The Electrochemical Society, Inc.

181st Meeting Program

Including:
State-of-the-Art Program on Compound Semiconductors XVI
Fullerenes: Chemistry, Physics, and New Directions
Quantum Confinement
Micromachining and Microstructures
Electronics/Dielectric Science & Technology Joint Recent News Papers

Adam's Mark Hotel
St. Louis, Missouri
May 17-22, 1992
ST. LOUIS, MO, MEETING ORGANIZATION

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Printed in the United States of America
Fullerenes: Chemistry, Physics and New Directions

K. M. Kadish and R. S. Ruoff

The Fullerene Symposium is organized with the intention of providing a forum for the presentation of latest developments on these fascinating new allotropes of carbon. Papers on recent advances or on the state of the field are solicited in the following areas; Fundamental understanding of the Physical Properties and Structures, Synthesis and separation, Chemical reactions and new derivative, Charge transfer reactions and electrochemistry, Conductivity and super conductivity, Possible applications and New directions.
St. Louis — The Gateway City

When the French fur trader Pierre Laclede came upon the swirling confluence of the Mississippi and Missouri Rivers in 1764, he instantly recognized its potential as a trading center. Landing at the first available prime spot south of the joining of the two rivers, he founded the village of St. Louis. In the 1800s St. Louis grew, and heavy river traffic between New Orleans and St. Paul made the city a major port. The city became known as the "Gateway West." It was from here that the explorers Lewis and Clark started out on their westward expedition.

The Gateway Arch, the 630 foot high stainless steel monument that dominates the downtown skyline, commemorates the role of St. Louis in the nation's surge westward. Beneath the Gateway Arch is the Museum of Westward Expansion which chronicles 19th century St. Louis history with displays of covered wagons, Texas longhorns, Indian ponies, and teepees. Nearby is the restored Old Courthouse, site of the famous Dred Scott decision.

St. Louis today is a bustling city of 2.5 million people, ranking in the top 15 in population among U.S. metro areas. It is the nation's second largest inland port, with barge connections to 29 American centers and, via the Mississippi and the Gulf of Mexico, to the rest of the world. It is also the nation's third largest rail center.

While not geographically located at the precise center of the U.S., St. Louis claims to be the population center of the nation. The city is accessible by air from almost anywhere in the 48 contiguous United States in three hours or less. Nearly 35 percent of the country's population lives within a 500 mile radius of St. Louis.

St. Louis has a rich musical tradition with something for everyone. For those who like classical music, there is the St. Louis Symphony, the nation's second oldest symphony orchestra. For those who like ragtime, a visit to the restored Scott Joplin house is a must. In addition to the second floor where the "King of Ragtime" lived between 1900 and 1903, there are exhibition galleries and a musical performance room. Blues and jazz buffs can hear their favorite music in the bohemian Soulard neighborhood or at Laclede's Landing.

Forest Park, a 1300 acre park (bigger than New York's Central Park) in the city proper, contains many attractions for the visitor. On its wooded grounds is the St. Louis Art Museum which was the Fine Arts Palace during the 1904 World's Fair. Also in Forest Park are the Muny Opera (an outdoor musical theater) and the Missouri Historical Society. One of the city's major attractions, the St. Louis Zoo, which is famous for displaying animals in their natural habitat, is also located in the park. The park is also home to the Missouri Botanical Garden, a 74 acre historical landmark founded by Henry Shaw in 1859. The Botanical Garden offers the Climatron, the world's first geodesic domed greenhouse which houses a tropical rain forest, the country's largest authentic Japanese garden, and an English woodland garden.

The city offers many other spots of interest. There is The Cathedral of St. Louis which is famous for its dazzling mosaic-covered domes, arches, wall panels, and ceilings. Also not to be missed is Laclede's Landing, the site of the original fur-trading settlement. Cobblestone streets and cast iron street lights surround dozens of century-old buildings housing offices, small shops and boutiques, unusual restaurants, and a collection of nightspots that make this one of the city's premiere entertainment districts. It is situated on the last remaining example of the street pattern laid out when St. Louis was founded as a French trading village in 1764.

Union Station, now a Historical Landmark, was built in 1894 when the railroad was king. In recent years the terminal fell into disrepair and was nearly torn down. The old station has been renovated from top to bottom, and the station building is now a hotel. The hotel has a breathtaking Grand Hall (the former station lobby) which features a 65 foot vaulted ceiling, gold leaf, stained glass windows, and sculptures. The Romanesque limestone facade resembles a medieval castle. The station's old train shed now contains myriad shops and restaurants. St. Louis has always been a big sports city. Busch Stadium, home to baseball's St. Louis Cardinals, contains the Sports Hall of Fame with displays on baseball, football, basketball, golf, soccer, tennis, and hockey. For those who prefer to watch ice hockey, there are the St. Louis Blues. The St. Louis Storm provides entertainment for indoor soccer fans. For those who would rather engage in sports than watch them, there are numerous parks in and around the city offering opportunities for boating, fishing, hiking, swimming, and horseback riding.

As the Gateway to the West in the 19th century, St. Louis offered travelers from the East their last opportunity for a fine meal as they prepared for the trek toward the Rocky Mountains. That tradition of great food continues today. In the many restaurants throughout the city, one will find a diversity of cuisine representing the rich and diverse cultural heritage of the area.

Your visit to St. Louis is one you are not likely to soon forget. It is a unique blend of old and new, a city rich in history and diversified entertainment. Come and enjoy all that the "Gateway City" has to offer you.
Representative George E. Brown, Jr., to Deliver The Electrochemical Society Lecture

The Honorable George E. Brown, Jr., Democrat of the 36th District (Riverside, San Bernadino, and Colton), California, has been selected to deliver The Electrochemical Society Lecture at the Plenary Session of the 181st Meeting of the Society in St. Louis, Missouri. His lecture entitled "Government Initiatives in Materials Science," will be presented at 9:00 a.m. on Monday, May 18, 1992 in Promenade Ballroom C and D, 2nd level of The Adam's Mark Hotel.

Representative Brown has been an aggressive proponent of the notions of open and free communication within the national and international science community, and a leader in our nation's efforts to broaden science and engineering career opportunities for women and minorities and to improve science literacy. In addition, his strong support for institutionalizing long-range planning and investment in science and technology distinguishes Rep. Brown as a consistently creative and supportive leader in advancing science.

First elected to the U.S. House of Representatives in 1962, he has worked to strengthen America's scientific and technological base. His participation in hearings and legislation led to the formation of the charter for the National Science Foundation in 1955. He also led the efforts to create the Office of Technology Assessment and the Office of Science and Technology Policy.

Long before it became an issue, Rep. Brown recognized that the United States needed to aggressively pursue alternative energy systems and nonpolluting technologies. He played an important role in developing a National Energy Policy in the wake of the oil embargo of 1973 and has chaired numerous hearings on solar, wind-powered, geothermal, and other forms of alternative energy systems. Most recently, legislation authored by Rep. Brown to develop new hydrogen research and development programs at the Department of Energy was signed into law by President Bush.

Representative Brown has introduced dozens of pieces of legislation to protect valuable natural ecosystems, remove cancer causing pesticides from our food supplies, and increase funding for federal environmental protection programs. He was instrumental in achieving passage of the nation's first Clean Air Act and helped to establish the Environmental Protection Agency in 1970.

A self-avowed space enthusiast, Rep. Brown has spent a considerable part of his congressional career promoting the civilian space program and working to prevent the militarization of space. Previously a member of the Science, Space and Technology Committee, Rep. Brown will serve as Chairman of this House Committee for the 102nd Congress.

Ernest B. Yeager to Receive the Vittorio de Nora Award

Ernest B. Yeager has been selected to receive the Vittorio de Nora Award for electrochemical engineering and technology. According to the citation: "The nomination of Professor Ernest B. Yeager is made in consideration of his leadership role in advancing the field of electrochemical technology as demonstrated through the foundation and growth of the Case Center for Electrochemical Sciences. In particular, his role in the development of fuel cell and battery technology through work in oxygen electrochemistry, electrocatalysis, and carbon electrochemistry is cited." He will be presented with the Award at the Vittorio de Nora Award Banquet on Tuesday, May 19, 1992 at 7:45 P.M. in the Rose Garden, 4th level of The Adam's Mark Hotel.

Dr. Yeager's address entitled "Opportunities for O2 Cathodes in Batteries, Fuel Cells, and Industrial Electrolytic Processes" will be delivered immediately following the presentation of awards at the Awards and Recognition Session to be held on Tuesday, May 19 at 4:30 P.M. in Promenade Ballroom C and D, 2nd level.

Ernest Yeager is a native of Orange, New Jersey. He received his B.A. (summa cum laude) from Montclair State College (New Jersey) in 1945 and his M.S. and Ph.D. in physical chemistry at Western Reserve University in 1946 and 1948, respectively. He then joined the faculty and advanced to the rank of Professor of Chemistry at what is now Case Western Reserve University.

From 1969 to 1972, he served as Chairman of the Chemistry Department and in 1972-1973 as Chairman of the University Faculty Senate. In 1978 he became the Director of the newly established Center for Electrochemical Sciences. In 1983 he was appointed to the Frank Hovorka Chair in Chemistry and in 1991 was given emeritus status.

Professor Yeager is a past President and Honorary Member of The Electrochemical Society which he joined in 1950. He is also a past President and Honorary Member of the International Society of Electrochemistry, a past Vice-President and Fellow of the Acoustical Society of America and a Fellow of the American Association for the Advancement of Science. His awards include the Technical Award (1954) and the Distinguished Service Award (1982) of the Cleveland Technical Societies Council, the Biennial Award of the Acoustical Society of America in 1956, the Navy Certificate of Commendation in 1972, the Acheson Medal of The Electrochemical Society in 1980, the Morley Medal of the Cleveland Section of the American Chemical Society in 1981, the Distinguished Achievement Medal of Western Reserve College in 1983, and an honorary doctorate from Montclair State College in 1985.

Professor Yeager's research publications involve many areas of electrochemistry including electrochemical kinetics, electrocatalysis, the electrochemistry of oxides, spectroscopic studies of electrochemical interfaces, lithium electrochemistry, the passivation of metals, underpotential deposition, the electrochemistry of single-crystal noble metals, and the relaxational properties of electrolytes and colloidal systems. In recent years, his research group has given particular attention to oxygen electrochemistry and to various electrocatalytic reactions including the oxidation of small organics. He is the editor or co-editor of sixteen books on electrochemistry and has published 250 papers.

His nonscientific interests include music, particularly the piano.
Ken Nobe to Receive the Henry B. Linford Award for Distinguished Teaching

Ken Nobe, Professor of Chemical Engineering at the University of California, Berkeley, has been selected to be the sixth recipient of the Henry B. Linford Award for Distinguished Teaching of The Electrochemical Society. The Award of a silver medal and a bronze replica and $1000 will be presented at the Awards and Recognition Session at the 181st Meeting of the Society in St. Louis, Missouri, Tuesday, May 19. The Linford Award is presented biennially to recognize excellence in teaching in subjects of interest to the Society. Funding is derived from an endowment by Samuel Rubin honoring the late Henry B. Linford, former Professor of Chemical Engineering at Columbia University.

Ken Nobe was born in Berkeley, California. After serving as an infantryman in Europe during World War II, he studied chemistry and chemical engineering at the University of California, Berkeley, receiving a B.S. in 1951. From 1951-1952 he was a chemical engineer at Air Reduction Research Laboratories in Murray Hill, New Jersey, working on the fabrication of copper catalysts for polyethylene for testing of its physical properties.

At the University of California, Los Angeles, where he received his Ph.D. in December 1956, he taught thermodynamics as an assistant in engineering (1955-1956), and in January 1957 became an Assistant Professor. During 1958-1959 he took an industrial leave of absence at the Ramo-Wooldridge Corp. (now TRW) in El Segundo, California. He was Chairman of the Chemical, Nuclear and Thermal Engineering Department (1978-1983), and Founding Chairman of the Chemical Engineering Department (1983-1984); he was also Chairman of the Faculty of the School of Engineering and Applied Science (1987-1988).

A member of the Society since 1962, he has served the Society as Chairman of the Southern California-Nevada Section (1965-1966); Chairman of the Corrosion Division (1978-1980); Chairman of the Corrosion Monograph Series (1980-1986) and a Corrosion Division Editor of the Journal (1967-1991). He was also Division Editor of Electrochimica Acta (1977-1985).

For the past thirty years the interests of his research group have been directed to kinetic studies of uninhibited and inhibited metal and alloy electrodissolution and, more recently, electrodeposition. He collaborated in repaired synthesis research with Manuel Baizer after the latter's retirement from Monsanto (1978) until his death in 1988. Their last collaboration was on bioelectrochemistry at the zym reactions, which was funded just before Dr. Baizer's death, and is still ongoing. In recent years, Dr. Nobe and his collaborators have focused on studies of nonlinear electrochemical instabilities during high current density breakdowns are enhanced by junctions, for trimming tunnel junction current density, and produce electrical active intermediates at the interface. He has found that MOS capacitor breakdowns are enhanced by field assisted transport of sodium ions into SiO2 when Si is negatively biased and has shown that the times to breakdown are proportional to the applied field as described by Peek's law. Based on interface studies with superconducting films, he has developed processes for preparing high quality, reproducible Nb/Nb oxide/Pb alloy Josephson tunnel junctions, for trimming tunnel junction currents by ion implantation, and for fabricating new three-terminal, nonequilibrium superconducting devices. He has studied conduction in granular NbN superconducting films using scanning tunneling microscopy at liquid helium temperatures and is currently evaluating the uniformity of deposited high Tc superconducting film properties using magneto-optic glasses.

