloring efficiency, optical density, switching time and current-voltage characteristics.


5:00 pm TF-Tha10 Properties of Transparent Conducting Zinc-Stannate Films Prepared by RF Magnetron Sputtering, T. Minami, S. Takata, H. Sato and H. Sonohara, Kanazawa Institute of Technology, 7-1 Ohigaoka, Nonoi, Ishikawa 921, Japan.
The preparation of newly developed transparent conducting oxide films by rf magnetron sputtering is demonstrated. The SnO₂-ZnO films were prepared on glass substrates using SnO₂ targets with a ZnO content of 0 to 55%. The resistivity of the deposited films markedly increased as the Zn/Sn ratio introduced into the films was increased. The minimum resistivity was obtained for films with a Zn/Sn ratio of about 0.2. It was found that the composition of the deposited films is ZnSnO₂ rather than Zn₂SnO₄. The electrical and optical properties of films deposited using Ar-O₂ mixture sputter gases were strongly dependent on the O₂ partial pressure as well as the substrate temperature. Resistivities as low as 4 × 10⁻³ Ω•cm and an average transmittance above 80% in the visible range were obtained for undoped zinc-stannate films prepared with substrate temperatures of RT to 300°C by controlling the O₂ partial pressure. Especially for the zinc-stannate films, thermal stability in a hydrogen environment was improved over that of SnO₂ films and chemical stability in acidic and basic atmospheres was improved over that of ZnO films.

We report the photoluminescence spectroscopy of etch-defined quantum box- and wire-like structures fabricated in GaAs/AlₓGa₁₋ₓAs quantum wells by a novel form of excimer-laser-excited neutral Cl atom etching. Our etching technique is suitable for the fabrication of these structures because it enables high resolution, noncrystallographic, dry etching, using neutral atom reactions. Such low-energy atom chemistry is not expected to damage the feature sidewalls, as is often the case for other high resolution, ion-based etch techniques. This damage has often been cited as a primary cause of the limited luminescence efficiency of etch-defined quantum wire- and box-like structures. In fact, comparison of our results with the luminescence of wet-etched samples shows little or no etch-induced damage to the feature sidewalls. In this connection, we have conducted a series of photoluminescence measurements at 10 K on Cl atom etched structures from our smallest features. As a result we have measured the photoluminescence efficiency and spectrum shape as a function of the feature lateral dimension from several micrometers to less than 0.25 μm. For smaller features, the chemical state of the surface is crucial for the luminescence efficiency, and so different surface chemical treatments, such as H-ECR passivation, have been used to enhance the luminescence efficiency to near that of the unprocessed material.

ELECTRONIC MATERIALS
Room A108 – Session EM-Tha

Materials for Nanostructures
Moderator: H. P. Gillis, Georgia Institute of Technology

2:00 pm EM-Tha1 Magnetotransport in Semiconductor Nanostructures, K. P. Martin, Georgia Institute of Technology, Atlanta, GA, 30332.
Semiconductor nanostructures have important device applications and present opportunities to investigate fundamental issues in condensed matter physics. This talk will discuss the use of magnetotransport as a tool to study these systems. Quite generally, the magnetic field allows the experimentalist to introduce an external scale for length, time, and energy through (respectively) the cyclotron radius, frequency, and energy. In turn, these B-induced quantities can be used to probe the material and transport properties related to phase coherent tunneling, elastic and inelastic scattering, charge buildup, and bandstructure. In some instances, magnetotransport can be used to evaluate properties related to growth (interface roughness) and processing (etch-induced damage) of these nanostructures. Supported by NSF grants DMR-92022879, ECS-8922512, and ECS-911866.
INVITED

We present the electrical and optical properties of nanostructures in III-V semiconductor devices. Interest in III-V semiconductors is of interest from both a fundamental viewpoint and for technological applications in quantum confined lasers and wires and dots for low power photonic and electronic device arrays. We have developed a simple deposition and etch-back technique to form narrow (~300 Å) masks for subsequent pattern transfer into the semiconductors. High ion density (5 X 10¹⁵ cm⁻³) ECR CH₂⁻/H₂(Ar (for In containing materials) or BCl₃/Ar (for Ga-containing materials) are used to give anisotropic low-damage etching. After initial resist patterning using conventional stepper lithography, conformal PECVD or reactive sputtering of a metal (W, Sn₃, or a low temperature dielectric (Si₃N₄, SiO₂) is followed by an anisotropic etch-back to leave a thin sidewall on the resist feature. The resist is then removed by dry etching, leaving the sidewall which can be used as a mask for pattern transfer into the semiconductor. Examples of optically-pumped quantum wire laser structures fabricated in GaN and InP-InGaAsP systems will be shown. The nano-laser structures can be integrated with microdisk lasers using combined dry etching and selective wet chemical etching. Low ion energies (≤ 100 eV) are required to avoid sputtering of the mask material and changes in the near-surface stoichiometry of the semiconductor.

We report the photoluminescence spectroscopy of etch-defined quantum box- and wire-like structures fabricated in GaAs/AlₓGa₁₋ₓAs quantum wells by a novel form of excimer-laser-excited neutral Cl atom etching. Our etching technique is suitable for the fabrication of these structures because it enables high resolution, noncrystallographic, dry etching, using neutral atom reactions. Such low-energy atom chemistry is not expected to damage the feature sidewalls, as is often the case for other high resolution, ion-based etch techniques. This damage has often been cited as a primary cause of the limited luminescence efficiency of etch-defined quantum wire- and box-like structures. In fact, comparison of our results with the luminescence of wet-etched samples shows little or no etch-induced damage to the feature sidewalls. In this connection, we have conducted a series of photoluminescence measurements at 10 K on Cl atom etched structures from our smallest features. As a result we have measured the photoluminescence efficiency and spectrum shape as a function of the feature lateral dimension from several micrometers to less than 0.25 μm. For smaller features, the chemical state of the surface is crucial for the luminescence efficiency, and so different surface chemical treatments, such as H-ECR passivation, have been used to enhance the luminescence efficiency to near that of the unprocessed material.
Our results will be explained in terms of standard models of carrier recombination. This work has been funded by ISEP, ARPA, and AFOSR.

3:20 pm Em-ThA5 Room Temperature Photoluminescence and Electroluminescence from Ge Nanocrystals in SiO_2 Formed by Ion Implantation and Precipitation. C. M. Yang, K. V. Sheveglov, S. S. Wong, H. A. Atwater, California Institute of Technology, Pasadena, CA 91125.

There is a growing interest in the luminescent properties of group IV semiconductor nanocrystals driven by a need for optoelectronic devices compatible with current VLSI Si-based manufacturing technology. Ion implantation and thermal annealing, standard Si processing methods, have been used to fabricate Ge nanocrystals in SiO_2 matrix. This configuration has an advantage over porous silicon in that it is mechanically robust and insensitive to environmental influences. It was found that by varying annealing conditions for the sample implanted with 6 atomic percent Ge, narrow size distributions can be obtained with mean size ranging from 3 nm for the 600°C anneal to 10 nm for the 1200°C anneal. Visible photoluminescence at room temperature has been observed from the above samples with peaks between 500 nm and 700 nm, depending on annealing conditions. Lastly, an electroluminescent device has been fabricated in a PIN configuration, the ‘intrinsic’ layer being Ge nanocrystal-containing SiO_2, with similar photoluminescence and electroluminescence spectra peaked at 700 nm. Comparison of photoluminescence and electroluminescence for this device with the particle-size distribution will be presented, as well as a systematic correlation of particle-size distributions with photoluminescence spectra for the above samples.

3:40 pm EM-ThA6 Direct Characterization of a 2 Dimensional Electron Gas in Heterostructure FETs with Scanning Tunneling Microscopy. Y. J. Skala, W. Wu, J. R. Tucker, K.-Y. Cheng, and J. W. Lyding, Department of Electrical and Computer Engineering, Beckman Institute and Microelectronics Laboratory, University of Illinois at Urbana-Champaign, Urbana, IL 61801.

Cross-Sectional Scanning Tunneling Microscopy (XSTM) has been used to directly image a 2 DEG in cleaved HFET devices. Variation in electron concentration within the 2 DEG can be measured as well as the location of the maximum of the first electron subband. We explore the uniformity of the 2 DEG as a function of material parameters such as barrier-channel interface quality and alloy fluctuations within a tertiary channel. Additionally, agreement between theoretically predicted characteristics of 2 DEG and direct measurements for a variety of device structures is determined.

Supported by NSF under contract NSF EDC 89-43166 and by ISEP under contract N00014-90-J-1270.

4:00 pm EM-ThA7 Temporal Evolution of Nanoscale Interfacial Phases Between GaAs and Metal Films. Tae-Jin Kim, Paul H. Holloway, Dept. of Materials Science and Engineering, University of Florida, Gainesville, FL 32611.

The electrical, thermal and mechanical properties of metal thin films on semiconductors depend on interfacial reactions. In the case of GaAs, multilayer films such as Au-Ge-Ni produce binary (AuGa, NiGa, NiAs) and ternary (Ni, GaAs) phases. Very often the phases extend irregularly into the semiconductor over distances on the order of 5–50 nm, leading to degradation of interfacial properties. To achieve regular nanoscale structures with desired properties, precise dimensional control based on a theory is essential. A “growth-and-etch” model will be suggested to explain the properties associated with the interfaces. It will be shown that control of the A and As phases from the binary or ternary phases “growth” into GaAs as a result of temporal evolution of the interfacial phases. It will be shown that the electrical and the other properties of the interfaces can be controlled by the regrowth.

4:20 pm EM-ThA8 Selective Chemical Vapor Deposition of Copper on Pd-Activated Self-Assembled Films, Stephen J. Potocki, David S. Y. Hsu, Jeffrey M. Calvert, and Pehr E. Persson, 1. Chemistry Division, 2. Center for Bio/Molecular Science and Engineering, Naval Research Laboratory, Washington, DC.

A process has been developed for selective chemical vapor deposition (CVD) of conductive, adherent copper films using self-assembled organosilane films to tailor substrate surface chemistry. Selective copper CVD was achieved on single-crystal diamond substrates at 423–453 K in a low-pressure, cold-walled reactor using hexafluoroacetylene-tributylammonium-Cu-trimethyl-vinylsilane (hfac-Cu-TMVS) in H_2 (1:1). Diamond was activated for copper deposition by covalent attachment of a ligating aminosilane film and a Pd-based catalyst; only isolated copper particles formed on unmodified diamond surfaces. Copper deposition on growth and nongrowth surfaces was characterized by Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), x-ray fluorescence (XRF), and scanning electron microscopy (SEM). Patterned copper features were formed on diamond substrates by exposing photosensitive aminosilane films to patterned ultraviolet (193 nm) radiation prior to Pd-catalyst addition and copper CVD.


Sequential surface chemical reactions for the atomic layer controlled deposition of Al_2O_3 were studied using transmission Fourier transform infrared spectroscopy (FTIR) and atomic force microscopy (AFM). A binary reaction for Al_2O_3 chemical vapor deposition (2Al(CH_3)_3 + 3H_2O → Al_2O_3 + 6CH_4) was separated into two half-reactions:

A) Al(OH)(CH_3)_3 → Al-O-Al(CH_3)_3 + CH_4
B) Al-O-Al(CH_3)_3 + 2CH_4 → Al-O-Al(OH)_2 + 2CH_4

The trimethylaluminium [Al(CH_3)_3] (TMA) and H_2O reagents were employed alternately in an ABAB... binary reaction sequence to achieve atomic layer controlled Al_2O_3 deposition. FTIR analysis of these surface reactions was performed in situ in an ultrahigh vacuum (UHV) chamber using high surface area alumina membranes. The ALOH and AlCH_3 surface species were monitored by the infrared absorbance of the Al-OH stretching vibration between 3800–2600 cm^-1 and the Al-H stretching vibrations between 2942–2838 cm^-1. The optimal conditions for controlled Al_2O_3 growth were observed using TMA and H_2O exposures at 0.3 Torr on substrates at 500 K. The analysis revealed that both the A and B reactions were self-limiting and complete. The atomic layer controlled deposition of amorphous Al_2O_3 thin films was demonstrated on Si(100) and sapphire using the ABAB... binary reaction sequence. Atomic force microscopy was employed to determine deposition rates versus temperature as well as examine the film conformity. A growth rate of 3 Å/AB binary reaction cycle was observed for the optimal Al_2O_3 growth conditions.


Polycrystalline CdTe thin films grown by different methods, including physical vapor deposition, sputtering, closed space sublimation, and MOCVD have been studied by atomic force microscopy (AFM). The results are compared with scanning electron microscopy (SEM) investigations. The films have been analyzed as-deposited and after heat and chemical treatments with CdCl_2, processing used to realize high-efficient thin-film (>15%) photovoltaic devices. It was found that the results for the AFM and SEM agree well up to the resolution of the later. At higher spatial resolutions (beyond the limit of the SEM) new structures have been observed for the first time in these films. For samples deposited by sputtering, AFM analysis showed that the micron-size features (observed also with the SEM) are actually formed by smaller grains, having few hundred of angstroms cross-sectional dimensions. These films, when treated with CdCl_2 at different temperatures show the nanograin structure coalescing, with complete recrystallization at temperatures of approximately 400°C. The nanograin structure has also been observed in some films grown by physical vapor deposition. A post-deposition heat-treatment of these films also changed this structure. The existence of the nanograin is a function not only of the deposition method, but also of the substrate on which the film is grown, and of the post-deposition heat-treatment. These nanoscale studies provide the first direct evidence linking the processing of the films to the change in nanostucture and the resulting efficiency improvement of the photovoltaic cells.
is generic in that the basic control framework and controller development results are applicable to VLSI manufacturing in general. The controller is multibranch as it utilizes multiple algorithms in complementary fashion to achieve process optimization and control. The current implementation utilizes three branches: (1) a linear approximation control algorithm, (2) an optimization algorithm that utilizes (real-time) data collected in-situ to determine optimal run-to-run process parameter settings, and (3) a statistical optimization algorithm that utilizes run-to-run data. The controller has been extended to incorporate an automated branch selection process that utilizes fuzzy logic to incorporate process engineer as well as optimization and control algorithms. The controller has also been extended to adapt to unforeseen events through utilization of a learning mechanism; this mechanism detects these unforeseen events, intelligently queries the process engineer, guides the engineer through the development of an event servicing scheme, and incorporates this new knowledge into its control knowledge base so that the event may be serviced automatically in the future. Implementation results of the controller (in the control of the etcher) confirm the robust control capabilities in the face of process shift and drift, and recipe change.

3:40 pm MSVT-ThA6 Real-Time Feedback for Sidewall Profile Control, Brian Rashap, Jim Freudenberg and Michael Elta, University of Michigan.

Real-time feedback control is being utilized in the Reactive Ion Etching process as part of a strategy to control sidewall profile. As with a number of etch characteristics, it is not possible to measure sidewall profile in-situ and in real-time. Therefore, an indirect method of controlling these characteristics is necessary. In previous work, we have shown that it is possible to stabilize etch rate by using feedback control to regulate various plasma characteristics.

Currently, we are investigating sidewall profile control, again using feedback to manipulate plasma properties. The sidewall shape produced by an etch is determined by the isotropic and anisotropic etch rate components. Free radicals diffusing to the surface determine the isotropic etch rate. Anisotropic etching is caused by an enhanced etch rate in areas exposed to ion bombardment and a reduced rate in areas where a passivation layer is present. This paper reports on the first step in the development of a strategy for controlling sidewall profile. Using a real-time controller for the plasma generation process in a C64/O2 chemistry, a response surface from plasma characteristics (self-bias voltage, pressure, and fluorine concentration) to vertical and horizontal etch rates is developed. A string model simulation, optimized to match scanning electron microscope images of the etch profile, is used to calculate etch rate components. This response surface will then be used to develop trajectories for the plasma environment that produce desired sidewall profiles. Finally, the real-time controller will be employed to actuate these trajectories.

4:00 pm MSVT-ThA7 Process Monitoring with Residual Gas Analyzers, Charles R. Tilford, National Institute of Standards and Technology, Gaithersburg, MD 20899.

Residual Gas Analyzers (RGAs), most commonly mass spectrometers of the quadrupole type, have long been used for qualitative vacuum-system diagnostics. Increasingly, they are also being used for quantitative process monitoring and control, often involving highly reactive contaminant and process gases. RGAs are complicated instruments and their performance is affected by a number of instrument and vacuum-environment variables, so that even "calibrated" instruments can behave significantly different than expected. The magnitude of the performance anomalies varies for different instruments, but can reach orders of magnitude in extreme cases. This talk will review the important factors affecting instrument performance (including ion-sourced and quadrupole-filter parameters, total pressure, and active gases), illustrate the magnitude of observed effects for different instruments, recommend instrument test and calibration procedures, and suggest operating parameters and procedures that can minimize anomalies.

3:20 pm MSVT-ThA5 Adaptive Extensions to a Multi-branch Run-to-Run Controller for Plasma Etching, James R. Moyné, Nauman Chaudhry, and Richard Yuan, Univ. of Michigan, DTN Center, Ann Arbor, MI 48109-2108.

Fuzzy logic and database learning mechanisms have been incorporated into a generic plasma etching run-to-run controller, resulting in a very dynamic, adaptable and robust system. The system features an Applied 8300 RIE controlled by a Techware II equipment controller. A TCP/IP connection links this equipment controller to the run-to-run controller residing on a SUN. The run-to-run controller environment


Deposition rate is a critical parameter that needs to be precisely controlled in all PVD processes. Atomic absorption spectroscopy (AAS) is a well established technique for determining the atomic density in vapor phase, and it has been tested for non-intrusive monitoring of deposition rates in PVD processes. However, previous AAS based monitors encountered serious problems during typical deposition processes.
due to changes in optical signal levels caused by viewport coating and temperature-induced movements in optical alignment, resulting in poor accuracy. In contrast, we have developed a novel optical scheme named COPACT (Common Optical Path for Automatic Correction of Transmission) which significantly improves the long-term stability of AAS deposition rate monitors. The emission from a broadband light source, with its central wavelength and bandwidth properly defined by a wavelength selection device, is utilized to compensate the overall optical system transmission change in real-time. This method enables us to fully utilize the advantages of AAS for deposition rate monitoring, which include absolute material specificity, high sensitivity, extended vapor sampling range, no background pressure limitation, uninterrupted operation and non-intrusive optical probing. Experimental results are presented to show that it is possible to accurately control very low deposition rates, on the order of one monolayer per minute, over an extended time period.

4:40 pm MSVT-ThA9 A Model Based Technique for Estimation of Fluorine in a CF4/Ar Plasma, P. D. Hanish, Jessy W. Grizzle, and M. D. Giles, University of Michigan, Ann Arbor, MI 48109.

A technique for quantitative interpretation of actinometric data to deduce bulk plasma fluorine concentration in a CF4/Ar plasma has been developed and tested on an RIE. This static, in-situ measurement is useful for monitoring fluorine in a manufacturing environment and, in particular, for application in real-time feedback control to plasma etching. Based upon a model of CF4 chemistry reaction pathways and products, it improves upon current fluorine estimation techniques by accounting for varying levels of argon dilution resulting from CF4 dissociation. A simple experiment was also developed in order to obtain an estimate of the actinometric scaling factor without an independent measurement of fluorine. Performance of this fluorine technique was compared to that of a standard technique by using time resolved etch rate measurements as an independent indicator of fluorine concentration, while a feedback control scheme decoupled the effects of physical etching by stabilizing the induced dc bias. The model based estimator reduced perturbations in the etch rate by more than 50% compared to those seen when using the standard estimator.

5:00 pm MSVT-ThA10 Role of Inert Carrier Gases in Modeling, Design, and Operation of a Single Wafer APCVD Reactor for Manufacturing, Prasad N. Gadgil, Department of Physics, Queen’s University, Kingston, Ontario, K7L 3N6, CANADA.

Atmospheric Pressure Chemical Vapor Deposition (APCVD) with inert carrier gases can offer several potential advantages in microelectronic device manufacturing. In a simple stagnation point flow configuration, lower viscosity of inert gases such as Argon and Nitrogen results in a lower pressure drop across the gas distributor and a smaller magnitude of undesirable entrance effects as compared to Hydrogen. In addition, lower thermal conductivity of N2 leads to the development of isotherms with a higher temperature gradient adjacent to the susceptor surface that are highly desirable to suppress homogeneous gas phase reactions. A hydrodynamically model of a single wafer stagnation point flow reactor is developed. An inverted APCVD stagnation point flow reactor with a novel flow distributor, optimized by flow visualization and fluid flow modeling is designed. The results of its operation with inert carrier gases are described. The suitability of mixing of inert gases with reactive gases such as H2, NH3 and O2 is evaluated for process chemistry of various microelectronic materials. Also, additional economic advantages in the back end of a CVD system in exhaust processing are outlined.

VACUUM METALLURGY
Room A106 - Session VM-ThA

Manufacturing Technology for Coatings
Moderator: D. C. Carmichael, Vacuum Technology Inc.

2:00 pm VM-ThA1 Environmentally Compatible Coating Technologies, Keith O. Legg, A. Adamski, C. West, P. Rudnick, F. Raasch, J. Schell,* A. Gonzalez, B. Sartwell*, BIRL Industrial Research Lab., Northwestern University, Evanston, IL; *Cummins Piston Ring Division; **GE Aircraft Engines; 'Corpus Christi Army Depot; Naval Research Lab.

This paper will discuss what is involved in replacing electroplated hard chrome with modern, clean alternatives. We shall illustrate the discussion with the results thus far of a program designed to evaluate various modern coating technologies as alternatives to hard chrome electroplate on military components. The primary uses of hard chrome in DoD are to hard-coat new components and to rebuild worn parts that come in a wide variety of geometries, sizes, and materials. Any alternative must fit into the total life cycle of the part, which usually includes teardown, inspection, and recoating several times during its service life.

We shall discuss the uses, capabilities, and performance of thermal spray, laser deposition methods, thin PVD coatings, and plasma nitriding for creating low-wear surfaces at the initial fabrication stage, and for building up worn parts in both military and commercial applications.

INVITED

1Supported by ARPA Grant #MDA972-93-1-0006.


This paper describes the development of an in-line reactive sputtering process to deposit AlN films on polycarbonate disks as barrier layers for magneto-optical recording media. Manufacturing requirements were for an equivalent static deposition rate of at least 2 nm/s and a temperature rise less than 40°C for the free-standing substrates. The 100 nm thick films had to have low absorption and adequate reflectivity close to 2.00, as well as be able to provide corrosion protection for the active TbFeCo layer. A dc rather than rf process was chosen in order to achieve the rates and low substrate temperatures needed. Factorial experiments were done to study the dependence of power, total pressure, and the Ar/N2 partial pressure ratio on the deposition process and film properties. Differences in substrate heating were found that were related to the nature of the dc supply used. Methods of arc suppression were also studied. A dark-space shield that confined the plasma to the racetrack region significantly reduced the number of arcs. However, we concluded that any arcing was unacceptable because it would eventually lead to a loss of control. In order to eliminate arcs completely, we used a 40 kHz power supply driving two cathodes out of phase with one another. Several methods of process control were compared. Two of them—either operating the target at constant power and using the target voltage to control the reactive gas flow or using an RGA to control the partial pressure of the reactive gas—produced adequate results as demonstrated by control charts of the thickness and optical properties. The effects of target wear on process drift were measured as well.

INVITED


Flow coating a protective coating onto a freshly deposited film on a web in situ in a vacuum chamber is very useful in many applications where the deposited film must be protected from scratches or reacting with the atmosphere when it is removed from the vacuum chamber. There are materials that will oxidize in the atmosphere very quickly resulting in undesirable changes in the film properties. When the laminated protective coating is used, it can be protected indefinitely if it is further processed or put in final use. The process combining the deposition and lamination in one pump-down involves the evaporation of highly reactive material such as bismuth onto a polycarbonate substrate on a roll-to-roll system. Immediately following the deposition, the coated substrate moves to a section of the vacuum chamber where a heat sealable cover film is bonded to the coated substrate and then rewound onto a take-up roll. A description of such a process and equipment will be presented. A comparison of the film properties with and without the in situ lamination will be presented.


The anode size and their spatial distribution affect the magnetron discharge characteristics and the deposition rate distribution. Reducing the anode size requires an increased voltage to maintain the same cathode current. Although the magnetron plasma is confined in the race track area, changing the position of small anodes redistributes the plasma density and changes the deposition rate distribution. Applying additional voltage between multiple small anodes allows one to vary the deposition rate distribution in a desired way. The potential
distribution in a magnetron plasma with small anodes and a floating power supply is discussed. Electron escape from the race track plasma of a rectangular planar magnetron in prevailing directions is shown.

4:00 pm  VM-ThA7 Recent Technology Advancements in Optical Thin Film Manufacturing, Paul M. Lefebvre, Bryant P. Hichwa, Robert W. Adair, Optical Coating Laboratory Inc., Santa Rosa, California 95407.

The optical thin film industry faces numerous challenges in today's marketplace. Films must be optically clear and pass stringent specifications for spectral performance, environmental resistance, and durability including abrasion resistance. SiO₂ is one of the few durable, low index of refraction materials, available for the design and manufacture of optical thin film interference coatings. In recent years, several critical technological advancements in the field of DC magnetron sputtering have led to reliable manufacturing systems for the high rate, arc free, sputter deposition of SiO₂. For high throughput, price sensitive products, cylindrical magnetrons, such as the Airco C-MAG®, and dual planar magnetrons, such as the Leybold TwinMag™, allow SiO₂ to be reliably and cost effectively deposited in "on-line" sputter coaters. In addition, novel machine geometries, such as the OCLI MetaMode® system, have led to the production of complex, high layer count, interference coatings on glass and plastic substrates. Currently, pulse modulated DC magnetron sputter deposition has further improved the rate and quality of SiO₂ films. In this paper, we compare these technology advances in SiO₂ sputter deposition and include examples of multilayer optical interference coatings such as anti-reflection, enhanced high reflectance, hot mirror and narrow band pass coatings.

4:40 pm  VM-ThA9 Sputter Deposition of Indium Tin Oxide (ITO) from a Cylindrical Ceramic Target, Kevin P. Gibbons, Tami Van Skike and C. K. Carniglia, Airco Coating Technology, 4020 Pike Lane, Concord, CA 94520.

ITO is usually sputtered from planar ceramic targets. The uptime of such planar targets is severely limited by the growth of "cones" in the racetrack area. In this paper we report on the sputter deposition of ITO from cylindrical rotating magnetrons. The use of rotary magnetron technology has significant process advantages. When a rotary ceramic target is used, the growth of target nodules or cones is significantly delayed. Using a 3 inch diameter by 15 inch long cylindrical target, ITO was sputtered for over 74 hours with no measurable decrease in rate or film conductivity. This result extrapolates to at least 6 days of running in a production size system before nodules or cones affect deposition rate. An ITO coating optimized for the CRT antistatic, antireflection market was developed. The effects of oxygen pressure, deposition temperature and deposition power on film properties are presented and discussed. Optical modelling was carried out using a three parameter fit to the refractive index in the visible spectral region. As expected, the films with the lowest resistivity and lowest visible absorption were produced at the highest deposition temperature.
Thursday Afternoon Poster Sessions

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room BR4 - Session NS-ThP

Nanoelectronics and Nanofabrication
Moderator: J. D. Dagata, National Institute of Standards and Technology.

NS-ThP1 Voltage Controlled Nanometer Scale Oxidation of Si(100) Surface by STM, F. Pérez-Murano, G. Abadal, N. Barniol and X. Aymerich, Dept. of Física Electrònica, Universitat Autònoma Barcelona, E-08913-Bellaterra, Spain.

In a previous paper we have presented a new technique to produce a local modification on Si(100) surface by STM (F. Pérez-Murano et al., J. Vac. Sci. Technol. B 11, 651 (1993)), consisting on locally oxidize the surface by controlling the electrical field between tip and sample. In order to improve the resolution of the technique and also to improve the velocity at which the modification can be produced, we have revised the technique. As it has been shown by other authors (E.S. Snow et al., Appl. Phys. Lett. 63, 749 (1993)), when the n-type silicon sample is polarized at a positive bias, the surface becomes more oxidized, being possible to produce local modifications, but the surface quality is degraded. In this communication, we show how the modification can be better controlled if positive voltage pulses are applied to the sample while scanning. As the feedback condition is fixed by the negative voltage applied to the sample, both the tip and surface quality are preserved. Moreover, adjusting the value of the positive voltage and the time duration of the pulse, the resolution is improved and the modification can be performed at selected points of the sample. Finally, we will show that this technique can be used not only for n-type silicon samples but also for p-type silicon sample if the surface is kept under illumination.

NS-ThP2 Local Electrical Switching Effects in CuTCNQ Films Studied by Surface Modification with STM, L. Wang, C. W. Yang, J. W. Liang, H. Chen, and Y. Wei, National Laboratory of Molecular Electronics, Southeast University, Nanjing 210018, People's Republic of China.

Local threshold electrical switching phenomena and reversing process of CuTCNQ films have been investigated by electronic beam etching and mechanical lithography with STM. A 5.5 nm thick polycrystalline organometallic semiconducting CuTCNQ film was etched directly on a copper plate by reacting neutral TCNQ in acetonitrile solution with the copper metal. When triggered by a 20 ms wide, 3V electrical pulse 10 times in 5 minutes with STM tip, a convex of 10 nm diameter and 5 nm high appeared on the STM images. By continuously observation, we found, for the first time, this protuberance was spontaneously diminishing and disappeared at last in ca. 5 minutes. The STM analyses showed the protuberance was due to the conductivity increase at the triggered portion of the film. It is revealed that the local high-conductivity state on the CuTCNQ film can automatically restored to the low-conductivity state after electrical pulse was removed. Through the mechanical etching by STM, a concave of ca. 50 nm in diameter and 20 nm high was formed on the film and it could not be remedied. The hole is probably due to the local destruction in structure on the film surface rather than the change of conductivity.

NS-ThP3 Deep-Etch Silicon MM-Waveguide Structure for the Relativistic Acceleration of Electrons, T. L. Wilke and A. D. Feinerman, EECS Department (M/C 154), University of Illinois at Chicago, Chicago, IL 60607-4348.

A UHV compatible planar corrugated waveguide structure possessing bi-fold symmetry and a beam aperture has been simulated with MAFIA software, micro machined, and initially characterized. The waveguide was fabricated for use at 120 GHz (λ = 2.5 mm) with 2ϕ/3 phase advance operation [1]. In order to provide a semi-closed conducting surface to serve as the waveguide and free-space aperture, two opposed composite Si/Pyrex metallized substrates patterned with the waveguide's corrugated geometry (see figure) were vertically stacked with Pyrex fibers to an accuracy of ~1 μm. Feinerman et al. has shown that precision-drawn optical fibers can be anodically bonded to Si substrates to provide fixed orientation of two or more object planes [2].

The structures upper- and lower topographies consist of rectangular anisotropically etched Si muffin-tin patterns and are electrically conductive to a few skin depths. The most critical aspects are the muffintin dimensions and the upper-to-lower die orientation error. The alignment mechanism keeps the two periodic structures in phase spatially while maintaining a 600 μm uniform gap (which determines the impedance and efficiency of the waveguide) to the accuracy that the corrugated geometry meets.