Stanley I. Raider to Receive the Thomas D. Callinan Award of the Dielectric Science and Technology Division

Stanley I. Raider has been selected as the recipient of the 1992 Thomas D. Callinan Award of the Dielectric Science and Technology Division of The Electrochemical Society. The award will be presented at the Awards and Recognition Session on Tuesday, May 19, 1992, at 4:30 P.M. in the Promenade Ballroom C and D, 2nd level of the Adam's Mark Hotel. His award address, "Carbon Impurities at a Si/SiO2 Interface," will be given on Wednesday, May 20 at 8:15 a.m. in the Promenade Ballroom F, 2nd level.

Dr. Raider received a B.S. degree in chemistry from Brooklyn College in 1955 and a B.Ch.E. degree from Brooklyn Polytechnic Institute in 1957. During employment by the U.S. Naval Powder Plant in 1956 and by Hooker Chemical Company which he joined in 1958, he was involved in applied polymer research on coatings and rigid foams. In 1967, he earned his Ph.D. in chemistry at the State University of New York at Stony Brook. His doctoral research involved the synthesis and spectroscopic analysis of new silyl-aluminum hydride and germi hydride compounds.

In 1967, Dr. Raider joined the IBM Components Division at East Fishkill, New York. He transferred to the IBM T.J. Watson Research Center in 1975 where he is a Research Staff Member. He is presently on temporary assignment in IBM at Thornwood, New York.

Dr. Raider's research at IBM has primarily focused on novel device properties and processing relate to the chemistry that occurs at interfaces between thin insulator films and semiconducting or superconducting substrates. He was among the first to use x-ray photoelectron spectroscopy as a tool to characterize interfaces and has applied this technique to study the Si-SiO2 interface, the oxidation of silicon nitride, and the Nb/Nb oxide interface formed during Josephson tunnel barrier processing. From studies of Si-SiO2 interfacial reactions involving N2 or carbon impurities, processes were identified which affect Si oxidation kinetics, initiate interfacial reactions at local sites, and produce electrically active intermediates at the interface. He has found that MOS capacitor breakdown are enhanced by field assisted transport of sodium ions into SiO2 when Si is negatively biased and has shown that the times to breakdown are proportional to the applied field as described by Peek's law. Based on interface studies with superconducting films, he has developed processes for preparing high quality, reproducible Nb/Nb oxide/Pb alloy Josephson tunnel junctions, for trimming tunnel junction currents by ion implantation, and for fabricating new three-terminal, nonequilibrium superconducting devices. He has studied conduction in granular NbN superconducting films using scanning tunneling microscopy at liquid helium temperatures and is currently evaluating the uniformity of deposited high Tc superconducting film properties using magneto-optic glasses.

Dr. Raider has received two IBM Outstanding Contribution Awards for his work on time-dependent breakdown of metal oxides semiconductor capacitors and for developing the process that IBM used in their Josephson program to produce reproducible Josephson tunnel junctions. He is
Gottlieb S. Oehrlein to Receive the Electronics Division Award

Gottlieb S. Oehrlein, a physicist at the IBM T.J. Watson Research Center, has been selected to receive the 1992 Electronics Division Award of The Electrochemical Society at the Awards and Recognition Session to be held at 4:30 p.m. on Tuesday, May 19, 1992 in Promenade Ballroom C and D, 2nd level of the Adam's Mark Hotel.

Dr. Oehrlein will present his award address, "Surface Studies of Reactive Ion Etch Processes in Silicon Technology: From Silicidation to High Resolution Depth Profiling," at 2:00 p.m. on Monday, May 18, 1992, in the Promenade Ballroom C, 2nd level of the Adam's Mark Hotel.

Dr. Oehrlein is a Research Staff Member in the Silicon Technology Department at the IBM T.J. Watson Research Center. He received a Vordiplom degree in physics from Wuerzburg University in West Germany in 1976 and his M.S. and Ph.D. degrees in physics from the State University of New York at Albany in 1978 and 1981, respectively. He subsequently joined IBM at the T.J. Watson Research Center, where he has worked on exploratory materials and plasma-based processes.

His early research interests have included nucleation of oxygen precipitates and oxygen-related thermal donors during heat treatment of Czochralski-grown silicon, rapid thermal processing of ion implanted silicon, and enhanced diffusion mechanisms of dopants. He also has worked on the deposition and characterization of $\text{Ta}_2\text{O}_5$ for applications as the storage capacitor dielectric in DRAMs.

More recently, Dr. Oehrlein has been interested in understanding fundamental aspects of reactive ion etching (RIE), in particular the plasma-surface interactions responsible for the achievement of etch selectivity and etch directionality, which may also result in dry etch damage. He and his coworkers characterized and elucidated the mechanistic details of silicon near-surface defects and surface contamination in selective oxide RIE and other plasma processes.

A new approach to spatially resolve in situ analysis of semiconductor microstructures (using blanket surface analysis techniques) was demonstrated which exploited the topographic and insulating characteristics of the structures. This approach enabled the study of the surface chemistry of contact hole etching processes using actual structures and also made possible the first in situ studies of the chemistry of sidewall passivation layers formed in Si trench etching using fluorine-, chlorine-, and bromine-based plasmas. Additionally, using real-time ellipsometry during RIE of Si, $\text{SiO}_2$, SiGe, etc., the kinetic aspects of etch selectivity could be studied.

The combination of RIE and ellipsometry also has enabled high-resolution compositional depth profiling of ultra-thin films, e.g., oxide-nitride-oxide dielectric stacks, or determiningGe profiles in epitaxial SiGe alloy/Si superlattices.

Dr. Oehrlein has been a Fellow of the Institute of International Education (New York) and received the SUNY at Albany Chancellor's "Honors Convocation Award for Academic Excellence and A Distinguished Dissertation" in 1982. Dr. Oehrlein was awarded the 1986 Solid-State Science and Technology Young Authors Award of The Electrochemical Society. He received several IBM internal awards, including an IBM Outstanding Technical Achievement Award for his work in the area of reactive ion etching. He is a member of the American Vacuum Society, The Electrochemical Society, and the American Physical Society, and served on the program committee of the 1983 Electronic Materials Conference and the 1990 Gordon Research Conference on Plasma Chemistry. He was a Visiting Scientist at the Forsvaret Forskiningsanstalt, Linkoping, Sweden, and the Centre National d'Etudes des Telecommunications, Grenoble, France, during the summers of 1981 and 1989, respectively. In 1988 he was appointed the first visiting professor of the SUNY Albany/RPI Joint Laboratories of Advanced Materials.

Dr. Oehrlein's research has resulted in over 100 publications, numerous invited lectures at conferences and universities, and several patents. Currently, he is working on the characterization of high-density plasma surface interactions and integration of plasma-based processes in vacuum cluster tools.

Robert A. Osteryoung to Receive the Max Bredig Award in Molten Salt Chemistry of the Physical Electrochemistry Division

The Max Bredig Award will be presented to Robert A. Osteryoung at the Awards and Recognition Session to be held on Tuesday, May 19, 1992, at 4:30 p.m. in Promenade Ballroom C and D, 2nd level of the Adam's Mark Hotel. "Through the Years and Temperatures: Adventures in Molten Salt Land" is the title of his award address which he will deliver after dinner (approximately 8 p.m.) Wednesday, May 20, in the St. Louis Ballroom H, 4th level of the Adam's Mark Hotel.

Robert Osteryoung was born in Cleveland, Ohio, in 1927. He served in the U.S. Navy and then obtained the degree of B.S. in Chemistry in 1949 from Ohio University. In 1951 he received his M.S. from the University of Illinois and obtained his Ph.D. in 1954 from the same institution. From 1951 to 1952 he worked for the Harshaw Chemical Company. From 1954 through 1959 he was Assistant, then Associate, Professor of Chemistry at Rensselaer Polytechnic Institute, Troy, New York. In 1959 he joined the staff of Atomic Energy International Division of what is now Rockwell International, Los Angeles. He moved to Rockwell's Science Center Laboratory as Group Leader of Physical Chemistry in 1966 and in 1968 was named Associate Director. He also served, from 1968 to 1968, as Director of the Materials and Process Laboratory of Rockwell's Autonetics Division. He was also a Visiting Associate in Chemistry at the California Institute of Technology from 1967 to 1968.

In 1968 he was named Professor and Chairman, Department of Chemistry, Colorado State University, Ft. Collins, Colorado, where he served as Chairman through June
1978. From July 1977 to June 1978 he was on leave as a Program Manager at the Air Force Office of Scientific Research in Washington, DC. He joined the faculty of the State University of New York at Buffalo in 1979. He will move to North Carolina State University in Raleigh, North Carolina, as Research Professor in July 1992.

His research interests are in molten salt chemistry and electrochemistry, in electroanalytical chemistry, with emphasis on fast pulse voltammetric methods, and in the online use of computers in electrochemistry. He has published over 200 research papers in these areas. A past Chairman of the Gordon Research Conference on Electrochemistry, he has been an invited speaker at Gordon Research Conferences in electrochemistry, analytical chemistry, and molten salt chemistry. He has also been an invited speaker at numerous other scientific meetings in the United States and abroad.

Having joined The Electrochemical Society in 1969, he has served as Secretary-Treasurer, Vice-Chairman, and Chairman of the Physical Electrochemistry Division, and member of the Board of Directors, and currently serves on the Finance Committee. He was twice nominated as Vice-President of the Society. From 1979 to 1986 he served as a Divisional Editor of the Journal of the Electrochemical Society.

He is a past member of the Advisory Board of Analytical Chemistry, a past Chairman of the Division of Analytical Chemistry of the American Chemical Society, and is currently an Alternate Councilor of that Division to the ACS Council. As Chairman of the Division of Analytical Chemistry's Professional Status Committee, he operated the Division's Summer Intern Program for thirteen years. He was Program Chairman of the Division's 1984 Summer Symposium. He currently serves as an Associate Editor of Analytical Chemistry for electroanalytical chemistry.

He was the 1978 recipient of the Distinguished Service Award of the Colorado Section of the American Chemical Society, the 1987 recipient of the Charles N. Reilley Award in Electroanalytical Chemistry of the Society for Electroanalytical Chemistry, was the winner of the 1990 Schoelkopf Medal of the Western New York section of the American Chemical Society, and, in 1991, received the American Chemical Society's Division of Analytical Chemistry Award in Electrochemistry. He was selected as a Fellow of the Electrochemical Society in 1990.

His other professional affiliations include: founding member and past member of the Board of Directors of the Society of Electroanalytical Chemistry, the International Society of Electrochemistry, the American Chemical Society's Division of Analytical Chemistry, Phi Beta Kappa, Sigma Xi, and fellow of the American Association for the Advancement of Science.
SCHEDULE OF SYMPOSIA/SESSIONS BY DIVISIONS AND GROUPS

BATTERY

Monday, May 18, 1992
62
Joint General Session ........................................................... 1-13
Direct Electrochemical Oxidation of Methanol and Small Organic Molecules .......... 19-28

Tuesday, May 19, 1992
Joint General Session ........................................................... 14-18
Direct Electrochemical Oxidation of Methanol and Small Organic Molecules .......... 29-40

Wednesday, May 20, 1992
High Temperature Sensors ..................................................... 355-365

CORROSION

Monday, May 18, 1992
Fractals in Electrochemistry ................................................... 308-320

Tuesday, May 19, 1992
Cathodic Protection Systems .................................................... 54-61
Fractals in Electrochemistry .................................................... 321-326

Thursday, May 21, 1992
General Session ............................................................... 41-53

DIELECTRIC SCIENCE AND TECHNOLOGY

Monday, May 18, 1992
Ninth Symposium on Plasma Processing ............................................. 62-76
Second International Symposium on Reduced-Thermal-Budget Processing for the Fabrication of Microelectronic Devices .................................................. 133-144
Fifth International Symposium on Silicon-on-Insulator Technology and Devices .......... 153-161
Materials and Processing Issues for Large Scale Integrated Electronic and Photonic Arrays ............................................................. 196-204
Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface .......... 210-222
Synthesis and Processing for High-Temperature Materials in the Year 2000 .................. 341-347
Fullerenes: Chemistry, Physics and New Directions ..................................... 613 FUL-626 FUL

Tuesday, May 19, 1992
Ninth Symposium on Plasma Processing ............................................. 77-88
Second International Symposium on Reduced-Thermal-Budget Processing for the Fabrication of Microelectronic Devices .................................................. 145-152
Fifth International Symposium on Silicon-on-Insulator Technology and Devices .......... 162-173
Materials and Processing Issues for Large Scale Integrated Electronic and Photonic Arrays ............................................................. 205-209
Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface .......... 223-235
State-of-the-Art Program on Compound Semiconductors XVI ................................. 565 SOA-568 SOA
Synthesis and Processing for High-Temperature Materials in the Year 2000 .................. 348-354
Fullerenes: Chemistry, Physics and New Directions ..................................... 627 FUL-638 FUL
Micromachining and Microstructures ........................................ 596 MIC-598 MIC