Eventually each two-surface waveguide section will be horizontally arrayed to form a structure 4.95 meters in length. Results of RF feedback testing a 70 GHz microfabricated waveguide section will be presented and compared with modeling data.


NS-ThP4 Measurement of the Piezoelectricity of Thin Films with STM, R. Winters, M. Reinermann, C. Enss, G. Weiss*, Institut für Angewandte Physik, Universität Heidelberg, Albert-Ueberle-Str. 3-5, 69120 Heidelberg, Germany, *present address: Physikalisches Institut, Universität Karlsruhe Engesserstr. 7, 76131 Karlsruhe, Germany.

With its extraordinary vertical and lateral spatial resolution the scanning tunnelling microscope (STM) is preeminent apt for measurements of the piezoelectric constant of thin films, especially if high lateral resolution is required, e.g. for the imaging of domains. For STM measurements the surface of the piezoelectric film has to be metalized. A variation in thickness due to an electric field applied across the film causes a vertical displacement of the surface relative to the tunnelling tip: Therefore the piezoelectric effect can directly be monitored by the STM. With its vertical resolution of less than 1Å even weak piezo-effects can be studied. Test measurements with X-cut quartz in air at room temperature show qualitatively correct behavior, but surprisingly the measured value of the piezoelectric constant varied both temporally and spatially. These fluctuations originate from the non-ideal characteristics of the tunneling contact, the physical properties of which are not well understood hitherto. Our experiment clearly shows that this is a principal problem of STM measurements under ambient conditions. To investigate piezoelectric films we circumvented this difficulty using a novel bridge technique, where the unknown piezoelectric is mounted on a X-cut quartz. We present measurements of polymer layers and sputtered ZnO-films that show the ability of measuring value and sign of piezo constants and the potential of imaging domains. As an additional benefit this technique allows the in-situ calibration of the z scale of the STM.


A focused ion beam (FIB) can sputter submicron features directly into a substrate, without the need for masks or lithography. However, the amount of material that can be removed in this way is limited. When an etch-enhancing gas is introduced at the surface being sputtered (e.g., Cl2 at a GaAs surface), the material removal rate can increase by an order of magnitude. An etch-enhancing gas speeds the removal process by increasing the formation of gaseous byproducts. The point of this work is to explore the utility of several candidate etch-enhancers for diamond, which is a technologically important material for precision edge tools, and, conceivably, for future
electronic materials applications. FIB sputtering in the presence of a halogen gas (commonly used to assist in the removal of Si) produced no significant increase in etch rate relative to that observed when no gas was present. However, preliminary results indicate a factor of 3-4 improvement in the etch rate of diamond when using H2O as the gaseous species. Given the positive results observed with H2O, other gases chosen for this investigation include the constituents of H2O, O2 and H2. Oxygen ion beams have already been observed to etch diamond (100) at = 10 times the rate of Ar ion beams, so O2 seems a likely candidate for a diamond etch-enhancer. Hydrogen is of interest as it may encourage the production of volatile hydrocarbon compounds as byproducts of FIB-induced reactions of H2 gas with the diamond substrate.


NS-TP9 Nanometer-Size Features Produced by Highly Charged Ions, Daniel C. Parks, "Martin P. Stockli, Robert W. Schmidt, and Robert J. Bastian, University of North Carolina, Chapel Hill, NC 27599 USA.

While many studies have explored the feasibility of using an STM or AFM, few have focused on the modification of thin films with the intent of creating isolated metallic features on insulating substrates. We present our results on defining nanometer scale conducting regions and devices in gold films deposited on insulating substrates (mica, SiO2) using the AFM. Our results demonstrate the utility of the AFM for conveniently creating structures for research purposes that would otherwise be limited to the extensive processing and hardware requirements of e-beam lithography. These results also bear on the eventual use of the AFM as a nanometer scale machining tool. We have successfully created isolated wires 70 nm wide by 1 μm by machining 15 nm thick gold films using Si cantilevers with nominal force constants of 30 N/m. These cantilevers allow nondestructive contact force and "tapping mode" imaging, while also providing the large forces (>100 nN) necessary for nanometer scale plastic deformation. We have defined holes, isolation trenches and cleared areas and determined their parameters as a function of applied force, tip velocity, machining technique, tip orientation and substrate compliance. We will present our progress in AFM/STM imaging of these structures and electrical measurements on the fabricated wires and tunnel junctions.

NS-TP10 The Self-Assembled Ordered Structures for Nanoelectronics: Stability and Local Modification, A. V. Emelyanov, V. V. Protasenko, V. N. Ryabakov, Zelenograd Research Institute of Physical Problems, Moscow, 103460, Russia.

Solution stability and accuracy problems in manufacturing nanoelectronic elements (NE) have been proposed by the use of self-assembled ordered structures (SAOS), because they permit to form NE with atomic accuracy. The work on simulation of formation processes on molecular level local nonuniformity of atomic scale with application pointed probe has been carried out. It makes possible to extract from necessary characteristics received on base modified SAOS nanostructures and to put forward the definite requirements to SAOS parameters and influencing factors. Particularly, using the activation model of the processes of local modification on atomic scale for SAOS has been considered, their cells contain the adsorbed fragments, teared off with help of STM probe for account of polarization interaction. This model, for example, in case of consideration of storage medium of large density, permits to connect the reliability and definite quantity of reading cycles, storage time, resolution and so on with parameters of SAOS and influencing tool.

NS-TP11 Mechanical Nanofabrication Based on Scanning Force Methods, S. Lukas, M. Löhndorf, C. Hahn, U. D. Schwarz and R. Wiesendanger, University of Hamburg, D-20355 Hamburg, Germany.

Nanofabrication based on the mechanical interaction between a sharp tip and a substrate offers a reliable and well controllable method to obtain nanometer-scale devices. We have used a scanning force microscope (SFM) to pattern a variety of different thin film substrates, including polycarbonate films, high-T, superconducting and metallic thin films (see figure). This has been achieved by using a dynamic mode of SFM operation at high loading forces. To address the problem of connecting the nanoscopic to the macroscopic world, we have combined photolithographic techniques with SFM nanofabrication. This allowed to perform transport measurements at low temperatures on some of the nanofabricated devices.

High-T, superconducting thin film microbridge

Gold thin film

NS-TP12 New Design of STM-CVD Nanofabrication, S. Rubel, X.-D. Weng, and A.L. de Lassalle, Department of Physics, University of Austin, Texas 78712-1081, USA.

For the past few years we have made transport measurements of pure nickel wires made by decomposing nickel carbonyl with the electrons from an STM, which is a form of chemical vapor deposition (CVD). The need to align the tip with four micron-size contact pads resulted in a design incorporating a non-UHV SEM, and a concomitant
low throughput of samples. To improve throughput and reliability, we have finished a novel design that utilizes a high power optical microscope instead of an SEM. Two key features of this design are a custom window which is positioned directly on the side of the chamber and a very compact x-y positioner. The 2-inch sapphire window, which protrudes into the vacuum chamber, makes it possible to have the tip as close as 6 mm from the atmosphere side. This gives an excellent view of the whole STM and allows us to use a 40x (or 20x) objective lens with 14 mm working distance (or 19 mm). The extra distance on the objective side is used to be able to have different viewing angles with the objective. This, together with the technique of Rapid Field Emission Imaging which we have demonstrated earlier (J. Vac. Sci. Technol. B, May/June 1994, to appear), will make it possible to locate any desired feature on the surface. The x-y positioner is built into a standard fine- 

screen tripod. It provides 200-300 μm of orthogonal motion by tilting a 0.25-inch ball bearing that holds a 0.125-inch ball bearing in an off-center position. We will present results from both STMs.

NS-ThP13 Submicron Pattern Transfer in HgCdTe and GaAs by Reactive-Ion Etching Through Nanochannel Glass Lithographic Masks, C. R. Eddy, Jr. and R. J. Tomoczek, Naval Research Laboratory, Washington, D.C. 20375.

A new technique has been developed for parallel patterning of submicron high definition features on HgCdTe and GaAs substrates. The technique utilizes reactive ion etching through nanochannel glass (NGC) array structures. Nanochannel glass materials contain a uniform array of submicron parallel channels or capillaries arranged in a 2-dimen- 
sional hexagonal closed packed configuration. Thin wafers of the glass are placed on the surface of the substrate forming the basis for a contact lithographic mask. A low pressure plasma of methane and hydrogen is used to reactive-ion etch HgCdTe substrates biased from 50 to 200 volts and held at room temperature. The addition of argon to this mixture is used to pattern GaAs. Both positive and negative feature replication of the NGC pattern has been demonstrated by this parallel processing technique. The relevance of channel glass aspect ratios to plasma processing parameters, especially pressure and ion energy anisotropy, on the definition of patterned structures will be discussed.

NS-ThP14 The Technological Aspects of Cluster Film Application in Nanoelectronics with Use of ETM, P. N. Luskinovich, E. E. Gutman and I. A. Ryzhikov, "Deltc" R&D Institute, Stichelkovskoje shosse, 2, 107122 Moscow, Russia, Karpov Institute of Physical Chemistry, 10 Obukha ul., Moscow 103464, Russia.

We describe the investigations on the reversible processes of stable potential relief features by the way of the local electrical influence on the surface of the cluster conducting films with the help of STM. In particular we discuss the results of investigation of the films which have the property of selective sensor in respect of various gases (CO, N and so on). The investigations are made on condition of the low pressure and the controlled medium. It’s shown that the stable features of potential relief with character size of several nanometers are achieved. The variants of application of such technological method are offered.


Previously, we have reported on the addition and electron beam exposure of various forms of carbon on silicon dioxide surfaces. We observe localized rate enhancements in the HF vapor oxide etching process where such exposure has occurred and have shown this route (Carbon Enhanced Vapor Etching, CEVE) to be a viable alternative approach for fine pattern delineation in silicon dioxide. This work is a continuation of this study in which we examine carbon-containing coatings as a means of generating nanoscale features on silicon dioxide using a scanning tunneling microscope (STM) as an electron beam source. A Digtal Instruments Nanoscope III was used to expose oxide films of ca. 5 nm thickness (insulator coatings >10 nm inhibit tunneling), with tip voltages and currents varied between 4 to 10 V and 0.1 to 1 nA respectively and ambient hydrocarbons used as the carbon source. Careful selection of voltage and current settings allowed the controllable production of oxide trenches of width from 10 to 75 nm following HF vapor exposure. Evidence eliminating STM oxidation of the underlying silicon as the trench forming mechanism will be presented. Additional work on the use of controlled ambients as a means of further reducing trench width and possible reaction pathways for the rate catalysis will be discussed.

NS-ThP16 Ambient Dependence of Nanometer Scale Writing on Silicon (100) Surfaces by an Atomic Force Microscope,* L. Tsou, Dawn Wang, and K. L. Wang, Department of Electrical Engineering, University of California at Los Angeles, Los Angeles, CA 90024-1594 USA.

Nanometer scale writing has been performed on Si(100) surfaces by using an atomic force microscope. The writing work was firstly demonstrated in a regular atmospheric environment. By biasing a p-type heavily doped silicon AFM tip negatively above ~3 V, patterns as small as ~10 nm could be generated. There were evidences showing that local oxidation takes place within a few atomic layers below the silicon surface during the AFM writing process. It was also found that no pattern could be generated for positive bias voltages up to 10 V. In order to investigate the mechanism of the tip-surface reaction during writing, similar work was performed in a controlled ambient environment. Firstly, it was found that, in a dry nitrogen or dry oxygen environment, it was difficult to generate patterns for bias voltages up to ~10 V. Further, the writing efficiency for each bias voltage (ranging from ~3 V to ~10 V) was examined by introducing water vapor into the box at various flow rates. The experimental results indicate that the moisture content plays an important role in the tip-surface reaction. A strong dependence of the writing speed upon the humidity was observed.

*This work was in part supported by JSEP and AFOSR.


A scanning tunneling microscope (STM) was utilized to form a nanometer-scale molecular memory. First, n-alkane molecules (C19H40) were adsorbed on a highly orientated pyrolytic graphite (HOPG), and then electric pulses with various pulse heights and duration times were applied through the STM tip to the molecular layers. After many trials, it was first revealed that some pulses, for example 2.0 V in height and 0.3 μs in duration, can cause the morphological changes of nanometer sizes to the organic layer, as depicted schematically in the figure. The diameter of the column created by such the electric treatment was very small in the range of a half nanometer. Since this kind of molecular column may be utilized to get on/off or 1/0 signals, a present result proves the possibility for the creation of a novel and extremely high density molecular memory with about 10^4 bits/cm^2, which is about 10^4 times higher than a conventional CD.

[Memory density : 1 0 1 4 bit/cm^2]

0.5 nm

Molecular Layer

HOPG substrate

Fig. Schematic illustration for the nm-scale memory unit formed by applying electric pulses to a molecular layer using a STM tip.

NS-ThP18 Using of Scanning Tunnelling Microscopy for Control and Revision of Strong Rifle Surfaces of Plane Carbon Field Emitters Modified by Ion Beams, A. L. Suvorov, V. V. Prasenko, V. G. Stolyarova, E. P. Sheshin, Institute of Theoretical and Experimental Physics, B. Cheremushkinskaya 25, Moscow, Russia.

Here is offered and realized method of preparing plane carbon field emission cathodes with 105 cm *cm) density of emission centres. At first stage this method include modification of initial plane surface of monolithic carbon half-finished products. In this work, La3+, Ar tested, found and optimized energy of ions (En = 35 keV), at an angle of 45 deg, irradiation intensity is 5x10^14 [ions/cm *s] and a field is about 1017 to 1018 [ions/cm *s].

The quality control of obtained field cathodes surfaces is carried out with using of air STM (original construction). This instrument (air STM) used for the final precision processing of surface formed by ion beams. Volt-Amper and other emission characteristics of prepared field cathodes is carried out.
Using this method is useful for realizing of such basic goal of vacuum microelectronics as creation of planar display screens and effective light sources (on the base of field electron emission of carbon materials).

NS-ThP19 Negative-Differential Conductivity Observed on a Germanium Layer on Si(001), H.-J. Müssig, D. Krüger, S. Hinrich, Institut für Halbleiterphysik, Walter-Korsging-Str, 2, D-13220 Frankfurt (O), Germany, and P. O. Hansson, Max-Planck-Institut für Festkörperforschung, Heisenbergstr. 1, D-70569 Stuttgart, Germany.

During the last years there has been a great interest in Si$_x$Ge$_{1-x}$/Si layered heterostructures, considering both basic material issues and applications in high performance devices. The morphology and the surface electronic structure of heteroepitaxial germanium layers grown pseudo-morphically by solution epitaxy on Si(001) has been investigated by scanning tunneling microscopy (STM) and -spectroscopy (STS). The Ge layer thickness has been estimated to be 4 to 5 monolayers by Auger electron spectroscopy. This thickness is near the critical thickness of strain relaxation by coherent island nucleation. By means of STS the tunneling current is measured as a function of sample voltage at a constant distance between tip and the sample on certain surface atom positions. A significant decrease of tunneling current at a sample voltage of 1.5 V is observed in areas of 0.5 nm diameter between dimer rows. This decrease is due to a negative differential conductivity at a tunnel diode configuration consisting of a surface defect structure of the germanium layer and the STM tunneling tip. A negative differential conductivity is a characteristic of quantum-well type structures. The same effect is utilized in the Esaki tunnel diode which is widely used in high-speed electronics applications.


Centre for Interdisciplinary Studies of Molecular Interactions, Chemistry Department, University of Copenhagen, Fruebjergvej 3, DK-2100 Copenhagen, Denmark.

By increasing the force when scanning with an atomic force microscope (AFM), it is possible to scratch holes in a Langmuir-Blodgett (LB) monolayer of behenic acid, and “write” lines with a width of 50 to 90 nm. A molecular electron acceptor and a molecular donor have been synthesized and investigated as monolayers by synchrotron $x$-ray diffraction and as multilayer LB-films by AFM. The number of defects and the degree of order show that these functionalized molecules may be suitable for application relying on the potential defect-free periodic structure of LB-films. In this talk it is shown that nanometer scale structures of 100 nm to 500 nm, and stable over at least 24 hours, can be written on these functionalized molecules. Progress on forming more narrow lines and defect free areas suitable for electrical contact, using very sharp tips, will be reported. The prospect of this technique to form prototype functionalyzed electronic devices using alternating layer of molecular electron donors and acceptors will be discussed.


The single crystal epitaxial layer ohmic contacts are very promising in the fabrication of shallow junction nanoelectronic and mesoscopic devices based on III-V compounds. Several metals and semiconductor epitaxial layers (e.g. Au, Ge or Pd, Ge) are deposited at a temperature range of 300–450°C on GaAs or InGaAs using an Ultra High Vacuum (10$^{-6}$–10$^{-7}$ Torr) Electron Beam (UHV E-Beam) deposition system. The interface between the metal and semiconductor is almost atomically abrupt, smooth and oxide free, and there is a minimum of disruption of the underlying layers. The in-situ cleaning of the III-V substrate at a temperature range of 500–650°C is achieved without any Au or P over pressure which is a very common practice in a molecular beam epitaxial (MBE) deposition system. A detailed analysis of interface quality, crystal structure and defect propagation in GaAs/Ge/Au, GaAs/Pd, InGaP/Ge structures are presented. High resolution Transmission Electron Microscope (HRTEM), Double Crystal X-ray Diffraction (DXRD), and Auger Electron Spectroscopy (AES) are used to characterize the materials.

NS-ThP25 Nanoscale Lithography and Pattern Transfer on Si(100) with a UHV Scanning Tunneling Microscope, G. C. Abeln, T.-C. Shen, C. Wang, J. R. Tucker, and J. W. Lyding, Department of Electrical and Computer Engineering and Beckman Institute, University of Illinois, Urbana.

We demonstrate nanoscale patterning of the hydrogen passivated Si(100) surface using a UHV STM, with linewidths as low as 1 nm.

This work is supported by NSF grant ECS-9303397.
being achieved. H atoms saturating the Si dangling bonds are desorbed by low energy electrons from the tunneling probe—it is shown that desorption occurs when the classical electron energy exceeds the Si-H bond energy (3.5-3.8 eV). Oxidation of the patterned areas is also demonstrated. The effect of NH3 exposure on the oxidation of the patterned areas will also be discussed.

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ELECTRONIC MATERIALS
Room BR4 – Session EM-ThP

Aspects of Electronic Materials and Processing II
Moderator: A. Rockett, University of Illinois, Urbana.


The use of W plugs for interconnect has become a widely used technology. There are several different process flow which can be used to achieve W plugs coupled with the AI interconnect. To reduce the number of processing steps the TiN adhesion layer for the CVD W can be left intact after W etch for use as the barrier layer for the AI. Previous workers have found that this process scheme can affect the EM lifetime of the interconnect.1 They determined that the crystal structure of the AI was affected by damage to the underlying TiN layer during etch. We extended that work by looking at the damage systematically and quantitatively. Thus, we extended the process parameters and at the effect the extent of the damage has the subsequent AICu sputter deposition.

The surface roughness due to etch damage of the TiN adhesion layer was studied using AFM. This roughness was compared to the structures of the as-deposited TiN and W films. We find that the roughness of the TiN film is influenced by the etch process and is not due only to the transfer of the W roughness into the TiN film during etchback.

The roughness of the AICu film was also studied by AFM. We determined that there were significant differences in the roughness and grain size of the AICu film as a result of the structure of the TiN film. These differences are discussed in light of the roughness of the TiN film and related to the etchback process parameters.


EM-ThP2 Low-Resistivity Ohmic Contacts to Moderately Doped n-GaAs with Low Temperature Processing, Michael L. Lovejoy, Arnold J. Howard, Peter A. Barnes,1 Dennis J. Rieger, Kevin R. Zavadil, Randy J. Shull and John C. Zolper, Sandia National Laboratories, Albuquerque, NM 87115,1 Auburn University, Department of Physics, Auburn, AL.

Low temperature ohmic contact processing is required for a variety of novel material systems such as II-VI materials that are grown at temperatures as low as 300°C which limits processing temperatures. In addition, for some photonic device applications where free carrier absorption degrades device performance, low-to-moderately doped substrates are desirable. PdGe contacts which are formed at intermediate growth rate on n-GaAs are most suited for this metallization scheme has focused on improving thermal properties to make the contact suitable for subsequent high-temperature processing. In this paper we report a comprehensive study of PdGe ohmic contacts to moderately-doped n-GaAs with the lowest possible anneal temperature and time.

The mechanism believed to be responsible for the excellent ohmic property of the GaAs surface by Ge that diffuses through the Pd. During the Ge-diffusion PdGe is formed and the film is highly non-ohmic and resistivity is high. Variation in composition is characterized by Auger and XPS. Surface morphology is mapped out with AFM and is compared to common Au/Ge alloyed contacts which suffer from non-uniformity and surface roughness which makes interconnect metallization difficult. The Cox and Strack method is used to measure the contact resistivity. For 30 minute anneals, a minimum specific contact resistance of ~ 2 μΩ-cm² is obtained at 250°C which is 75°C lower than previous investigations considered at this doping level. To investigate the implication of incomplete PdGe formation on electrical, parasitic, wave measured, and reported resistance of the PdGe contact metal as a function of anneal temperatures from 200-400°C. The degree of PdGe formation determined by spectroscopy is correlated to the sheet resistance properties.

EM-ThP3 Excimer Laser Induced Deposition of Tungsten on GaAs from WF6 and SiH4, Maleek Tabbal, Michel Menauer, Ricardo Izquierdo and Arthur Yelon, Groupe des Couches Minces and Departement de Genie Physique, Ecole Polytechnique de Montreal, Quebec, Canada, H3C 3A7.

The excimer laser direct projection patterning of W Schottky contacts on GaAs to form, in one step, self-aligned gates for MESFETs could prove to be an advantageous process compared to the conventional sputtering, patterning and etching process now used in the industry. We present recent results on large area excimer laser induced deposition of tungsten using WF6. Silane gas is used as the reductant, permitting the deposition of W at a temperature as low as 200°C and thus limiting the thermal budget on the fragile GaAs substrates. Typical operating pressures were ~140 torr for a mixture of WF6, SiH4, H2 and Ar, having flow rates of 1.3, 30 and 140 sccm, respectively. Using a KrF excimer laser (248 nm) at 25 mJ/cm², metallic W films (as determined by XPS), are deposited with an averaged growth rate of 1 Å/pulse. The tungsten layers have a diamond structure, as evidenced by SEM. Auger Electron Spectroscopy profiling shows that no impurities (such as F, C and O) are detected above the noise level (~1 at%). The absence of Si suggests that the SiH4 reduction is complete. X-ray diffraction spectra show that the pure and stable α-W is deposited and that very little β-W is present. Resistivities are 20 μΩ-cm, in agreement with the observed predominance of α-W. Possible deposition mechanisms are discussed in terms of gas phase chemistry and surface reactions.

EM-ThP4 The Effect of Chemical Etchants on Back Contact Formation to CdTe-Based Solar Cells, Xiaonan Li, Dave Niles, Falah Hasoon, and Peter Sheldon, National Renewable Energy Laboratory, 1617 Cole Boulevard, Golden, CO 80401.

CdTe is an important material for a variety of thin film electronic applications. For example, Cds/CdTe photovoltaic devices have reached conversion efficiencies of up to 15.8%. Forming a low resistance ohmic contact to CdTe thin films is critical to the success of this technology. Pre-treatment of these materials are used to modify the surface chemistry to facilitate ohmic contact formation. In this study, CdTe thin films, deposited by close spaced sublimation (CSS), are characterized using cathodoluminescence (CL) and light and dark I-V measurements both before and after chemically etching in H3PO4:HNO3:H2O solution. CL measurements of CdTe films etched for 1 minute had a ~2 μm thick low emission layer. This low emission layer was found to result from conversion of the near surface region from stoichiometric CdTe to a Te-rich layer. The Te-rich layer is thought to improve the series resistance, however, light and dark I-V measurements show that the conductive Te-rich layer introduces shunt paths into the CdS/CdTe device. These shunt paths are found to significantly degrade device performance. We demonstrate that shunting can be reduced by varying the surface treatment. Finally, devices etched in bromine methanol, prior to contact application, are made for comparison.

EM-ThP5 STM Observations of Sub-Surface Donor and Acceptor in GaAs, Jun-Fi Zheng, D. Frank Ogiele, Elke Weber and Miguel Salmeron, Materials Sciences Division, Lawrence Berkeley Laboratory, and Materials Science Department, University of California, Berkeley, California 94720.

The scanning electron microscope has been widely used to study the structure and electronic properties of semiconductor surfaces. Recently we have used this technique to investigate the bulk electronic properties of point defects in GaAs. When cleaved, GaAs(100) shows a crystalline reconstruction and no new states are created in the gap. As a result perturbations in the STM tunneling current due to sub-surface features can be observed. We have investigated Si doped n-type and Zn doped p-type GaAs. Sub-surface donor and acceptor sites can be identified in the top six layers of the crystal. Unlike dopant atoms at the surface, which induce localized states in the gap, the sub-surface dopants appear in the STM image as de-localized states. They are a few nm wide superposed on the normal atomic lattice of the surface. Both donors and acceptors produce positive protrusions in filled and empty state images, circular for Si and Zn in filled state images and triangular for Zn in empty state images. The protrusions decrease in height as the
STM bias voltage moves away from the band edge and for dopants further below the surface. Imaging mechanisms, and evidence for sub-surface vacancies will be discussed.


Most measurement techniques of Schottky barrier heights and heterojunction band discontinuities are based on an inherent assumption of homogeneity. The recent development of new microscopic-scale experimental techniques enabled us to reveal strong lateral variation of these interface parameters.

In particular, our scanning photoemission spectromicroscopy data revealed substantial inhomogeneities in the band lineup of the interface between the two heterolayers GaAs and Ge. These inhomogeneities lead to valence band discontinuity changes from place to place, whose magnitude is at least of 0.4 eV.


The evolution of the interface between Bi and polycrystalline, ceramic ZnO has been studied in situ with photoelectron spectroscopy (PES) under ultra-high-vacuum conditions. This system is a model varistor (variable resistor) compound. Evaporation of Bi on highly n-doped (Al) doped ZnO layers, fractured in situ and held at room-temperature, results in a considerable upward band-bending. After evaporation of Bi at room temperature, a Bi-induced bandbending of 0.43 eV, can be observed from the energy-shift of the Bi 5d emission in PES. Valenceband and bandgap studies using ultraviolet PES, directly reveals the states responsible for the observed bandbending: Bi induces states in the ZnO bandgap at 0.9 eV above the valenceband maximum. The filling of this high density of bandgap states yields a pinning of the Fermilevel which makes the bandbending proportional to the Bi coverage, with a rapid increase during the formation of the first monolayer and a markedly slower thereafter. These results clearly show the importance of Bi in the formation of the varistor Schottky barriers.


We report internal photoemission measurements of the low temperature band-offset of dilated magnetic semiconductor (DMS) heterojunctions as a function of magnetic field (0-2 tesla). Two photon sources were used covering the wavelength range from 4 nm to 10 μm: the Vanderbilt University Free Electron Laser which produces intense 2-10 μm infrared and a monochromatized quartz halogen lamp covering the visible to 3 μm range. The optical nature of the measurements allows us to determine the photoconductivity threshold associated with the band-offset to an accuracy of better than 5 meV. Tuner effects have been corrected by varying the bias voltage. The light was brought to the interface at grazing incidence so that both s and p polarizations could be used to determine the k-space dependence of the band-offset. Other interfaces to be discussed include diamond Schottky barriers and GaAs homojunctions with Si layers.

EM-TP9 Interface Exciton Luminescence: Indication of Interface Inhomogeneities in Single GaAs/GaAs Heterostructures, V. N. Bessolov, V. V. Evtovtsov, M. V. Lebedev, V. V. Rossin, A. F. Ioffe Physico-Technical Institute, St. Petersburg, 194021, Russia.

A specific luminescence band originating from heterointerface (H-band) has been observed in the photoluminescence spectrum (at 4.2 K) of a single GaAs/GaAs heterostructure. Threshold effect of interface formation conditions (epitaxial temperature and growth rate) on interface luminescence has been found. The H-band is observed if growth temperatures are above some critical value and is absent if those temperatures are below. The threshold emergence of the H-band is interpreted as consequence of the GaAs surface roughening transition observed earlier only for homoepitaxial growth. Inhomogeneities on interface critically depend on growth temperature, drastically decreasing above critical temperature. The model has been proposed which attributes interface luminescence to annihilation of interface exciton which is composed from 2D-electron (2D-hole) in the notch at the interface and 3D-hole (3D-electron) in GaAs bulk at the interface. In the case of large inhomogeneities the interface potential fluctuations prevent formation of such exciton and corresponding H-band is not observed in spectrum. Thus, interface exciton luminescence can be used for characterization of the interface inhomogeneity.

EM-TP10 The Nature of Ambient (100) GaAs Surfaces. O. J. Glembocki, J. A. Tuchman, K. K. Ko,* S. W. Pang,* J. A. Dagata,** R. Kaplan and C. E. Stutz,** U.S. Naval Research Laboratory, Washington, DC 20375, USA.

Photoelectrolyte (PR) spectroscopy has been used to study the Fermi-level pinning position of chemically modified (100) GaAs surfaces. The stoichiometry of the GaAs/oxide interface was obtained by Auger electron spectroscopy and time of flight SIMS. PR shows that there are two pinning positions for the semiconductor GaAs on the Fermi level. For GaAs, the Fermi level pinns near midgap, while for p-GaAs the Fermi level pins near the valence band. An Ar/C12 plasma generated by an electron-cyclotron resonance (ECR) source and a P25 chemical passivation treatment were used to change the stoichiometry of the surface. ECR etching resulted in an As rich GaAs/oxide interface, while P25 passivation produced a thin Ga rich oxide. The Fermi-level pinning position for the As rich oxide is shown to be near midgap while for the Ga rich oxide it is near the valence band. These results allow us to relate the Fermi-level pinning position to the stoichiometry of the GaAs/oxide interface and to test various models for the Fermi-level pinning position of ambient (100) GaAs surfaces.

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EM-TP11 Surface Studies of MBE Grown GaAs(111)A by X- and UV Photoelectron Spectroscopy. F. R. Hreels, XPS, UPS and LEED, J. Wu and G. J. Lapeyre, Physics Department, Montana State University, Bozeman, MT 59715.

P-type GaAs(111)A(2\times2), grown by molecular-beam-epitaxy (MBE) method, has been studied by high-resolution electron energy-loss spectroscopy (HREELS), X-ray and ultra-violet photoelectron spectroscopy (XPS and UPS), and low-energy electron diffraction (LEED). During atomic hydrogen exposure, XPS spectra of Ga 3d (-126.5 eV) and As 3d (-106.5 eV) revealed no significant changes of surface stoichiometry, and the band bending ranges from 0.4ev to 0.6eV from the UPS data. At 500L hydrogen exposure, (1\times1) LEED pattern with very high background develops due to the surface disorder. The LEED results from the clean surface shows the surface optical phonon energy (~35meV) with a large broad background, which is caused by small hole mobility and the dependence on the incident electron energy. With increasing hydrogen exposure, characteristic changes in the intensities of surface optical phonon and the hole plasmon excitation, features strong Ga-H stretching mode (~360 cm^-1) is observed. At 2000L hydrogen exposure, double surface optical phonon show up. Those are interrupted by the changes in the space charge regime and GaAs(111)A vacancy buckling model.

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Research supported by NSF.

EM-TP12 Geometric and Electronic Structure of Ni Silicide on Si(100). Y.-H. Kang, D. Jeon* and Young Kuk, Department of Physics, Seoul National University, Seoul 151-742, Korea, *Department of Physics, Myong-ji University, Kyunggi-do 449-728, Korea.

It is now understood that NiSi2/Si(111) is a model system in which the Schottky barrier heights are determined by the atomic structure of the interfaces. In NiSi2/Si(100) system, it was reported that the measured Schottky barrier heights varies with samples having different dopant concentrations, annealing times and temperatures.

We have studied this system by scanning tunneling microscopy/ spectroscopy and tunneling electron interferometry. Besides the previously reported the (1×1)-NiSi2 phase in TEM and STM studies, phases such as the (2×1)-NiSi2(√2×√2)R45° and (111) facets have been observed under various growing conditions. We propose a model for the (1×1) phase structure, in which the inverse-pyramidal patch structure with stacking fault is considered. The lower interface energy along the (111) direction causes the pyramidal shape, resulting in uniformly spaced antiphase boundaries. The observed multiple phases are due to several local minima in the interfacial phase diagram.
as suggested by a theoretical calculation. By measuring the differential conductivity in a Fowler-Nordheim regime, the structure of N/Si interface was imaged together with the surface topography. The observed interfacial structure will be compared with the results by BEEM.


Epitaxial iron silicides have been actively investigated in recent years due to their possible applications and their interesting phenomenology. Out of the many existing phases, β-FeSi 2 is the only bulk stable, semiconducting phase. It presents an orthorhombic structure. When epitaxially grown on Si(111), a metastable phase called γ-FeSi 2 can be obtained under suitable conditions. γ-FeSi 2 crystallizes in the fluorite structure, and is metallic. It has been proposed that it might be spin polarized. On the other hand, a 2 x 2 surface reconstruction is observed when this phase is grown on Si(111). Two important issues remain to be clarified concerning the electronic structure of γ-FeSi 2 nature of the states giving rise to the Fermi edge, nature of the electronic states associated to the 2 x 2 reconstruction (which depends on its detailed geometric structure). We have answered both questions by performing a detailed angle-resolved photoemission-spectroscopy study. We have mapped the band structure of γ-FeSi 2 along the QL direction, and the dispersion of the surface states along the high-symmetry directions of the surface unit cell. The symmetry of the states was determined by taking polarization-sensitive measurements. The results were compared to existing theoretical calculations. We have measured the dispersion of bulk states, in particular for the band crossing the Fermi level, whose shape significantly differs from theoretical predictions. The dispersion of the observed surface states supports an adatom-based model for the 2 x 2 reconstruction.


Atomic hydrogen is a very important constituent for processes ranging from amorphous silicon and diamond deposition to the cleaning of silicon wafers. Because measurement of the actual atomic species fraction in highly dissociated hydrogen is very challenging, a specially designed catalytic sensor was developed. The sensor is simple and inexpensive to fabricate. It also inhibits thermal runaway, which occurs when atomic species density is high enough to impart significant recombination energy to a non-catalytic surface to substantially raise its temperature. While recombination coefficients for such surfaces are very low near room temperature, they increase nearly exponentially with temperature unless actively cooled. The sensor comprises a cooled quartz diffusion tube that is open at one end to sample the highly dissociated gas. The other end is terminated by a catalytic surface of nickel. This surface is actually one end of a nickel rod that is thermally anchored at its other end. Temperature is measured at two points along the rod to determine power deposited by recombining hydrogen on the rod tip. This is employed with the known recombination coefficient of nickel to determine atomic species fraction. With the use of a straightforward calibration scheme to determine the nonlinear variation in species fraction along the tube, the atomic species fraction at the tube opening is then determined. For example, the atomic hydrogen species fraction at the output of a flow tube operating at a flow of 100 sccm, pressure of 400 mTorr, and at power input of 200 W was measured to be 0.8 decreasing to 0.5 when pressure was increased to 1.0 Torr. Details of sensor design, calibration, and application will be discussed.


EM-Thp15 Spectroscopic Ellipsometry Studies of a-Si 1-x C x:H Film Growth by Reactive Magnetron Sputtering, Y. H. Yang, M. Katar, and J. R. Abelson, Coordinated Science Lab. and Materials Science Department, U. Illinois, Urbana, IL 61801.

Hydrogenated amorphous silicon carbide (a-Si 1-x C x :H) films with x ≅ 0.3 are suitable for use as the active layer in visible-light-emitting diodes, as the dielectric in metal-insulator-semiconductor devices, and as mechanically hard coatings. We deposit a-Si 1-x C x :H films by dc reactive magnetron sputtering of a Si target in a mixture of Ar + CH 4 + H 2. This process produces dense and homogeneous material. We demonstrate that real-time, in-situ spectroscopic ellipsometry (2.0-4.5 eV) provides information on the structure of films and interfaces. Films are deposited at temperatures of 100-400°C with 0.2 mTorr CH 4 on native-oxide-covered silicon substrates. Below 170°C, a layer with high refractive index is formed between the substrate and film; the thickness of this layer diminishes at higher temperatures. The dielectric functions of the films deposited above 230°C have the same values for the imaginary part, while the real part values increase with temperature. This indicates that the films are denser at higher temperatures. Ex-situ IR measurements show an increased absorption around 780 cm -1 , corresponding to Si-C stretching mode. Study of the films deposited under different CH 4 or H 2 pressures is underway.

EM-Thp16 Numerical Ellipsometry: Application of a New Algorithm for Real-Time, in-situ Film Growth Monitoring, John Craig Comfort and Frank K. Urban III, School of Computer Science and Department of Electrical Engineering, Florida International University, Miami, FL 33199.

The equations associated with the popular models of film deposition of a material on a substrate tend not to be invertible in practical situations. Thus development of numerical techniques for obtaining approximate solutions to these equations becomes necessary. At present, real time, in-situ thin film growth ellipsometry data is limited to a single angle of incidence due to the deposition system windows. The 2C algorithm, developed by the authors, represents a new approach to solve this problem. This algorithm is fast enough for reliable real time solutions without requiring either accurate initial estimates or any off line processing. Algorithm 2C employs the techniques of dynamic step adjustment, overrelaxation, and reduction in dimension, as well as curve following and curve intersection location techniques developed by the authors. In this paper, the algorithm has been extended to solve the equations for silicon, silicon and gold depositions on BK7 glass. An example of the results generated by the algorithm is shown at the right for nickel deposited on BK7 glass.


The main puzzle in oxidation of hexagonal SiC is the slower rate of the Si-terminated surface as compared to the C-terminated surface, which is blamed on an unknown interface compound. ARXPS is a versatile method to identify minor amounts of interface compounds, especially for smooth surfaces. Our ARXPS analysis of oxidized Si(001) and C(001) surfaces of 6 H SiC reveals the interface oxide Si 3 C x O y (x < 2, likely a reaction product of a peroxide O 2 - bond to a Si-C double layer. Si 3 C x O y occurs in larger thickness (≈ 1 nm) at the slowly oxidizing Si(001) surface, whereas the C(001) surface shows smaller amounts, only, diminishing fast with oxidation above 700°C. Evidence is presented that with increasing amount of Si 3 C x O y the oxidation of SiC to SiO 2 is reduced. ARXPS is consistent with a layer of SiO 2 containing less than 3% Si 3 C x O y being an oxidation product of Si 3 C x O y. At the surface of SiO 2, graphite and some Si 3 C x O y exist, aside from standard adsorbates.

EM-Thp18 Adsorption and Co-adsorption of Boron and Oxygen on Ordered a-SiC Surfaces, F. M. Bermudez, Naval Research Laboratory, Washington, DC 20375.

Boron layers have been grown (by thermal decomposition of B 2 H 6) on ordered Si- and C-terminated surfaces of a-SiC and studied using AES, ELS, and LEED. Adsorption of O 2 on the clean and B-adsorbed surfaces has also been studied. Cleaning the Si-face by annealing in
a flux of Si vapor yields a \((3 \times 3)\) structure due to a layer of chemisorbed Si. Annealing the \((3 \times 3)\) in UHV produces Si vacancies, giving a \((\sqrt{3} \times \sqrt{3})R30^\circ\) structure. A \((1 \times 1)\) structure is seen for the C-face. Boron interacts only weakly with the \((3 \times 3)\) Si- and \((1 \times 1)\) C-faces. On the \((\sqrt{3} \times \sqrt{3})R30^\circ\) Si-face, structure in the Si L23VV and B KLL lineshapes suggests compound formation, and an incommensurate \((1 \times 1)\) overlayer appears in LEED. On B-free surfaces, room-temperature chemisorption of \(O_2\) is slow on the \((1 \times 1)\) C-face than on the \((3 \times 3)\) Si-face, but the rates of O uptake become comparable as coverage increases and chemisorption gives way to oxidation. On the \((\sqrt{3} \times \sqrt{3})R30^\circ\) Si-face, chemisorption is rapid but oxidation slow. Pre-adsorbed \(O_2\) suppresses \(O_2\) chemisorption on all surfaces but has little or no effect on oxidation. The implications of this work for "16-doping" of a-SiC will be discussed.


Traditionally hot atoms can be used to overcome many problems traditionally associated with processing, passivation, and oxide growth on compound semiconductor materials. The unprecedented growth of thick, uniform, and extremely high-quality oxide layers on (100) and (110) GaAs wafers has been demonstrated by exposing the substrate \((T_e<250^\circ C)\) to a high kinetic energy \((2.5 \text{ eV})\) beam of neutral atomic oxygen at flux levels of ~50 monolayers/second. Oxide layer characterization was performed using x-ray photoemission spectroscopy (with \(Ar^+\) ion sputter profiling), Raman spectroscopy, ATR infrared spectroscopy, x-ray diffraction, and photoluminescence. XPS shows the oxide layer to be \(>200 \text{ Å}^\circ\) thick and uniform in composition, with the Ga and As species formed in their highest oxidation states (implying the formation of either Ga\(_2\)O\(_3\) or As\(_2\)O\(_3\); or GaAs\(_2\)O\(_4\)). Raman spectroscopy indicates that there is no metallic As (amorphous of crystalline) in the oxide or at the interface between the oxide and substrate and that there is no appreciable oxidation induced disorder of the substrate as is seen in high temperature thermal and plasma oxidation processes. Photoluminescence measurements also show no degradation of the oxide/substrate interface and are used to evaluate oxide quality. The effects of GaAs substrate temperature and beam exposure time on the oxide layer growth kinetics will be discussed. GaAs samples flown on the Space Shuttle and exposed to ambient atomic-O will be compared to results from our laboratory. Applications of this new non-thermal materials synthesis technology to semiconductor processing will also be discussed, and results for other semiconductor materials will be presented.

**EM-THP20** Oxidation-Induced Roughening and Oxide Nucleation on Si(001)-2×1 Surfaces: An STM Study, J. P. Polit and J. F. Selpke, The Ohio State University, Department of Physics, Columbus, OH 43210.

Room temperature STM measurements were used to study surface roughening on Si(001)-2×1 samples exposed to \(O_2\) from pressures of \(1 \times 10^{-8}\) to \(5 \times 10^{-6}\) torr and temperatures from 500 to 700°C. At \(T=600^\circ C\) and \(1 \times 10^{-6}\) torr, a plate step-recessed \(B_{\Delta}\) structure is observed, but single A-domain formation is prevented due to step pinning. This pinning roughens the surface via step "fingering" and the formation of multi-layer Si islands. The strong dependence of pinning density with oxidation pressure and the characteristic nature of the pinning defects indicate that the pinning sites are nucleated oxide clusters. These pinning sites were counted over large areas to estimate an oxide nucleation rate, \(J_{\text{OX}}\). This nucleation rate was found to scale with the oxide pressure as \(J_{\text{OX}} \propto P_{\text{OX}}^{m}\), with \(m>2\). This dependence is consistent with a simple model in which two diffusing oxygen species are required to nucleate a stable oxide cluster. The critical \(O_2\) pressure (at a dose of 65L) for the transition between the etching dominated and oxide-growth dominated regimes is about \(1 \times 10^{-7}\) torr at \(600^\circ C\) and \(4 \times 10^{-6}\) torr at \(700^\circ C\), but is sensitively dependent on the starting defect density of the surface. Preliminary results regarding the temperature dependence of \(J_{\text{OX}}\) will also be reported.

**EM-THP21** Growth of Layered Semiconductors by Molecular Beam Epitaxy: Formation and Characterization of GaSe, and MoSe\(_2\), Layers on Sulfur Passivated GaP(111) and GaAs(111), C. Hammond(1)*, M. Lawrence(1), K. N. Neuberger(1), P. Lee(1), R. Schlapf(1) and N. R. Armstrong(2)*, (1) Department of Chemistry, University of Arizona, Tucson, Arizona 85721; (2) Department of Chemistry, Concordia University, Montreal, Canada; (1) Hahn Melnier Institute, Berlin, Germany.

Layered compound semiconductors are attractive for the growth of heterojunctions and multilayers with molecularly sharp interfaces, which are "tailored" to optimize their electronic and photonic device properties. Weak interactions between adjacent layers may permit the formation of ordered heterojunctions from materials with a large lattice mismatch. In this first of two papers, we discuss the MBE growth and characterization of ordered layers of GaSe, MoSe\(_2\), and related semiconductors, on sulfur-passivated GaP(111) and GaAs(111). Simple solution passivation procedures allow formation of a III-V semiconductor surface which will readily support the growth of highly ordered layers of GaSe (\(E_{\text{bandgap}} = 2.0\text{ eV}\)). These thin layers then permit the further growth of ordered layers of a semiconductor like MoSe\(_2\) (\(E_{\text{bandgap}} = 1.9\text{ eV}\)). RHEED and angle resolved XPS data confirm the molecularly sharp nature of the heterojunctions formed and complement the spectroscopic characterization of ordered GaSe/MoSe\(_2\) ("quantum-well-like") multilayers.

**EM-THP22** An Atomically-Resolved STM Study of the Interaction of Trimethyl Gallium with Si(001), M. J. Bronikowski and R. J. Hamers, Dept. of Chemistry, University of Wisconsin, Madison, WI 53706.

Using Scanning Tunneling Microscopy (STM) and tunneling spectroscopy, we have studied the interaction of trimethyl gallium with Si(001). At room temperature we observe the formation of gallium-induced reconstructions, indicating that at least some of the TMG molecules completely dissociate. We also observe a corresponding number of fragments which we identify as adsorbed CH\(_3\) fragments as well as some larger fragments, attributed to Ga(CH\(_3\))\(_n\) molecules. By studying the images at successively higher decomposition temperatures we have identified the sequential decomposition of CH\(_3\) species on the surface. A statistical analysis of the number and types of fragments produced as a function of temperature provides insight into the mechanism of TMG decomposition.

This work is supported by the National Science Foundation and by the U.S. Office of Naval Research.

**EM-THP23** Reactions of Tetraethoxysilane (TEOS) Vapor on Titanium Dioxide (TiO\(_2\)) Surface, A. Jurgens(1)*, G. S. Herman(1), T. T. Tran(2), S. A. Chamber(3), C. H. F. Peden(1), and J. W. Rogers, Jr.(4), (1) University of Washington, Seattle, WA 98195 and (2) Pacific Northwest Laboratory, Richland, WA 99352.

The reactions of tetraethoxysilane (TEOS) vapor on different TiO\(_2\) surfaces were studied using Fourier transform infrared spectroscopy (FTIR), temperature programmed desorption (TPD), Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS) and x-ray photoelectron diffraction (XPD). Adsorption of TEOS on dehydroxylated or water pre-dosed polycrystalline TiO\(_2\) produces a monothiy silj ligand and ethoxide species, which decompose in the range 450 to 650 K as gas phase ethanone and ethylene and yield surface bound SiO\(_2\). On the water pre-dosed surface, FTIR shows vibrations characteristic of a hydroxylated silica species. TPD, XPS, and AES were used to characterize the reactions of TEOS on model TiO\(_2\) films deposited on a W(110) substrate. The intermediate silj ligand formed by dissociative adsorption of TEOS on a water- and hydroxyl-free TiO\(_2\) single crystal surface was examined by XPD.

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**EM-THP24** Synchrotron Radiation Induced Decomposition of closeo-1,2-dicabado-decarbocane, Donglin Byun(1)*, Seong-don Hwang(1), Jindu Zhang(2), Hong Zeng(1), P. Keith Perkins(1), G. Vidali(2), and P. A. Dowben(2), (1) Center for Materials Research and Analysis, University of Nebraska, Lincoln, NE 68588-0111, (2) Solid State Science and Technology Program and Dept. of Physics, Syracuse University, Syracuse, N.Y. 13244-1130, (3) Electronic Science Technology Division, Code 6864, Naval Research Laboratory 4555 Overlook Ave., SW, Washington, D.C. 20375-5320.

We have observed that molecular films of closeo-1,2-dicabado-decarbocane (C\(_2\)B\(_2\)H\(_{12}\)) decompose to form a heterogeneous intermediate phase between associative molecular fragments and solid, thin film boron-carbide. This heterogeneous phase has an observed electronic structure that is an admixture of the electronic structure observed for molecularly condensed orthocarborane and the electronic structure anticipated for rhombohedral boron-carbide (based on the B\(_3\)icosahedral "building block"). With the synchrotron radiation ex-
EM-THP25 Interaction of Tungsten Hexafluoride with W(100), Wei Chen and Jeffrey T. Roberts, Department of Chemistry, University of Minnesota, Minneapolis, MN 55455.

Tungsten hexafluoride (WF₆) is a precursor for the selective chemical vapor deposition (CVD) of tungsten, an important material for ultra-large-scale integrated circuit (ULSI) applications. Deposition occurs on conducting and semiconducting substrates, but not on insulating substrates. To gain insight into the basis of the selectivity, it is crucial to investigate the initial nucleation, surface reactions, and formation of surface subfluorides of WF₆ on model growth surfaces. We have studied the interaction of WF₆ with W(100) using temperature programmed reaction spectroscopy (TPRS), Auger electron spectroscopy (AES), and x-ray photoelectron spectroscopy (XPS). A multilayer of WF₆ condenses on W(100) at 100 K. Upon heating to 700 K, WF₆ is the sole gaseous product. Besides sublimation of the multilayer, two more thermal desorption features, attributed to the reactions of surface subfluorides (WF₅, WF₄, WF₃, W₂F₁₀, and W₃F₁₆) are observed at 500 and 430 K. The formation of the subfluorides begins below 200 K, and is evidenced by XPS as shifts of the N(1s) and W(4f) binding energies. One of the subfluorides is probably tungsten pentfluoride. The fluorides dissociate to atomically adsorbed fluorine around 700 K. Remaining fluorine can be removed by the electron bombardment heating.

EM-THP26 Chemisorption and Thermally Activated Etching of Iodine on Si(100)-2 x 1, D. Rios, F. Stephan, R. J. Pechman, and J. H. Weaver, University of Minnesota, Department of Materials Science and Chemical Engineering, Minneapolis, MN 55455.

Halogen-semiconductor systems are interest as models of surface-adsortate interactions, and a systematic study of adsorption and etching across the halogen family reveals the influence of electronegativity and size. These simple systems also yield insight into the complex plasma etching processes commonly used in device manufacturing. Scanning tunneling microscopy (STM) and photoemission spectroscopy were used to investigate the adsorption and thermally activated etching of I₂ on Si(100)-2 x 1. STM indicates that room temperature adsorption was dissociative on dimers dangling bonds without disruption of the dimers. Steric hindrance influenced the surface structures so that 4 x 2 domains were created when alternating dimers were iodine-terminated. Photoemission spectra revealed the presence of SiI, species where x = 1 was the majority species, in support of the STM-derived adsorption model. Exposure at temperatures in the range 600 to 900 K resulted in etching. Single-layer etch pits formed on terraces and step edges were roughened. Regrowth structures on the terraces were characterized by 2D islands and dimer chains. Etching at 900 K resulted in iodine-free areas where dimer vacancy interactions yielded a network of line defects perpendicular to the dimer row direction. At 800 K, etching produced 4 x 2 domains of iodine-terminated dimers, whereas mixed 2 x n domains dominated at lower temperatures. Etching resulted in layer-by-layer removal at the temperatures investigated here.

THIN FILM
Room BR4 – Session TF-ThP

Thin Film Characterization and Sensors Applications Moderator: A. C. Wall, IBM Corporation.

TF-ThP1 Auger Electron Spectroscopy as a Real-Time Probe of Film Composition During MBE Growth, S. A. Chambers and T. T. Tran, Pacific Northwest Laboratory,* Richland, WA 99352.

We have incorporated a commercially available, high-speed Auger electron spectrometer into an MBE system for the purpose of obtaining real-time compositional information on evolving epitaxial films. We have used the same primary electron beam as is used to generate RHEED patterns. The range of detected electron emission angles is such that Auger electron attenuation lengths are less than or equal to the penetration depth of the primary beam. We have used the system to explore incident-beam diffraction and its effect on measured Auger intensities, and to monitor composition during the epitaxial growth of MgO/MgO(001) and Al₂O₃/Al₂O₃(001). By using well-defined standard specimens and cross-calibrating with x-ray photoelectron spectrometry, we have been able to obtain spectral intensities at the rate of ~10 seconds per Auger transition, leading to composition determinations that are accurate to within ~±2%. Incident beam diffraction effects are dramatic, producing Auger intensity enhancements of up to ~50% when the primary beam is aligned with the [100] and [110] low-index directions in the MgO(001) surface. These enhancements are due to strong elastic scattering and constructive interference (forward focusing) that occurs when the primary beam is aligned with chains of atoms in the surface plane. The Auger ratio varies by ~10% with incident angle, making primary beam alignment critical in generating reproducible intensities.

*Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RL01830.


B-doped Si(001) 2 x 1 films were grown on Si(001) substrates by gas-source molecular beam epitaxy (GS-MBE) using SiH₄ and B₂H₆. B concentrations C₈ (5 x 10⁻⁵ to 5 x 10⁻³ cm⁻²) were found to increase linearly and to increase exponentially with increasing flux ratio J_B₂H₆/J_SiH₄ (3 x 10⁻³ to 2.5 x 10⁻³). At constant film growth temperature T = 600°C to 950°C and to decrease exponentially with 1/T, at constant J_B₂H₆/J_SiH₄ ratio. The B₈H₆ re-active sticking probability ranged from 6 x 10⁻⁴ at T = 600°C to 1.4 x 10⁻¹ at 950°C. The difference in the overall activation energies for B and Si incorporation at T = 600-950°C is 0.34 eV. A comparison of quantitative secondary-ion mass spectrometry (SIMS) and temperature-dependent Hall-effect measurements showed that B was incorporated into substitutional electrically-active sites over the entire B concentration range investigated. SIMS B depth-profiles from modulation-doped samples were abrupt with no indication of surface segregation to within the instrumental resolution limit and initial 8-doping experiments were carried out. Structural analysis by in-situ reflection high energy electron diffraction combined with post-deposition plan-view and cross-sectional transmission electron microscopy showed that all films were high-quality single crystals with no evidence of dislocations or other extended defects. Temperature-dependent (20-300 K) hole carrier mobilities were equal to the best reported bulk Si:B values and in good agreement with theoretical maximum values.

TF-ThP3 Mass Spectroscopy of Recoiled Ions(MSRI) as a Tool Both for In-Situ Analysis of Boron Nitride Thin Films During Deposition and Ex situ, Laura Woolworth, J. Albert Schultz, Ketti Epers-Smith, and Kelly Waters, IONWERKS, 2472 Bolsover Suite 255, Houston, TX 77005.

MSRI is performed by impinging a pulsed keV ion beam onto a surface at grazing incidence and mass analyzing the subsequently produced forward recoiled keV ionized surface atoms at time of flight. The ionized recoils are collected in specialized shaped electrostatic analyzers which provide time focusing so that moderate resolution can be obtained (400 at mass 238). The spectra are entirely free of molecular interferences because of the absence of the binary collision and the speed with which the recoils leave the surface.

Recent developments in instrumentation will be illustrated in our study of boron nitride surface chemistry. The dependence of the recoiled ion intensity upon the chemical state of the boron and upon the ambient pressure of the growth reactor will be discussed. We report a 15/1 relative sensitivity of boron/nitrogen. This relative sensitivity is good when compared to that obtained by SIMS of 10⁻¹. It will be also shown that in addition to nitrogen, MSRI is also good for several other elements which are poorly detected by SIMS.

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sitivity of the resonant fluorescence. These experiments and prospects for other high brightness experiments will be discussed.

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Science Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098 for the Advanced Light Source, Lawrence Berkeley Laboratory and under Contract No. W-7405-ENG-48 for the Lawrence Livermore National Laboratory.


Thin films of hydrogenated titanium carbide are prepared by reactive magnetron d.c. sputtering in mixed atmosphere of argon and methane or argon and acetylene. The deposition process is followed by optical emission spectroscopy (OES). The films are characterized by various techniques: scanning electron microscopy, transmission electron diffraction and microscopy (high resolution), atomic force microscopy, X-ray diffraction, Auger electron spectroscopy, X-ray photoelectron spectroscopy, and Fourier transform infrared spectroscopy.

The influence of the total and partial pressures, the current density and voltage on the structure and composition of the films is examined. The morphology and crystallinity of the deposits are strongly dependent on the carbon content in the films. The presence of amorphous carbon phase results in a decrease of the TiC grain size. The proportion of Ti-C bonds in the films is correlated to the decomposition of the carbon precursor in the discharge as shown by optical emission spectroscopy. This is especially strong in the case of C<sub>H</sub><sub>2</sub>-Ar mixture.


Ion beam-induced crystallization of SiC in Si(100) was investigated by valence band Auger electron spectroscopy and electron energy loss spectroscopy in combination with sputter depth profiling. After pre-amorphization by 200 keV Ge<sup>+</sup> ion bombardment and 25 keV C<sup>+</sup> ion implantation at room temperature and various doses, recrystallization of the amorphous carbon/silicon layer was induced by a subsequent 300 keV Si<sup>+</sup> ion bombardment at 700 K. The depth dependent composition, the different chemical states and the crystal order were obtained by a thorough analysis of the plasmon losses and the Auger line shape with Factor Analysis and least-squares fitting. This study discloses the migration of the recrystallization front from the underlying amorphous silicon substrate into the carbon-rich layer in dependence on the Si<sup>+</sup> ion dose.


The growth of 1 to 4 monolayers of iron on the (100) face of single crystal, synthetic diamond substrates has been studied with low energy electron diffraction (LEED) and angle resolved Auger electron spectroscopy (ARAES). The film thickness was measured using an in-situ fluorescence flux monitor which was calibrated against a quartz crystal monitor and an ex-situ x-ray fluorescence thickness monitor. The 4 monolayer films are shown to be in the metastable fcc phase, which is stabilized by the lattice match to the diamond substrate (2.59 Å, extrapolated from the high temperature fcc-phase of Fe, as compared to 3.57 Å for diamond). This is expected to put a compressive in-plane strain on the epilayer of Fe-Fc films. A single scattering simulation of the ARAES data was performed using a 216 atom cluster, and it is found that the films have a tetragonal distortion which is due to an ≈3% expansion of the lattice perpendicular to the plane of the film. In agreement with previous estimates [1], the LEED patterns of these films have been measured, and the reconstructions observed are compared to those observed from metastable fcc-Fe films grown on a Cu(100) substrate (a = 3.61 Å). Finally, the adsorption site of the first monolayer of Fe onto the diamond lattice has been determined by modeling the carbon ARAES data.

TF-ThP10 Study of Adsorption and Desorption of 1,5-Cycloocta-
diene on Cu(111) Surface, Q. Cheng, K. Griffiths, S. Serghini, Z. Yan, P. R. Norton,* and R. J. Puddeepeh, Department of Chem-
istry, The University of Western Ontario, London, Ontario N6A 5B7, Canada.

The adsorption and desorption of 1,5-cyclooctadiene (COD) on the Cu(111) surface has been studied under ultra-high vacuum (UHV).
Although the Cu(111) surface does not appear to exert a strong catalytic
effect on the molecule, it does influence the surface adsorption of
COD. Temperature programmed desorption (TPD) shows that both
chemisorption and physisorption occur on the Cu(111) surface. It also
indicates that most of the desorption from the surface occurs as parent
ions and the peak desorption temperatures are at ~175K and ~240K,
from the chemisorbed layer and the physisorbed layers respectively.
FTIR spectra indicates a parallel orientation of the C=C double bonds
via the absence of 1650 cm⁻¹ vibration feature in the chemisorbed
layer and its presence in multi-layer. The AES spectrum shows no
contamination from the adsorbed molecule on the surface after the
desorption.

TF-ThP11 Spatial Gratiating Formation in Amorphous Chalcogénide Thin Films,* E. López-Cruz, J. Gonzalez-Hernández, F. Ruiz,**
C. Vázquez-López, and E. Haro-Poniatowski,*** Programa Multi-
disciplinar de Materiales Avanzados, CINVESTAV-Satillo, Coahuila,
Mexico, **On leave from Universidad Autónoma de San Luis Potosí,
***Universidad Autónoma Metropolitana-Iztapalapa, México-D.F.

During the measurements of the phase conjugation via degenerate
diffraction in amorphous Se, laser induced gratings were formed.
We used Scanning Force Microscopy (AFM) to characterize the grats.
It is found that the periodicity of the grating is a film-thickness-
dependent parameter. Several possible mechanisms which may result
in atomic displacement, including bond softening due to electronic
effects, are discussed. It will be reported the dependence of gratating
formation on several parameters such as film thickness and laser power.

* Work partially supported by CONACyT-Mexico.

TF-ThP12 Synchrotron Radiation Photoelectron Emission Micro-
scopy of Natural and Chemical Vapor Deposited Diamond Surfaces,
J. D. Shovlin, M. E. Kordesch, Ohio University, Athens, OH 45701,
D. Dunham, B. P. Toner, Synchrotron Radiation Center and Uni-
versity of Wisconsin-Milwaukee, Milwaukee, WI 53201, and W. Engel,
Fritz Haber Institute, Faradayweg 4-6, Berlin, Germany, D-14195.

Natural and chemical vapor deposited diamond surfaces have been
一幕ized using a photoelectron emission microscope and synchrotron
radiation in the 4-18 eV and 250-350 eV ranges. Both images and
spectra by resolved electron yield spectra could be acquired simulta-
nenously. Near-edge spectra at the carbon 1s edge show a resonance
due to graphite; extended fine-structure due to diamond was also
observed. The image intensity varies above and below the C 1s edge.
Contrast variations between individual crystallites are very sensitive
to the alignment of the diamond surface to the microscope axis.
In the 4-18 eV range, no photoemission threshold was observed in the
electron yield spectra on any of the specimens. The image contrast
was not strongly dependent on the illumination energy. Natural type
IIa diamond showed severe charging effects.

An applied electric field of 20 kV/cm was present during observation.
No spectroscopic or image related evidence for field emission from
the surfaces tested was found.

Work supported by NATO and ONR-BMDO Grant N00014-91-1596.