Wednesday, May 20, 1992
Ninth Symposium on Plasma Processing ............................................. 89-105
Fifth International Symposium on Silicon-on-Insulator Technology and Devices .......... 174-184
Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface .......... 236-252
State-of-the-Art Program on Compound Semiconductors XVI ................................. 569 SOA-581 SOA
Joint General Session ........................................................... 269-293
Electrochemical Characterization of Thin Solid Films ....................................... 327-334
Fullerenes: Chemistry, Physics and New Directions ..................................... 639 FUL-668 FUL
Quantum Confinement .......................................................... 589 QUA-595 QUA
Micromachining and Microstructures ........................................ 599 MIC-612 MIC
<table>
<thead>
<tr>
<th>Date</th>
<th>Event</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thursday, May 21, 1992</td>
<td>Ninth Symposium on Plasma Processing</td>
<td>106-123</td>
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<tr>
<td></td>
<td>Fifth International Symposium on Silicon-on-Insulator Technology and Devices</td>
<td>185-195</td>
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<tr>
<td></td>
<td>Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface</td>
<td>253-268</td>
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<td>State-of-the-Art Program on Compound Semiconductors XVI</td>
<td>582 SOA-588 SOA</td>
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<td>Joint Recent News Paper Session</td>
<td>608 RNP-709 RNP</td>
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<td>Fullerenes: Chemistry, Physics and New Directions</td>
<td>669 FUL-684 FUL</td>
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<td>Fullerenes: Chemistry, Physics and New Directions</td>
<td>585 FUL-697 FUL</td>
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<td>ELECTRODEPOSITION</td>
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<td>Monday, May 18, 1992</td>
<td>Micromorphology in Electrocrystallization</td>
<td>465-474</td>
</tr>
<tr>
<td>Tuesday, May 19, 1992</td>
<td>Micromorphology in Electrocrystallization</td>
<td>475-481</td>
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<tr>
<td>ELECTRONICS</td>
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<td>Monday, May 18, 1992</td>
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<td>62-76</td>
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<td>133-144</td>
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<td>Fifth International Symposium on Silicon-on-Insulator Technology and Devices</td>
<td>153-161</td>
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<td>Materials and Processing Issues for Large Scale Integrated Electronic and Photonic Arrays</td>
<td>196-204</td>
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<td>Fullerenes: Chemistry, Physics and New Directions</td>
<td>627 FUL-638 FUL</td>
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<td>Micromachining and Microstructures</td>
<td>596 MIC-598 MIC</td>
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<tr>
<td>Wednesday, May 20, 1992</td>
<td>Ninth Symposium on Plasma Processing</td>
<td>89-105</td>
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<td></td>
<td>Fifth International Symposium on Silicon-on-Insulator Technology and Devices</td>
<td>174-184</td>
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<td>Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface</td>
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<td>State-of-the-Art Program on Compound Semiconductors XVI</td>
<td>589 SOA-581 SOA</td>
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<td></td>
<td>Joint General Session</td>
<td>269-293</td>
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<td>Electrochemical Characterization of Thin Solid Films</td>
<td>327-334</td>
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<td>Fullerenes: Chemistry, Physics and New Directions</td>
<td>639 FUL-668 FUL</td>
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<td>Quantum Confinement</td>
<td>589 QUA-595 QUA</td>
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<td>Micromachining and Microstructures</td>
<td>599 MIC-612 MIC</td>
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<td>Thursday, May 21, 1992</td>
<td>Ninth Symposium on Plasma Processing</td>
<td>106-123</td>
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<td>Fifth International Symposium on Silicon-on-Insulator Technology and Devices</td>
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<td>Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface</td>
<td>253-268</td>
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<td>State-of-the-Art Program on Compound Semiconductors XVI</td>
<td>582 SOA-588 SOA</td>
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<td></td>
<td>Joint Recent News Paper Session</td>
<td>698 RNP-709 RNP</td>
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<td>Fullerenes: Chemistry, Physics and New Directions</td>
<td>669 FUL-684 FUL</td>
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</tbody>
</table>
Friday, May 22, 1992

Ninth Symposium on Plasma Processing ........................................... 124-134
Fullerenes: Chemistry, Physics and New Directions ......................... 685 FUL-697 FUL

ENERGY TECHNOLOGY

Monday, May 18, 1992

Joint General Session .......................................................... 1-13
Direct Electrochemical Oxidation of Methanol and Small Organic Molecules ............................................................. 19-28
Fractals in Electrochemistry .................................................... 308-320

Tuesday, May 19, 1992

Joint General Session .......................................................... 14-18
Direct Electrochemical Oxidation of Methanol and Small Organic Molecules ............................................................. 29-40
Fractals in Electrochemistry .................................................... 321-326

Wednesday, May 20, 1992

Surface Processing in Energy Technologies .................................... 294-307
Electrochemical Characterization of Thin Solid Films ....................... 327-334

HIGH TEMPERATURE MATERIALS

Monday, May 18, 1992

Synthesis and Processing for High-Temperature Materials in the Year 2000 ............................................................. 341-347
Eighth International Symposium on Molten Salts .............................. 482-496

Tuesday, May 19, 1992

Synthesis and Processing for High-Temperature Materials in the Year 2000 ............................................................. 348-354
Eighth International Symposium on Molten Salts .............................. 497-510

Wednesday, May 20, 1992

Stability of Refractory Materials ............................................. 335-340
High Temperature Sensors ..................................................... 355-365
Eighth International Symposium on Molten Salts .............................. 511-529

Thursday, May 21, 1992

Eighth International Symposium on Molten Salts .............................. 530-543

INDUSTRIAL ELECTROLYSIS AND ELECTROCHEMICAL ENGINEERING

Monday, May 18, 1992

Electrochemistry in Mineral and Metal Processing III ...................... 366-374
Industrial Electro-Orgnic Processes .......................................... 401-407

Tuesday, May 19, 1992

Cathodic Protection Systems .................................................. 54-61
Electrochemistry in Mineral and Metal Processing III ...................... 375-383
Industrial Electro-Orgnic Processes .......................................... 408-413

Wednesday, May 20, 1992

Electrochemistry in Mineral and Metal Processing III ...................... 384-394

Thursday, May 21, 1992

Electrochemistry in Mineral and Metal Processing III ...................... 395-400

ORGANIC AND BIOLOGICAL ELECTROCHEMISTRY

Wednesday, May 20, 1992

Electro-Orgnic Synthesis with Homogeneous and Heterogeneous Catalysts ................. 414-423

Thursday, May 21, 1992

Electro-Orgnic Synthesis with Homogeneous and Heterogeneous Catalysts ................. 424-433
PHYSICAL ELECTROCHEMISTRY

Monday, May 18, 1992

Direct Electrochemical Oxidation of Methanol and Small Organic Molecules .................. 19-28
Fractals in Electrochemistry ................................................................. 308-320
General Session ......................................................................................... 447-455
Micromorphology in Electrocrystallization .............................................. 465-474
Fullerenes: Chemistry, Physics and New Directions ........................................ 613 FUL-626 FUL
Eighth International Symposium on Molten Salts ........................................ 462-496

Tuesday, May 19, 1992

Direct Electrochemical Oxidation of Methanol and Small Organic Molecules .................. 29-40
Fractals in Electrochemistry ................................................................. 321-326
General Session ......................................................................................... 456-464
Micromorphology in Electrocrystallization .............................................. 475-481
Fullerenes: Chemistry, Physics and New Directions ........................................ 627 FUL-638 FUL
Eighth International Symposium on Molten Salts ........................................ 497-510

Wednesday, May 20, 1992

Electrochemical Characterization of Thin Solid Films ........................................... 327-334
Electrochemistry of High Temperature Superconductors ..................................... 434-441
Fullerenes: Chemistry, Physics and New Directions ........................................ 639 FUL-668 FUL
Eighth International Symposium on Molten Salts ........................................ 511-529

Thursday, May 21, 1992

Electrochemistry of High Temperature Superconductors ..................................... 442-446
Fullerenes: Chemistry, Physics and New Directions ........................................ 669 FUL-684 FUL
Eighth International Symposium on Molten Salts ........................................ 530-543

Friday, May 22, 1992

Fullerenes: Chemistry, Physics and New Directions ........................................ 685 FUL-697 FUL

SENSOR

Monday, May 18, 1992

Electrochemical Sensors in Medical Science ................................................... 544-556

Tuesday, May 19, 1992

Electrochemical Sensors in Medical Science ................................................... 557-564
Micromachining and Microstructures .......................................................... 596 MIC-598 MIC

Wednesday, May 20, 1992

High Temperature Sensors ........................................................................... 355-365
Micromachining and Microstructures .......................................................... 599 MIC-612 MIC

NEW TECHNOLOGY SUBCOMMITTEE

Tuesday, May 19, 1992

Micromachining and Microstructures .......................................................... 596 MIC-598 MIC

Wednesday, May 20, 1992

Quantum Confinement .................................................................................. 589 QUA-595 QUA
Micromachining and Microstructures .......................................................... 599 MIC-612 MIC
ST. LOUIS, MISSOURI MEETING
Symposium and General Session Organizing Chairmen

Battery/Energy Technology Joint General Session

E. Gagnon
A. Landgrebe

Direct Electrochemical Oxidation of Methanol and Small Organic Molecules

J. Fenton
V. Jalan
W. O’Grady
P. Ross, Jr.

Corrosion General Session

H. Isaacs

Cathodic Protection Systems

K. Nisancioglu
R. White

Ninth Symposium on Plasma Processing

D. Hess
G. Mathad

Second International Symposium on Reduced-Thermal-Budget Processing for the Fabrication of Microelectronic Devices

J. Osenbach
G. Schwartz

Fifth International Symposium on Silicon-on-Insulator Technology and Devices

W. Bailey
S. Cristoloveanu
G. Cullen
P. Hemment
K. Izumi

Materials and Processing Issues for Large Scale Integrated Electronic and Photonic Arrays

D. Buckley
R. Enstrom
R. Levy

Second Symposium on the Physics and Chemistry of SiO₂ and the Si-SiO₂ Interface

B. Deal
C. Helms

State-of-the-Art Program on Compound Semiconductors

D. Buckley
B. Etienne
T. Kamijoh
A. Katz
V. Swaminathan
G. Valco

Electronics/Dielectric Science & Technology Joint General Session

A. Harrus
R. Jaccodine

Electronics/Dielectric Science & Technology Joint Recent News Paper Session

L. White

Surface Processing in Energy Technologies

A. Czanderna
A. Landgrebe

Fractals in Electrochemistry

J. Kaufman
J. Talbot
M. Tomkiewicz

Electrochemical Characterization of Thin Solid Films

M. Orazem
P. Sides

Stability of Refractory Materials

D. Jacobson
K. Shin

Synthesis and Processing for High-Temperature Materials for the Year 2000

M. Allendorf
J. Dismukes
J. Wagner, Jr.

High Temperature Sensors

A. Khandkar
M. Liu
W. Worrell
Electrochemistry in Mineral and Metal Processing III

P. Richardson
R. Woods

Industrial Electro-Organic Processes

C. King
P. Pintauro
N. Weinberg

Electro-Organic Synthesis with Homogeneous and Heterogeneous Catalysts

D. Peters
J. Toomey

Electrochemistry of High Temperature Superconductors

J. McDevitt

Physical Electrochemistry General Session

B. Conway

Micromorphology in Electrocrystallization

T. Hepel
G. Whitney

Fullerenes: Chemistry, Physics and New Directions

K. Kadish
R. Ruoff

Eighth International Symposium on Molten Salts

G. Blomgren
R. Gale
H. Kojima

Electrochemical Sensors in Medical Science

D. Harrison
R. Wightman

Quantum Confinement

E. Nicollian
R. Tsu

Micromachining and Microstructures

P. Barth
H. Gray
J. Zemel

PROGRAM

ST. LOUIS, MISSOURI

The Electrochemical Society, Inc.

May 17 - 22, 1992
Sunday through Friday

HOTEL INFORMATION

The 1992 Spring Meeting will be held at the Adam's Mark Hotel, Fourth and Chestnut, St. Louis, MO 63102. The telephone number for the hotel is 314-241-7400. The standard convention rates are $105 for single rooms and $118 for double rooms.

Hotel reservation cards have been sent to all members, authors of papers and Technical Session Chairmen and Vice-Chairmen. It is suggested that you use the special reservation envelope. If you call for a reservation you MUST mention that you are attending The Electrochemical Society Meeting to obtain these special convention rates. DEADLINE FOR HOTEL RESERVATIONS IS APRIL 10, 1992.

TRAVEL INFORMATION

In order to reduce expenses for attendees, TWA has been named the official carrier for this Spring Meeting. Arrangements have been made for attendees to obtain a special reduced airfare. This special airfare, which is not available elsewhere, will be guaranteed to all registrants who wish to use the special Fugazy International Travel meeting service telephone number listed below.

To make a reservation at the special reduced airfare, which can be as low as a 45% discount, you can call Fugazy International Travel directly. The phone numbers are: 800-828-4488 (anywhere in the Continental US) or 908-828-4488 in the state of New Jersey.

Call between the hours of 8:30 AM and 5:30 PM Eastern Time, Monday through Friday or 10:00 AM to 2:00 PM on Saturday. Please mention that you are attending The Electrochemical Society Meeting in St. Louis, Missouri.

REGISTRATION

The registration area will be located on the Fourth Level in the Coat Room Foyer of The Adam's Mark Hotel.

Advance Registration

Advance registration is suggested if you plan to attend the Spring Meeting. Forms are available from The Electrochemical Society, Inc., 10 South Main Street, Pennington, New Jersey 08534-2894, phone number 609-737-1902, or FAX: 609-737-2743. DEADLINE FOR RECEIPT OF ADVANCE REGISTRATION, INCLUDING PAYMENT, IS MAY 1, 1992. Written requests for refunds will be honored only if received at Society Headquarters in Pennington before May 8, 1992.