TF-ThP13 Surface Spectroscopic Studies of the Deposition of Tin
Thin Films From Tetraakis-(dimethylamido)-titanium and Ammonia,
P. J. Chen, C. M. Truong, J. S. Cornell, W. S. Oh and D. W.
Goodman, Department of Chemistry, Texas A&M University, College
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The transmission reaction of [Ti(CH₃)₄N] (TDMAT) in excess
NH₃ has shown to induce carbon-free TiN thin films. However,
due to the rapid reaction in the gas-phase, it remains a challenge to
simultaneously achieve good film conformality and film purity.
The results of this investigation indicate that pure TiN thin films can still
be deposited via a growth mechanism which is completely surface-
mediated. At the low pressure threshold (< 10⁻³ Torr) where the direct
reaction between TDMAT and NH₃ in the gas-phase becomes negli-
gible, and film growth is controlled by the surface reactions between
adsorbed TDMAT and NH₃ species. In this work, X-ray photoelectron
(XPS) and Auger spectroscopic (AES) analyses show that TiN thin films
possessing low carbon-content can be deposited at pressures below
10⁻¹ Torr. In order to detail the basic surface chemistry which is
relevant to the deposition process, the adsorption of nitrogen on
Ti on Ti and TiN, surfaces were also examined using XPS,
infrared reflection absorption (IRAS) and thermal desorption (TDS)
spectroscopy.

TF-ThP14 Phase Stability and Al Solubility in Epitaxial Nb₅Al₃N Films
Grown on MgO (001) by Reactive Sputtering, T. I. Seneller, D. J.
Miller, K. E. Gray, L. Hultman,* and M. R. Sardela, Jr.,
Argonne National Laboratory, MS 0223, 9700 South Cass Ave., Argonne,
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koping, Sweden.

Ternary Nb₅Al₃N films were grown on single-crystal MgO (001)
substrates by reactive triode sputtering in Ar + N₂ gas mixtures.
A phase spread deposition technique was employed to fabricate films
with a composition gradient. For a substrate temperature of 500°C,
x-ray diffraction (XRD) showed that the compound films had cubic
Bi structure for Al fractions in the range from 0 to 0.62. The lattice
parameter decreased with increasing x, from 4.40Å at x = 0 to ap-
proximately 4.27Å for x = 0.62. Moreover, determination of elastic
strain, crystallographic tilt and microstructural quality was performed
by high resolution XRD mapping. The films were found to exhibit
pseudomorphic growth up to at least x = 0.50, for x > 0.56 the films
were two-phase, and finally, pure AlN had the Wurtzite structure.
The growth modes and phase decomposition paths of the metastable Bi
films at x = 0.50-0.62 were assessed by planar and cross sectional
transmission electron microscopy (TEM). The results are discussed in
the light of results from previous work on the related transition-metal
nitride based system Ti₆Al₃N which exhibits similarities in Al solub-
ility and formation regime of the metastable Bi structure.

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76CH00016. L.H. and M.R.S. acknowledge support from the Swedish
National Research Council (NFR).

TF-ThP15 Growth and Structure of Silicon Oxide Thin Films on
Polymers by AFM and XPS, H. Li, S. Krommenhoek and E. Esse,
BEC Group Technical Center, Murray Hill, NJ 07974, and F. Jansen,
Airco Coating Technology, Concord, CA 94524.

The structure and chemistry of silicon oxide films were studied by
Atomic Force Microscopy and X-ray Photoelectron Spectroscopy. Films
were deposited at room temperature on plastics, like, polyethylene
terephthalate), by plasma enhanced chemical vapor de-
position from hexamethyldisiloxane and oxygen gas. At low oxygen
flow rate, the film consists of silicon sub-oxide as well as a significant
amount of carbon. With increasing oxygen flow rate, the film structure
becomes more like silicon oxide. Increasing oxygen flow rate also
leads to a denser film: for example, clusters of the silicon oxides are
more closely packed and more uniformly distributed. The gas barrier
quality of the film is shown to be directly related to the film chemistry
as well as morphology. The influence of the plastic substrate rough-
ness on the growth and structure of the films is also discussed.

TF-ThP16 Characterization of Aluminum Based Oxide Layers
Formed by Microwave Plasma, Z. Katz, Dept. of Mater. Eng., Ben-
Gurion-University, A. Raveh, NRC-Negev, P.O.B. 9001, Beer-Sheva,
Israel.

Intensive efforts have been invested in solid state surface modifi-
cations by various techniques. These, intensify the requirements for
appropriate layer assessments. In this context, microwave plasma ox-
ides has been applied to polycrystalline commercially pure alu-
nium 1100 and 7075-Alloy, at relatively low temperature (400 ±
50°C) under non-equilibrium low pressure (0.5-2.5 mbar) plasma.
Chemical composition and the oxidation profiles were established by
Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy
(XPS) and Fourier Transform Infrared (FTIR) spectroscopy. In the
1100-Al alloy, typical oxide layers thickness of 1 μm in scale were
obtained. Here, the AES/XPS spectra revealed that the layer was
composed from aluminium oxide and aluminium metal. The FTIR
spectra showed sharp and strong absorption bands at 940-960 cm⁻¹ due
to stoichiometric composition with high crystallinity of the γ-Al₂O₃ with
peaks at 1345 cm⁻¹ and 732 cm⁻¹ indicating Al₂O₃ and Al₂O₃-stretch
bands, respectively. In contrast, the 7075-Al under similar oxidation
conditions, Al₂O₃ was absent while sharp and intense peak at 732 cm⁻¹,
as well as broad absorption bands at 870 cm⁻¹ and 1100 cm⁻¹ were
observed. Thus, magnesium segregation and the formation of MgO thin layer in the uppermost oxygenation layer was here manifested. However, the inner layer was composed from Al-O and Al metal due to oxygen penetration into aluminum under ion bombardment. Atomic Force Microscope (AFM) was supplemented for fine scale surface topography features in different phase conditions. The role of micro-alloying affects and plasma dominant parameters are considered in order to provide some rationale to the current findings.

**TF-TP17**

Pyroelectric PbTiO	extsubscript{3} thin films deposited on Si(100) substrates (Pt/Ti/SiO	extsubscript{x}/Si) by sol-gel processing have been characterized in some detail to determine the usefulness of these films for uncooled detector applications. The films have been deposited without substrate heating. The amorphous films were then annealed in the range of 600-750°C in the oxygen atmosphere. X-ray diffraction patterns taken on these films showed single phase tetragonal structure with a slight c-axis preference. The grain size was in the range of 0.2-0.4 micron. The D-E hysteresis loop characteristics at room temperature, as well as measurements of the dielectric and pyroelectric properties versus temperature and frequency, were performed. The influence of poling treatment on the dielectric and pyroelectric properties was also investigated. The results of measurements show that PbTiO	extsubscript{3} thin films exhibit a relatively high pyroelectric coefficient relative to the PbTiO	extsubscript{3} thin film contributing to a very high figure of merit for voltage responsivity that is competitive with PbTiO	extsubscript{3} single crystal and PZT ceramics.

**TF-TP18**
ReSi	extsubscript{2} Thin Film Infrared Detectors, James P. Becker and John E. Makan, Department of Electrical Engineering, Colorado State University, Fort Collins CO 80523; Robert G. Long, Hewlett-Packard Co., Fort Collins, CO 80525.

For the first time, infrared sensing devices have been demonstrated using the narrow bandgap semiconductor, ReSi	extsubscript{2} (E\textsubscript{g} \approx 0.1 eV). They are the ReSi	extsubscript{2}/n-Si heterojunction internal photoemission (HIP) detector, and the ReSi	extsubscript{2} thin film photodetector. Both devices were characterized with monochromatized infrared radiation using phase-sensitive detection. The HIP detector's spectral response was found to obey the power law with a long wavelength cutoff of \( \approx 2 \mu \text{m} \) (0.55 eV) at room temperature. Considering the energy band diagram of the HIP structure, this behavior suggests that the detection mechanism is free carrier absorption leading to internal photoemission of hot electrons from the silicide into the substrate. The HIP detector's cutoff wavelength falls well short of the theoretical limit, \( \approx \lambda_\text{Si} \), for a ReSi	extsubscript{2}-based photonic detector. In hopes of approaching this ultimate limit, the photodetector device was used to explore a second mechanism of detection, the photogeneration of excess free carriers in ReSi	extsubscript{2} as manifested by intrinsic band-to-band photod conductivity. It was found that the spectral response of the ReSi	extsubscript{2} photocathode (normalized to photon flux and measured at 10 K) extends to 6 \mu m (0.21 eV), the present limit of our measurement equipment with no indication of a detection cutoff. In conclusion, these first-ever demonstrations of infrared detection with ReSi	extsubscript{2}, using unoptimized devices, suggest that this narrow bandgap semiconductor may lend itself to practical applications as a silicon-integrated detector element for long wavelength infrared radiation. (This research was supported by the National Science Foundation through Grant No. DMR-9021507.)

**TF-TP19**
Detection of Chlorinated Hydrocarbon Vapors Using 1,3-bis-(1-pyrrene)propane in a Poly(vinyl alcohol) Film, Susan L. Rose-Petersen, John Kroch, Naval Research Laboratory, Washington, DC 20275.

Development of remote sensors for hazardous materials is an important part of the Navy's Environmentally-Sound Ships program. 1,3-bis-(1-pyrrene)propane (bpp) has been incorporated into a thin film of cross-linked poly(vinyl alcohol) (PVA) on a glass slide and used to detect chlorinated hydrocarbon vapors in the vapor phase. Changes in the fluorescence emission from bpp in the film have been followed upon exposure to dichloromethane, chloroform, or carbon tetrachloride vapors. The spectra contains emissions from pyrene monomer (377, 382, 388 and 397 nm) and excimer (480 nm) units. The intensity ratios R1732/R388 and I\textsubscript{excimer}/I\textsubscript{monomer} can be used to examine the polarity and micro-viscosity of the bpp's local environment. In addition, the measuring point is different to distinguish them. Of the polymer matrices that have been investigated, poly(ethylene-vinyl alcohol), poly(ethylene oxide), OV-1 and others, PVA has given the best results. It is felt that the porous network and the polar environment of the hydrogel contribute to the initial ease of excimer formation and the subsequent interactions of bpp with the vapors. The PVA/bpp film is presently being investigated for chlorinated hydrocarbon detection in an aqueous environment.

**TF-TP20**

Despite tremendous environmental interest in monitoring chlorinated organic (R-Cl) solvents such as trichloroethylene (TCE) in contaminated groundwater and water systems, R-Cl sensors do not exist until now an inexpensive, portable R-Cl detector. We have synthesized a sodium-based silicate compound whose conductance at elevated temperatures is sensitive to ppm levels of R-Cl vapors, but not to elemental chlorine or hydrocarbons. Gas sensing mechanisms were investigated by studying the material's conductance during gas exposures while heated inside a tube furnace. Temperature studies indicate an activation energy of about 0.2 eV for R-Cl vapor sensing. Current-voltage scans follow non-ohmic behavior with significant hysteresis. Both capacitive and "inductive" components were measured by a.c. impedance spectroscopy, suggesting the material is a mixed conductor. X-ray diffraction patterns of gas tested material demonstrate R-Cl exposure transforms the bulk structure into a pyrosilicate, while electron micrographs clearly show NaCl crystal growth on the outer surface. Chemical analysis of the exhaust gas from the tube furnace shows no Cl\textsubscript{2} molecules, indicating the gas-sensing reaction involves chlorine atoms or radicals. We discuss the electronic and chemical nature of this novel material and its potential as an R-Cl sensor.

**TF-TP21**
Surface Characterization of Ultra-Thin Metallic Sensing Films, H. Liu, J. L. Gland, J. W. Schwanke\textsuperscript{*}, K. Wise, Department of Chemistry, Department of Chemical Engineering\textsuperscript{*}, The University of Michigan, Ann Arbor, Michigan 48109.

Various pretreatment protocols for ultra-thin Pt/Ti and Pt/AI films used for oxygen sensing have been investigated. The pretreatments are meant to obtain a stabilized film structure that is capable of interacting with certain types of gases. The sensing films are evaporated onto a silica substrate. They are studied as a 1 cm\textsuperscript{2} macroscopic model of the sensing element in an ultra-thin film conductivity-type gas sensor.

Adsorption and desorption of gases such as oxygen and hydrogen from such thin films at different temperatures have been studied using TPD. Results from characterization by ex situ ESCA and STM will also be presented. These results give us a better understanding of the chemical compositions and structures of the films. Results from these studies will also be combined with the electrical responses from the gas sensors to determine the influences of different pretreatment protocols on the sensing performance of such gas sensors and find effective windows for real time gas detection.

**TF-TP22**

The copper on oxide interface is of general interest in many different domains including catalysis and microelectronics. It is also relevant as a model for the copper/superconductors oxides interfaces formations. We investigate here the formation of the interface between copper on two different surface preparation of SrTiO\textsubscript{3} namely a reduced form prepared by high temperature heating under vacuum (1000°C) and an stoichiometric form obtained after re-oxidation of the reduced form.

Using simple growth model to calculate the intensity evolution of the XPS of Sr, Ti, O atoms in core level lines, we were able to show that copper grows on SrTiO\textsubscript{3} with the simultaneous formation of several monolayers followed by the formation of island on the surface. It was also shown that the growth mode is similar for both forms of SrTiO\textsubscript{3} but that the number of monolayers formed is higher for the reduced form, which is explained by the higher number of defects on the surface. UPS and Auger parameter studies of the interface formation showed a strong interaction between copper and the substrate, bringing copper in a CuO-like electronic configuration.

HREELS studies confirmed the growth model developed for the XPS intensities. In addition, we were able to show the importance of
the impact interaction mode in the HREELS spectra obtained for the metal/oxide interface.

TF-ThP23 AIN Thin Film Structure Development and Quality During Magnetron Sputter Deposition on Si(100) and Si(111), Studied by XRD, AES, and FE SEM. Barbara Ladwa, Marc Chason, Steve Voight, Motorola, ILO/Rm 1014, 1301 E. Algonquin, Schaumburg, IL 60196.

AlN is a material of great interest for its properties as a piezoelectric, a wide band-gap semiconductor, and a protective thin film. The development of the AlN thin film structure, during sputter deposition on the Si substrate, was investigated to understand the structure-properties relationship. XRD was used to determine the film crystallinity and orientation. AES was used to analyze the film surface composition and the elemental concentration changes within the film. SEM was used to analyze the film grain structure, thickness and uniformity. It was found that AlN films as thin as 200 A can have oriented, columnar grain structure. The grains grow in diameter as well as in height during the film deposition while the film density increases. The AlN/Si interface was found to be clean and well defined and it didn’t change with the film growth. The AlN (0002) XRD peak intensity, representative of the degree of the oriented grain structure, increased with the film thickness and the grain structure quality.


Atomic force and scanning tunneling microscopes have been used to characterize the microstructure of metal films grown on wide-band semiconducting substrates at 300K and at a cryogenic temperature. Previous work has shown a significant increase in the barrier heights of resulting Schottky Diodes as the deposition temperature was decreased. Because of the importance of the structure-property relationship, structural differences were expected to shed some light on the factors responsible for this increase in barrier height.

Pd and Ag were thermally evaporated onto InP and InGaAs, respectively, at RT and 77K. The films were grown to thicknesses of 14 and 85 nm. Grain size surface roughness were measured with the scanning probes. Not surprisingly, thicker films have larger average grain size and RMS roughness. The low temperature Pd films were found to possess significant cracking while both all Ag films were characterized by varying densities of holes.
Friday Morning, October 28, 1994

SURFACE SCIENCE
Room A205 – Session SS1-FrM

Surface Electronic Structure
Moderator: R. Hwang, Sandia National Laboratories.

8:20 am SS1-FrM1 Theory of Clean and H-Covered Be(0001), Roland Stumpf and Peter J. Feibelman, Sandia National Labs, Albuquerque, NM 87185-0344.

Be is a "marginal metal." The stable phase, hcp-Be, has a low Fermi-level density of states and very anisotropic structural and elastic properties, similar to a semiconductor's. At the Be(0001) surface, surface states drastically increase the Fermi-level density of states. The different nature of bonding in bulk-Be and at the Be(0001) surface explains the large outward relaxation (ca. 3% according to our LDA total energy calculations) in the presence of a large tensile stress (0.2 eV/Å).

We have calculated the energy of H/Be(0001) at different coverages and various geometries. For coverages <1/2 ML, H atoms sit in 3-fold hcp sites which are separated by a barrier of ~0.2 eV. The low T phase will therefore be disordered at 300 K, which explains the experimentally observed 1 x 1 surface state dispersion. At higher coverages the dilute phase will consist of two condensed phases of novel adsorption geometry: H-atoms sit on tilted bridge sites, next to Be vacancies. At 2/3 ML, H adopts a 1 x 3 structure, in which every third row of Be surface atoms is missing. At 1 ML the stable structure has a √3 x √3R30° unit cell with the Be surface atoms forming a honeycomb lattice. In both H-induced vacancy structures the H atoms are particularly close to the surface so that the H-H repulsion is especially low. Additionally the Fermi-level density of states at the surface is efficiently quenched, i.e., the near-covalent bonding of bulk Be is recovered for the H-covered surface. We discuss the energetics of the H-induced vacancy structures in terms of the vacancy formation energy of the clean surface.

Work supported in part by US-DOE under contract DE-AC04-94AL85090.

8:40 am SS1-FrM2 Core-Level Spectroscopy of Metal Monolayers and Interfaces, N. D. Shinn, B. Kim, K. J. Kim, T.-H. Kang, and J. L. Erskine, (1) Sandia National Labs., Albuquerque, NM 87185-0344; (2) Pohang Univ. of Science and Technol., Pohang 790-784, Korea; (3) Dept. of Physics, Kon-Kuk Univ., Seoul 133-701, Korea; (4) Dept. of Physics, Univ. of Texas, Austin, TX 78712.

The electronic, chemical and mechanical properties of ultra-thin-film and metal-multilayer structures are largely determined by the electronic structure at the heterointerface. For epitaxial interfaces, substrate atoms in the bulk and at the interface have geometrically equivalent coordination spheres; hence the interface-atom core-level shifts are a measure of only the local bonding between the dissimilar metals, mediated by final state screening effects of the photoemission process itself. Using monochromatized soft X-ray synchrotron radiation, we have obtained high-resolution W(4f)/2 photoemission spectra (AE = 100 meV) from M(W(110)) interfaces, where M = Fe, Ni, Pd, Pt, Cu, Ag, and Au. By rigorous Doniach-Sunjic lineshape deconvolution methods, we can determine and compare the W(4f)/2 binding energy shifts for the single layer of interface tungsten atoms at these structural interfaces. Systematic W(4f)/2 core-level shifts are found that, in many cases, follow expectations based upon bulk electronegativities, i.e., there is no anomalous "charge transfer" between metals as proposed by others [1]. However, we show that for other bi-metallic interfaces this correlation with electronegativities breaks down, thereby invalidating a simple "charge-transfer" interpretation of core-level shifts at bi-metallic interfaces.


Work performed at the NSLS and supported by DOE-BES under contract DE-AC04-94AL85000 with Sandia, the NSF under contract DMR93-03091 with U. Texas, and by the user program of the Pohang Light Source.
Gurney-model. The increased covalency is characterized by a shift of the 4s resonance towards E\textsubscript{F}, a distortion of the 4p orbitals, and a steady increase in the 4s transfer rate. The K\textsubscript{4}s-substrate bonding is, however, still considered to be primarily ionic for all the studied systems, since the main part of the 4s resonance is situated above E\textsubscript{F}.

10:00 am SS1-FrM6 Photoemission Study of Alkali Metal Adsorption on the Passivated Si(111)×1-As Surface, M. C. Häkansson, M. Johansson, and L. S. O. Johansson, Department of Synchrotron Radiation Research, Institute of Physics, University of Lund, Sölvegatan 14, S-223 62 Lund, Sweden.

We have studied potassium adsorption on the Si(111)×1-As surface with angle-resolved photoemission and core-level spectroscopy. The As-terminated Si(111) surface is well known for its ideal bulklike termination of the Si lattice, as well as for its passivated nature. The electronic structure of the surface is dominated by the filled lone-pair state near the valence band edge and an empty state close to the conduction band edge, with no electronic states in the fundamental bandgap.

Deposition of small amounts of K on the surface led to an abrupt shift in the Fermi level position and to the occupation of the minimum of the empty state. This state showed up as a sharp peak at the Fermi level near the surface Brillouin zone boundary at the M point, as expected from quasiparticle band structure calculations. This allowed us to determine the surface band gap to 1.7 eV, in good agreement with calculations.

In the core-level spectra from the surface after K deposition, large K-induced shifts were observed. In the As 3d spectra a new component at 1.1 eV lower binding energy emerged, whereas in the Si 2p spectra a K-induced peak was found at higher binding energy. This indicates significant charge rearrangements in the surface layer as a result of the K adsorption.

These results are very different from previous core-level studies of alkali adsorption on clean Si surfaces, where generally only small shifts in the Si 2p level have been seen.


One of the most intriguing aspects of early transition-metal carbides is that they often show electronic and catalytic properties similar to those of platinum-group metals. In an attempt to understand the fundamental correlation between the electronic and catalytic properties of this important class of materials, we have chosen vanadium carbide films on a vanadium (110) surface as model systems for our experimental and theoretical study. By using HREELS and NEXAFS, we determine that the electronic properties of vanadium, especially the characteristics of the d-orbitals, are essentially modified by the formation of carbides. We detect a charge transfer from vanadium to carbon, with approximately 1.3 electrons per vanadium, as a result of C\textsubscript{4}V bond formation. Such an electronic modification is in turn responsible for the catalytic properties. For example, by using XPS, we find that monoxide and ethylene as probing molecules, we observe a strong similarity between the surface reactivities of carbide-modified V(110) and platinum-group metals. These results can be explained by a narrowing of the d-orbitals in vanadium carbide and therefore a less d*-type interaction. More interestingly, we observe that the formation of carbide has opposite effects on the selective activation of C\textsubscript{4}C and C\textsubscript{4}H bonds, based on a comparative study of reactivities of several saturated and unsaturated C\textsubscript{4}M molecules with clean and carbide-modified V(110) surfaces. Our results indicate that, although the formation of carbide reduces the decomposition probabilities of C\textsubscript{4}C bonds, it selectively enhances the activation of C\textsubscript{4}H bonds. These observations will be explained by comparing the different roles of p- and \sigma-type orbitals in the decomposition of C\textsubscript{4}H and C\textsubscript{4}C bonds on transition metal surfaces. These results represent the first experimental study of hydrocarbon chemistry on clean and carbide-modified vanadium surfaces.

10:40 am SS1-FrM8 Electronic Transitions and Excitations in Solid C\textsubscript{60} Studied by HREELS and XPS C1s Shakeup Structures, B. Y. Han, L. M. Yu, K. Hevesi, G. Gensterblum, P. Rudolf, J. J. Pireaux, P. A. Thiers, R. Caudano, Laboratoire Interdisciplinaire de Spectrométrie de Spectroscopie Électronique pour la Recherche des Matériaux et des Surfaces, Facultés Universitaires Notre-Dame de La Paix, B-5000 Namur, Belgium.

The electronic transition and excitation properties of highly ordered C\textsubscript{60} films grown on lamellar substrates MoO\textsubscript{3}(001) and GeS\textsubscript{3}(001) have been studied by means of High Resolution Electron Energy Loss Spectroscopy and XPS C1s shakeup structures. From the HREELS study, a total of eleven features were observed in the energy loss range 1-30 eV. These include the lowest electronic excitation ever observed in C\textsubscript{60} fullerene, a peak at 1.53 eV loss energy, assigned to a Frenkel-type molecular excitation; the double peak at 5.1 eV and 5.9 eV corresponding to the two split levels of the π*-plasmon of C\textsubscript{60} induced by its lower symmetry compared to C\textsubscript{60}. A broad hump at ~28 eV corresponds to the ω-plasmon. In XPS (hv = 1486.6 eV, instrumental resolution 0.3 eV) C1s shakeup structures, several features (out of a total of seven within 30 eV from main C1s line) find their counterparts in the HREELS spectra. A prominent peak at ~2.5 eV, present in both spectra, appears to reveal the fundamental gap (exempt of the off-site correlation effect) in C\textsubscript{60} fullerene. Two prominent peaks at ~5.4 eV and ~27 eV are also present. A few weak features were resolved for the first time, thanks to the high energy resolution of our SCIENTA ESCA-300 instrument.

11:00 am SS1-FrM9 Inverse Photoemission Study of CO Chemisorption on Metallic Quantum Wells, F. G. Curti, R. A. Bartynski, Rutgers University, Department of Physics and Astronomy and Laboratory for Surface Modifications, Rutgers, The State University of New Jersey, Piscataway, NJ 08855.

We have used isochromat inverse photoemission spectroscopy to study the chemisorption of CO on Cu/fccCO/Cu(100) and fccCo/Co(100) layered structures. Under optimal growth conditions, these systems exhibit metallic quantum well (MQW) states in the region of the Fermi level. Upon chemisorption of CO at 140 K a c(2x2) LEED pattern forms on Cu/fccCO/Cu(100). The two peaks in the first MQW state take place on adsorption of 1 ML of CO and it is no longer observed in inverse photoemission. A shift to lower energies of the CO derived peaks also occurs. The chemisorption behavior of the Cu overlayers, a weak chemisorption system, will be contrasted with that of the more strongly interacting Co overlayers. The change in the density of states in the region of the Fermi level will be related to changes in both the electronic interaction and thermal desorption properties of the CO/metallic quantum well systems.

11:20 am SS1-FrM10 A Core Level Spectroscopy Study of Different Adsorption States of Ethylene on Pt(111), R. Hernaiz, A. Nilsson, O. Karis, C. Puglia, A. Sandell and N. Mörrensson, Dept. of Physics, Uppsala University, Box 530, S-751 21 Uppsala, Sweden.

We have studied the C1s/Pt(111) system by means of core level spectroscopies, such as x-ray photoelectron spectroscopy (XPS), x-ray absorption spectroscopy (XAS) and deexcitation electron spectroscopy (DES also denoted resonant Auger). Three different temperature phases were studied; monolayer at 20K, monolayer at 90K and the high temperature phase at 300K where a 2x2 ethylene phase forms. In the 90K state, XPS intensities of C1s and Pt 4f surface core level shifted components a bridge adsorption site can be derived i.e. as denoted di-σ configuration. This is consistent with a strong modification of the π intensity in XAS. The 300K monolayer has been suggested to contain only π-bonded molecules, has shown to be a mixture of two different adsorbed ethylene species. One with the earlier proposed σ bonding configuration, and the second, which is weakly adsorbed with a standing up configuration. The latter one clearly shows that an intact π*-orbital, i.e. this orbital does not participate in the bond to the surface. It is a higher lying orbital that hybridizes with the surface and thereby creates the chemisorption bond. Also the ethylene species that is created on this surface has been studied, and we present a new way of interpreting the XAS spectra for this species. Furthermore, coverage measurements using the C1s and Pt 4f core level lines suggest domains of 2x2 structure with a coverage of 0.41±0.05.

11:40 am SS1-FrM11 The Auger Relaxation of C2Cl\textsubscript{2} Following Electron Capture Decay of Cl\textsuperscript{37}Ar, L. Zhu, R. Avci, G. J. Lapuyere, Montana State University, M. M. Hindi, R. L. Kozub, S. J. Robinson, Tennessee Technological University.

We developed an experimental system to study the Auger relaxation process following the 37\textsuperscript{Ar}+Cl\textsuperscript{−} electron capture (EC) decay. A mixture of 5 x 10\textsuperscript{-5} parts 37\textsuperscript{Ar} and one part 35\textsuperscript{Ar} is physisorbed on a flat cold substrate kept at 16K under UHV conditions. Using time of flight and coincidence technique we, for the first time, measured the energy dependence of the probability distribution for one of the two electrons emitted as a result of double Auger process. The two electrons share the total energy with one of the electrons prefers to have the most of the energy, which agrees with the theoretical prediction.

a cylindrical mirror analyzer (CMA), we also measured the kinetic energy distribution of the regular LMM Auger electrons following the EC decay. We observed a sharp Auger peak at \( \sim 193 \) eV which does not appear in any known Ar or Cl Auger data. We believe that this extra feature is due to the particular initial state configuration with one hole in the K-shell produced by the EC decay process.


**SURFACE SCIENCE**

Room A201 – Session SS2-FrM

**Group IV Semiconductor Surface Structure**

**Moderator:** R. Hamers, University of Wisconsin.


We introduce the use of multivariate image classification techniques in determining surface electronic structure from multiple-bias scanning tunneling microscope (STM) images. Multiple measurements at each site are used to distinguish and categorize inequivalent electronic or atomic species on the surface via a computerized classification algorithm. Then, comparison with theory or other suitably chosen experimental data enables the identification of each class. We demonstrate the technique by analyzing dual-polarity constant-current topographs of the Ge(111) surface. Utilizing no prior knowledge of the atomic structure of the surface, we find that the two measurements, negative- and positive-bias topography height, enable pixels to be separated into seven different classes. In order to assess the validity of our classifications and determine the identity of each class, we compare the results with the atomic structure of the \( c(2\times8) \) reconstruction of Ge(111). If we label four of the classes as adatoms, first-layer atoms, and two inequivalent rest-atom sites (R1 and R2), we find that our results are in very good agreement with the \( c(2\times8) \) structure. The remaining classes are associated with structural defects and contaminants. This work represents the first step towards developing a general electronic/chemical classification and identification tool for multispectral scanning probe microscopy imagery.

This work was supported by the U.S. Department of Energy under contract DE-AC04-94AL85000.

8:40 am SS2-FrM2 High Resolution TEM Determination of the \((5\times2)\) Au/Si(111) Structure, R. Plass and L. D. Marks, Dept. of Mat. Sci. and Eng., Northwestern Univ., 2225 North Campus Drive, Evanston, IL 60208.

Among the numerous unsolved metal semiconductor interfaces the submonolayer gold on silicon (111) system is of interest as it displays a linear \( 5 \times 2 \) surface structure between .1 and .5 monolayers of metal coverage. Recent x-ray diffraction and high resolution STM studies have yielded new information about this structure, yet the placement of the gold atoms remains unclear [1, 2]. We present here a solution for this structure found using off-axis plan view high resolution transmission electron microscopy and digital image restoration.

Si(111) TEM samples were prepared by ion sputter cleaning and electron beam annealing cycles in ultrahigh vacuum conditions to obtain fairly flat \( 7 \times 7 \) reconstructed surfaces. Approximately .4 ml of gold was evaporated onto the top surface of the sample followed by annealing to produce the \( 5 \times 2 \) structure. A ten member 250K off zone high resolution through focal series was taken, digitized, and analyzed using Wien and Schiske filters and a Chi-squared form of the Schiske filter to obtain the atomic potential map.

The proper analysis of the HREM data requires the \( 1 \times 1 \) and bulk spacings, omitted in the x-ray diffraction study, be included and gives a simple result: two gold atoms have a spacing near-coincident with the silicon \( 1 \times 1 \) spacing. This interpretation is supported by diffraction patterns which show that in near kinematical diffraction conditions the \( 1 \times 1 \) and bulk diffraction spots are always the strongest set of spots in the pattern. Further details of atomic structure will be presented.


9:00 am SS2-FrM3 FT-IRAS of Adsorbed Alkioxides: Methoxide and Ethoxide on Cu(111), Shane C. Street, University of Illinois Urbana-Champaign 61801, and Andrew J. Gelman, Carnegie Mellon University, Pittsburgh, PA 15213.

The surface chemistry and structure of methoxide and ethoxide adsorbed on Cu(111) has been studied by FT-IRAS and TPD. The structure of methoxide on Cu(111) has been a matter of debate. Previous IRAS results were interpreted to show that the \( C_{\text{o}} \) axis of methoxy at saturation coverage lies perpendicular to the surface. Subsequent X-ray diffraction and backscattering photoelectron diffraction experiments support this orientation. However recent work with methoxy cluster compounds has led to the proposal that at low coverages methoxy is tilted, and that steric crowding reorients the molecular axis towards the surface normal at saturation. We present high resolution spectra for methoxy at a range of coverages. For the more complex ethoxy adsorbate we present the FT-IRAS spectra for a set of seven deuterated and isotopically \((^{13}\text{C})\) labelled ethoxides. This is by far the most detailed labelling study to assign vibrational modes of an adsorbate. The \( C_{\text{o}} \) axis of the methyl group of ethoxy is found to lie roughly parallel with the surface.

9:20 am SS2-FrM4 Boron Induced Structures on the Si(111) Surface, C. Wang, T. C. Shen, J. W. Lyding and J. R. Tucker, University of Illinois at Urbana-Champaign, Urbana, IL 61801.

It has been known that B-saturated Si(111) surface will be \( \sqrt{3} \times \sqrt{3} \) R 30° reconstructed. It is not clear, however, how the transition from the B-free \( 7 \times 7 \) surface to the B-rich \( \sqrt{3} \) surface occurs. We have used STM to study the boron-doped Si(111) surface as a function of annealing times and temperatures. The surface structure is found to be determined by the concentration of B. When the substitutional B concentration is less than 1% of the top 1 x 1 bilayer atoms, the surface is largely \( 7 \times 7 \) but surrounded by adatom-covered 1 x 1 regions (which have higher B concentration). When the B concentration is more than 3%, the whole surface will be adatom-covered 1 x 1 including \( \sqrt{3} \) structures. The \( \sqrt{3} \) domains will increase with the B concentration. The effect of the surface B on \( 7 \times 7 \) growth is hence deduced. Rows of the \( 7 \times 7 \) domain preferentially grow along the 3 equivalent [112] directions. The adatom-covered 1 x 1 regions are bounded by faulted halves of the \( 7 \times 7 \) domains. The domain size of \( 7 \times 7 \) or \( 9 \times 9 \) can be as small as a faulted half of a unit cell. The energy relations and the transition temperatures between the \( 2 \times 2 \), \( c(4 \times 2) \), \( 7 \times 7 \) and \( 9 \times 9 \) reconstructions are discussed. The dark sites of \( 7 \times 7 \) are observed and counted. They are further interpreted in terms of a B substitution model. The pattern of bright and dark atoms in \( \sqrt{3} \) domains is analyzed and a criterion for a B stabilized Si-\( \sqrt{3} \) structure is obtained.

Supported by the Office of Naval Research URI:N00014-92-J-1519.

9:40 am SS2-FrM5 Surface-Induced Optical Anisotropies of Single-Domain (2×1)-Reconstructed (001) Si and Ge Surfaces, L. Mantese, T. Yasuda, and D. E. Aspnes, North Carolina State University, Raleigh.

Above-bandgap surface-induced optical anisotropy (SIOA) is becoming increasingly important in elucidating the electronic structure of non-ohmic surfaces and interfaces, yet the accurate theoretical calculation of SIOA spectra remains a difficult challenge. To provide an opportunity for critically testing theoretical approaches, we have performed SIOA measurements on clean and oxygen-exposed single-domain (2×1) reconstructions of (001) Si and Ge from 1.5 to 5.5 eV using Reflectance-Difference Spectroscopy (RDS). Single-domain surfaces are obtained by heating chemically cleaned vicinal wafers cut 4° off [001] toward [110] in u,v, and were assessed by in situ Auger and LEED. The clean (2×1) Ge surface exhibits two broad peaks at 1.8 and 3.1 eV, while the Si surface shows a relatively sharp peak at 3.4 eV and broader features at higher energy. By exposing the surface to oxygen and analyzing the resulting LEED patterns, we separate the individual SIOA contributions from dimers and steps. Only dimers contribute to the Ge spectra, whereas both dimers and steps contribute to Si. The (2×1) Si spectrum disagrees with published calculations.
not only in lineshape but also by nearly an order of magnitude in amplitude, suggesting the importance of these results in critically testing theoretical models.

*Supported by ONR Contract N-00014-93-1-0255.

10:00 am **S22-FrM6** High-Temperature Structural Phases of the Si(111) Surface Studied with High-Resolution Energy-Resolved HeLIum Atom Scattering, C. A. Meli, Naval Research Laboratory, Washington, DC 20375 (USA); G. Lange and J. P. Toennies, Max-Planck-Institut für Strömungsforschung, 37073 Göttingen (Germany).

The structure of the Si(111) surface in the temperature range 900-1550 K has been investigated using high-resolution thermal-energy helium atom scattering with energy resolution. The well-known 7 x 7 to 1 x 1 transition near 1140 K was confirmed by the loss of all fractional-order diffraction peaks, as well as sharp changes in the intensity of all integral-order diffraction peaks. An additional order-disorder transition occurring near 1410 K was unequivocally confirmed by the loss of all all integral-order diffraction peaks above this temperature. Measurements of the thermal attenuation of diffraction peaks, hysteresis of peaks through the phase transition region, relative disorder, and inelastic scattering over the entire high-temperature range were performed. These results will be discussed in light of possible models of the Si(111) structure above 900 K, and in relation to the high-temperature behavior of related surfaces.

10:20 am **S22-FrM7** Two-Dimensional Surface Interactions and Dynamics of Benzene on Cu(111), M. M. Kamana, S. J. Stranick, and P. S. Weiss, Department of Chemistry, The Pennsylvania State University, University Park, PA 16802.

We seek to understand the interactions that determine the chemistry, structure, and dynamics of surface adsorbates. We have used ultrahigh vacuum scanning tunneling microscopy to study the low temperature behavior of benzene adsorbed on Cu(111). Electron standing waves are apparent in STM images of Cu(111) at low temperature due to scattering of surface states from step edges, defects, and benzene molecules. The resulting charge density modulations influence the binding of the benzene molecules. We find that at low coverage, adsorbed benzene binds along Cu step edges and at other high charge density sites. The molecules form a tightly bound 2-D solid at the step edges while benzene molecules on terraces move across the surface as a 2-D molecular gas. At the interface between the solid and gas we observe 2-D adsorption and desorption in real time. We discuss the relative strengths of interactions at various step and terrace sites due to these nanoscale step variations in the surface electronic structure.

This work was supported by BRDC, NSF, and ONR.

10:40 am **S22-FrM8** Strain-induced Surface Morphology on Si(111)-(7 x 7), Y. Wei, L. Li and I. S. T. Tsong, Arizona State University, Tempe, AZ 85287.

A novel strain stage was designed for a scanning tunneling microscope (STM) to apply an isotropic tensile stress on a Si(111) surface at elevated temperatures. After straining, the surface was allowed to cool to room temperature and STM images of the strained surface were taken. Glide steps along (110) directions with single atomic layer height of 3.1 Å were observed. These steps could be easily distinguished from the normal wandering single-steps on the (111) surface by their linearity over large distances and their alignment with the (110) directions. At some of the intersections between a glide step and a normal step, or between two glide steps, "kissing" sites were formed. The glide steps were determined to be caused by slips of (111) planes intersecting the (111) surface. Despite the application of ~1% isotropic strain, the anticipated (5 x 5)-(7 x 7) phase transition was not observed on the Si(111) surface because the strain was taken up by the slippage of (111) planes occurring in the bulk.

11:00 am **S22-FrM9** A Novel Face Reconstruction of Si(112) Revealed by STM, A. A. Baski and L. J. Whitman, Naval Research Laboratory, Code 6177 Washington, DC 20375.

Si(112) has attracted attention as a potential substrate for electronic devices due to its large vicinity. It is a (111) surface "missing-out" by (1, 1, 0). The idealized projected surface would consist of short (111) terraces separated by monoatomic-height steps running along the (110) direction (unit cell = 9.4 Å [111] x 3.8 Å [110]). In contrast, our STM images reveal that the surface has a sawtooth-like morphology, with upward-sloping, 50-100 Å-wide terraces oriented 4-5° steeper than the (112) plane and downward-sloping, relatively short regions. The dominant, upward terraces consist of a periodic arrangement of bright rows oriented along [110] with an inter-row spacing of ~16 Å along [111]. To maintain the net orientation, the upward terraces are broken up by a variety of downward-sloping structures, including short reconstructed (111) terraces, disordered multiple step bunches and other types of terrace reconstruction. A quantitative analysis of the STM images indicates that the upward faceted terraces are (337) planes; a plane tilted 23.5° away from the (111) surface with a [111] unit cell length of 15.7 Å. We have also investigated the atomic-scale structure of this surface by acquiring high-resolution images. Atomic corrugations occur along the bright rows with a 1.0 Å (0.84 Å) periodicity in the [110] direction. Other surface features include dim rows located adjacent to the dominant bright rows and bright protrusions (~10 Å dia.) located between the periodic row structures. Models of this surface incorporating both the observed atomic-scale structures and the facet reconstruction will be discussed.

11:20 am **S22-FrM10** Structural Determination of the Dimerized Si(100) Surface, Y. Ojan, M. J. Bandzuk, G. E. Franklin, S. Tang and A. J. Freeman, Argonne National Lab and Northwestern University, Argonne, IL 60439.

The atomic structure of Ga chemisorbed on the Si(100) 2 x 1 surface at low coverage has been studied by a combination of X-ray scattering wave (XSW) measurements and molecular cluster total energy calculation. For various Ga coverages below 1/2 ML, the Ga ad-dimer is found to be 1.03 ± 0.01 Å above the Si(400) diffraction plane. The measured Ga ad-dimer bond length is 2.50 ± 0.06 Å, which matches the prevalent bond length of gallium. By comparing experimental results with the theoretical calculation, we conclude that the Ga ad-dimers are oriented parallel to the underlying Si dimers, which is consistent with a previous total-energy calculation. This parallel dimer geometry is in contrast to the orthogonal dimer model proposed by previous LEED, AES, and STM studies. Our measurement is the first experimental confirmation of this parallel Ga ad-dimer model.

This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences, under contract W-31-109-ENG-38.

11:40 am **S22-FrM11** Fluctuations of Monatomic Steps on Si(001), H. J. W. Zandvliet and B. Poelsema, Faculty of Applied Physics, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands; H. B. Elswijk, Philips Research Laboratories, P.O. Box 80.000, 5600 JA Eindhoven, The Netherlands.

The motion of monatomic steps on Si(001) is studied on an atomic scale at elevated temperatures with scanning tunneling microscopy. The kinks in the step edges move in units of 2 dimers alongside the monatomic A-type step edge and perpendicularly to the monatomic B-type step edges. The overall time dependencies of the equilibrium step fluctuations of A- and B-type step edges were found to be both proportional to the square root of time but with different prefactors for the different steps. The fluctuations of long kinks in the B-type step edge are however much larger and exhibit initially a more t-like dependence, i.e. one-dimensional random walk behavior. Both time dependencies can be understood in terms of the Langen equation.

**NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY**

**Room A209 – Session NS1–FrM**

**Novel Materials and Methods for Nanofabrication**

**Moderator:** Y. Kuk, Seoul National University, Korea.

8:20 am **NS1-FrM1** Chemistry and Applications of Self-Assembled Films, Jeffrey M. Cate, Center for Bio/Molecular Science & Engineering (Code 6900), Naval Research Lab, Washington, DC 20375-5348.

Precise positional control of surface chemical functionalities and physical properties such as wetting can be obtained by direct patterning of organosilane self-assembled (SA) monolayer films with lithographic
exposure tools. A variety of transformations have been investigated for film irradiated with deep UV photons, x-rays, ions, and low energy electrons. Patterned SA film surfaces have been used as a tool for controllable selectivity by using reactivity for selective deposition, attachment, and orientation of a wide variety of materials to surfaces including catalysts, electroless and CVD metal films, proteins, cells, nucleic acids, and liquid crystals. Selectively metallized, patterned SA films have been produced with linewidths to 20 nm using STM lithography, and high resolution metal patterns have been used to fabricate electron test structures. The utility of patterned SA films for microelectronics, sensors, displays, and other applications will be discussed.

INVITED

9:00 am  NSl-FrM3 Preparation and Characterization of Highly Organized Self-Assembled Monolayers as Substrates for Nanoelectronics, C. W. Sheen, A. N. Purik and D. L. Allara, Department of Materials Science and Engineering, University Park, PA 16802; M. J. Lercel and H. G. Craighead, School of Applied and Engineering Physics, Cornell University, Ithaca, NY 14853.

Self-assembled monolayers have been prepared on a variety of substrates for the purpose of ultrahigh resolution electron-beam lithography and etching of features in the substrate. The major emphasis has been on maximizing the density of the film in order to maximize the etch resistance. Film stability has been designed by utilizing both direct chemical bonding to the substrate and film crosslinking. For GaAs(100), film formation was accomplished by reactions with octadecanethiol. X-ray photoemission data show that an oxide-free, S-GaAs interface is formed. For TiO2 and SiO2 films, films were formed using octadecyltrichlorosilane and the highest degree of organization was obtained by the film from the substrates by ultrathin water films. In these cases the high stability is due to intermolecular crosslinking. Characterization of the molecular structure using infrared spectroscopy and ellipsometry show that the molecules are highly oriented and densely packed and thus quite suitable as etch resists.

This work was sponsored by the Advanced Research Projects Agency ULTRA program.

9:20 am  NSl-FrM4 Nanometer Resolution Near-field Optical Lithography, M. Rudman, A. Shekemelitov, K. Lieberman and A. Lewis, Hebrew University of Jerusalem, Department of Applied Physics, Givat Ram Campus, 91904 Jerusalem, Israel.

In this work we develop and present results on a new method of optical lithography which can write structures with a resolution that is normally associated with electron beam techniques. This method is based on near-field optics, which involves illuminating a substrate with a subwavelength aperture that is maintained above the surface at a height of a few hundred Angstroms. The spot of light thus created corresponds to the dimension of the aperture rather than the wavelength of the light. In this paper we combine the methods of near-field optics with the capability of an argon fluoride (ArF) excimer laser to ablate materials without heating. Specifically, our method involves passing the deep ultraviolet wavelength of the 193 nm ArF laser through a hollow glass micropipette that can be readily drawn with heat to an aperture at the tip as small as a few nanometers. We describe an instrument which incorporates lateral atomic force feedback to track such a micropipette over an arbitrary surface with nanometer accuracy. We present results of patterns that have been produced on photoresists and other substrates with linewidths that are as small as 50-70 nm. By investigating the dimensionality of the structures drawn on the photoresist and on the substrate on which the photoresist was deposited, it appears that nonlinear characteristics of the ablation process may allow the near-field dimensionalities to be maintained at distances that are relatively remote from the tip of the pipette aperture.

9:40 am  NSl-FrM5 Fabrication of 10nm Holes on a 20nm Hexagonal Lattice in Si(100), T. A. Winningham,* T. T. Moore,* S. D. Williams,* K. Douglas,* D. Choutov,* J. D. Piper,* K. P. Martin,* and H. P. Gillis,* *Department of Physics, University of Colorado, Boulder, CO 80309-0390, School of Chemistry and Microelectronics Research Center, Georgia Institute of Technology, Atlanta, GA 30332-0400.

Using a biological template and Low Energy Electron Enhanced Etching (LEEED), we have fabricated a nanometer-scale hexagonal array of holes in Si(100). The holes are 10 nm in diameter with a lattice constant of 20 nm, and have been etched to depths approaching 10 nm.

The biological template (3-layer) is fixed on the Si substrate with an organosilane adhesion promotor and metal-shadowed with 12 A of Ti, which oxidizes to 35 A of TiO2. Ion milling transfers the pattern of the template into the TiO2 overlayer, exposing the Si only at the holes of the template. The TiO2 pattern is then transferred into the Si substrate by LEEED in an H2/He DC plasma, which inflicts minimal damage on the substrate. In previous attempts at fabricating, ion milling and reactive ion etching (RIE) have either destroyed the ultrattract mask or shown low selectivity between mask and substrate. LEEED leaves the mask intact while transferring the pattern to the substrate.

To evaluate the extent of etching, the samples have been examined with atomic force microscopy (AFM) before and after LEEED. The etch rate, anisotropy, and quality of the Si surface will be described as functions of current density and gas composition. During etching, the TiO2 mask is chemically modified, and becomes difficult to image after etching. The implications of surface chemical modifications on AFM imaging will be discussed.


Electron-beam induced deposition represents a new technology for production of nanoscale structures. Three-dimensional shapes can be obtained with nanometer accuracy, which may have impact on many applications in scanning probe microscopy or in vacuum microelectronics. The properties of the deposited material are determined by the precursor material as well as by the deposition parameters. At certain conditions, the deposits of very different organometallic precursor molecules show similar morphologies. The metal content of these deposits is strongly dependent on the beam current and the energy of the primary electrons. Using high beam currents and a power density of about 50 MW cm⁻² for deposition a polycrystalline material is build up, consisting of single crystallites with diameters of 1-4 nm embedded in an amorphous carbon containing matrix. The distance between the metallic crystallites is in the Ångström range. Thus electrical conductivity of the material is accomplished by tunneling between the nanocrystallites. Wires built up from these materials are able to carry current densities up to 2 MA cm⁻². Several materials have been characterized by TEM, EDX, and electrical measurements.

10:20 am  NSl-FrM7 Synthesis of Polymers at Highly Ordered Pyrolytic Graphite Template via Flow Injection Tunneling Microscopy, L. M. Morick, P. G. Van Patten, and J. D. Noll, Department of Chemistry and Biochemistry, University of South Carolina, Columbia, SC 29208 USA.

Highly Ordered Pyrolytic Graphite (HOPG) has step defects that are formed during the manufacturing process of the HOPG monocrystal. If we derivatize these steps with functional groups that will react with the polymer, we can use the HOPG monocrystal as a template to initiate the growth of a polymer at the nanometer scale. Recently, we have developed a method that will allow us to flow a solution over a surface while scanning with the Scanning Tunneling Microscope (STM). We can inject a reactant into an injector and watch the reactant hit the surface. The reactant flows out of the cell, and we can capture images of the surface before, during, and after the reaction. This Flow Injection Tunneling Microscopy (FISTM) technique allows us to inject the polymer and watch the polymer initiate and grow along the step of the HOPG. We show the initiation and growth of conducting polymers onto HOPG step defects.

10:40 am  NSl-FrM8 Nanoarchitectures of Poly(di-n-alkylsilylene), S. Sheiko, H. Frey and M. Möller, Organische Chemie III/ Molekularelektrokemie Chemie, Universität Ulm, D-89069 Germany.

As a conceptual intermediate between linear hydrocarbons and bulk silicon, catena Si polylsilylenes (PSi) demonstrate strong electro-optical activity with a possibility to tune the physical properties chemically. Due to encapsulating of a Si backbone in a shell of hydrocarbon side chains, the molecular properties of those polymers are strongly anisotropic. This forms a basis for well defined nanoarchitectures with electro-optical functions. In this report, novel techniques to fabricate...
fibres and films of polydi-n-alkylsiloxanes with controlled nanostructure will be demonstrated.

Fibres carrying highly oriented nanometer size domains of PSI were prepared by polystyrene gel crystallization and subsequent ultradrawing. The resulting fibres exhibit linear optical activity and anisotropy of the radiation induced conductivity. Orientation in a polymer host enables to combine the one-dimensional photo- and semiconducting properties of PSI with good stability and processability of the polystyrene.

Submicrometer thick films of different PSI showing a high degree of order were obtained by crystallization on a special substrate. Well defined 50 × 500 × 3000 nm³ crystals of extended and uniaxially oriented PSI molecules were grown on 50 nm in diameter polytetrafluoroethylene fibres. Morphology and growth of these "hetero-shish-kebab" structures were examined by scanning force microscopy. Microtopography on the prepared films was performed to realize conductivity measurements.


11:00 am NS1-FrM9 Carbon Nanotubes, Sumio Iijima, NEC R&D Group, 34 Miyukigakou Tsukuba, Ibaraki 305, Japan.

Carbon nanotubes are expected to show unusual electronic properties like a semiconductor according to the theoretical calculations. The properties depend on the tube diameter and helicity, and the tube diameters are required to be around 1 nm. We have successfully synthesized such nanotubes of a single-wall graphitic shell (Iijima et al. Nature 361, 603(1993)). In order to refine the tubes specimen, we extended the studies of the tubule growth. In particular we examined a structural relationship between single wall nanotubes and "single wall balloons" which are considered as giant fullerene. The latter structures often coexist with the nanotubes in our specimens. Their growth behaviors seem to be controlled by catalytic carbide particles (FeC3) which are used in our synthesis.

Other topics on nanotubes include TEM observations on atomic-scale mechanical deformation where individual tubules show a characteristic sharp bent figure, single-shell nanotubes enclosing nearly a single atom chain (or filament) of carbon atoms inside their central hollows, and an abnormal large separation (c-spacing) in double-shelled tubules in comparison with conventional graphite crystals.

INVITED

11:40 am NS1-FrM11 C60 on Silicon: Self-organization, Film Growth, and SiC Formation, D. Sarid and D. Chen, Optical Sciences Center, University of Arizona, Tucson, AZ 85721, USA.

We report on studies of UHV-prepared submonolayers, monolayers and multilayers of C60 molecules on silicon surfaces with the following results: (1) UHV-STM reveals that a single monolayer of C60 molecules, adsorbed on a Si(111)-7×7 surface, will self-organize in two ordered structural phases having different orientations on the Si surface. (2) UHV-STM and ambient AFM reveal the growth of crystalline C60 films on Si(111) and Si(100) surfaces that exhibit partially ordered layers and crystalline islands that differ in their morphologies. Annealing the samples to 600°C causes the C60 islands to evaporate, leaving a full layer of C60 molecules capped on the substrate. (3) UHV-STM reveals the interaction of deposited silicon atoms with C60 molecules already adsorbed on a Si(111)-7×7 surface. It is found that the interaction of the deposited silicon atoms with the adsorbed C60 molecules is rather weak. Also, the silicon atoms, when deposited on top of a monolayer of C60 adsorbates, form 3-dimensional clusters rather than wet the underlying surface. These results indicate that the adsorption of a C60 molecule on a Si surface reduces its reactivity with additional Si atoms. (4) Ambient AFM, SEM, and infrared spectroscopy were used to characterize thick SiC films, fabricated on Si(100) wafers held at 900°C, using C60 molecules as a carbon source.

NANO 3/NANOMETER-SCALE SCIENCE AND TECHNOLOGY
Room A207 – Session NS2-FrM

8:20 am NS2-FrM1 Applications of an AFM Probe with Ultrafast Time Resolution, B. A. Neehag, F. Ho, A. S. Huang, and D. M. Bloom, IBM T. J. Watson Laboratory, Stanford University, Stanford, CA 94305, USA.

We have developed a new measurement tool for probing ultrafast voltage waveforms on the nanometer length scale. This technique is based on mixing due to the nonlinear electrical force interaction between an atomic force microscope (AFM) tip and the sample-under-test. We have used this AFM probe for non-contact, electrical testing of internal nodes on VLSI and microwave circuits, demonstrating submicron spatial resolution and temporal resolution below 5 picoseconds. The probe also has the exciting capability for studying the transient physics within nanoelectronic devices.

In developing the ultrafast AFM probe, we microfabricated cantilevers on gallium arsenide with integrated microwave transmission lines, which allow picosecond sampling signals to propagate to the tip. The sampling signals are generated using nonlinear transmission line technology which produces voltage steps with sub-picosecond fall times. The result is a compact all-electronic system with measurement bandwidths beyond tens of gigahertz. In our presentation, we will discuss advances in instrumentation and our current research into the high-speed characterization of nanostructures.

8:40 am NS2-FrM2 Measurement of Ultra Fast Optoelectronic Phenomena in Organic Molecular Assembly by Scanning Maxwell Stress Microscopy, Takahito Inoue and Hiroshi Yokoyama, Molecular Physics Section, Electrotechnical Laboratory, 1-1-4 Umezono, Tsukuba-shi, Ibaraki 305, JAPAN.

The scanning Maxwell stress microscope (SMM) is an electric force microscope, designed to simultaneously image topography, dielectric constant, surface charge and potential, based only on harmonic analysis of oscillations of the cantilever driven at nonresonant frequencies [1]. In the SMM the nonlinear dependence of the Maxwell stress on the field strength can be utilized to image high frequency voltages running on the sample by the heterodyne force detection technique [2]. In the measurement of ultra fast optoelectronic phenomena, the high frequency voltages associated with sample irradiation by periodically modulated light are detected by heterodyne force detection. By changing the modulation frequency, furthermore, this technique allows observations of temporal responses of photovoltaic and photoconductive substances.

In the present paper we report observations of ultra fast optoelectronic phenomena, i.e. photoconduction and photoisomerization, in organic molecular assemblies by use of the SMM equipped with the heterodyne force detection system.

9:00 am **NS2-FrM3** High Frequency Pattern Extraction in Digital Integrated Circuits Using Scanning Electrostatic Force Microscopy, G. E. Bridges, R. R. Salama, M. Mitrai, D. J. Thompson, Department of Electrical and Computer Engineering, University of Manitoba, Winnipeg, RT2 2NZ, Canada.

The ability to perform logic analysis at the internal points of integrated circuits is crucial in the design and test of advanced microelectronics. As density and speed increases, conventional direct electrical contact probing methods are no longer adequate due to low spatial resolution and sensitivity of the circuit's operation due to pin counts and parasitic loading. We present a non-contact scanned probe technique for extracting the high-frequency digital patterns at internal points of an integrated circuit. The digital waveforms are determined by sensing the localized electrostatic force between a small probe and point on the circuit being measured. The force is monitored by detecting the deflection of the probe, using an optical interferometer. Higher frequency resolutions can be achieved if the interaction is localized and the probe can be scanned over the circuit with high precision. The temporal bandwidth of force measurements made using proximal probes are typically limited by the mechanical frequency response of the probe. In the presented instrument high frequency measurements are enabled by using a heterodyne technique. In conjunction with a nulling approach, the technique is capable of accurate measurements without complex calibration or probe positioning, and can be performed over passivated structures. Using a probe with a kHz resonant frequency, Mb/s patterns have been measured and digital logic levels with a voltage resolution less than 100 mV has been demonstrated.

9:20 am **NS2-FrM4** Room Temperature Quantum Mechanical Capacitance Measured With the Ultrafast Scanning Probe Microscope, D. Botkin, S. Weiss, D. F. Ogletree, M. Salerno and D. S. Chemla, Department of Physics, University of California at Berkeley, CA 94720, USA.

Recently, we proposed and demonstrated a general technique to obtain ultrafast laser spectroscopy and Scanning Probe Microscopy to obtain simultaneous picosecond and atomic space resolution. One of the most exciting prospects for this technique is the potential to create movies of surface dynamics on atomic scale, with atomic (ultrafast) time resolution. To this end, understanding the dynamic response of the quantum point contact formed between the STM tip and the sample surface is essential. Our measurements suggest that a large portion of the tunneling signal comes from a capacitive-like response. The tunneling gap height dependence of this response is very different from the one expected for the geometrical capacitance of the junction. In fact, the geometrical capacitance of the tip assembly contributes negligibly to the signal. These observations, which have been confirmed many times, suggest that "quantum mechanical capacitance", associated with the tunneling process itself, is responsible for the time resolved tunnel current data. The existence of an intrinsic capacitance associated with the tunneling process itself (quantum capacitance) has been discussed for instance in the context of the hotly disputed problem of "tunneling time" and in the context of photon assisted tunneling in SIS tunnel junctions. In both cases, quantum capacitance is the result of time lag between excitation and the tunnel current itself. A detailed study of the nature and the origin of the tunnel-gap capacitance is currently under way. Our current efforts are directed at comparing the predictions of the existing theories for quantum capacitance with our experimental results.

9:40 am **NS2-FrM5** Continuous Observation of the Motion of Single Adsorbed Atoms and Molecules with Picoscend and Sub-nanometer Resolution, G. M. McCllelland, F. Watanabe, and H. Heinzenmann, IBM Research Division, Almaden Research Center, San Jose, CA 95120, USA.

We have developed a new instrument, the femtosecond field emission camera (FFEC), which can follow continuously the motion of single adsorbed atoms and molecules adsorbed on a sharp metal tip. To detect motion through its effect on the field emission rate, the emitted electrons are tightly focused into a beam which is electrostatically swept across a detection screen. Very fast time resolution is possible, because the tip is an ideal point source, and the strong field near the tip minimizes the time of flight spread of the field emitted electrons. To observe the hopping of a Cs atom at sites, field ion microscopy methods are used to form a sharp W(111) tip on which the field emission is confined to a region \( \approx 1 \, \text{nm} \) across. A Cs atom is deposited onto a 90 K tip, and a field emission image is recorded. The tip is then pulsed negatively for 2 ns while the focussed emission beam is swept across the screen. The increased field emission excites the Cs, which hops to a nearby site. After the sweep, the new position of the Cs atom is observed in the field emission image. When a copper phthalocyanine molecule is adsorbed on the tip, 10(6) Hz oscillations in the field emission intensity are frequently observed. These are believed to result from the slow overall vibration of this heavy molecule with respect to the tip.

10:00 am **NS2-FrM7** Magnetic Resonance Detection and Imaging Using Force Microscope Techniques, O. Zueger, S. Hoen, C. S. Yannoni and D. Rugar, IBM Almaden Research Center, 650 Harry Rd., San Jose, CA 95120.

Electron spin resonance (ESR) and nuclear magnetic resonance (NMR) were detected via the magnetic force acting between the spins of the sample and a ferromagnetic particle. An oscillatory magnetic force was generated by polarizing the spins in a strong magnetic field and then modulating the sample magnetization using magnetic resonance techniques. The resulting femtowatt magnetic force was detected by sensing the angstrom-scale vibration of a micromechanical cantilever on which the sample was mounted. Detection of both ESR and NMR was demonstrated with nanogram sensitivity. Magnetic resonance imaging was performed with micrometer-scale spatial resolution by scanning the ferromagnetic particle with respect to the sample.

10:40 am **NS2-FrM8** Parallel Imaging with an Atomic Force Microscope, S. C. Minne, Ph. Flueckiger, H. Soh, C. F. Quate, Applied Physics, Stanford University, Stanford, CA 94305-4090.

If the atomic force microscope (AFM) is to become an effective production tool, its scan size and image acquisition rate must be improved. The current scan size is generally limited to about 100 \( \mu \)m by 100 \( \mu \)m because, at large sizes, the piezoelectric tube scanner becomes nonlinear. AFMs operate with a single probe and thus the entire image must be obtained serially by measuring each pixel in sequence. This is quite time consuming, even for scans smaller than the 100 \( \mu \)m by 100 \( \mu \)m limit. To address these problems of size and speed, we have fabricated and operated AFMs incorporating multiple probes formed by parallel arrays of piezoresistive cantilevers. We have obtained 400 \( \mu \)m by 100 \( \mu \)m images by operating four probes simultaneously. Thus, either the acquisition time is decreased or the scan size is increased by a factor equal to the number of atomic force probes acting in parallel. We anticipate that there will be many areas where parallel imaging will be highly beneficial; one that we are currently investigating is its incorporation into an AFM lithography system.