Note: Meeting registration fees and all tickets are discounted if purchased with Advance Registration.

Registration at the Meeting

The registration hours are:

<table>
<thead>
<tr>
<th>Day</th>
<th>Time</th>
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<tbody>
<tr>
<td>Sunday, May 17</td>
<td>2:00 P.M. to 7:00 P.M.</td>
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<tr>
<td>Monday, May 18</td>
<td>7:30 A.M. to 3:30 P.M.</td>
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<td>Tuesday, May 19</td>
<td>7:30 A.M. to 3:30 P.M.</td>
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<td>Wednesday, May 20</td>
<td>7:30 A.M. to 3:30 P.M.</td>
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<tr>
<td>Thursday, May 21</td>
<td>7:30 A.M. to 3:30 P.M.</td>
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<tr>
<td>Friday, May 22</td>
<td>8:00 A.M. to 10:00 A.M.</td>
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REGISTRATION FEES

All participants and attendees are required to pay a registration fee. Payment can be made by cash, check, or travelers checks in U.S. funds. Credit cards, only Mastercard or Visa, are accepted. The schedule of fees is as follows:

<table>
<thead>
<tr>
<th>Type of Registrant</th>
<th>ADVANCE Fee</th>
<th>AT MEETING Fee</th>
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</thead>
<tbody>
<tr>
<td>Members</td>
<td>$170.00</td>
<td>$190.00</td>
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<tr>
<td>Nonmembers</td>
<td>$255.00</td>
<td>$275.00</td>
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<tr>
<td>Student Members</td>
<td>$10.00</td>
<td>$10.00</td>
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<tr>
<td>Nonmember Students</td>
<td>$20.00*</td>
<td>$20.00*</td>
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<tr>
<td>Nontechnical Registrants Fee</td>
<td>$30.00</td>
<td>$40.00</td>
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<tr>
<td>Emeritus and Honorary Members</td>
<td>Complimentary</td>
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<tr>
<td>Last Day Registration (Friday only)</td>
<td>$40.00</td>
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*If a nonmember submits an Application for Admission within four months of the Meeting and is subsequently elected to membership in the Society, the difference between the nonmember and member registration fee will be applied to the 1992 dues, which are $85.00.

MEETING INFORMATION

Meeting Registration .......................... Coat Room Foyer, 4th Level
Information/Message Center................. Coat Room Foyer, 4th Level
Nontechnical Registrants Headquarters -- Rose Garden, 4th Level
Society Headquarters Office ............... Room 27, 2nd Level

GENERAL FUNCTIONS

Sunday, May 17

Sunday Evening Get-Together
An informal Sunday Evening Get-Together will be held in the St. Louis Ballroom D, Fourth Level from 7:30 to 9:30 P.M. All meeting registrants are invited to attend.

Monday, May 18

Monday--Plenary Session
At 9:00 A.M. the Plenary Session will be held in the Promenade Ballroom C & D, Second Level. The Honorable George E. Brown, Jr., California State Representative, will deliver The Electrochemical Society Lecture entitled, "Government Initiatives in Materials Science."

Monday Evening Mixer
An informal gathering will be held in the Pre-Convene, Second Level from 6:00 to 7:15 P.M. Beer, soft drinks, and snacks will be served on a complimentary basis. In addition, there will be a cash bar for those who prefer mixed drinks or wine. All meeting registrants are invited to attend.

Tuesday, May 19

Awards and Recognition Session
The Awards and Recognition Session will begin at 4:30 P.M. in the Promenade Ballroom C & D, Second Level.

At this session, the Thomas D. Callinan Award of the Dielectric Science and Technology Division will be presented to Stanley I. Raider. The Electronics Division Award will be presented to Gottlieb S. Gabriele. The Henry B. Linford Award for Distinguished Teaching will be presented to Ken Nobe.
**Committee Meetings**

Committee Meeting Rooms 21 through 29 are located on the Second Level to the left of the entrance to the Promenade Ballrooms. Committee Meeting Rooms 41 through 48 are located on the Fourth Level to the left of the entrance to the St. Louis Ballrooms.

**Sunday, May 17**

- 7:30 A.M. High Temperature Materials Division Executive Committee
- 7:30 A.M. Industrial Electrolysis and Electrochemical Engineering Division Executive Committee
- 7:30 A.M. Physical Electrochemistry Division Executive Committee
- 7:30 A.M. Dielectric Science and Technology Division Long Range Symposium Planning Committee
- 10:00 A.M. Solid State Science & Technology Award Subcommittee
- 10:30 A.M. Education Committee
- 11:30 A.M. Society Meeting Committee
- 1:30 P.M. Executive Session of the Individual Membership Committee
- 1:30 P.M. Technical Affairs Committee
- 4:00 P.M. Organic and Biological Electrochemistry Division Executive Committee

**Monday, May 18**

- 7:30 A.M. Symposium Subcommittee
- 9:00 A.M. Publication Committee
- 10:30 A.M. Executive Session of the Individual Membership Committee
- 1:30 P.M. Financial Policy Advisory Committee
- 2:30 P.M. Contributing Membership Committee
- 3:30 P.M. Organic and Biological Electrochemistry Division Mixer
- 6:00 P.M. Max Bredig Award in Molten Salt Chemistry Dinner

**Tuesday, May 19**

- 7:30 A.M. Financial Committee
- 7:30 A.M. Council of Past Presidents’ Breakfast
- 7:30 A.M. Solid State Monograph Committee
- 10:00 A.M. Ways and Means Committee
- 1:30 P.M. Contributing Membership Committee
- 3:30 P.M. Organic and Biological Electrochemistry Division Mixer
- 6:00 P.M. Max Bredig Award in Molten Salt Chemistry Dinner

**Wednesday, May 20**

- 9:00 A.M. Board of Directors’ Meeting
- 5:00 P.M. Ninth International Symposium on Molten Salts Planning Meeting

**Thursday, May 21**

- 7:30 A.M. Solid State Division/Group Chairmen’s Breakfast

**LUNCHEONS AND BUSINESS MEETINGS**

(Luncheon tickets are $13.00 in advance and $17.00 at the meeting)

**Monday, May 18**

- 12:15 P.M. Physical Electrochemistry Division (Northern Section)
- 12:15 P.M. Electronics Division
- 12:15 P.M. Industrial Electrolysis and Electrochemical Engineering Division (Northern Section)

**Tuesday, May 19**

- 12:15 P.M. Annual Society

**Wednesday, May 20**

- 12:15 P.M. Dielectric Science and Technology Division

**Monday, May 18**

- 12:15 P.M. Physical Electrochemistry Division St. Louis Ballroom D
- 12:15 P.M. Electronics Division Rose Garden
- 12:15 P.M. Industrial Electrolysis and Electrochemical Engineering Division St. Louis Ballroom E

**Tuesday, May 19**

- 12:15 P.M. Annual Society Rose Garden

**Wednesday, May 20**

- 12:15 P.M. Dielectric Science and Technology Division Rose Garden
TECHNICAL SESSION CHAIRMAN ORIENTATION

All Session Chairmen and Vice-Chairmen are urged to attend an orientation meeting on the day of their participation. The meeting is scheduled from 7:30 to 8:00 A.M., each day, in Room 24, Second Level of the Adam's Mark Hotel. All Session Chairmen and Vice-Chairmen have been sent the necessary information and materials needed to conduct their Technical Sessions. Should you have any questions or specific problems that you would like to discuss, a member of the Society Headquarters Staff will be available in Room 24 from 7:30 to 8:00 A.M. on the day of your participation.

NONTECHNICAL REGISTRANTS PROGRAM

For details of the evening social events, please refer to the category GENERAL FUNCTIONS.

The headquarters for the Nontechnical Registrants will be located in the Rose Garden Room, Fourth Level of the Adam's Mark Hotel, Monday, May 18 through Thursday, May 21. Those individuals registered as Nontechnical Registrants are cordially invited to a continental breakfast each morning from 8:30 AM to 10:00 A.M., Monday through Thursday.

During this period plans can be made with old and new acquaintances to enjoy one of the many sightseeing tours available through Images and Ideas, the exclusive tour operator for the Society's 181st Meeting. The Tour Registration Desk will be located in the Meeting Registration area on the fourth level adjacent to the escalators. The registration hours are: Sunday - 2:00 P.M. to 7:00 P.M., Monday through Wednesday - 8:00 A.M. - 10:30. Buses for all tours depart from the 4th Street Exit of the Adam's Mark Hotel.

Monday, May 18

IMAGES AND IDEAS ORIENTATION - 9:30 AM - 10:00 AM

The Images and Ideas Hostess will provide an orientation in the Rose Garden Room of all tours being offered during the week.

DISCOVER ST. LOUIS - 10:30 A.M. - 3:30 P.M.

Begin with a drive through Laclede's Landing where cobblestone streets and cast-iron streetlamps surround century-old buildings that house small shops, boutiques and unusual restaurants. Continue your tour along the historic riverfront passing boats and stern-wheelers on one side and the magnificent stainless steel Gateway Arch on the other. View St. Louis' oldest church, the Old Cathedral, recognized as the Oldest Church west of the Mississippi and the Old Courthouse, once the tallest building in St. Louis and the scene of the Dred Scott trial.

From the riverfront, your tour will proceed to St. Louis' Historic South Side, where the Anheuser-Busch, the last brewery in the United States, is located. Also in this area is the DeMenil ante-bellum Mansion, built in the 1800's and the Lemp Mansion, which Life Magazine named as one of the ten most haunted houses in the United States.

Your first stop will be Union Station. Built in 1894, this former train station is a National Historic Landmark. Enjoy lunch on your own and still have time to browse through some shops before reboarding your motorcoach to continue your tour to the Cathedral of St. Louis. A visit to this magnificent Church is definitely one of the highlights of any tour. Built in 1907, it contains one of the largest collections of mosaic art in the world.

The final stop will be the Missouri Botanical Gardens. May is the most spectacular month for viewing the gardens. This 79-acre Missouri State Botanical Garden is surrounded by old buildings and features the country's largest Japanese garden and a rose garden displaying 200 varieties of roses.

Discover some of the more popular sights of St. Louis on this incredible tour of the city. PRICE: $22.00 per person in advance, $24.00 after April 17, 1992.

Tuesday, May 19

FOX THEATRE AND SCIENCE CENTER - 10:00 A.M. - 3:00 P.M.

This tour begins with a short drive to "Fox Theatre" where arrangements have been made for tours of Fox Theatre. This tour will be led by Willy Fox, grand-nephew of his uncle. He has earned the name "The Fabulous Fox" due to the lavish interior decorations; a mixture of cultures that make it "beautifully bizarre". Once a movie palace, it is now used for entertainment featuring Broadway shows and Super Star variety shows. Riding form rock to classical, this theatre must be seen to be believed.

After lunch on your own in the Central West End, we continue the tour to St. Louis' newest jewel - the St. Louis Science Center. Opened in November of 1991, it is ranked among the top ten in the world. The four galleries represent Ecology and the Environment, Human Adventure, Technology and Space Science. Experience an earthquake or tornado, create your own rainbow, take a journey into the basic building block of life through a three-dimensional cell model, or launch a hot air balloon.

The Fox Theatre and St. Louis Science Center are spectacular sites that should not be missed by anyone visiting this wonderful city. PRICE: $24.00 per person in advance, $26.00 after April 17, 1992.

Wednesday, May 20

1904 WORLD'S FAIR REVISITED - 10:00 A.M. - 3:00 P.M.

This tour offers you the opportunity to spend the day in Forest Park, St. Louis' cultural center where our city's science center, history museum, zoo, art museum and the famous "Muny Opera" theatre are located. It was also the site of the 1904 World's Fair, one of the most famous World Fairs ever held.

The first stop will be the History Museum where the history of Missouri and St. Louis comes alive as you stroll through this museum which houses collections on the 1904 World's Fair, Charles Lindbergh, riverboats, antique guns, and period costumes.

You will also have the opportunity to visit the St. Louis Zoo and the Art Museum. The Zoo contains over 2800 animals in their natural setting and is considered one of the top zoos in the United States. See Big Cat Country, The Jungle of the Apes, and the newest addition to the zoo, "The Living World".

If you prefer the world of art, a visit to the Art Museum may be more to your liking. This impressive building sits atop Art Hill and is guarded by a 47-foot statue of St. Louis the Crusader. The museum was the Fine Arts Palace of the 1904 World's Fair, and is today considered among the top ten art museums in the country.

Enjoy lunch on your own at either the Painted Giraffe at the Zoo or in the Art Museum Cafes. PRICE: $20.00 per person in advance, $22.00 after April 17, 1992.

Thursday, May 21

HISTORIC ST. CHARLES - 10:00 A.M. - 2:00 P.M.

This tour's destination is historic St. Charles, located on the wide Missouri River. Founded in 1769, it is the oldest city on the Missouri River and was Missouri's first state capitol as well as the site of the beginning of the Lewis & Clark expedition.

Enjoy lunch on your own in one of the quaint restaurants located in this unique, historic area. You will still have time to stroll through the many craft and antique shops that line the cobblestone streets or relax in the garden of the old Missouri State Capitol. Tours can also be taken through Missouri's First State Capitol.

This tour has been designed to let you enjoy the ambiance of this historic area at your leisure. PRICE: $19.00 per person in advance, $20.00 after April 17, 1992.

DISCUSSION

No recording will be made of the oral discussions. Those contributing to the discussion of a paper and desiring their remarks to be published should send the discussion to the Publications Manager, JOURNAL OF THE ELECTROCHEMICAL SOCIETY, 10 South Main Street, Pennington, New Jersey 08534-2896. The discussion will then be referred to the author for a reply. Publication of the discussion and the comments of the author(s) depends on the publication of the paper in the JOURNAL.