11:00 am **NS2-FrM9** Detection of Sub-Femtowatt Forces, S. Hoen, O. Zueger, C. S. Yannoni, H. J. Mamin, K. Wago, and D. Rugar, IBM Research Division, Almaden Research Center, San Jose, CA 95120.

We have developed micromechanical cantilevers capable of detecting sub-femtowatt forces. These cantilevers have been used to detect nuclear magnetic resonance (NMR) signals from microscopic samples, inaugurating a technique which is several orders of magnitude more sensitive than conventional NMR schemes. The cantilevers are fabricated from low stress silicon nitride grown on (110) oriented silicon using low-pressure chemical vapor deposition. After an oxygen dry etch, the fixed end of the cantilever is determined by a vertical slow-etching (111) plane, eliminating the need for backside alignment or anodic bonding. The release process is completed by a critical point drying step which is necessary to protect these delicate structures from meniscus forces. Using this process, we have fashioned 200-A thick silicon nitride cantilevers with thickness to length ratios of 1:2500 and spring constants of \( \approx 10^{-3} \, \text{N/m} \). Their thermally limited force sensitivity is \( 2.4 \times 10^{-17} \, \text{N/Hz} \) at room temperature. Damping measurements have been made as a function of temperature for 600-A thick nitride cantilevers. These cantilevers may provide enough sensitivity for the detection of single electron spins and move this NMR technique significantly closer to the ultimate goal of detecting single nuclear spins.

11:20 am **NS2-FrM10** Atomic Force Microscope Tip Modification with Self-Assembled Monolayer Molecules, Taejoon Han, John Williams, T. P. Beebe, Jr., Department of Chemistry, University of Utah, Salt Lake City, UT 84112.

Atomic force microscopy (AFM) generates precise topographical information for both conductive and non-conductive materials. AFM is not without limitations however, and foremost among them is its lack of chemical specificity. Since the tip material most commonly used in AFM is Si3N4, AFM tips do not have much chemical variability without some international modifications. We discuss a method to modify AFM tips with self-assembled monolayers (SAM) of organic molecules in order to give the tip well defined chemical characteristics.
Alkanethiols on gold are used as the SAM system. Since both the surface and the tip can be chemically modified and have well known characteristics, several experiments and measurements are possible under this scheme. In this study, we discuss the artifacts arising from scanning probe microscopy measurements caused by the structural interaction of a finite-sized tip and small hollow surface structures ("pits" in the SAM). The analysis of SAM-modified surfaces with several spatially-averaging UHV techniques is also presented. We develop a statistical method which produces information about individual chemical bond forces and present evidence for detection of individual bond force changes which can be attributed to hydrophilic forces. Furthermore, it is possible to detect changes in the interaction force by varying the chemical environment without assuming which chemical bonds are involved.


We report a new type of focusing of far ultraviolet light, λ<1000 Å, using tapered capillary tubes similar to those first developed for the near-field scanning optical microscope (NSOM) which we call photon channeling. Silica tubes with taper-half-angles of ~0.5° to 6° have been tested. An array of capillaries mounted on a rotatable drum is placed in front of a collimated photon source obtained from a rare-gas discharge lamp, successively the capillaries can be brought into alignment, and the photomission detected by a channeltron-grid detector. The straight capillary intensity shows no significant light guide effect; it simply decreases as the area of the capillary. However, the tapered capillaries show an enhanced transmission (or photon channeling effect). In quantitative agreement with simple numerical estimates [1] we find that the transmission enhancement factor varies from ~10–700 for the largest transmission of a pinhole aperture of the same dimensions with an approximately linear dependence of channeling factor on inverse of the half angle. Useful intensity suitable for photoelectron energy analysis with capillary openings of ~0.7 μm has been demonstrated. The expected spatial resolution is comparable to that obtained with more complex lithographically formed Fresnel zone plates and has the important advantage that the image probe size is independent of photon wavelength. A scanning stage has been developed to form spectral energy-resolved images and requires a tip approach feedback control different from that of NSOM to operate in high vacuum. Preliminary calibration measurements and scanning tests will be presented.

**Present address: Advanced Light Source, Lawrence Berkeley Laboratory, Berkeley, CA 94720.


APPLICATION OF SURFACE SCIENCE

Room A101 – Session AS-FrM

Adhesion and Adhesive Bonding

Moderator: C. R. Anderson, Martin Marietta Laboratories.


Plasma polymerized films of monomers such as hexamethyldisiloxane (HMDS) and trimethylsilylane (TMS) have been deposited on aluminum substrates and evaluated as primers for adhesive bonding of aluminum. As-deposited films were characterized by a variety of techniques including X-ray photoelectron spectroscopy (XPS), ellipsometry, secondary ion mass spectrometry (SIMS), atomic force microscopy (AFM), and scanning electron microscopy (SEM). The results showed that the molecular structure of the films depended strongly on the nature of the carrier gas which was used as the carrier, films having siloxane-like structures were obtained but when oxygen was used as the carrier, silica-like films were obtained. The films were composed of spherically shaped particles whose growth seemed to be nucleated by the oxidized aluminum surface. The properties of the films as primers for adhesive bonding of aluminum were evaluated by preparing lap joints from coated aluminum substrates, applying a load to the joints, and measuring the time required for failure to occur during exposure to a corrosive environment. It was found that the silica-like films were excellent primers.


Conventional surface treatments for aluminum adherends commonly involve chromates and almost always involve strong acids or bases and significant quantities of waste water. Environmentally safe means to dispose of the wastes are costly and will become more expensive in the future. Plasma spray coatings can potentially reduce or eliminate much of the disposal and cleanup costs associated with chemical treatments. We have characterized and evaluated several different plasma spray coatings as surface treatments of aluminum adherends. In wedge tests, the various aluminum-rich aluminum/polyester and aluminum/PEEK coatings give better results than either constituent by itself. The best durability performances are better than that of FPL-treated bonds and equivalent to that of PAA-treated bonds for some adhesives. In blended coatings, the polymer is believed to toughen the coating and provide moisture-resistant chemical bonds to enhance durability. The aluminum in the coating provides coating strength and adherence to the substrate.

This work was supported by Wright Laboratory Materials Directorate under contract F36315-93-C-5324.

9:00 am AS-FrM3 Characterization of the Interphase Formed on Adhesion of Polymers to Metal Oxides, J. P. Wightman, Virginia Tech, Blacksburg, VA 24061.

The interphase region at polymer/metal oxide interfaces may be critical in determining the strength and durability of adhesive bonds. Methods to investigate these regions are limited however and this research has focused on the complementary techniques of infrared spectroscopy (IRS) and x-ray photoelectron spectroscopy (XPS) to investigate the molecular structure of the interphase. Dielectric thermal analysis was used in earlier work to probe the properties of thin polysulfone films on pretreated aluminum surfaces. A significant increase in the activation energy associated with segmental motion in the backbone of polysulfone occurred for films between 1–2 μm thick deposited on an acid-etched aluminum substrate. A new technique, variable temperature reflection absorption infrared spectroscopy, was developed recently as a method to investigate the thermal relaxation behavior of ultra-thin polyphenylene sulfide (PPS) films on a variety of substrates. PPS films on copper showed a loss in crystallizability in contrast to results observed for chromium and aluminum. Loss of curcupoxide film underlying the PPS film was also observed. Bonded PPS/copper laminates showed that the particular surface chemistry was critical in determining the peel strength observed. For example, after a simple thermal oxidation pretreatment for copper foil, an order of magnitude increase in peel strength was observed in agreement with XPS and IRS results.

INVITED

9:40 am AS-FrM5 Practical Multi-Technique Analysis of Metal Surfaces for Adhesive Bonding Applications, A. P. Dwiranji, A. R. Madura, R. Martin, LORD Corporation, Erie, PA 16514.

Metal components used in adhesive bonding applications are often subjected to machining and forming processes. These processes leave residue to contamination on the bonding surface. Contamination is one of the most insidious factors affecting adhesive bond performance and reliability in metal-to-rubber, structural bonding and coating applications. Contamination is generally removed by cleaning treatments to yield a clean surface. A clean surface is one that contains no significant amount of undesired materials. The nature and composition of these undesired materials can vary depending on bonding applications.

Metal production and cleaning treatments are continuously changing with new applications, developments in materials technology and regulatory requirements. This necessitates ongoing characterization of both contaminated and cleaned surfaces to assess the nature of the contamination and the effectiveness of current cleaning treatments.

In this study, processed metal alloys were cleaned by methods including acid pickling, alkaline cleaning and vapor degreasing. Multi-technique surface investigations including ISS, SIMS and XPS were used to characterize the effectiveness of the treatments and determine 324
The composition and chemistry of the bonding surfaces. These results are compared to results from methods such as wettability determination and evaporation analysis that use thermal analysis techniques. To address the issue of surface cleanliness, data from these techniques correlate well with each other and reveal the presence of both organic and inorganic surface contaminants that may be significant to bond performance.

10:00 am  AS-FRM9  Strained Siloxane Rings in the Surface of Silica: their Reaction with Organosiloxanes, Organosilanes, and Water*  A. Grabbe, T. A. Michalske, and W. L. Smith  Sandia National Laboratories, Dept. 1114, Albuquerque, NM 87185.

The glass network on the surface of highly dehydroxylated silica contains strained siloxane defects. The SiO dimer defects are edge shared tetrahedra, some of which have lessened reactivity due to an attached hydroxyl. Using Infrared spectroscopy we show that dimer defects react with siloxanes or with water at rates that can be comparable, depending on the structure of the organosiloxane. A polar bond component is a necessary condition for a rapid reaction with dimer defects, since non-polar organosiloxanes react orders of magnitude slower than do water or organosiloxanes. The important features of the reacting bond that control the rapidity of the reaction are its polarity, steric accessibility, and bond strain. The reactions require 6 orders of magnitude of gas exposure to go from one to ninety-nine percent completion, showing that the surface is highly heterogeneous. We have applied a simple model of the surface's heterogeneity to our data, incorporating a linearly distributed activation energy in the defect population. A fit using this model indicates that the spread in activation energy is approximately 10 kcal/mole in all cases. The organosiloxane reaction with dimer defects produces hydrolytically stable couple points for adhesion to polymers, which cannot be readily synthesized by conventional silane chemistry.

*Work supported by D.O.E. contract DE-AC04-94AL8500 and AT&T Bell Laboratories.


The ability of a metal-ceramic bond to resist fracture is a function of the interfacial adhesion and toughening mechanisms due to metal plasticity. We have developed a sensitive method of detecting and quantifying the extent of metal plasticity on the atomic scale produced at such interfaces during fracture. It involves the detection of emitted electrons with intensity directly related to the number of mobile atoms exposed during fracture. Plastic deformation greatly increases this number due to surface roughening, which in turn order is proportional to the extent of slip. A description of the experimental arrangement, absolute calibration of the electron emission intensities vs. surface area, and results from a series of interfacial failure experiments will be presented. Comparisons with post-fracture AFM measurements performed on the metal surface will be made; limitations of the AFM results are discussed. Our model systems are 1 μm, thick Mg and Ti films detached from soda-lime glass in a Chevron notch geometry. We have established a linear relationship between electron emission intensity and fracture toughness.

*This work supported by AFOSR and NSF-DMR.

10:40 am  AS-FRM8  Improved Adhesion at the Copper/Polymide Interface Using an Organometallic Additive  D. Coulman, The DuPont Company, Central Research and Development, Wilmington, DE 19880-0326.

Polymide Kapton® films containing an organo-tin additive have been found to improve adhesion of direct metallized copper-polymide clads. The peel strength of the polymide films which contain the organo-tin additive, after 500 hours at 85% RH and 85°C, are substantially higher 4-7 lb./in compared to 2-3 lb./in for the comparable films without the additive. More importantly the normally marked decrease in peel strength after temperature humidity aging is not observed with the organo-tin additive. Changes in the surface chemistry and structure of these films has been investigated using XPS, RBS, and TOF SIMS analysis. RBS analysis clearly indicates the segregation of the tin additive to the near surface region of the polymide film. Atomic force microscopy (AFM) has been used to study the surface morphology of the Kapton® films. A factor of two improvement in peel strength has been observed even with no surface pretreatment.

The effect of plasma etching on the peel strength will also be presented. These results on copper-Kapton® film laminates will be correlated with ultrahigh vacuum studies of copper deposition on the organometalic containing film.


Benzocyclobutene (BCB) polymers are used for dielectric layers in new generation multilayer interconnect devices (multipitch modules). In this metal/polymer application it is important to understand the nature of the bonding and complexing which occurs at the metal/polymer interface in order to optimize adhesion. We have observed the relative orientation and chemistry of polymer functional groups in several buried metal [Al, Ti, Cu, and Au (10-100 nm thick)]/BCB polymer interfaces using the bond specific polarization anisotropy of carbon K fluorescence yield NEXAFS.

The depth sensitivity (about 200 nm) and the non-destructive nature of our fluorescence yield NEXAFS technique are particularly useful in studying the buried metal/BCB interface. The aromatic ring of BCB appears to undergo a dramatic enhancement in orientation after the formation of the metal polymer interface for the Al and Au overlayers compared to free standing BCB surfaces. These preliminary results highlight the use of fluorescence yield NEXAFS as a probe of chemical structure at buried interfaces.

11:20 am  AS-FRM10  Adhesive Bonding between Self-Assembling Monolayer Films Measured by Interfacial Microscopy, R. C. Thomas, R. M. Crooks, 1,2 Taisun Kim, 2 J. E. Houston, 2 and T. A. Michalske 1 Department of Chemistry, University of New Mexico, Albuquerque, NM 87131; 2Department of Chemistry, Texas A&M University, College Station, TX 77843-3255; 2Surface and Interface Sciences Department, Sandia National Laboratories, Albuquerque, NM 87185-0355.

We have used the interfacial force microscopy (IFM) to study adhesive bonding between n-alkanethiol monolayer films that are strongly adsorbed to both a Au probe and a Au sample. Our results demonstrate that force-versus-displacement curves can be used as "fingerprints" to identify specific bonding mechanisms between monolayers having either the same or different endgroups. For example, we have measured the magnitude of the van der Waals interactions between two methyl-terminated, surface-confined monolayer films over the entire range of interfacial separation. Moreover, the force curves are continuous (e.g., no jump-to-contact) and show no hysteresis between loading and unloading. By measuring the adhesive pull-off force and using DMT theory, we can calculate surface energies that are in excellent agreement with literature values for these low-energy hydrocarbon films. We will contrast these results to IFM studies between surface-confined monolayer films modified with the chemically reactive end groups, COOH and NH2, which have additional adhesive contributions arising from acid-based and hydrogen-bonding interactions. The force curves for these data are also continuous, but show a distinct hysteresis between loading and unloading. We have found that it is possible to calculate bond energies for specific types of chemical interactions by integrating the area under the unloading curves and estimating the contact area at zero load. We will compare our results to those recently obtained using both the surface forces apparatus and the atomic force microscope.

11:40 am  AS-FRM11  Micromachining Induced Adhesion Enhancement Between High Performance Polymer and Silicon Dioxide Substrate, Hye-Soo Jeong, Institute for Advanced Engineering, C.P.O. Box 2849, Seoul, Korea, and Jack Jiang, Thomas Electronics, Inc., Wayne, NJ 07470.

It is well known that polymers do not adhere to silicon dioxide (SiO2). Surface modification is required for adhesion enhancement. The surfaces of thermal oxide films have been fabricated by silicon micromachining technology in order to enhance adhesion between polycarbonate and SiO2 substrate. An artificial interface is thus formed between the two materials. The micromachined mushroom structure (on the order of 100 A) act as mechanical hooks between these interfaces, contributing to enhanced bonding strength via mechanical interlocking mechanism between the two materials.

Our results show that the fracture energy (Gf) of the micromachined
THIN FILM
Room A105 - Session TF-FrM

In Situ Thin Film Characterization
Moderator: C. R. Aita, University of Wisconsin, Milwaukee.


A multi-wavelength in-situ ellipsometer has been developed for in-situ thin film deposition monitoring and control. This instrument acquires ellipsometric data \( \psi \) and \( \Delta \) at 44 different wavelengths simultaneously in spectral range from about 410 to 750 nm. Typical data acquisition rate is about 1 measurement per second with maximum rate of 25 measurements per second. The in-situ ellipsometer was mounted at a 70° angle of incidence on a deposition. The typical chamber vacuum was about 10⁻⁶ Torr. A multilayer stack of quarter-wave ZnS and Cryolite was thermally evaporated onto BK7 glass substrate. Quarter-wave thicknesses were determined by reading transmitted light intensity maxima or minima recorded using a photomultiplier tube. The in-situ ellipsometer acquired data during the deposition process and the deposited layer thickness without interaction with the deposition process. The ellipsometric data were analyzed after the deposition. Ex-situ ellipsometric measurements, spectral transmission measurement, and XRD were also performed for post-deposition analysis and characterization. Thicknesses of each layer and optical constants of ZnS and Cryolite were obtained. Optical constants were found to have changed after the sample was exposed to air.

9:00 am TF-FrM3 Surface Analysis at Low to Ultra-High Vacuum by Ion Beam Recoil Spectroscopy, M. S. Hammond, SI Diamond Technology Inc., Houston, TX 77098, J. A. Schultz, Ionwerks Inc., Houston, TX 77005.

An overview of widely used ex situ ion beam surface analysis techniques will be given. These techniques will be compared to the recently developed ion beam recoil spectroscopies which are capable of in situ surface analysis at pressures ranging from tens of milliTorr to UHV. Ion beam recoil techniques, by using forward scattering in the keV range, enhance surface selectivity and enable the beam and the detected constituents to be detected at vacuum levels far higher than ordinary surface spectroscopy techniques. In addition, the energy range used results in higher detector efficiencies, thereby relaxing constraints imposed by ion scattering cross sections. Time of flight detection is used to provide both energy and mass information from the scattered ions. Two variations of this technique exist. Direct Recoil Spectroscopy (DRS) uses line of sight detection and detects both neutrals and ions, resulting in high detection efficiencies. DRS has the added advantage of being able to detect H, C, O, and N surface stoichiometries at pressures up to 100 mTorr per cm of pathlength. Mass Spectroscopy of Recoiled Ions (MSRI) uses a mass and charge filtering scheme to detect only ions and is applicable to a wide variety of elements and isotopes. The higher resolution afforded by the use of mass and charge filtering enables the use of isotopic labeling techniques during adsorption experiments. Examples of data taken during the CVD growth of diamond, high Tc superconductors and Group III-nitrides will be presented, along with H-D exchange experiments on silicon and diamond.

9:40 am TF-FrM5 Deposition and Characterization of Transition-Metal Nitride Superlattices, Scott A. Barnett, Northwestern University, Evanston, IL 60208.

In this talk, the role of in situ monitoring and control as well as ex situ characterization is described for the case of epitaxial and poly-crystalline nitride superlattices on thin films that exhibit extremely high uniformity. The use of in situ Auger electron spectroscopy for determining nucleation mechanisms during epitaxial growth of various nitride layers is described, particularly the effect of lattice mismatch. The use of in situ nitrogen gas pressure monitoring during superlattice deposition for maintaining high-rate reactive sputtering conditions and for obtaining the desired stoichiometry of both layer materials is described. Post-deposition characterization is also critical for determining both the nature of the layers and the properties. The use of x-ray diffraction combined with simulations of the resulting scans yields information on crystal structure, interface widths, and layer coherency strains; factors that are critical for determining properties. Cross-sectional and plan-view TEM provide critical information that is largely complementary to the x-ray results, including direct observation of dislocation relaxation of coherency strains, porosity, and layer morphology. Finally, the results of Vickers microhardness and nanoindenter measurements are described and compared with models of superlattice properties.


Real time, in-situ spectroscopic ellipsometry is a powerful tool for characterizing thin film growth or surface modification, and thus providing process control. Virtually all studies to date have examined thin layers on a semi-infinite substrate, i.e., the transmitted beam is completely absorbed (as in crystalline silicon) or scattered at the back of the substrate (as with roughened glass). This situation is required since the coherent optic formulae are used in modeling. However, the requirement for a special substrate has limited spectroscopic ellipsometry to experimental conditions which are not typical for industrial device-making.

Here, we propose an explicit analytical formalism for rotating analyzer ellipsometry which permits the analysis of spectroscopic ellipsometry data for thin films on transparent substrates such as glasses, where the transmitted beam becomes phase-incoherent and reflects back toward the sample surface. We show that the incoherent optical limit can be incorporated into the first-level data reduction routine. As an example, we present data and analysis for hydrogenated amorphous silicon growth on transparent conductive oxides, and show that film nucleation and coalescence, and chemical reduction of the oxide can reliably be determined.
10:40 am TF-FrM8 Optical Emission Spectroscopy of Plasmas for Diamond Growth, R. Manukonda, R. Dillon, Electrical Engineering Department and Center for Microelectronic and Optical Materials Research, University of Nebraska, Lincoln, NE 68588 and T. Furtak, Physics Department, Colorado School of Mines, Golden, CO 80401.

Our radio frequency chemical vapor deposition system has been used to deposit diamond films with water-alcohol and the traditional hydrogen-carbon-oxygen source gases. The water-alcohol source gases yield high atomic hydrogen and electron densities. Optical emission spectroscopy (OES) was performed with both source gases with a 1/2 meter monochrometer in the spectral range from 200 to 800 nm. OES is only sensitive to emitting species and therefore does not provide fundamental information on diamond growth. However, a low ratio of CH to atomic H peaks has been found by ourselves and others to be an empirical indication of increased diamond growth for the traditional source gases. Thus, OES has excellent potential as an inexpensive and non-intrusive diagnostic for quality control in diamond growth. In this paper, we compare the OES spectra for diamond growth from the water-alcohol and the traditional hydrogen-carbon-oxygen source gas and discuss correlations between the spectra and film characteristics such as growth rate and Raman spectra.

11:00 am TF-FrM9 Optical Second Harmonic Generation (SHG) as an in Situ Technique to Monitor the Growth and to Analyse the Structure of Thin Films, M. Buck, Ch. Dressler, M. Grunze, F. Träger*, Angewandte Physikalische Chemie der Universität Heidelberg, Im Neuenheimer Feld 253, 69120 Heidelberg, Germany, *Fachbereich Physik der Universität Kassel, Heinrich-Plett-Str. 40, 34132 Kassel, Germany.

To analyse thin films and their interface to the substrate a none intrusive characterization is desirable. Ellipsometry and infrared spectroscopy are two examples of linear optical techniques that are widely used for thin film investigation. SHG as a nonlinear technique well established for interface analysis has also been applied recently to study thin film growth.

Polymeric acid films on metal substrates have been investigated. Pyromellitic dianhydride (PMDA) and various diamines were used as constituents of the polymer. The shape of the thickness dependent SHG-signal is found to vary with the monomers. A theoretical model shows that the phase sensitive superposition of contributions generated in the film and in the substrate strongly affects the shape of the SHG-signal. Most importantly, the thickness dependent SHG-signal bears information about the orientation of the molecules and therefore can serve to infer on the structure of the films.


Infrared spectroscopy is ideally suited for real time studies of chemical bonding on thin film surfaces during processing in gas ambient, e.g., chemical vapor deposition, reactive etching, or surface modification. The vibrational spectra reveal the identity, configuration, and local environment of bonds, both on the physical surface and in the bulk.

Since infrared absorption is relatively weak, optical enhancement is necessary to observe the signals from a surface layer or very thin film in real time. Here, we critically review five techniques which permit analyses during processing. Optical enhancement in a reflection-mode geometry can be obtained using (i) metal substrates, (ii) a thin dielectric layer on a metal substrate, (iii) multiple internal reflection substrates, (iv) infrared ellipsometry, or (v) multilayer optical cavity substrates (the latter two are referred to as cavity and ringdown techniques, respectively). The choice of technique depends on the material system, IR modes of interest, and experimental constraints. We calculate the electric fields in each structure and the quantity that is measured, normally the change in reflectance. We consider the effect of experimental variables on the measured signal, e.g., substrate material, incident angle, polarization and wavelength of light, dielectric constants of the film, and thickness of the film. Examples are drawn from systems of current interest, e.g., H on and in Si. For future practitioners, we compare these techniques in terms of their ability to identify bonds on the physical surface vs. the bulk, reliability in measurement conditions, and ease of data analysis.


The use of comprehensive, non-destructive, in situ methods for the characterization of thin film growth phenomena is key to obtaining both a better understanding of thin film growth processes and to developing more reliable deposition procedures, especially for complex layered structures involving multi-phase materials. However, surface characterization methods that utilize either electrons (e.g., AES or XPS) or low energy ions (e.g., SIMS) for the signal require an UHV environment and utilize instrumentation which obstructs line-of-sight access to the substrate. These methods are therefore incompatible with thin film deposition processes which introduce gas, either for the deposition process or the production of the desired phase such as an oxide, and require line-of-sight deposition. We have developed a means of differentially pumping both the ion beam source and the detector of a TOF ion beam surface analysis spectrometer that does not interfere with the deposition process and permits compositional and structural analysis of the growing film at pressures up to several tens of mTorr.

In order to quantify the sensitivity of Ion Scattering Spectroscopy (ISS), Direct Recoil Spectroscopy (DRS) and Mass Spectroscopy of Recoiled Ions (MSR) we have measured the signal intensity for stabilized clean metal and well-characterized oxide surfaces in a variety of gas environments as a function of the ambient gas species and pressure, and ion beam species and kinetic energy. The results are interpreted in terms of collisional cross sections which are compared with known gas phase scattering data, and provide an apriori basis for the evaluation of the method for various industrial processing environments which involve both inert and reactive gases at pressures up to several hundred mTorr.

*Work supported by the Department of Energy Division of Basic Energy Sciences under contract W-31-109-ENG-38.

**ELECTRONIC MATERIALS**

Room A108 – Session EM-FrM

**Interface Characterization**

**Moderator:** S. A. Chambers, Pacific Northwest Laboratory.


X-ray specular reflectivity has become a standard tool for analysis of interface abruptness in thin film microstructures. However, low resolution specular reflectivity scans only yield information about the average density profile. High-Resolution X-ray Reflectivity allows measurement of diffuse scattering that contains additional information about the amplitude and lateral wavelength of the roughness and its evolution from interface to interface.

We have performed High Resolution X-ray Reflectivity on Si/Ge short-period and Si/Ge, Si, , long period superlattices. Roughness from at least two different physical origins can be observed. The first is an interface corrugation with an upper limit on the lateral wavelength of 1.2 microns. The corrugation is asymmetric and oriented along the plane of miscut and is highly periodic, producing characteristic peaks in reflectivity rocking scans. A second roughness exists on a much shorter length scale, and exhibits a strong dependence on growth temperature. The amplitude of this roughness is highest for very low (250°C), and very high growth temperatures (650°C).

The degree of vertical correlation can also be analyzed with this technique. Very low and intermediate growth temperatures produce highly correlated roughness, while growth at temperatures above 650°C produces only partially correlated roughness. This suggests that random noise is introduced in the growth front at higher temperatures causing a fluctuation of the roughness from interface to interface.

Other techniques such as cross-sectional electron microscopy and double crystal x-ray diffraction have been performed on the same long and short period superlattices for comparison.
9:00 am  EM-FrM3 Buffer Layer-Superlattice Interactions in the AlAs/GaAs Superlattice System, J. G. Pellegrino, 'S. B. Quadri, 'B. Roughani, 'A. Richter, D. Chandler-Horowitz, N. V. Nguyen, P. M. Amirtharaj, NIST Semiconductor Electronics Division, Gaithersburg, Maryland 20899, 'Naval Research Lab, 'GMI Engineering and Management Institute, 'NRC Research Associate.

An issue of crucial materials concern in producing high quality reproducible layered semiconductor devices is whether roughness originates at the substrate and propagates up the layer versus growth-induced roughness. We present results which clearly indicate that the thickness of the buffer layer affects the roughness observed in a subsequently grown (AlAs)\textsubscript{n}/GaAs\textsubscript{m} superlattice test structure where m = n = 3 and p = 18. A comprehensive set of samples grown on both (001) and (111) substrates were examined. Growth rates of 0.1 \textmu m/hr and 0.2 \textmu m/hr were used for the AlAs and GaAs sublattices. High resolution x-ray reflectivity was used to examine the interfacial structural properties for typical scans \( Q = (0.7-1.7) \text{ Å}^{-1} \). These results were correlated with optical properties obtained by Raman, photoconduct, and ellipsometric measurements. Results indicate that roughening is promoted by thin GaAs buffer layers (10-100 nm). Smoother interfaces were observed in samples with buffer layer thicknesses 250 nm and greater. The analysis of roughness in the (AlAs)/GaAs system of interest because the sublattices are sufficiently thin such that a strict step-flow growth mode cannot be assumed. The implications of these results on electron transport properties were investigated in a series of modulation doped heterostructures.

9:20 am  EM-FrM4 Interfacial Properties of Metal-Insulator Semiconductor Capacitors on GaAs(110), L. J. Huang, R. Krishnamurthy, W. M. Lau, Department of Materials Engineering, University of Western Ontario, London, Ontario N6A 5B9 Canada; J. Inger, Bell Northern Research, P.O. Box 3551, Station C, Ottawa, Ontario K1Y 4H7 Canada; D. Landheer, and J.-P. Noel, Institute for Microstructural Sciences, National Research Council of Canada, Ottawa, Ontario K1A OR6 Canada.

Metal-insulator-semiconductor capacitors were fabricated on cleaved n-GaAs (110) facets and on n-GaAs(110) wafers using remote plasma deposited silicon nitride as gate insulator. The interface properties of the capacitors made on this surface were analyzed by capacitance-voltage (CV) measurements. X-ray absorption near-edge structure (XANES) spectroscopy and x-ray photoemission spectroscopy were also used to investigate the structures of the buried interface. Prior to the insulator deposition, the cleaved facets were processed with different surface treatments including HF-etch of native oxide, passivation with an ammonium sulfide solution, passivation with hydro- gen polysulfide, and passivation with a silicon interface control layer. It was found that while the passivation procedures with the sulfur compounds did improve the CV data when compared with the HF oxide etch, the silicon interface control layer technique led to the best CV results. By comparing the quasi-static and high frequency (1 MHz) CV data, we found that the minimum interface state density of the fabricated capacitors was about 10\textsuperscript{13} eV\textsuperscript{-1} cm\textsuperscript{-2}. The results were compared with those obtained from GaAs(100) and the differences addressed with respect to the surface geometry and the electronic structures.


During the heteroepitaxy of strongly dissimilar materials, the complex growth kinetics and interface reactions of the first few molecular layers can control the structures and morphology of the next several hundred layers. Within these first few layers, the atomic and electronic structure continuously evolves as the bare substrate reacts to form a new interface compound, which in turn serves as the substrate for the nucleation and coalescence of subsequent layers. The interface structure may also change as it is covered. Using the powerful in-situ combination of X-ray photoelectron spectroscopy and diffraction, we have monitored this evolution of the atomic and electronic structure when the insulator CaF\textsubscript{2} is deposited on Si. We observe and explain changes in the metal and core electron emission energies for 8 different atomic layers, and exploit energy-resolved diffraction to separately determine their local structure. We also determine the extent of interface reaction and average film morphology as a function of growth kinetic conditions, and have successfully modeled the results. We have also observed changes in the buried interface structure when the reacted layer is covered by additional fluoride or when the surface of the film is modified by oxidation or by deposition of Si.

Work supported by the U.S. Dept. of Energy.

10:20 am  EM-FrM7 Modified Surface Charge Spectroscopy for the Characterization of Insulator-Semiconductor Structures, W. M. Lau and L. J. Huang, Department of Materials Engineering, University of Western Ontario, London, Ontario, Canada N6A 5B9; and R. W. McKoy and G. Jin, Department of Chemistry, Chinese University of Hong Kong, Shatin, Hong Kong.

The operation of all thin-gate-insulator/semiconductor structures relies on two basic properties, charge maintenance by the insulator and the response of the semiconductor surface potential to the insulator charging potential. In surface charge spectroscopy (SCS), external negative/positive charge is put on the insulator surface in vacuum. The breakdown and leakage characteristics as well as the relationship between the semiconductor surface potential and insulator charging potential are then measured using an x-ray photoelectron spectrometer. Once the relationship between the semiconductor and insulator surface potential is determined, the interface state distributions are calculated using a typical space-charge analysis model. High throughput, SCS has only been applied with low energy electron flooding for negative surface charging, and thus no data corresponding to positive gate-insulator voltages have been shown. The present study shows that by collecting secondary electrons emitted from the sample surface, positive charging of the insulator could be obtained and controlled. The modified SCS, which covers both negative and positive charging, has been successfully applied to the studies of SiO\textsubscript{x}/Si and SiN\textsubscript{x}/SiP. The interface state density data were found to be consistent with those from the structural capacitance-voltage analyses of the equivalent metal-insulator-semiconductor structures.

10:40 am  EM-FrM8 Buried Contaminant Structure Determination with Component-Resolved X-Ray Photoelectron Diffraction in CaF\textsubscript{2}/Si(111) Heterostructures, 'M. Leskovar, U. Hessinger, and Marjorie A. Olmstead, Dept. of Physics, Univ. of Washington, Seattle, WA 98195.

Contamination during heteroepitaxial growth or subsequent processing may alter the chemical and structural properties of buried interfaces and other subsurface structures. The technique of component-resolved X-ray photoelectron diffraction (CR-XPD) enables simultaneous, in situ determination of both the chemical identity and the local structure of buried contaminants through the energies and diffraction intensities, respectively, of the photo-emitted electrons. We use CR-XPD to investigate an ultrathin CaF\textsubscript{2}/Si(111) heterostructure exposed both to residual gases in an UHV environment and to atmosphere. While a stoichiometric CaF\textsubscript{2} surface should be stable under these conditions, in both cases at least two oxygen species were incorporated in the CaF\textsubscript{2} layer at room temperature. This room temperature incorporation dramatically reduces the energy splitting between Ca emission from the interface and bulk layers, indicating structural changes well below the surface. Subsurface incorporation of oxygen is confirmed by the presence of O K-S P diffraction peaks. These results may explain the differences observed in situ and ex situ measurements of CaF\textsubscript{2}/Si(111) interface properties.

Work supported by the U.S. Dept. of Energy.

11:00 am  EM-FrM9 A Study of Thermal Oxidation of Rough Silicon Surfaces, Q. Liu, L. Spanos, and E. A. Irene, Department of Chemistry, University of North Carolina, Chapel Hill, NC 27599.

As the thickness of the gate oxide is projected to be on the order of 5 nm, the magnitude of interface roughness at the nm scale becomes a large fraction of the device dimensions. Therefore it is crucial to control the atomic scale structure of an interface. We studied the oxidation of rough silicon surfaces by Spectroscopic Immersion Ellipsometry (SIE) and Atomic Force Microscopy (AFM). Essentially, SIE uses liquids that refractive index match to the films, thereby optically removing the films and consequently increase the sensitivity at the interface. We found that as the thickness of the thermally grown SiO\textsubscript{2} overlayer increases, the average radius of the crystalline silicon protrusions (roughness) at the interface decreases as measured using both SIE and AFM. A fractal analysis which characterizes the irregularity and complexity of a surface is concordant and furthermore shown a simpler surface resulting from oxidation. The frequency spec-
traction of AFM images give important information about how the interface roughness changes with oxidation, and these results are in agreement with the Kelvin equation which predicts that small features are more reactive.


Interfacial studies and growth initiation studies are necessary to help predict the morphology and structure of the resulting epilayer and to determine under which conditions heteroepitaxial growth is favored. In this study we investigate the evolution of the growth morphology of AlN thin films on sapphire substrates. The growth initiation and interface structure are correlated with the surface morphology of the AlN film. Thin films of aluminum nitride (AlN) were grown on sapphire (0001) and sapphire (1102) substrates by plasma source molecular beam epitaxy (PSMBE). AlN film quality is correlated with substrate temperature and bias effects. The substrate temperature was varied between 400°C and 800°C. The acceleration bias was varied between 0 V and −20 V dc. Interface reconstruction and growth kinetics of thin overlayers are investigated using transmission electron microscopy (TEM). Periodic measurements during the growth stages of AlN were performed by high resolution atomic force microscopy (AFM) by transferring under vacuum to an AFM analysis chamber. Local structure at the interface is correlated with growth mode. Our current AFM and TEM studies on c-plane and a-plane AlN films grown at 600°C indicate that the growth mode is a 3-D mechanism. Layer-by-layer (2D) growth is highly desirable and favored at lower substrate temperatures (400°C) under bias conditions of approximately −10 V dc. AFM and TEM studies of the temperature and bias effects on interfacial growth will be presented.

VACUUM METALLURGY
Room A106 – Session VM-FrM

Surface Engineering for Wear and Corrosion Protection

8:20 am VM-FrM1 Friction and Wear of Gas Lubricated SiC/ Mo Couples in Sliding Contact, J. L. Singer, Th. le Mogne, Ch. Donnet and J. M. Martin, *U.S. Naval Research Laboratory, Washington, DC, USA* and **Centre de Lyon, Ecoilly, France.**

Friction tests were performed in a UHV chamber with a SiC pin against a Mo flat exposed to various gases. At p = 13 Pa (10⁻¹ Torr), friction coefficients were between 0.1 and 0.2 with SO₂ and O₂ gas and less than 0.01 with H₂S gas. Tests performed at p < 10⁻³ Pa (<10⁻² Torr) after H₂S exposure gave friction coefficients of 1. In-situ XPS analysis indicated the gas reacted layers on Mo were less than 1 nm thick. In-situ Auger analysis of Mo wear tracks showed tribo-reacted surface films of Mo oxysulfide, Mo oxide and Mo sulfide for the three gases, respectively, and transfer films of Si oxide for SO₂ and O₂ exposures. In SiC wear tracks, transfer films of Mo oxide were seen after sliding in SO₂ and O₂; in addition, Si oxide and Si sulfide were detected. Microscopy of wear tracks showed more wear debris in the SO₂ and O₂ tests than in the H₂S tests; EDX and TEM identified the debris as metallic Mo. Gas/solid reactions and lubrication processes responsible for up to three orders of magnitude reduction in the friction coefficient will be discussed.

8:40 am VM-FrM2 Microstructural Effects on Tribological Properties of Electron Enhanced Magnetron Sputtered Coatings, J. M. Schneider, A. C. Rebholz and A. Matthews, The Research Centre in Surface Engineering, The University of Hull, Hull, HU6 7RX, UK, D. B. Lewis, Materials Research Institute, Sheffield Hallam University, Sheffield S1 1WB, UK.

X-ray diffraction measurements on tungsten carbo-nitride and titanium carbo-nitride coatings produced in an electron enhanced unbalanced magnetron system were performed. The films were deposited onto steel substrates with an Argon background pressure in the range of 3 × 10⁻³ mbar and introducing a gas mixture of nitrogen and acetylene in different ratios, which alters the carbon to nitrogen ratio in the coating. The chemical composition of the film was analysed by means of GDOES. The peak positions, integral breadth and shape parameters were determined by X-ray diffraction. The tribological performance under abrasive and adhesive wear have been determined and correlated to micro structure and phase composition.

9:00 am VM-FrM3 Coatings Defects on Corrosion Behavior of Hard Nitride Coatings on Steel, Y. B. Wang, M. S. Wong, R. Krueger, W. D. Sproul, and T. J. Barlow, BIRL Industrial Research Laboratory, Northwestern University, Evanston, IL 60201.

The corrosion behavior of reactively sputtered niobium nitride (NbN) on 1018 carbon steel was investigated in a deaerated solution of sulfuric acid and sodium chloride using electrochemical corrosion tests. In general, NbN coatings, similar to other hard nitride coatings such as TiN and ZrN, exhibited high corrosion resistance and reduced the corrosion rate of the base metal by a factor of 1000. The defects in the coatings have a dominant effect on the corrosion behavior of the coated samples. By studying the variation of the corrosion potentials and corrosion current densities of coated samples with time, the influence of the coating defects on the corrosion behavior became evident. The results showed that the corrosion process consisted of three stages related to the initial interaction and nucleation and growth of corrosion pits. All of the observed corrosion pits on the tested coated samples corresponded to the positions of the preexisting defects on the coating surfaces before the test. The effects of several types of defects were studied.

9:20 am VM-FrM4 Thermo-Mechanical and Chemical Properties of SiC-C Functionally Gradient Coatings on C/C Composites, M. R. Richardson*, A. C. Richards, M. Taya*, and F. S. Otuchi, Department of Materials Science and Engineering, and *Department of Mechanical Engineering, University of Washington, Seattle, WA 98195.

Functionally Gradient Coatings (FGC) represent a new class of composite materials in which the material composition and/or structure has been intentionally graded to overcome interface discontinuities. When depositing FGCs for applications requiring a homogeneous coating over the entire surface of the material, conventional CVD techniques produce gaps which compromise the coating integrity. We have developed an innovative cold-wall Chemical Vapor Deposition (CVD) apparatus specifically designed to overcome these problems. A key feature of the design is to employ electromagnetic fields to simultaneously heat and levitate the substrate. This technique has been applied to the fabrication SiC-C functionally gradient coatings on graphite and C/C composites. By controlling the flow rate of the reactant gases, SiCl₄, CH₄ and H₂, a gradient layer can be grown with any compositional profile, to any desired thickness. Thermo-mechanical, chemical, and oxidation properties of the SiC-C FGCs are then characterized in order to assess the coating reliability for use in high temperature oxidizing environments.

9:40 am VM-FrM5 Plasma-based Surface Engineering Processes for Wear and Corrosion Protection, A. Matthews, A. Leyland, B. Dorn, P. R. Stevenson, M. Bin-Sudin, C. Rebholz, A. Voevodin, J. Schneider, Research Centre in Surface Engineering, University of Hull, HULL, HU6 7RX, UK.

In a study of wear and corrosion coatings usage in an industrial economy (Ref. 1) it was shown that the advanced plasma or ion-assisted PVD and CVD methods account for less than 0.5% of the market by value. This is surprising, given the widely reported benefits of these new coating methods. The lack of market penetration by these processes is due to several factors, amongst which is their perceived unsuitability for cheaper substrate materials such as low alloy steels, and their relatively higher cost. This paper discusses how recent developments in plasma-based coating technologies are beginning to extend these processes into new applications sectors. These developments include hybrid methods, in which two or more processes are combined (e.g. PVD plus plasma diffusion treatment or PVD plus electroless nickel coating), or the development of new tougher PVD or CVD coatings which can be applied to relatively soft substrates (e.g. thick Ti/TiN multilayer coatings or multilayer ceramic/DLC coatings). Wear and corrosion data obtained with these coatings is reported, and new applications sectors identified.

INVITED


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Conventional CVD and PVD processes for deposition of wear-resistant coatings have been shown not to be highly effective for improvement of service life of TiC or TiCN based cerments with a Ni-Mo binder. A new technology for deposition of multilayer wear-resistant coatings onto cerments of the TiCN-Ni-Mo or TiCN-WC-Ni-Mo systems is developed. The technology is based on an interaction between the cerments and chromium vapors carried out under a special regime in vacuum. The coatings deposited by use of the process are composed of an upper hard carbide layer and an intermediate thin metal layer. The carbide layer is composed of a mixture of (Cr, Ni) 7C3 and (Cr, Ni) 23C6 and provides improved wear-resistance of coated cermet cutting inserts. The interlayer is composed of Ni-based solid solution and serves as a barrier for penetration of a crack from the carbide layer into the cermet substrate; it also reduces a level of residual stresses in the carbide layer and eliminates surface defects. Because of formation of a large diffusional zone between the coating and the substrate the coating has extremely high adhesion and cannot be removed from the substrate without failure of the coating itself. Substantial improvement in tool life of cermet indexable cutting inserts coupled with maintenance or even slight increase of transverse rupture strength, toughness and performance strength of the TiCN-based cerments is achieved by use of the technology developed. The coated cerments have more than an order of magnitude higher corrosion resistance and far better high-temperature oxidation resistance compared with the uncoated ones. The new technology has been introduced in industry on a large scale and the coated cermet cutting inserts have up to 3 times higher tool life being tested under real operation conditions.

10:40 am VM-FrM8 Fluoroplastic Coating of Cold Drying—“TETRON”, V. Ju. Demin, V. I. Rakhovsky.

The new type of protection coating has been developed. It possesses all the advantages of ordinary fluoroplastic coatings, but it has better adhesion and does not require any thermal treatment. “TETRON” is a composition based on organosoluble fluorocopolymers with specific modifiers—anhesion promoters. The coating is applied on cleaned metal surface (iron, carbon steel, aluminum) by dipping, spraying or brush and dried at room temperature.

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A manufacturer and OEM supplier of standard and custom High Vacuum systems. Systems include Evaporation; Thermal and Electron Beam, Sputtering; Magnetron and Ion Beam, Ion Beam etch, and combinations. Systems are complemented by CHA’s full line of Fixturing and other components.

COMDEL INC.
126 Sohier Road
Beverly, MA 01915
Contact: Theodore E. Johnson Jr.
Tel: 508-927-3144
Fax: 508-922-8205


COMMONWEALTH SCIENTIFIC
500 Pendleton Street
Alexandria, VA 22314
Contact: Claire Moss
Tel: 703-548-0800
Fax: 703-548-7405

Will be displaying its complete line of ion beam sources and systems. Applications include Ion-Assisted Deposition, Sample PreCleaning, Ion Beam Etching, and Reactive Ion beam etching. CSCs gridded sources range from 3 to 38cm in size to accommodate any application. New products to be displayed this year include the Cathodic Arc Source and the 1 Meter Linear Gridless Ion Source.

COOKE VACUUM PRODUCTS
13 Merritt Street
S. Norwalk, CT 06854
Contact: Jerry P. Santucci
Tel: 203-853-9500
Fax: 203-838-9553

Displayed this year will be Model CV301, a Thermal Evaporation System, high vacuum valves, traps, feedthroughs and other associated vacuum components.

CTI-CRYOGENICS
Nine Hampshire Street
Mansfield, MA 02048
Contact: Susan Pieroni
Tel: 508-337-5104
Fax: 508-337-5169

The On-Board® vacuum control system expands upon traditional ideas about vacuum management. Its unique, total integration of microprocessor-controlled pump operations with the overall vacuum system, significantly improves performance and economics. Real-time pump monitoring and control maximizes process repeatability and uptime, reduces regeneration time significantly, and provides predictive diagnostics, for single and multiple pumps. Modularity supports easy upgrade and process-specific applications flexibility for cryopumps, water-vapor pumps, and turbopumps.

CVC PRODUCTS
525 Lee Road
P.O. Box 1886
Rochester, NY 14603-1886
Contact: Tim Sladden
Tel: 716-458-2550
Fax: 716-458-0424

Supplier of MESC cluster tools for integrated thin film processes. Also offers systems for physical vapor deposition (PVD), plasma enhanced chemical vapor deposition (PECVD), rapid thermal processing (RTP). CVC offers the Connexion™ a fully MESC compliant modular cluster tool for VLSI fabrication. Founded in 1934, CVC continues to serve the world semiconductor and data storage markets from our base in Rochester, NY, and regional technical centers in Fremont, CA, Leuven, Belgium, Hong Kong and Osaka, Japan.
CVI INC. #526, 528
P.O. Box 2138
Columbus, OH 43216
Contact: Kenneth F. Wilson
Tel: 614-876-7381
Fax: 614-876-5648

A designer and manufacturer of vacuum and cryogenic sys-
tems and components. Included are cryopumps (6" to 48"
diameter), Mini-refrigerators, vacuum jacketed cryogenic pip-
ing and valves, high pressure cryogenic pumps and LNG fuel
systems. We also include turnkey thermal vacuum test facili-
ties, helium refrigerator/liquefiers and miscellaneous systems.

DANIELSON ASSOCIATES #817, 819
1989A University Lane
Lisle, IL 60532
Contact: Antonio Garza
Tel: 708-960-0086
Fax: 708-960-0546

Manufactures innovative products for the high vacuum indus-
try, designed to solve common vacuum technology problems
in an uncommon way. Products include 27 models of oil-free
TriBody, Barodym, Spiradyr, and Cyclodym vacuum pumps,
six models of the high and ultra-high vacuum Sorbodyn bulk
getter pumps, two models of the ultra-high vacuum Sorbion
good getter pump, Omnibar portable leak sensor, and the
patented Phototron device.

DENTON VACUUM, INC. #837, 839
1259 N. Church Street
Moorestown, NJ 08057
Contact: Sharron Williams
Tel: 609-439-9100
Fax: 609-439-9111

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from its laboratory line of economical evaporation and sput-
tering systems to its powerful, flexible and fully automated
load-lock systems and production coating systems. On dis-
play will be a broad array of subsystems such as the CC-102R
cold cathode ion source, the SD-10 optical monitor, as well as
an exciting assortment of electrical and mechanical feedthroughs. Stop by and peruse valuable technical literature
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DIGITAL INSTRUMENTS #511, 513
520 E. Montecito Street
Santa Barbara, CA 93103
Contact: Terry Mehr
Tel: 805-899-3380
Fax: 805-899-3392

Manufactures a complete line of NanoScope® scanning probe
microscopes (SPMs). The Dimension™ 3000 Atomic Force
Microscope (AFM) offers the complete range of AFM tech-
niques for small or large samples and analyzes samples up to
8" in diameter in air or liquid with manual and automated
stepping for scanning multiple areas of your sample. It's new
TrakScan™ scanning system makes setup simple and pro-
vides superior images for the full range of scanning tech-
niques including Tapping Mode™ magnetic force, lateral
force, electrochemistry and others. The MultiMode™ AFM is
a high resolution scanning probe microscope. It supports both
AFM and scanning tunneling microscopy (STM) in conven-
tional and electrochemical operation. Samples can imaged in
different modes including contact, non-contact, lateral force,
LiftMode™, magnetic force and Tapping Mode™.

DUNIWAY STOCKROOM #524
1600 N. Shoreline Blvd.
Mountain View, CA 94043
Contact: Bob Friedrich
Tel: 415-969-8811
Fax: 415-965-0764

Replacement parts, both new and rebuilt, for pumps, leak
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Bakeable leak valve, sapphire seat type - in stock. Ion pumps
new and rebuilt, also rebuilding service. Catalog available.
The last 6 pages show surplus equipment, rebuilt equal to
new, including prices.

DYNAVAC #927
30 Woodrock Road
Weymouth, MA 02189
Contact: Liz Claflin
Tel: 617-337-4111
Fax: 617-337-5145

Designs and manufactures systems for a wide range of process
requirements. Our major product line includes systems for
space simulation, optical coating, metallizing, sputtering,
evaporation and medical labware processing. Retrofitting ser-
vice is also offered to refurbish existing systems or upgrade
process capabilities with our supervisory control system.

EBARA TECHNOLOGIES #1020, 1022
3560 Bassett Street
Santa Clara, CA 95054
Contact: Kathy Baro
Tel: 408-496-2825
Fax: 408-496-2801

Manufacturer of clean, dry vacuum products including dry
roughing pumps for CVD, etch, and load lock applications
and Dry Gas Scrubbers. Cryopumps for Sputter, Ion Implant,
MBE, and general vacuum applications. Magnetically levitat-
ed turbos and CryTurbo™ with high water pumping. Data
acquisition and diagnostics for all products.
EDWARDS HIGH VACUUM INT’L. #700, 702
301 Ballardvale Street
Wilmington, MA 01887
Contact: Lisa Doody
Tel: 508-658-5410
Fax: 508-658-7969

Displaying its full line of RV Mechanical Vacuum Pumps, Scroll Drypump, Active Vacuum Gauges, EXT Turbopumps, STP Turbopumps, Barocel Pressure Transducers, Leak Detectors, Vacuum Coating Systems, and a complete array of Vacuum Hardware and Accessories.

ELNIK SYSTEMS #617, 619
3 Edison Place
Fairfield, NJ 07004
Contact: Claus J. Joens
Tel: 201-882-8033
Fax: 201-882-8037

Will exhibit: 100% Oil Free Roots Pump Stations that contain Diaphragm Pumps with pumping speeds from 6 to 30 CFM and ultimate vacuums from 10 - 2 and 10 - 4 Torr. 100% Oil Free Diaphragm Pumps with pump speeds from 1.4 to 4.6 CFM and ultimate vacuum ranges of 1.5 Torr. These pumps are available in Aluminum/Vitron or chemically resistant PTFE versions. High Temperature/High Vacuum Furnaces with ultimate vacuum capability of 10 - 9 Torr and fully integrated computer operating system.

ELSEVIER SCIENCE INC. #648
655 Avenue of the Americas
New York, NY 10010
Contact: Sandra Pierre-Lys
Tel: 212-633-3758
Fax: 212-633-3764

Will exhibit journals and books dealing with physics and chemistry of surface and analytical tool, vacuum processing of materials and surface technology. Free sample copies are available of journals including Surface Science, Applied Surface Science, Surface Science Reports, Progress in Surface Science and Vacuum. Detailed information about the alerting service in Condensed Matter Science called CoDAS, will be available.

ENGELHARD CORPORATION #842
101 Wood Avenue
Iselin, NJ 08830-0770
Contact: D.A. Toenshoff
Tel: 908-205-5772
Fax: 908-205-7476

Specialists in precious metal sputter targets for all segments of science and industry. Worldwide producers and suppliers of precious metals. Provides full service capability from refining, reclamation, working, and financing. Introducing a newly expanded sputter coating capability for the production of enhanced precious metal coatings on wire, ribbon, and foil. This capability brings to all industries an opportunity to benefit from the unique characteristics of precious metal with tailor-made physical properties associated with base metals.

ENI #547
100 Highpower Road
Rochester, NY 14623
Contact: Philip A. LaTulipe
Tel: 716-292-7437
Fax: 716-427-7839

Will display a DC plasma generator designed for high-power sputtering of hard disks and flat panel displays. Also ideal for semiconductor wafer sputtering and deposition, the DCG-100 features major industry advances in repeatability, and arc control. Also on display: 13.56MHz RF generators for plasma etching, sputtering and deposition.

E.T. SYSTEMS/ELECTROTECH #1023
3350 Scott Blvd., Bldg. 8
Santa Clara, CA 95054
Contact: Lynn Tocci
Tel: 408-727-5501
Fax: 408-988-6385

300 Series, Multiplex Cluster Plasma Systems will be displayed. These systems provide plasma processing solutions for R&D, pilot production, and full scale production. Available in PECVD, PECVD, and PVD.

EVAC INTERNATIONAL #918
215 River Vale Road
River Vale, NJ 07675
Contact: Ruth Tschudin
Tel: 201-666-8558
Fax: 201-666-8470

Exhibiting our chain clamps, flanges, fittings and seals for high vacuum and UHV. Our chain clamps are designed to provide high, even clamping force with time-saving one- and two-fold convenience. They are especially suitable for metal seals, i.e. aluminum knife-edge seals, bakeable to 150-degrees C. New product: glass components NW 10…125.

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Buys and sells all types of vacuum related equipment as well as all types of related oils. We sell and service leak detectors and equipment utilized in glass and semiconductor manufacturing. Please call us for a free catalogue.
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Tel: 412-967-5763
Fax: 412-963-6578

Featuring quadrupole mass spectrometers and systems including: quadrupole power supplies with mass ranges up to 4000 amu; quadrupole mass filters - 9.5 mm, 16 mm and 19 mm diameter quadrupole rods; Electron impact ionizers - axial crossbeam and direct inlet; and the electronics of quadrupole mass spectrometers. All are used in applications such as: thermal desorption, surface analysis, molecular beam and cluster chemistry. Also exhibiting the MS-250™ - high performance gas analyzer. This high performance quadrupole mass spectrometer is designed for general purpose analytical requirements.

FEI CO. #430, 432
7451 NE Evergreen Pkwy.
Hillsboro, OR 97124
Contact: Andree Kraker
Tel: 503-640-7500
Fax: 503-640-7502

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FERRAN SCIENTIFIC #1017,1019
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San Diego, CA 92121
Contact: Brian Sutherland
Tel: 619-792-2332
Fax: 619-792-0065

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6 Pinckney Street
Boston, MA 02114
Contact: Paula L. Becker
Tel: 617-227-1133
Fax: 617-742-0686

Offers savings, with new, lowered pricing, on the following vacuum system supply items: Quartz crystals, single and dual sensor heads with shutters, electron beam gun replacement parts, FT704 and DC704 diffusion pump fluids, Du Pont Krytox and other mechanical pump fluids, ionization gauge tubes, and thermocouple gauge tubes.

FISON'S INSTRUMENTS #500,501,502,503,504,505
55 Cherry Hill Drive
Beverly, MA 01915
Contact: Jacky Kieras
Tel: 508-524-1000
Fax: 508-524-1019

Manufacturer of equipment designed to meet applications from research to quality control: includes surface analysis spectrometers for ESCA, Auger, SIMS and complete MBE systems.

FUJI SEIKI INC. #900
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N. Chelmsford, MA 01863
Contact: Eckhard Beier
Tel: 508-251-1365
Fax: 508-251-1365

Total Dry oil-free vacuum pumps are designed to replace small oil-sealed vacuum pumps. Their double sealed design, guarantees that no gases can leak into or out of the pumps, even when not operating. Gases can be pumped or circulated without contamination from oil, air, or nitrogen. They are suitable for leak detectors, mass spectrometers, electron microscopes, instruments, load-locks, and general vacuum systems.

GELLER MICROANALYTICAL #806
One Intercontinental Way
Peabody, MA 01960-3885
Contact: Joseph D. Geller
Tel: 508-535-5595
Fax: 508-535-7653
Offers products and analytical services to the technical community. Products include NIST and NPL traceable magnification reference standards for atomic force, optical and scanning electron microscopy; and reference materials for surface analysis and microprobe techniques; high vacuum dessicators; and metallographic equipment. Analytical services include Auger electron spectroscopy, SEM, X-ray, Electron Microprobe, profilometry and metallography.

**GFE GES. FUR ELEKTROMETALLURGIE**

45 Hoefener Str.
Nuernberg D-90431
GERMANY
Contact: Kalb Werner
Tel: 49 911 9315 311
Fax: 49 911 314 980

As an integrated producer of high purity metals, alloys, ceramics and cermets GfE offers a wide variety of coating materials for thin film technology. The targets, cathodes, slugs and granules are used in electronics, optics, flat glass coating and for wear and decorative applications.

**GNB CORPORATION**

29393 Pacific Street
Hayward, CA 94544
Contact: Marsha Braaten
Tel: 510-537-4722
Fax: 510-537-4729

Designs and fabricates application-specific and standard vacuum equipment: Chambers -- Custom and Semi-custom, round, square, in-line and cluster. Valves -- Custom and standard, 0.5" to 144" free aperture, slat, gate and poppet. Components -- Bellows, viewportis, spools, baseplates, and manifolds. Integrated Components -- Load-locks with valves, valves with traps.

**HAUZER VAC-TEC**

5400 Spine Road
Boulder, CO 80301
Contact: Robert S. Clarke
Tel: 303-530-0144
Fax: 303-530-0262

Manufactures a full range of PVD production coating systems. Representing Hauzer Techno Coating Europe, B.V. of The Netherlands, Hauzer Vac-Tec addresses both wear-resistant and decorative coating markets. Hauzer's proprietary Arc Bond Sputtering® (ABStm) unit combines cathodic arc and unbalanced magnetron processes to enhance both adhesion and finish quality.

**HELICOFLEX CO.**

P.O. Box 9889
Columbia, SC 29290
Contact: Ryan McCall
Tel: 803-783-1880
Fax: 803-783-4279

Specializes in high performance sealing systems for Ultra-High Vacuum and high purity chemicals. The Helicoflex Delta® Seal is an all metal, low load spring energized seal that provides Helium sealing at 1x10^-12 cc/sec from -272°C to 700°C. The seal fits standard ISO flanges and is available in custom shapes and sizes. Applications include: quartz windows, cryogenics, radioactive environments and high power RF joints. The Helicoflex Quick Disconnect System: QDS® is available for ISO KF fittings.

**HIDEN ANALYTICAL LTD.**

231 Europa Blvd.
Warrington, WA5 5TN
United Kingdom
Contact: Peter Hatton
Tel: 44 925 445225
Fax: 44 925 416518

Manufactures quadrupole mass spectrometers for Vacuum, Gas, Surface and Plasma analysis. Hiden's exhibit includes the EQP plasma analyser, the new auto-analysis Langmuir probe, live RGA demonstrations and details of a new range of quadrupoles for the scientific user. U.S.A. distribution and service by AVC, Inc. headquarter in Pittsburgh, PA.

**GRANVILLE-PHILLIPS**

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Tel: 303-443-7660
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Featuring the new STABIL-ION® Vacuum Measurement System, which helps remove significant waste in vacuum processing caused by widely used Bayard-Alpert and inverted magnetron type ionization gauges. The STABIL-ION® gauge provides stability of calibration approximately ten times better than older technology gauges—giving dependable process repeatability and reduced defects.
HUNTINGTON MECHANICAL LAB. #201,203
1040 L'Avenida
Mountain View, CA 94043
Contact: Bruce Harley
Tel: 415-964-3223
Fax: 415-964-6153

An integrated supplier of UHV components hardware including valves, roughing components and viewports as well as positioners, feedthorugh and custom chambers. Also offering prototype and high volume production support services for custom or standard UHV requirements including engineering design assistance.

IBM ANALYTICAL SERVICES #825
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Hopewell Junction, NY 12533
Contact: Kevin Hutchings
Tel: 914-892-4556
Fax: 914-892-2003

A world-leading technological resource now available to analyze, evaluate, and help improve your company's products or industrial processes. Our professionals have the first-hand knowledge it takes to understand your needs, solve your problems, and work with you as technical peers. For your general analytical requirements, we perform: chemical, structural, and surface analysis; metallurgy and microscopy. Stop by and see us at booth #825 to discuss your needs.

INLAND VACUUM #841
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P.O. Box 373
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Tel: 716-293-3330
Fax: 716-293-3903

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Tel: 805-522-9040
Fax: 805-522-6017

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INTEGRATED VACUUM TECHNOLOGY #647
505 Violet Street
Golden, CO 80401
Contact: William L. Goldsworth
Tel: 303-278-7271
Fax: 303-278-4922

Designs, and manufactures cathodes for sputter deposition, they produce custom cathodes, their standard cathodes, or subcontract manufacture other designs. They own and use sputtering cathode technology which was formerly that of Vac-Tee systems. Offering proven cathodes for sputtering in RF and DC and magnetic materials.