Written discussion of a published paper should be submitted within two months following the publication of the article.

EMPLOYMENT SERVICES

There will be a special bulletin board in the Registration Area for employment posters. Companies desiring to recruit employees are requested to place their announcements on this special board. Please note that these announcements should be no larger than 8 1/2" x 11".

NOTE

Photographing of slides, charts, etc., will be permitted unless specifically prohibited by the speaker. PHOTO FLASH AND PHOTO FLOODS ARE PROHIBITED. TAPE RECORDINGS, EXCEPT ON BEHALF OF THE SOCIETY, ARE PROHIBITED.
<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Location</th>
<th>Speaker(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:00</td>
<td>Experimental Determination of the Transport Number of Water in Nafion 1179 Membrane</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
</tr>
<tr>
<td>10:20</td>
<td>High Utilization Gas Diffusion Electrodes for Proton Exchange Membrane Fuel Cells</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
</tr>
<tr>
<td>10:40</td>
<td>Temperature and Pressure Dependence of the Electrode Kinetics of Oxygen Reduction at the Platinum Microelectrode/Kiln Interface</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
</tr>
<tr>
<td>11:00</td>
<td>Spontaneous Hydrous Oxide Formation on Platinum and Its Relevance in Oxygen Gas Reduction</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
</tr>
<tr>
<td>11:20</td>
<td>Effect of Platinum Loading on Proton Exchange Membrane Fuel Cell Performance</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
</tr>
<tr>
<td>11:40</td>
<td>The Hydrogen Diffusion Porous Electrode Catalyzed by Tungsten Carbide</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>2:00</td>
<td>Optimization of Electrode Structure to Further Minimize the Platinum Loading in Proton Exchange Membrane Fuel Cells</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>2:20</td>
<td>A Thin Film Solid Oxide Fuel Cell Prepared Using Reactive DC Magnetron Sputtering</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>2:40</td>
<td>Effects of a Sputtered Film of Pt on Performance of Proton Exchange Membrane Fuel Cells: Electrode Kinetic and Morphological Characteristics</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>3:00</td>
<td>Mechanism of Mixed Fuel/Oxidant Solid-State Thin Film Fuel Cells</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>3:20</td>
<td>Ten-minute intermission</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>3:30</td>
<td>New Aspects in the Development of Proton Exchange Membrane Electrolyzers</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>3:50</td>
<td>Calorimetric Concentration and Mass Flow Monitor for Gas Phase Ozone</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>4:10</td>
<td>SPECF Development at the Centre for Electrochemical and Energy Research</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<tr>
<td>4:30</td>
<td>The Influence of Surface Acid-Base Properties of Pt/C Catalysts on the Electrochemical Oxidation of Methanol</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<tr>
<td>4:50</td>
<td>Carbon Supported Platinum Alloy Catalysts for Methanol Oxidation</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>5:10</td>
<td>Methanol Oxidation on CNT - R. Manoharan and J. B. Goodenough</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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**NINTH SYMPOSIUM ON PLASMA PROCESSING**

Dielectric Science and Technology/Electronics

T. O. Mantei, Chairman; S. Butler, Vice-Chairman

Promenade Ballroom C, 2nd Level

**Modeling and Mechanisms (cont’d)**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Location</th>
<th>Speaker(s)</th>
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<tbody>
<tr>
<td>10:00</td>
<td>Feature Scale Simulation of Oxide Plasma Etching</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<tr>
<td>10:20</td>
<td>Use of Overhang Test Structure to Understand RIE Lag in Oxide Etching</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>10:40</td>
<td>Modeling of Plasma Etching Reactors Including Water Heating Effects</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>11:00</td>
<td>Optimally Uniform ECR Plasma Generation for Precise Patterning</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>11:20</td>
<td>Variation of Ion Energy and Ion Flux in Various Gas Plasmas with 13.56 MHz Cathode Coupled Parallel-Plate Plasma Equipment</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<tr>
<td>11:40</td>
<td>Dependencies of Negative Ions from Pulsed Radio-Frequency Discharge</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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<td>12:00</td>
<td>Direct Electro-Oxidation of Methanol in Acid Medium at Modified Electrodes with Low Precious Metal Loadings</td>
<td>St. Louis Ballroom D, 4th Level</td>
<td>E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-Chairman</td>
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**ELECTRONICS DIVISION AWARD ADDRESS:**

Surface Studies of Reactive Ion Etching Processes in Silicon Technology: From Surface Damage to High-Resolution Depth Profiling - G. S. Oehrlein
1:00 Introduction by N. F. Schmitt, S. C. Pantelides, and R. C. Albrecht

7:30 The Effect of Wafer Backside on RTA Processing - J. W. Butler and K. Brankaer

3:00 Proximal Plasma Diagnostics for the Etching of Silicon Nitride - M. R. M. Dvornikov, K. Ikeda, and T. Tsukada

5:00 Diffusion Laser Endpoint for Trench Etch Applications - M. B. Rang and P. Ebbing

6:00 Diagnostics of an ECR Plasma Using the Langmuir Probes - Y. Nakaegawa, K. Ikeda, and T. Tsukada

SECOND INTERNATIONAL SYMPOSIUM ON REDUCED-THERMAL-BUDGET PROCESSING FOR THE FABRICATION OF MICROELECTRONIC DEVICES

Dielectric Science and Technology/Electronics

J. W. Osenbach, Chairman; G. C. Schwartz, Vice-Chairman

St Louis Ballroom G, 4th Level

10:00 Defect-Free Rapid Thermal Processing - Z. Nenyei, H. Wath, and T. Knarr

10:20 The Impact of the Wafer Back Side on RTA Processing - B. Lojek


11:00 An Estimation of Thermal Budget for Wafer Cooling from Impurity Diffusion - K. Yamaie, K. Imai, H. Kawasaki, Y. Suzui, and K. Okumura

11:20 Reliability Characterization of RTO and O2-Diluted Thin Gate Oxides - L. A. Fonseca and F.Campbellad

G. C. Schwartz, Chairman; J. W. Osenbach, Vice-Chairman

2:00 A Study of the Effect of Deposition Parameters on the Growth Rates and Microstructure of Silicon Homopitaxial Films Grown by ArF Laser-Enhanced Chemical Vapor Deposition - S. Krishnan, S. Lian, B. Fowler, L. Jung, C. Li, D. Samara, I. Manne, and S. Banejee


3:00 Fifteen-minute Intermission

3:15 A Low Resistivity Polysilicon Film Fabricated with a SiH4/H2Mixture at 350°C - J. Shizowawa, K. Yamabe, Y. Kasai, S. Miyasa, and Y. Miki


FIFTH INTERNATIONAL SYMPOSIUM ON SILICON-ON-INSULATOR TECHNOLOGY AND DEVICES

Electronics/Dielectric Science and Technology

W. Bailey, Chairman; G. W. Cullen, Vice-Chairman

Promenade Ballroom E, 2nd Level

10:00 Introductory remarks

10:10 Silicon-On-Insulator Technology and Devices - H. H. Hosack

10:50 Manufacturing of VLSI CMOS on SIMOX Substrates - J. Yue, B. Urte, K. Kueng, R. Roisen, P. Fehmer, G. Dougal, and M. Liu

11:30 High Performance Submicron CMOS/SOI for Logic and SRAM Applications - N. Haddad and L. K. Wang

J. Gautier, Chairman; S. Cristoloveanu, Vice-Chairman

Novel Device Concepts

2:05 The Implementation of a Commercial Thick Film SOI Process - K. Yallup

2:45 Body-Contacts for SOI MOSFETs - M. Mattubian

3:15 Ten-minute intermission

3:25 A 0.5 µm CMOS/SOI Technology Using Accumulation Mode Device Design - L. K. Wang, J. Salskard, A. J. Edenthal, O. Spencer, and N. Haddad

3:45 An Intelligent 500 V Power Vertical DMOS on SIMOX Substrate - F. Vogt, B. Mutterlein, and H. Vogt

4:05 Parasitic Capacitances of SOI MOSFETs - J. Chen, R. Solomon, T. Y. Chan, P. K. Ko, and C. Hu

4:25 Transient Behavior of SOI NMOS at Liquid Helium Temperatures - C. Cleaves and E. Simonen

MATERIALS AND PROCESSING ISSUES FOR LARGE-SCALE INTEGRATED ELECTRONIC AND PHOTONIC ARRAYS

Electronics/Dielectric Science and Technology

D. N. Buckley, Chairman; R. A. Levy, Vice-Chairman

Room 43, 4th Level


10:40 Microfabrication of Ultra-Small Optical Cavities - A. Soherer, E. Yablonovitch, J. L. Jewell, B. P. Van der Gun, and E. D. Beebe


N. Bousdame, Chairman; R. E. Enstrom, Vice-Chairman

2:00 Laser and Photodetector Arrays for Parallel Optical Data Link Applications - N. K. Dutta and P. R. Berger


3:00 Anisotropic Photocasting of GaAs - E. Mannheim, R. L. Sani, and R. C. Allaire

3:20 Fifteen-minute Intermission

3:35 Two-Dimensional Vertical to Surface Transmission Electro-Optinic Device Array for Optical Interconnection - K. Kasahara

4:15 Long Wavelength Infrared 129 x 128 Staring Array from AllGaAs/AllGaAs Multiquantum Well Detectors - Growth, Array Performance - V. Swaminathan
MONDAY CONTINUED


SECOND SYMPOSIUM ON THE PHYSICS AND CHEMISTRY OF SiO₂ AND THE Si-SiO₂ INTERFACE

E. A. Irene, Chairman; M. Hirose, Vice-Chairman
Promenade Ballroom F, 2nd Level

Thermal Oxidation Mechanisms and Modeling

10:00 Silicon Oxides and Silicon Oxidation - A. M. Stoneham 210
10:50 Strain Dependent Diffusion during Dry Thermal Oxidation of Crystalline Si - C. H. Bjorkman and G. Lucovsky
11:10 Oxidation of Silicon in Oxygen: Measurement of Film Thickness and Kinetics - S.-C. Kao and R. H. Doremus
11:30 Modeling Process-Dependent Thermal Silicon Dioxide (SiO₂) Films on Silicon - H. Wei, A. K. Henning, J. Stinkman, and J. Rogers

S. Rigo, Chairman; B. E. Deal, Vice-Chairman

Novel Oxidation Methods and Characterization

1:45 New Approach to Chemically Enhanced Oxidation - A. I. Jacobson 215
2:15 Kinetics of Oxidation of Silicon by Electron Cyclotron Resonance Plasma - J. Joseph
2:35 Mechanisms of Oxidation Rate Enhancement in Negative-Point Oxygen Corona Discharge Processing of SiO₂ Films on Si - L. M. Landsberger
2:55 High Pressure, Low Temperature Oxidation of Si,Ge; Promise for MOS-Quality Passivation - C. Garapartis, Y. Shigesato, and D. C. Paine
3:15 Ten-minute intermission
3:25 A New Ellipsometry Technique for Interface Analysis - M. V. Paulino, E. A. Irene, and V. A. Yakovlev
3:55 Infrared-Dichroism on a Thin Silicon Oxide Layer - S. Fujimura, K. Ishikawa, and T. Ogawa
4:35 TEM investigations of the Oxidation Kinetics of 222 Amorphous Silicon Films - M. Reiche

FRACTALS IN ELECTROCHEMISTRY

Energy Technology/Physical Electrochemistry/Corrosion

J. H. Kaufman, Chairman; M. Tomkiewicz, Vice-Chairman
Room 40, 4th Level

10:10 Introductory remarks by M. Tomkiewicz
10:15 Effects of Anisotropy on Pattern Formation in Electrochemical Deposition - F. Family and K. C. B. Chan
10:35 Interfacial Dynamics and Induced Convective Transport in Electrodeposition - D. P. Bartley
10:55 Diffusion to Patterned Electrodes - Y. Dassas and P. Duby
11:15 Electro-Convection Around Two-Dimensional Ramified Copper Aggregates - V. Fleury, J. N. Chazalviet and M. Rosso
B. Sapevol, Chairman; T. Paljosey, Vice-Chairman
2:00 Morphology Transitions in Rapid Electrodeposition - L. M. Sandor

3.00 Probing Electrocrystallization Mechanisms of Molecular Solids - A. C. Hillier and M. D. Ward
3.20 Fifteen-minute intermission
3.35 Multifractal Fluctuations in Diffusion in Disordered Systems - S. Havlin
4.00 Fractals in Electrochemical Photovoltaics - A. J. McEvoy and M. Gratzel
4.20 Morphology and Kinetics of Fractal Growth - D. B. Hibbert and S. N. Atkins
4.40 Photocatalysis on Porous Substrates - M. Tomkiewicz and H. Wang

SYNTHESIS AND PROCESSING FOR HIGH-TEMPERATURE MATERIALS IN THE YEAR 2000

High Temperature Materials/Dielectric Science and Technology

J. B. Wagner, Jr., Chairman; M. D. Allendorf, Vice-Chairman
Promenade Ballroom A, 2nd Level

10:30 Introductory remarks
10:40 Materials Research in the U.S.: The Development of a National Agenda - L. H. Schwartz
11:20 Expanding Horizons for Chemical Vapor Deposition Synthesis - K. E. Spear