ION TECH INC. #829, 831
2330 East Prospect
Fort Collins, CO 80525
Contact: Gerald Isaacson
Tel: 303-221-1807
Fax: 503-493-1439

Available equipment ranges from linear, round DC and filamentless RF ion beam sources, which can be fitted to existing systems; to complete optical deposition or DLC systems. Systems can be designed to meet any specific customer requirements in producing various high quality thin films.

JEOL USA, INC. #311
11 Dearborn Road
Peabody, MA 01960
Contact: Mike Kersker
Tel: 508-535-5900
Fax: 508-536-2205

Manufactures a full line of surface analysis instrumentation including UHV-STMs featuring a high temperature sample heating stage, Scanning Auger Microprobes with ESCA attachments and ultra high resolution, low voltage, field emission SEMs.

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Nesconset, NY 11767
Contact: A.J. Kozyrski
Tel: 516-360-3970
Fax: 516-360-3973

Manufacturers of high vacuum equipment and components. Standard catalog items include: portable pumping stations, valve controllers, foreline traps, ASA, CF, NW, ISO flanges. Featuring a wide selection of valves, the stainless steel family 3/8" thru 2" having a mtbf of 500,000 cycles on stem seal. Custom chambers, manifolds and systems quoted to customer specifications.

KIMBALL PHYSICS #810, 812
311 Kimball Hill Road
Wilton, NH 03086
Contact: Faye M. Bigarel
Tel: 603-878-1616
Fax: 603-878-3700

UHV electron sources, ion sources, Systems: 5 eV to 100 keV. 10-15 A to 1 A; _m to m diameters. Applications: surface physics, RHEED, ESD, MBE, heating, space physics, semiconductor processing, microscopy, etc. UHV components: cathodes, cathode mounts, Wehnelt, and cartridges; Faraday cups, multi-CF fittings, phosphor screens, eV parts, insulators, materials.

KRATOS ANALYTICAL #ISLAND B
535 East Crescent Avenue
Ramsey, NJ 07446
Contact: David Surman
Tel: 201-825-7500
Fax: 201-825-8659

Developer and supplier of surface analysis instrumentation. On display will be the latest in high performance, small area x-ray photoelectron and Auger electron spectroscopy equipment. Also on display will be the Vision Data System for instrument control and data processing.

L.D.S. VACUUM PRODUCTS #429
9807 Sunshine Lane
Altamonte Springs, FL 32714
Contact: A. Greer
Tel: 407-862-4643
Fax: 407-862-8723

Introducing the 911AM Industrial helium leak detector. This rugged leak detector is designed to test products in continuous production for both gross and fine leaks during a single test. See our line of calibrated leaks including our exclusive Cal-Link data tracking program. A complete line of vacuum components is featured.

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2840 Bay Road
P.O. Box 371
Redwood City, CA 94064
Contact: Chuck Craft
Tel: 415-369-6734
Fax: 415-369-0728

Manufacturer of viewports, glass-to-metal tubular seals, double-ended or domed adaptors, stainless steel bellows-to-glass, and fiberoptic and electrical feedthroughs, usable in vacuum systems and bakable to 400°C. Flanges, accessories and viewport repairs offered. Custom glass-to-metal seal requests welcome.

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Tel: 412-233-4200
Fax: 412-233-4275

USA agents for Vacuum Generators' high precision, UHV manipulators; VG Microtech's excellent surface science components (XPS, AES, UPS, ARUPS, LEED & RHEED); and VG Quadrupoles' complete line of quadrupoles. Additional displays include: sputter systems, sources and materials; electrical and motion feedthroughs; flanges and hardware; multi-sensor vacuum gauges and Inficon deposition monitors.

LEYBOLD INFICON INC. #737,739,741,743
Two Technology Place
East Syracuse, NY 13057
Contact: Betty Ann Kram
Tel: 315-434-1126
Fax: 315-437-3803
Transpector® AGM (Aggressive Gas Monitor) offers dependable process monitoring for aggressive gas environments. It provides valid data from in-situ gas analysis for improved process control, increased yield, higher quality, and reduced downtime. Designed specifically for chemical vapor deposition (CVD) processes, the Transpector AGM uses a patented sampling method the Virtual Value™.

LEYBOLD TECHNOLOGIES  #740, 742
120 Post Road
Enfield, CT 06082
Contact: Tim Day
Tel: 203-741-2267
Fax: 203-741-0267

Manufacturer of vacuum coating systems employing EBPVD, sputtering, evaporation, PECVD, for applications including microelectronics, decorative and functional coatings, optics, architectural and automotive glass, data storage media and thin film heads, web coating, CD mastering, metallizers, and replication lines, and the deposition of active matrix LCD's.

LEYBOLD VACUUM PRODUCTS  #736, 738
5700 Mellon Road
Export, PA 15632-8900
Contact: Eugene Fuselier
Tel: 412-327-5700
Fax: 412-733-5960

New product Sogevac SV25 Single Stage pump vacuum. Other products include ceramic ball bearing and magnetic bearing TMP's and TMP pumping systems for instruments and research, rotary vane pumps for standard and corrosive applications, Cluster tool video demonstrates vacuum technology capabilities. Ask about worldwide service.

LK TECHNOLOGIES  #904
3910 Roll Avenue
Bloomington, IN 47403
Contact: Greg Hepfer
Tel: 812-332-4449
Fax: 812-332-4493

High Resolution Electron Energy Loss Spectrometers (HREELS): New Model ELS3000 based on the KFA, Julich design at 1 meV resolution and LK2000 at 3 meV resolution. Also exhibiting our line of ion guns, Auger spectrometers and scanning tunneling microscopes.

LUXTRON CORPORATION  #328
2775 Northwestern Parkway
Santa Clara, CA 95051-0941
Contact: William Kolbeck
Tel: 408-727-1600
Fax: 408-727-1677

Optical fiber temperature measurement and control instrumentation. Offers non-contact temperature measurement from -200 to 4,000°C with resolution to 0.01°C.

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Tel: 203-792-8797
Fax: 203-792-7097

Will be exhibiting it's line of RF and microwave power systems and components. Our display will include the Series PS-3 & PB-3 low cost RF plasma power systems, the Series AT automatic impedance matching networks, the Delta Glow™ high energy plasma source and, the American Industrial Microwave™ line of 915 MHz/2.45 GHz microwave power generators, tuners, and components.

MASS-VAC, INC.  #823
247 Rangeway Road
N. Billerica, MA 01862-0359
Contact: Herb Gatti
Tel: 508-667-2393
Fax: 508-671-0014

MV products manufactures a complete line of vacuum inlet traps, oil mist eliminators, oil filtration systems, degassing systems and central source vacuum systems. A variety of filter and trapping elements are available for laboratory and production applications.

MAT-VAC TECHNOLOGY  #920
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Flagler Beach, FL 32136
Contact: Mary Sales
Tel: 904-439-7003
Fax: 904-439-7004
Offers a complete line of vacuum related products including: sputtering targets, evaporation sources, target bonding, backing plates, ETERNA™ 2", 4" & 8" planar magnetron sputtering cathodes for conventional and self-sustaining sputtering. New AC/DC magnetron power supply will be introduced. Sputtering cathode upgrades/retrofits. Sputtering process optimization.

MATERIALS RESEARCH GROUP
12441 West 49th Avenue, Suite 2
Wheat Ridge, CO 80033-1927
Contact: Pawan K. Bhat
Tel: 303-425-6688
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A thin film technology company, MRG specializes in the design and fabrication of UHV systems for various processing applications with guaranteed materials performance. MRG's products include PECVD and sputtering cluster tool systems, PV lamination, large area light sources, PV production plant design and integration, custom coatings, and product specific research.

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Tel: 510-887-6100
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Tel: 609-924-3011
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#309

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Tel: 415-965-8205  
Fax: 415-965-8207

Provides innovative product solutions to meet the needs of ultrahigh vacuum, semiconductor manufacturing, and surface analysis customers. Surface/Interface specializes in "ultra-clean" components for vacuum and manufacturing applications, analytical systems and software for surface analysis and highly pure, characterized reference materials for materials characterization and failure analysis laboratories. S/I also offers custom products and consulting services to industry and the materials science community.

SURFACE SCIENCE SPECTRA  
150 West Iowa Avenue  
Suite 104  
Sunnyvale, CA 94086  
Contact: Melissa Beers  
Tel: 408-737-0285  
Fax: 408-737-9529

An official journal of the AVS published by the American Institute of Physics, is devoted to archiving XPS, AES, and SIMS spectra of technological and scientific interest. Published in both a hard copy journal and in a digitized data format on disk, the journal is an evolving database of standardized, peer-reviewed spectra and related sample and instrument data.

SWAGELOK CO.  
31400 Aurora Road  
Solon, OH 44139  
Contact: John Burton  
Tel: 216-349-5934  
Fax: 216-349-5843

Tube fittings, valves, quick connects, flexible hoses and fluids & gas system components rated from ultra-high vacuum to 10,000 p.s.i. Temperatures from cryogenic to 1,200_F.

SYCON INSTRUMENTS  
6757 Kinne Street  
East Syracuse, NY 13057  
Contact: Gwen Stell  
Tel: 315-463-5297  
Fax: 315-463-5298

Displaying its complete line of Thin Film Deposition Monitors and Controllers utilizing the quartz crystal sensing techniques. A complete line of HV and UHV sensors and shutters for these products will also be displayed. A new deposition monitor based on the principle of Atomic Absorption will also be displayed for continuous monitoring of film deposition. A multi-pocket E-Beam Source Indexer for the control of 4 x 6 pocket E-Beam Guns will also be displayed.

T-M VACUUM PRODUCTS INC.  
630 S. Warrington Ave.  
Cinnaminson, NJ 08077  
Contact: John Strada  
Tel: 609-829-2000  
Fax: 609-829-0990

Presents equipment available from thin film deposition (sputter E-beam, etc.) and from the vacuum oven and furnace systems line including inert gas glove box/purification systems. Controls and new advancements can be seen and discussed along with custom and modified standard systems.

TARGET MATERIALS, INC.  
1145 Chesapeake Avenue  
Columbus, OH 43212  
Contact: J.R. Gaines  
Tel: 614-486-0261  
Fax: 614-486-0912
Manufactures high quality sputtering targets including indium tin oxide, aluminum, tantalum, niobium, zirconium, zinc oxide and high temperature superconductors. We also provide bonding, backing plates and toll deposition services. Visit our booth to pick-up our new price list and display magnets featuring our '800' numbers, or call 1-800-292-8639.

TECHNICAL INSTRUMENT CO. #1000
348 Sixth Street
San Francisco, CA 94103
Contact: Francis E. Lundy
Tel: 415-431-8231
Fax: 415-431-6491

The Rasterscope® from DME is an ultra-high vacuum scanning tunneling microscope for the fundamental study of both static and dynamic structures. The Rasterscope UHV-STM was developed in collaboration with leading scientists, and is designed to be flexible and uncomplicated in a compact rigid system. It is extremely insensitive to vibration, and has a simple damping system. Typically, images are recorded in two to ten seconds.

TECHNOTRADE INTERNATIONAL #447,449,451
P.O. Box 543
Londonderry, NH 03053-0543
Contact: Albrecht Auwaerter
Tel: 603-437-2991
Fax: 603-437-3091

Introduces the MECO® Chain, an innovative chain clamp for fast, reliable and economical connection of pipes, and Pink GmbH, a German vacuum engineering firm, with products ranging from standard vacuum components to the management and implementation of special projects according to customer specification, and Wolf feedthroughs for Sub-D connections.

TECHWARE SYSTEMS #319, 321
#100-12051 Horseshoe Way
Richmond, BC V7A 4V4
Canada
Contact: Mark Hanna
Tel: 604-271-2000
Fax: 604-275-8572

Showing their latest hardware and software products for advanced equipment control of thin film process applications. Cluster tool controls, and updated graphical user interface, PC-based process programming and networking capabilities will be featured. Our controls are designed for single chamber, multi-chamber and in-line systems applications.

TELEDYNE BROWN ENGINEERING #607, 609
804 Newcombe Avenue
Hampton, VA 23669
Contact: James F. Pouchot
Tel: 804-723-6531
Fax: 804-723-3925

Displaying the newly introduced miniaturized silicon based thermal vacuum sensor and gauge which covers the range of 760 Torr to 1x10^-4 Torr with a linear output signal and digital readout. This sensor can be mounted in any orientation, is very rugged and possesses a very fast response to pressure variations. In addition, our complete line of mass flow meters and controllers will be available.

TELEMARK #923
51 Whitney Place
Fremont, CA 94531
Contact: Gerald Henderson
Tel: 510-770-8700
Fax: 510-770-8879

Produces electron beam guns, power supplies and other pvd components. New products include low cost 3kw sources, 6kw UHV sources and related power supplies. An affiliate, SC Technology, produces multi-wavelength in situ monitors that measure and control film thickness and develop rate of photoresist at multiple locations on track coaters; in situ film thickness measurement systems for cd's, FPD's; full spectral OES diagnostic systems that characterize and identify spectral signatures of plasmas and light sources.

TELEVAC #324
2400 Philmont Avenue
Huntingdon Valley, PA 19006
Contact: Richard Glazewski
Tel: 215-938-4444
Fax: 215-947-7464

Will the introduction of their new line of Intelevac™ active gauges, continues its tradition of dependable innovation. The Intelevac™ Extended Range Cold Cathode Gauge operates over the 1.3 x 10^-2 to 10.1 Torr range. The Televac Convection Gauge covers the 10.3 to 10.13 Torr vacuum range.

TEMESCAL #408
2700 Maxwell Way
P.O. Box 2529
Fairfield, CA 94558
Contact: Brian J. Kaemmer
Tel: 707-423-2620
Fax: 707-425-1706

Manufactures high quality thin film components and deposition systems. They will be introducing and exhibiting several
new products this year including: SuperSource2 Modular Electron Beam Source; TRC-3460, Turret Electron Beam Source Rotation Controller; VSW-1090, Variable Wave From Sweep Controller; Temescal's Patent High Performance Electron Beam Emitter Assembly.

TENCOR INSTRUMENTS
2400 Charleston Road
Mountain View, CA 94043
Contact: Gail Nishimura
Tel: 415-988-4313
Fax: 415-968-9482

Surface profiling systems for a variety of applications and budgets providing comprehensive surface analysis on even very soft films. Guaranteed repeatability ensures highly accurate measurements. Large sample profiler for flat panel displays. Thin film stress measurement systems for analysis at temperatures from -65 to 900°C and automated film stress measurement system with radial stress mapping.

TERRANOVA SCIENTIFIC
5841 Bell Road
Auburn, CA 95602
Contact: Ron Paitch
Tel: 916-889-1100
Fax: 916-269-2877

High-performance, low-cost vacuum gauge controllers include: new Model 934 and 934-UHV Vacuum Gauge Controller with three digital displays, for ion gauge and two CONVECTRON™ gauges; covering 10-11 torr to 1000 torr. Also showing are Model 935 Ion/TC Gauge Controller, Model 914A digital TC gauge, measuring 10-3 torr to 1000 torr and Model 916 CONVECTRON™ Gauge Controller. CONVECTRON™ is a trademark of Granville-Phillips Company.

TFS TECHNOLOGIES
3700 Osuna NE
Suite 605
Albuquerque, NM 87109
Contact: John Hornkohl
Tel: 505-344-3979
Fax: 505-344-1976

Carries a large inventory of used and rebuilt vacuum equipment in New Mexico. We service, rebuild, and sell evaporation systems, sputtering systems, turbo pumps, cryopumps, vacuum valves, baffles, bell jars, collars, fittings, ion gauges, TC gauges, and leak detectors. Large coating chambers, mechanical pumps, from 1-7000 cfm.

THERMIONICS LAB.
22815 Sutro Street
P.O. Box 3711
Hayward, CA 94540
Contact: John Brooks
Tel: 510-538-3304
Fax: 510-538-2889

Vacuum systems, components and hardware with a 5 year guarantee. UHV manipulators; sample introduction, handling, and transfer, with heating and cooling, differentially pumped rotary seals and mechanical feed throughs. MBE systems, components for PLD, MBE and PLD/MBE combined processes. R-HEED (15-50 KeV), new CCD based R-HEED imaging and analysis system. Hanks HMe e-guns, 3-20kW, evaporation systems, power supplies, ion pumps and systems. PyraFlat flanges, gate valves, all-metal valves, T.C. gauges and digital/analog controllers.

TOPOMETRIX
5403 Betsy Ross Drive
Santa Clara, CA 95054
Contact: Tim Van Slambruck
Tel: 408-982-9700
Fax: 408-982-9751

Manufactures and distributes worldwide a complete line of scanning probe microscopes, including the Aurora Near-Field Scanning Optical Microscope, the Explorer LifeSciences SPM (for integration with an inverted optical microscope) and the Voyager Semiconductor Wafer Analyzer. These complete research workstations feature TopoMetrix' Windows based SPMLab acquisition and analysis software.

TYLAN GENERAL
9577 Chesapeake Drive
San Diego, CA 92123
Contact: Leslie Helsel
Tel: 619-571-1222
Fax: 619-576-1703

Produces precision gas flow & pressure measurement and control instrumentation. Displaying capacitance diaphragm gauges, adaptive pressure control systems, motorized throttling valves, mass flow controllers and meters, and ultra clean gas distribution systems.

U-C COMPONENTS, INC.
410 Logue Avenue
Mountain View, CA 94043-4019
Contact: Ron Anderson
Tel: 415-964-3827
Fax: 415-964-0216

All vented and non-vented fasteners are 300 series stainless steel and chemically cleaned for high vacuum use. We offer
stainless, MoS₂, W₅₉, and graphite coatings, as well as nickel and silver platings. Over 5,000 different items in stock. Special orders are available upon request. Fully illustrated, free catalog available.

ULVAC TECHNOLOGIES
6 Riverside Drive
Andover, MA 01810
Contact: Karen Blandford-Anderson
Tel: 508-686-7550
Fax: 508-689-6300

Will be displaying the company's new 8'' RIE and downstream microwave asher technology used in the Phoenix which renders residuals on the wafer surface DI water soluble. Also exhibiting will be information on the Z-1000 Multi-chamber sputtering system, which has been designed for advanced VLSI and ULSI production applications. In addition, Ulvac's patented DI water treatment technology and Materials including: sputter targets, evaporation sources and ultra-fine powders will be promoted.

US THIN FILM PRODUCTS
1999 S. Bascom Avenue
Suite 405
Campbell, CA 95008
Contact: Blair D. Kott
Tel: 408-371-6900
Fax: 408-359-1739

Manufacturer of 1" through 4" diameter planar magnetron sputtering sources; also provides special sources and multiple source systems on a common flange. High temperature (950°C) resistive substrate heater for use in UHV to 0₂ atmosphere. Gen-A-Torr, a cryopump regeneration gas purge monitor.

UTI INSTRUMENTS
2030 Fortune Drive
Suite C
San Jose, CA 95131
Contact: Glynis Finsterbusch
Tel: 408-428-9400
Fax: 408-428-0823

Quadrupole mass analysis systems from UHV to atmospheric pressure for equipment monitoring, process monitoring, process control, process development, and cylinder/house gas analysis. Featuring QualiTorr Remote™ for simultaneous monitoring of multiple stations/process systems at any combination of pressures analyzed. Key feature: Parallel operation of multiple analyzer stations, independent data output style (rate of rise, baseline, calibration, leak check, selected peak monitoring, etc.) at each display station, any combination of open or closed ion-source analyzers, and remote 486 computer control.

VACUUM ENGINEERING & MATERIALS
P.O. Box 4480
Santa Clara, CA 95056
Contact: Jack Kavanagh
Tel: 408-986-8900
Fax: 408-986-8980

Manufactures sputtering targets, evaporation materials, evaporation cones, hearth liners and backing plates. We furnish a large selection of high purity metals, intermetallics, dielectrics and cerments. Sputtering targets are manufactured to customers exact specifications to size and purity. Purities are available from 99 to 99.999% upon request.

VACUUM INC.
5541 Central Avenue
Boulder, CO 80301
Contact: Ted Van Vorous
Tel: 303-444-8750
Fax: 503-444-0104

Introduce a number of new products for 1994 including Random Arc Sources, the Ohmega-10 Barrel Coater, added cathodes in the Micro-PM/3 line of magnetrons; revised UHV cathodes for R/D, the Ohmega-SM Surface mount Resistor Coater and a line of throttle plates.

VACUUM RESEARCH CORP.
2401 Smallman Street
Pittsburgh, PA 15222
Contact: John F. Hartnett
Tel: 412-261-7630
Fax: 412-261-7220

Small size, low cost Dry Roughing Pumps. 5 to 32 CFM, 20 mTorr ultimate pressure. Bellows sealed aluminum gate valves with metal bonnet seal, Viton gate and Con Flat ports up to 10 inch. diaphragm, Pirani and thermocouple gauges from 10⁻³ to 1500 Torr digital display, analog output. Explosion proof gauge tubes.

VACUUM TECHNOLOGY
1003 Alvin Weinberg Drive
Oak Ridge, TN 37830
Contact: George Solomon
Tel: 615-481-3342
Fax: 615-481-3788

Manufactures the AERO VAC 1000TM, an innovative and rugged mass spectrometer/RGA featuring our AERO SCAN™ system for true computer control and data acquisition/analysis using either a PC or Mac. AERO SCAN™ provides all the functions you want, including storage, recall, true calibration, library, and spectral interpretation. Our new AERO SCAN 1200 and 1600 systems with all these functions plus process-parameter controls, turn the UTI, Extrel, VG and other quadrupoles into versatile PC or Mac controlled RGA's.
VARIAN ASSOCIATES  
121 Hartwell Ave.  
Lexington, MA 02173  
Contact: Ron Stanton  
Tel: 800-882-7426  
Fax: 617-860-5437

Will exhibit maintenance free, ceramic bearing turbo pumps in a wide range of models. Varian's UHV products will include ion pumps and controllers, valves and other hardware. A complete line of vacuum instrumentation is on display measuring pressure from two atmospheres down to 5 x 10^-12 Torr. Also on display will be a host of leak detectors including dry portables and cabinet models as well as the ultra-portable helium sniffer, Helitest®.

VSW LIMITED  
Graeme House, Chorlton  
Manchester M21 1AQ  
ENGLAND  
Contact: Mike Brayford  
Tel: 44 61 881 6213  
Fax: 44 61 881 4624

Will be featuring their wide range of surface science components. These will include the well established CLASS range of hemispherical analysers, the HIB1000 EELS system, ARIES components for photoemission studies and excitation sources. As well as the existing components the latest developments in surface science systems, detectors, and software will be shown.

VAT, INC.  
600 West Cummings Park  
Woburn, MA 01801  
Contact: John Freeman  
Tel: 617-935-1446  
Fax: 617-935-3940

Will display vacuum valves for pump-isolation, load-locks, throttling, soft-starts and other applications from atmosphere to extreme UHV. Included are aluminum and stainless steel gate valves, angle valves, and valves for fast cycling, 2 million cycles, low-vibration, or particle sensitive applications.

WELCH VACUUM TECHNOLOGY  
7300 N. Linder Avenue  
Skokie, IL 60077  
Contact: Joseph A. Saxe  
Tel: 708-676-8800  
Fax: 708-677-8606

Vacuum pumps for instruments and laboratory applications. Introducing Chemstar™ vacuum pump for pumping corrosive gases such as HCl, C12, HNO3, etc. in laboratory applications. Also featuring a complete line of belt and direct-drive vacuum pumps and accessories.

VEEKO INSTRUMENTS  
Terminal Drive  
Plainview, NY 11803  
Contact: Fran Brennan  
Tel: 516-349-8300  
Fax: 516-349-8321

Featuring surface measurement and precision ion etching for microelectronic and industrial applications. Worldwide sales/service offices are located in the United States, Europe and Asia Pacific.

JOHN WILEY & SONS, INC.  
605 Third Avenue  
New York, NY 10158  
Contact: Sally Sanford  
Tel: 212-850-6000  
Fax: 212-850-6799

Publishers since 1807, offers a diverse selection of professional & reference books & journals. Stop by our booth to browse through our latest vacuum science publications.

VSI VACUUM SCIENCE INSTRUMENTS GMBH  
Auguste-Viktoria-Strabe 16  
Wiesbaden 65185  
Contact: Karin Neukirchen  
Tel: 49 611 990450  
Fax: 49 611 376145

Will be exhibiting our reverse view ErLEED optics with digital controlled power supply for highest performance in LEED and AES applications, the real time image processing system for quantitative LEED measurements. AIDA-PC and the ErEELS 31 toroidal electron loss spectrometer (resolution <2meV at 8pA guaranteed) with rotatable analyser and large scattering chamber.

WILLIAMS ADVANCED MATERIALS  
2978 Main Street  
Buffalo, NY 14214  
Contact: Richard Sager  
Tel: 716-837-1000  
Fax: 716-833-2926

Fully integrated manufacturer specializing in high purity precious metal sputtering and evaporation materials. The company has full analytical capabilities including: Mass Spec, ICP, SEM, Atomic Absorber and modern chemical laboratory. A complete on site chemical refining facility supports quick turnaround and customer material recycling programs.
Specializing in Ametek/Dupont helium leak detectors - new, used, upgrades & components. Stainless steel vacuum chambers - custom, to your specifications. Glass/stainless steel bell jars, new, repair, or recalibration of your helium leak standards, technical glassblowing, vacuum bake-out, heliarc welding, helium leak testing.

Multilayer and multi-constituent materials analysis by spectroscopic ellipsometry. In situ real-time data acquisition and analysis, as well as research grade ex situ variable angle spectroscopic ellipsometry (vase). Both fully automated, computer controlled, 250 nm to 1700 nm spectral range. Measure film thickness, optical constants, alloy fractions, surface and interfacial roundness.

A selection of new books on display include: Monte Carlo Methods in AB Initio Quantum Chemistry (Hammond), Physical Chemistry of Solids (Franzen), and Microwave Properties of Magnetic Films (Vittoria). Sample copies of our new journal Surface Review and Letters (SRL) will be available. World Scientific will discount all conference book orders.

Displaying vacuum equipment support instruments, such as electron beam sweep controls, analog vacuum gauge controls, and microprocessor based valve controller electronics. Also showing electron beam guns, expendable items for electron beam guns, crystal monitors and other vacuum equipment.
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FUTURE SYMPOSIUM LOCATIONS

1995	October 16–20
    Minneapolis Convention Center
    Minneapolis, MN

1996	October 14–18
    Philadelphia Convention Center
    Philadelphia, PA

1997	October 20–24, 1997
    San Jose Convention Center
    San Jose, CA

1998	November 2–6
    Baltimore Convention Center
    Baltimore, MD

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<td>MoM</td>
<td>AS Imaging and Small Area Analysis</td>
<td>VT Total Pressure Gauging</td>
<td>TFVM Cubic Boron Nitride and Other Ultra-Hard Films I</td>
<td>BI Cell-Solid Surface Interactions</td>
<td>EM Materials for Device Integration</td>
<td>PS Plasma Etching &amp; Deposition</td>
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<tr>
<td>MoA</td>
<td>AS Data Processing and Reference Methods</td>
<td>VT Partial Pressure Analysis, and Leak Detection</td>
<td>TFVM</td>
<td>BI</td>
<td>EM Thin Film Heterostructures</td>
<td>PS Plasma Deposition</td>
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<tr>
<td>TuA</td>
<td>ASSS Electrochemistry and Liquid/Solid Interfaces</td>
<td>VT Vacuum System Outgassing and Cleaning</td>
<td>TFVM Diamond, Cubic Boron Nitride and Other Ultra-Hard Films II</td>
<td>BI The Biosensor-Biology Interface</td>
<td>EM Silicon-based Optoelectronics</td>
<td>PS Advanced Plasma Reactors</td>
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<td>TuP</td>
<td>WeM Quantitative Analysis, Emphasizing Angle-Resolved XPS</td>
<td>SSEM Semiconductor Surface Reactions I</td>
<td>TF Optical, Piezoelectric and Ferroelectric Films</td>
<td>BINS Artificial Cellular Assemblies</td>
<td>EM Heterostructures for Optoelectronics</td>
<td>PS Plasma Diagnostics</td>
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<td>WeA</td>
<td>AS Depth Profiling</td>
<td>SSEM Semiconductor Reactions II</td>
<td>TF Deposition &amp; Characterization Techniques of Nanostructures in Thin Films</td>
<td>VM Thin Film Microstructure Evolution</td>
<td>EM Wide-bandgap Nitrides</td>
<td>PS Plasma Induced Charging and Conformational Effects</td>
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<td>WeP</td>
<td>ThM Self-Assembled Monolayers</td>
<td>EMSS Semiconductor Surface Reactions III</td>
<td>TFVM Energetic Condensation: Processes, Properties and Products</td>
<td>VM Pulsed Laser &amp; Pulsed Ion Tech. for Film Deposition &amp; Surface Modification</td>
<td>EM Optical Diagnostics for Materials Processing</td>
<td>PS Charge Free Processing</td>
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<tr>
<td>ThA</td>
<td>AS Polymer/Organic Surfaces</td>
<td>PS2 Target Fabrication for Inertial Confinement Fusion</td>
<td>TF Thin Films for Energy Conversion and Efficiency/Active Films</td>
<td>VM Manufacturing Technology for Coatings</td>
<td>EM Materials for Nanostructures</td>
<td>PSI Plasma Surface Interactions</td>
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<td>FrM Adhesion and Adhesive Bonding</td>
<td>TF In Situ Thin Film Characterization</td>
<td>VM Surface Engineering for Wear and Corrosion Protection</td>
<td>EM Interface Characterization</td>
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**NS1** NANO 3 Plenary Session

**POSTER SESSIONS**

**MS** Manufacturing Overview and Environmental Issues

**MS** Manufacturing Equipment - A

**MS** Manufacturing Equipment - B

**MS** Diagnostics, Sensors, and Control

**MS** Micro-Contamination and Defects

**MSV** Vacuum Process Control for Manufacturing

**NS1** Nanostructure Fabrication and Atomic Scale Manipulation of Surfaces

**NS2** Novel Materials and Methods for Nanofabrication

**NSB1** Biology at the Nanoscale: I
The American Vacuum Society is pleased to welcome you as a participant in the 41st Annual Symposium. We hope that you will be satisfied by the content of the meeting whether your main interest lies in the technical sessions, which contain over 1,000 papers, the short courses, the equipment exhibit, the companions’ program or the special events and functions which are scheduled throughout the week. The Board of Directors of the Society, through the Program and Local Arrangements Committees, strives to make the National Symposium meet the needs of the community which utilizes all aspects of vacuum science and technology. To assist the Board in meeting this objective, suggestions for improvement in any aspect should be directed to Patricia Thiel, Iowa State University, Department of Chemistry, Ames, IA 50011, 515/294-8985, or Harry Meyer, Martin Marietta Energy Systems Inc., PO Box 2009, Oak Ridge, TN 38831, 615/576-3866 who are respectively chairs of the Program and Local Arrangements Committees for the

42nd NATIONAL SYMPOSIUM
to be held at the
MINNEAPOLIS CONVENTION CENTER
MINNEAPOLIS, MN
OCTOBER 16–20, 1995

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