J. Dismukes, Chairman; S. C. Singhal, Vice-Chairman
2:00 Chemical Vapor Deposition Process Aspects for the Year 2000 - J.-O. Carlson
2:40 Computational Analysis of Fluid Flow and Chemical Kinetics in Chemical Vapor Deposition - R. J. Kee, G. H. Evans, and M. E. Coltrin
3:20 Ten-minute intermission
3:30 Processing and Fabrication of Ceramic Composites in the 21st Century - R. W. Rice
4:10 Chemical Vapor Infiltration - T. M. Besmann, D. P. Stinton, and R. A. Lowden
4:50 High-Tech Fibers - G. G. Shibetta

ELECTROCHEMISTRY IN MINERAL AND METAL PROCESSING III

Industrial Electrolysis and Electrochemical Engineering

R. Woods, Chairman; I. Iwasaki, Vice-Chairman
St. Louis Ballroom A, 4th Level

10:00 Requirements for Industrial Collectorless Flotation - N. J. Arbiter and J. E. Gebhardt
10:30 Surface Electron Structures of Galena and Pyrite Related to Collectorless Flotation - S. Sun, D. Wang, and B. Li
11:00 Electrochemical Aspects of Cast Iron Grinding Media Wear and its Effect on Flotation - I. Iwasaki and V. Rajagopal
11:30 An Electrochemical Study of Sulfide Mineral-Grinding Medium Contact and Its Relevance to Flotation - X. Li and I. Iwasaki

G. H. Kelsall, Chairman; D. R. Nagaraj, Vice-Chairman

2:00 Mechanism of Thionocarbamates Interaction in Flotation Systems - R. H. Yoon and C. I. Basilio
2:30 The Role of Pulp Redox Potentials and Modifiers in Complex Sulfide Flotation with Dithiophosphinates - A. Gorkhen, D. R. Nagaraj, and P. J. Riccio
3:00 The Mechanism of Sulfide Depression with Functionalized Synthetic Polymers - D. R. Nagaraj, C. S. Basilio, and R. H. Yoon
3:30 Fifteen-minute intermission
4:15 Interpretation of Electrode Responses with the Help of a Multivariate Technique - B. I. Palsson and E. Oberg
MONDAY CONTINUED

INDUSTRIAL ELECTRO-ORGANIC PROCESSES
Industrial Electrolysis and Electrochemical Engineering
P. N. Pintoarao, Chairman; C. King, Vice-Chairman
Promenade Ballroom B, 2nd Level

2:00 Organic Electrolysis at Extended Area Nickel Electrodes - C. J. Brown and D. Fletcher
2:25 Oxidation of Methanol on a Metallized Polymer Electrolyte Membrane - R. Liu and P. Fedtkov
2:50 The Electrocatalytic Hydrogenation of Soybean Oil - G. Yusem and P. N. Pintoarao
3:15 Ten-minute intermission
3:25 Direct and Indirect Electrochemical Epoxidation of Olefins in a Sieve Plate Reactor - C. F. Oduoza and K. Scott
3:50 A Comparison of Some Insoluble Oxide Catalysts in the Electro-Oxidation of Thioethers in Aqueous Sulfonate Suspensions - T. C. Franklin, R. Nondinehe, and R. C. Duty
4:15 Electrochemical Oxidation of Organic Pollutants for Waste Water Treatment - Ch. Commellis

GENERAL SESSION
Physical Electrochemistry
B. E. Conway, Chairman; R. P. Buck, Vice-Chairman
Room 25, 2nd Level

10:00 The Effect of Solvent on the Simultaneous Adsorption of Anions and Cations - M. Anbu Kulandainathan and S. Venkatakrishna Iyer
10:30 Manipulation of Double Layer in Metal Insulator Electrolyte - K. Ghose
11:00 Molecular Recognition at Interfaces: Specific Binding of an Electroactive Tetrathiafulvalene (TTF) Derivative to Organosulfur Monolayers by Hydrogen Bonding - L. M. Frostman and M. D. Ward

R. P. Buck, Chairman; B. E. Conway, Vice-Chairman

2:00 Effects of Monosubstituted Phenol Additives on the Conductivity of Electrochemically Synthesized Poly(pyrrrole - M. Fukuyama, Y. Naito, T. Kojima, Y. Kudoh, and S. Yoshimura
2:30 Impedance and Voltammetric Characterization of Electrochemically Deposited (Poly)anilime Conducting Films - P. Vanysek and G. Sandl
3:00 A Cathodically PolymerizedBinuclear Cobalt Complex and its Electrochemical Reduction of Carbon Dioxide - X. Ren, S. K. Mandal, and P. Pickup
3:30 Fifteen-minute intermission
3:45 The Electrochemical Reduction of CO2 on Ag and Au Electrodes: The Effect of Trace Impurities - R. Kostecki, P. Kedzierzewski, and J. Augustynski
4:15 Redox Potential of N-Hexadecyl-N'-Methyl Viologen (d1+1)
Solubilized in Cetyltrimethylammonium Chloride Micelle - C.-W. Lee and M.-K. Oh

MICROMORPHOLOGY IN ELECTROCRYSTALLIZATION
Physical Electrochemistry/Electrodeposition
T. Hellen, Chairman; G. Whitney, Vice-Chairman
St. Louis Ballroom F, 4th Level

10:00 Introductory remarks by T. Hellen
10:10 Study of Unidirectional Crystallization of 1-D Quantum Conduction - A. R. Hillman, D. C. Lovelady, and M. Hellen
10:38 Electrodeposition of Epitaxial Films of Ag(Ag2O)2(NO3) - B. E. Breyfogle and J. A. Switzer
11:00 Ten-minute intermission

11:10 In Situ Characterization of p-Type Copper Thiocyanate Films by Raman Spectroelectrochemistry - Y. Son, N. R. de Tacconi, and K. Rajeshwar
A. R. Hillman, Chairman; G. Whitney, Vice-Chairman

2:00 Introductory remarks by A. R. Hillman
2:05 Electroosmosis, Characterization, and Modeling of Highly Efficient Polypyrrole/Pt Nanocomposite Catalysts - C. S. C. Bose, C. C. Chen, and K. Rajeshwar
2:30 Electrodeposition of Metals on Polypyrrole Coated Au/Quartz Piezoelectrodes - M. Helf, S. Perkins, and T. Hellen
2:55 Localized I/V and IZ Measurements on Conducting Poly-n-methyl Pyrrole Thin Films Performed with a Scanning Tunneling Microscope - S. Cramer
3:20 Fifteen-minute intermission
3:35 Factors Affecting Electrochemical Metallization of Insulating Substrates Precoted by Conducting Polymer Films - P. A. Ulbre, A. J. Rudge, and S. Gottlesfield
4:00 When Anodic Polymerization of Dibenzo-18-Crown-6 Leads to a New Artificial Membrane: Changes of Micromorphology upon Different Modes of Doping - J. Simonet, J. Rault-Berthelot, V. Questaligne, and L. Angely
4:25 Formation and Removal of p-SnS Patterns and Characters on Transparent, Conductive Oxide Glass by Mask-Defined Photoelectrodeposition and Dissolution - R. D. Engelen, C. Brinkley, L. N. Chang, and L. Yu

FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS
Physical Electrochemistry/Dielectric Science and Technology/Electronics
R. S. Ruoff, Chairman; P. Eklund, Vice-Chairman
St. Louis Ballroom C, 4th Level

10:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

K. Kadish, Chairman; R. Malhotra, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

EIGHTH INTERNATIONAL SYMPOSIUM ON MOLTEN SALTS
Physical Electrochemistry/High Temperature Materials
G. Mamantov, Chairman; D. Newman, Vice-Chairman
St. Louis Ballroom B, 4th Level

10:00 On Charging Palladium in an Al/LI/Al2O3 Eutectic, Excess Li(D)/Pd Cell - B. Y. Liew, P.-I. Tao, and B. E. Liebert
10:50 Electronic Conduction in Molten KBr-K Solutions - G. M. Haarberg and j. A. Eggers
11:10 Electronic Polarizabilities of LiCl-CaCl, LiI-AI and LiI-Ca Binary Melts - Z. Hongmin, Y. Sao, and T. Yamamura
11:30 Computer Assisted Data Acquisition and Analyses of Britouin Spectra of ZnCl2, Single and ZnCl2-NaCl Binary Melts - Z. Hongmin, Y. Sao, T. Yamamura, and K. Sugimoto

D. Newman, Chairman; G. Mamantov, Vice-Chairman

2:00 Development of Raman Spectroscopic Sensors for the Determination of Magnesium in a Molten Salt System - G. Mamantov, S. Dal, G. M. Begun, J. P. Young, and J. E. Coffield
MONDAY CONTINUED

2:20 Structural Properties of Zinc Halide Melts - E. A. 489
Pavlitsou and G. N. Papatheodorou

2:40 Structural Investigation of Molten Lithium Bromide - S. 490
Rossetti

3:00 Use of Raman Spectroscopy for Determining the 491
Corrosion of Ceramics in Molten Fluorides - C. G.
Kontoyannis and N. S. Tzilas

3:20 Raman and Infrared Spectroscopic Studies of the 492
Platinum Electrode-Molten Nitrates Interface - M. M.
Gaphurov, and V. D. Prisayzhnyi

3:40 Ten-minute intermission

3:50 Selective Precipitation of Oxide Superconductors from 493
Molten Hydroxide and Molten Nitrate - A. M. Stary, S. L.
Stoll, L. N. Markewitz, and S. W. Keller

4:10 A New Series of Complex Metal Oxides: Crystalization 494
of $\text{AB}_6\text{MO}_4$ ($\text{A} = \text{Na, Li}; \text{B} = \text{Ba, Sr}; \text{M} = \text{Bi, Sb}$) from
Hydroxide Melts - V. A. Carlson and A. M. Stary

4:30 Investigation of a Molten Salt Extraction/Electrolysis 495
Process for Converting Ilmenite Iron and a High
Grade TiO$_2$ Feedstock - K. J. Leary

4:50 New Principles for the Processing of Carnallites in the 496
Domain of Molten Salt Hydrates - H.-H. Emons and T.
Fanghein

ELECTROCHEMICAL SENSORS IN MEDICAL
SCIENCE
Sensor
D. Belanger, Chairman; D. J. Harrison, Vice-Chairman
St. Louis Ballroom E, 4th Level

10:10 Introductory remarks by D. J. Harrison

10:15 Development of an Implantable Glucose Sensor - G. S. 454
Wilson, Y. Zhang, D. Moatti-Siral, V. Poilout, G. Reich, and
D. R. Thewenot

10:40 Permeability of Glucose and Other Neutral Molecules in 545
Nafion Films Cured at 25°C and 120°C - J. D. Harrison
and Z. Fan

11:00 Amperometric Glucose Sensors Based on Glucose 545
Oxidase Immobilized in Nafion® - T. A. Zawodzinski, Jr.,
S. Gottesfeld, and J. Rishpon

11:20 Development of a Transdermal Electrochemical Sensor 547
for the Continuous Monitoring/Recording of Alcohol
Vapor - L. Sweitl, N. Kackley, A. B. LeConti, and R. M.
Swift

11:40 Progress in the Development of Amperometric Sensors 548
for Measuring the Partial Pressure of Oxygen in Blood -
J. S. Foos

D. J. Harrison, Chairman; K. Seiler, Vice-Chairman

2:00 Moving Sensors from the Journal to the Clinical Lab: 549
Some Real World Consideration - R. W. Mason

2:20 Improved Adsorption of Hydrophilic Membranes on Planar 550
Silicon Based Oxygen Sensors - E. Aw, J. Lee, C.-Y. Aw,
and J. C. Patton

2:40 A Thin Platinum Island Film Glucose Sensor - B. 551
Kasapbasioğlu, P. J. Hesketh, W. C. Hanly, and J.
Maclay

3:00 Evaluation of Sensing Surfaces for Use in LAPS-Based 552
Biosensor Devices - L. Bousse, B. van der School, and
N. F. de Rooij

3:20 Ten-minute intermission

3:30 Design, Fabrication and Testing of Flexible Ion 553
Microsensors for Cardiovascular Applications - R. P.
Buck, E. Lindner, V. V. Cosofret, R. P. Kusy, T. A.
Johnson, and M. P. Neuman

4:00 Measurement of Guinea Pig Heart Intracellular 554
Potassium Ion Concentration with Potassium
Microelectrodes - Y. M. Liu, Z. Q. Huang, J. S. Xiao, and
S. Z. Yan

4:20 Carrier Based Optodes - K. Seiler and W. Simon

4:40 Towards Reversible Sensors Based on Photochemical-
Electrochemical Switching - M. J. Preigh and S. G.
Weber

TUESDAY, MAY 19, 1992

12:15 P.M. Annual Society Luncheon and Business Meeting, Rose Garden, 4th Level.

4:30 P.M. Awards and Recognition Session, Promenade Ballroom C and D, 2nd Level. Ernest B.
Yagster will deliver the Vittorio de Nora Award Address, "Opportunities for O$_2$ Cathodes in
Batteries, Fuel Cells, and Industrial Electrolytic Processes.

JOINT GENERAL SESSION
Battery/Energy Technology
E. G. Gagnon, Chairman; A. R. Landgrebe, Vice-
Chairman
St. Louis Ballroom D, 4th Level

9:00 A Model for the Deliverable Capacity of the TiS$_2$
Electrode in a Li/TiS$_2$ Cell - Z. Mao and R. E. White

9:20 The Rechargeable Li/TiS$_2$/LiClO$_4$/Li$_2$CO$_3$ Solid-State
Cell - W. K. Bahl, E. J. Pichta, D. Vujic, H. S. W. Wang,
and D. M. Schleich

9:40 Electrochemical Properties of Polyaniiline and 16
Substituted Derivatives - P. V. Madsen, T. O. Poehler, J.
Gopal, D. O. Cowan, and P. C. Pearson

10:00 Ten-minute intermission

10:10 Investigations on a Novel Zn/KOH/DDH Primary Cell 17
System - R. Udhayan, D. P. Bhatt, and P. B. Mathur

10:30 Low Temperature Removal of Hydrogen Sulfide from
Sour Gas and its Utilization for Hydrogen and Sulfur
Production - K. M. Petrov, S. Srinivasan, and A. J.
Appleby

DIRECT ELECTROCHEMICAL OXIDATION OF
ETHANOL AND SMALL ORGANIC MOLECULES
Battery/Energy Technology/Physical Electrochemistry
P. Rose, Jr, Chairman; W. E. O'Grady, Vice-Chairman
St. Louis Ballroom H, 4th Level

8:30 In Situ FTIR Characterization of Electro catalysts for the
Oxidation of Methanol - W. Vielsisch, T. Iwasita, F. C.
Nart, and B. Lopez

8:55 Adsorption and Electro-Oxidative Pathways for Small 30
Organic Molecules on Gold and Transition-Metal-Coated
Gold Electrodes as Probed by Real-Time Surface-
Enhanced Raman Spectroscopy - Y. Zhang and M. J.
Weaver

9:20 Variable Angle In Situ FTIR Reflectance Spectroscopy of
High Surface Area Electrodes: A New Method for Studying Electro catalytic Systems - P. W. Faguy

9:45 In Situ Infrared and Electrochemical Studies of the
Oxidation of Ethylene on Single Crystal and
Polycrystalline Platinum Electrodes - Q. Zhao and E. B.
Yagster

10:10 Applications of Real-Time Infrared Spectroscopy to
Electrocatalysis at Bimetallic Surfaces: Electro-Oxidation
of Ethylene Glycol on Bismuth-Modified Pt (111) - X.
Jiang and M. J. Weaver

10:35 Ten-minute intermission

10:45 Potential Step and NMR Investigation of the Oxidation
and Adsorption of Methanol on Platinum Surfaces - A.
Wieckowski, K. Franaszczuk, P. Slezak, C. K. Rhee, and
P. Zellner

11:10 XAS Studies of UPD Metal/Pt Electro catalysts - J. 35
McBreen

11:35 Premonolayer Formation of Active Oxides and the Role
of the Latter in the Oxidation of Small Organic Molecules
- L. D. Burke, D. T. Buckley, and J. K. Casey
TUESDAY CONTINUED

V. Jalan, Chairman; W. E. O'Grady, Vice-Chairman

2:00 Direct Methanol Fuel Cells with Aqueous Carbonate Electrolytes - E. J. Caifornia, F. R. McLarnon, and B. R. Rahe

2:25 A Methanol-Aqueous Carbonate Fuel Cell - J. A. Kosek, S. Saranganapari, and J. Giner

2:50 Ten-minute intermission

3:00 Direct Electrochemical Oxidation of Methanol Vapor on Au-FEM Electrode - A. S. Lin and W. E. O'Grady


CATHODIC PROTECTION SYSTEMS
Corrosion/Industrial Electrolysis and Electrochemical Engineering

K. Nisancioglu, Chairman; R. E. White, Vice-Chairman
Room 29, 2nd Level

9:00 Influence of Al,F, Intermediate Inclusions on Depassivation of Al-5051 Alloys - C. H. Paik and R. C. Alkire

9:20 Oxygen Reduction on Steel in Basic Aqueous Solution with Relevance to Disbonding of Coatings from Cathodically Protected Steel - D. Gervasio and J. H. Payer

9:40 Formation of Calcium Deposits on Cathodically Protected Steel in Seawater - R. E. White, J. F. Yan, T. V. Nguyen, and R. B. Griffin

10:00 Some Results of Cathodic Polarization Experiments - M. de Lourdes, M. Maga/per, and L. Sathier

10:20 Ten-minute intermission


10:50 Cathodic Protection Design with Time-Dependent Boundary Conditions - K. Nisancioglu and P. O. Gartland


11:50 Closing remarks

MICROMACHINING AND MICROSTRUCTURES
New Technology Subcommittee/Sensor/Electronics/Dielectric Science and Technology

H. F. Gray, Chairman; J. N. Zemel, Vice-Chairman
Room 29, 2nd Level

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

NINTH SYMPOSIUM ON PLASMA PROCESSING
Dielectric Science and Technology/Electronics

G. S. Oehlerlein, Chairman; S. J. Fonash, Vice-Chairman
Promenade Ballroom C, 2nd Level

Diagnosics and Measurements (cont'd)

9:00 Electrical Measurement of Etching Parameters in an Oxide RIE System - S. I. Dohme and J. P. McVittie

9:20 Gate Oxide Breakdown Phenomena in Magnetron Plasma - M. Setine, K. Horioke, H. Okano, Y. Matsunaga, T. Matsushita, K. Hishioka, and Y. Yoshida

10:00 Gate Edge Effects on Oxide Damage during Polycide Etching - C. Gabriel


10:40 A Model for Thin Oxide Damage in Nonuniform Discharges - S. Fang and J. P. McVittie

11:00 Ion Bombardment Effects on Silicon Surface Properties in Plasma Etching - G. S. Oehlerlein, D. Vender, and Y. Zhang

11:20 Reactive Ion Etch Induced Device Characteristics Changes in Thin Film Transistor - Y. Kuo


D. W. Hesse, Chairman; J. I. McOmer, Vice-Chairman

Plasma Particles

2:00 A Model of Particulates in Glow Discharge Plasmas - D. B. Graves, M. G. Kilgore, J. E. Daughterty, and R. K. Porteous

2:40 Detection and Control of Particle Contamination in Production Plasma Equipment - G. S. Selwyn, T. E. Patterson, and K. L. Haller

3:20 Trapped Contamination Particulates in an RF Processing Plasma - R. N. Carile and S. G. Geha

3:40 Particle Contamination on Silicon Wafers Etched in RF Plasmas - M. M. Smadi, G. Y. Kong, R. N. Carile, and S. E. Beck

SECOND INTERNATIONAL SYMPOSIUM ON REDUCED-THERMAL-BUDGET PROCESSING FOR THE FABRICATION OF MICROELECTRONIC DEVICES
Dielectric Science and Technology/Electronics

J. W. Osenbach, Chairman; G. C. Schwartz, Vice-Chairman
St. Louis Ballroom G, 4th Level

9:00 A Highly Reliable Rapid Thermal Sintering Process for Aluminum Alloy Metallization - A. Hussain


9:40 Effects of Dopants in Polysilicon on Titanium Silicide Degradation - T. Koch

10:00 Fifteen-minute intermission


10:35 Water Trapping and Detrapping in Thin Film Dielectrics: Temperature Dependence and Water-Trap Dynamics - J. N. Cox, J. Z. Ran, J. M. Van Horn, and K. W. Kwok


11:35 Autodoping of Boron from BSG Glass - K. D. Beyer, G. Fitzgibbon, and P. A. Romshaim

FIFTH INTERNATIONAL SYMPOSIUM ON SILICON-ON-INSULATOR TECHNOLOGY AND DEVICES
Electronics/Dielectric Science and Technology

S. Cristoloveanu, Chairman; J. Gaulter, Vice-Chairman
Promenade Ballroom E, 2nd Level

Floating Body Effects

8:50 Introductory remarks
TUESDAY CONTINUED

9:00 Improved Physical Modeling of Bipolar Effects in SOI 162
Transistors - G. A. Armstrong and W. D. French
9:20 A Simple Subthreshold Model for Floating Body SOI
MOSFETs - G. Liu, P. Liu, Y. H. Chang, G. P. Li, and J. White
9:40 Voltage Limitations of Submicron CMOS on Thin SOI -
J. Sellskast, F. Brady, L. K. Wang, and N. Haddad
10:00 Study of the Kink-Related Excess Low-Frequency Noise
in SOI NMOSFETs at Room Temperature and 77 K - C.
Claeyts and E. Simon
10:20 Ten-minute intermission
M. Malloubian, Chairman; G. Campisi, Vice-Chairman
Reliability Issues
10:30 Advantages and Limitations of Thin Film SIMOX MOSFETs - J. Gautier and G. Reimbold
11:10 Hot-Carrier-Induced Degradation in Partially and Fully
Depleted SIMOX MOSFETs - S. M. Guwadi, S.
Cristoloveanu, D. E. Ioannou, G. Campisi, and H. L. Hughes
11:30 Back-Channel Hot-Electron Effect on the Drain
Breakdown Voltage in Thin-Film SOI MOSFETs - B.
Zhang and T. P. Ma
11:50 Numerical Analysis of Short-Channel and Drain
Engineering Effects for Fully Depleted SOI MOSFETs in a
Radiation Environment - J. H. Smith, R. Lawrence, and
G. Campisi
G. Campisi, Chairman; M. Malloubian, Vice-Chairman
Device-Based Characterization
2:00 Device-Based Electrical Characterization for SOI 170
Technology Development - D. E. Ioannou
2:40 Evaluation of ZMR SOI Films for BiCMOS Application by
Low Frequency Noise Investigations - B. Titlack, R.
Banisch, F. Januschewski, A. Chovet, K. Hoeppner, and
H. H. Richter
3:00 Measurements and Analysis of Random Telegraph
Signals in Small Area SOI MOSFETs - P. Rout dit
Buisson, G. Ghibaudo, J. Brini, and T. Ouisse
3:20 A New Transient Drain Current Technique for Interface
Characterization in SOI MOSFETs - S. Cristoloveanu, H.
Haddara, and M. T. Elwah
MATERIALS AND PROCESSING ISSUES FOR
LARGE SCALE INTEGRATED ELECTRONIC AND
PHOTONIC ARRAYS
Electronics/Dielectric Science and Technology
S. Yamkoshi, Chairman; D. N. Buckley, Vice-Chairman
Room 43, 4th Level
9:00 Integrated Optoelectronic Device Technology - S. R. 205
Forrest
9:40 Devices and Processes for Integrated Photoreceivers on
InP - A. Savennelec, L. Giraudet, P. Blanchonier, G. Post,
J. P. Praseuth, and A. Temmar
10:20 Fifteen-minute intermission
10:35 GaAs-on-InP, Receiver-Transmitter Optoelectronic
Integrated Circuit - P. J. O'Sullivan and D. A. Allan
11:15 Computer Simulation and Characterization of MBE
Growth of AlGaAs/GaAs Heterostructures - P. B. Kosel
and T. R. Krishna
11:35 Formation of Epitaxial CoSi2 Films on (001) Silicon Using
Ti-Ge Alloy and Bimetal Source Materials - S. L. Hsiu, T.
Y. Tan, P. L. Smith, and G. E. McGuire
STATE-OF-THE-ART PROGRAM ON COMPOUND
SEMICONDUCTORS XVI
Electronics/Dielectric Science and Technology
V. Swaminathan, Chairman; J. P. Villcot, Vice-Chairman
Room 43, 4th Level
2:00 The program and abstracts will be published in the April
issue of the Journal and included in the program booklet
distributed at the Meeting.
SECOND SYMPOSIUM ON THE PHYSICS AND
CHEMISTRY OF SiO2 AND THE Si-SiO2 INTERFACE
Electronics/Dielectric Science and Technology
D. Griscom, Chairman; S. I. Raider, Vice-Chairman
Promenade Ballroom F, 2nd Level
Deposition and Properties of SiO2
8:30 Thermal and X-Ray Production of Point Defects in
Vitroous SiO2 - F. L. Galeener
9:10 Nonstoichiometry and Defects in Bulk a-SiO2 - H.
Kawazoe and K. Awazu
9:50 The Paramagnetic Defects in Crystalline SiO2 - J. A.
Weil, R. J. McEachern, and M. M. Mombourquette
10:30 Ten-minute intermission
10:30 Formation of Si/ SiO2 Heterostructures by Low-
Temperature, Plasma-Assisted Oxidation and Deposition
Processes - G. Lucovsky, Y. Ma, T. Yasuda, and S.
Habermehl
11:00 Growth and Characterization of SiO2 Thin Films
Deposited by DECR-PECVD at Low Temperature - B.
Agius, M. C. Hugon, N. Jiung, M. Puech, G. Ravel, and
F. Plais
11:20 Low Temperature Synthesis and Characterization of
Silicon Dioxide Films - G. S. Chakravarthy, R. A. Levy,
and J. M. Grow
11:40 Fundamental Spectroscopic Studies of SiO2 Deposition
from TEO2 - J. E. Crowell, H. C. Cho, and L. L. Tedder
T. Ohmi, Chairman; G. Lucovsky, Vice-Chairman
Chemical Properties of Si Surfaces Related to Oxidation
and Oxide Deposition
1:30 Native Oxide Growth and Hydrogen Bonding Features
on Chemically Cleaned Silicon Surfaces - M. Hirose, M.
Takakura, T. Yasaka, and S. Miyazaki
2:00 Understanding the Surface Chemical and Structural
Implications of HF Solution Cleaning of Silicon - G. S.
Higashi
2:30 Pre-Gate Oxide Si Surface Control - M. Morita and T.
Ohmi
3:00 Chemical Structures of Native Oxides Formed during
Wet Chemical Treatments on Atomically Flat Si(111)
Surface - H. Ogawa and T. Hattori
3:20 Silicon Surface Analysis and Very Thin Silicon Oxide
Characterization after HF/Ethanol Pretreatment Silicon
Cleaning - J. R. Morante, B. Garrido, J. Samitier, F.
Geslin, J. L. Prom, and G. Sarrabayrouse
3:40 Effects of Metallic Impurities upon Thin Gate Oxide
Integrity and Related Bulk Electrical Properties in CZ Si -
K.-C. Cho, J.-G. Park, Y.-S. Kwak, D.-J. Lee, C.-S. Lim,
C.-K. Shin, and S. Hahn
FRACTALS IN ELECTROCHEMISTRY
Energy Technology/Physical Electrochemistry/Corrosion
S. Havlin, Chairman; C. Thompson, Vice-Chairman
Room 45, 4th Level
9:00 Equivalent Circuit Study of Fractal Electrodes - B. 321
Sapoval
9:20 The Double Layer Impedance: Effects of Electrode
Roughness - T. C. Halsey
9:40 Theory of Interfacial Constant Phase Element in
Electrode-Electrolyte Systems - S. H. Liu
10:00 Fifteen-minute intermission
10:15 Noise Analysis Applied to Electrochemical Systems - U.
Bertocci
10:35 Surface Morphology Characterization with X-Ray
Scattering - C. Thompson
11:05 Electrochemical Fractal Dimension Measurement on
Rough Surfaces - T. Pajosassy, A. Imre, and L. Nyitok
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<tr>
<th>Time</th>
<th>Session</th>
<th>Location</th>
<th>Speaker(s)</th>
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<tr>
<td>10:00</td>
<td>The Interaction of Ethyl Xanthate with Pyrite</td>
<td>J. Raiston, D. F. A. A. K. Y. Rambaksh, and R. Woods</td>
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<td>10:30</td>
<td>The Effect of EDTA on Collectorless Floation of Pyrite</td>
<td>J. S. A. A. A. K. Y. Rambaksh, and R. Woods</td>
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<td>10:45</td>
<td>An Electrochemical Study of Selective Deactivation/Depression of Cu(II)/Activated Pyrite and Arsenopyrite</td>
<td>X. H. W. and C. L. Jiang</td>
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<td>12:00</td>
<td>Surface Layer Structure of Sulfide Mineral Treated in Thiol Collector Solutions</td>
<td>E. S. A. A. A. K. Y. Rambaksh, and R. Woods</td>
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<td>12:30</td>
<td>The Interaction of Diethyl Dithiophosphate with Freshly Exposed Galena and Chalcolite Surfaces</td>
<td>A. N. B. K. Y. Rambaksh, and R. Woods</td>
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<td>14:00</td>
<td>Hydrodimerization of Dimethylmaleate I: Scouting</td>
<td>J. H. W. A. A. A. K. Y. Rambaksh, and R. Woods</td>
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**GENERAL SESSION**

- **Physical Electrochemistry**
  - A. Lasia, Chairman; P. Vanysek, Vice-Chairman

- **Physical Electrochemistry/Electrodeposition**
  - R. C. Alkire, Chairman; T. Hapal, Vice-Chairman

- **Physical Electrochemistry/Electrodeposition**
  - R. C. Alkire, Chairman; T. Hapal, Vice-Chairman

- **Micromorphology in Electrocrystallization**
  - Physical Electrochemistry/Electrodeposition

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  - Physical Electrochemistry/Electrodeposition

- **Micromorphology in Electrocrystallization**
  - Physical Electrochemistry/Electrodeposition
TUESDAY CONTINUED

QUANTUM CONFINEMENT
New Technology Subcommittee/Electronics/Dielectric Science and Technology
E. Nicollan, Chairman; R. Tsu, Vice-Chairman
St. Louis Ballroom F, 4th Level

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS
Physical Electrochemistry/Dielectric Science and Technology/Electronics
J. Bernholc, Chairman; B. Dunlap, Vice-Chairman
St. Louis Ballroom C, 4th Level

9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

D. M. Cox, Chairman; D. Tomanek, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

EIGHTH INTERNATIONAL SYMPOSIUM ON MOLTEN SALTS
Physical Electrochemistry/High Temperature Materials
E. J. Cairns, Chairman; D. G. Lloving, Vice-Chairman
St. Louis Ballroom B, 4th Level

9:00 Molten Carbonate Fuel Cell Reaction Mechanisms - K. Hemmes, R. C. Makkus, R. Woever, and J. H. W. de Wit

9:20 IMHEX® Molten Carbonate Fuel Cell Program Status - G. L. Kaznich and R. Donelson


10:00 High Temperature Corrosion of Nickel Coated with Molten Alkaline Carbonates at 500°-750°C - J. Derynck, G. Moutiers, M. Cassir, and B. Tremillon

10:20 Ten-minute intermission

10:30 Galvanic Corrosion of Coupled Pt-Ni System in Molten Sodium Carbonate at 1173 K - Y. Shinata, M. Hara, and S. Hashimoto


11:10 Application of Molten Salts in Pyrochemical Processing of Reactive Metals - B. Mishra, D. L. Olson, and W. A. Averill


11:50 Voltammetric Study of a Planar Electrode with Superemiscuous Film in Molten Carbonate - G. L. Lee and J. R. Selman

D. G. Lloving, Chairman; E. J. Cairns, Vice-Chairman

2:20 Material-Deposition Processes in the Separator of Li-Alloy/Fe/Si Thermal Cells - M. C. Hash, J. A. Smiga, R. A. Guidotti, and F. W. Reinhardt


3:00 The Modification of Film Membranes for Use in Energy Generating Devices - D. S. Newman, S. Li, and T. Howard

3:20 Molten Salt Regular Mixture Membranes for Transfer in Ag2SO3-Li2SO4 - A. Grimaldetti and S. K. Ratjke

3:40 Theory of Thermocells: Transported Entropies and Heat of Transfer in Ag2SO3-Li2SO4 - A. Grimaldetti and S. K. Ratjke

ELECTROCHEMICAL SENSORS IN MEDICAL SCIENCE
G. Wilson, Chairman; L. Bousse, Vice-Chairman
St. Louis Ballroom E, 4th Level

9:00 Electrochemical Methods for Quantitation of Peroxidase and Alkaline Phosphatase - H. Song and D. G. Hafeman

9:20 Bionzyme Amperometric Sensors Using a Polymeric Electron Transfer Mediator - A. C. Michael and M. G. Garguillo


10:00 Glutamate Dehydrogenase-Modified Carbon Fiber Microelectrodes with Millisecond Response Times - W. G. Kuhn and P. Pantano

10:20 Ten-minute intermission

10:30 Application of w-Thiocarboxylic Acid Monolayers for the Detection of Dopamine in the Presence of High Concentration of Acetic Acid - D. Mandler and F. Malem

10:50 Controlled Binding and Electroluminescence of Metal Ions, Drugs, and Neurotransmitters from Cation Gate Composite Polymer Films Studied by Piezoelectric Sensor Technique - M. Hafeman and L. Dentrone

11:10 The Effect of Oxidation on the Electrochemical Behavior of Pyrpyrrole Films Doped with Various Anionic Species - D. Belanger and F. Provencher

11:30 Polyprpyrrole-Enzyme Film Electrode Mediated by Electropolymerized Catalytic Polymer Film - H. Tachikawa, Z. Sun, and H. Ge

WEDNESDAY, MAY 20, 1992

12:15 P.M. Dielectric Science and Technology Division Luncheon and Business Meeting, Rose Garden, 4th Level.

12:15 P.M. Energy Technology Division Luncheon and Business Meeting, Promenade Ballroom D, 2nd Level.

12:15 P.M. Organic and Biological Electrochemistry Division Luncheon and Business Meeting, St. Louis Ballroom H, 4th Level.

NINTH SYMPOSIUM ON PLASMA PROCESSING
Dielectric Science and Technology/Electronics
R. N. Caslile, Chairman; D. W. Hess, Vice-Chairman
Promenade Ballroom C, 2nd Level

Plasma Cleaning

9:00 Plasma and Surface Diagnostics of ECR Hydrogen Cleaning of Native Silicon Oxide - W. Tsai, M. Delfino, B. C. Chung, T. Sheng, and S. Salimian

9:20 Process and Module for Low Temperature Hydrogen Cleaning for Silicon Wafer - F.-P. Steiner, E. Beck, and J. Ramm

9:40 In Situ Chamber Dry Cleaning for HBr RIE - K. Iizuka and M. Nakamura

10:00 Ion Beam-Assisted Etching of Si (111) with Fluorine at 77K - J. W. Coburn and C. S. Mulhine

Low Temperature Etching
WEDNESDAY CONTINUED

10:40 A New Method of Sidewall Protection for Anisotropic Etching Using Sulfur Deposition - T. Tatsumi, T. Nagayama, and S. Kadomura

11:00 Cooling Wafer Stage for Low Temperature Dry Etching - M. Kanetomo, T. Kure, K. Tsujimoto, and S. Tachi

11:20 Problems of Low Pressure Single Crystal Silicon Etching - M. Engelhardt

11:40 Highly Selective Low Pressure Polysilicon Etching in High Density, Low Energy RF Plasmas - M. Engelhardt

G. S. Mathad, Chairman; S. Salimian, Vice-Chairman

SiC Etching (cont'd)

2:00 Selective High-Rate Etching of Polysilicon in Microwave ECR Discharges: A Comparison of ECR Configurations - T. O. Mantel, D. Dane, and P. Gadgil

2:20 Quarter-Micron Deep Trench Etch for ULSI - Y. T. Li and T. V. Rajavalkamar

2:40 MRE 0.1 μm Polysilicon Lines by Using HBr - Y. T. Li, C. M. Reeves, D. A. Danner, P. J. Coane, and L-K. Wang

3:00 Fundamental Processing Limit of Gate Oxide Thickness for Polysilicon Gate Definition - A. S. Yapsir

Aluminum/Alloy Etching

3:20 200 nm Aluminum Etch in MERIE System - S. Mak, S. Arias, and C. S. Rhoades

3:40 Dry Etching of Al Alloy Films Using HBr Mixed Gases - K. Fujino and T. Oku

4:00 One-Step Electron Cyclotron Resonance Etching of Submicron Al-Si-Cu - D. X. Ma and C.-H. Chen

4:20 Low Energy Ion Etching of Aluminum Oxide Films and Native Aluminum Oxide - M. E. Day, S. Salimian, and M. Delfino

4:40 Analysis of Post-Metal Etch Processes Causing Submicron Technology Corrosion Defects - K. E. Mautz

G. S. Selwyn, Chairman; D. B. Graves, Vice-Chairman

Plasma Generated Particles

7:00 Informal discussion

FIFTH INTERNATIONAL SYMPOSIUM ON SILICON-ON-INSULATOR TECHNOLOGY AND DEVICES

Electronics/Dielectric Science and Technology

P. L. F. Hemment, Chairman; J. Margail, Vice-Chairman

Promenade Ballroom E, 2nd Level

Characterization of Materials I

9:00 Introductory remarks


10:10 Screen Oxide Effects on the SIMOX Material Quality and Interface Properties of SOI - J. Margail, J. R. Morante, E. Martin, J. Jimenez, J. Margail, and A. M. Papan

10:30 Fifteen-minute intermission


11:15 Novel Approach to Defect Etching in Thin Film SOI - H. Gasselt, J. Peter-Weidemann, and H. Vogt

J. Margail, Chairman; P. L. F. Hemment, Vice-Chairman

Characterization of Materials II

2:00 Correlation Between X-Ray Moire' Pattern and Dislocation Density in SIMOX - M. K. El-Ghor, K. A. Joyner, and G. A. Rozgonyi


3:00 Ten-minute intermission

3:10 The Effects of Carrier Lifetime of SIMOX Anneal Process Parameters: A Designed Experiment - K. A. Joyner


SECOND SYMPOSIUM ON THE PHYSICS AND CHEMISTRY OF SiO₂ AND THE SiO₂/Si INTERFACE

Electronics/Dielectric Science and Technology

T. Hattori, Chairman; C. R. Helms, Vice-Chairman

Promenade Ballroom F, 2nd Level

Chemical, Structural, and Microroughness Effects at the Si-SiO₂ Interface

8:15 T. D. CAILLARNE AWARD ADDRESS: Carbon Impurities at Si-SiO₂ Interfaces - S. I. Raiker

8:50 Microscopic Structure of the Si/SiO₂ Interface - F. J. Himpsel


9:50 Dependence of Surface Microroughness on Types of Silicon Substrates - T. Ohmi, T. Tsuga, and J. Takano

10:20 Ten-minute intermission

10:30 The Effect of Surface Roughness on Gate Oxide Leakage Currents - M. Chonko and V. Kaushik

10:50 A Double Sacrificial Oxide Process for Smoother 150 Å SiO₂ Gate Oxide Interfaces - H.-H. Tsang and P. J. Tobin

11:10 Effect of Solidification Induced Defects in CZ Silicon upon Thin Gate Oxide Integrity - H. Suga, H. Abe, H. Koya, T. Yoshihira, H. Yohshioka, and N. Kagawa

11:30 Effects of D-Defects in CZ Silicon upon Thin Gate Oxide Integrity - J. G. Park, S.-P. Choi, G.-S. Lee, Y.-J. Jeong, Y.-S. Kwak, C.-K. Shin, S. Hahn, and H. Macbush

11:50 Oxidation-Induced Changes in the Si Surface Microroughness - V. Nayar, A. J. Pidduck, and C. Pickering

A. Goodman, Chairman; W. T. Lynch, Vice-Chairman

Novel Structures, Processes, and Phenomena

1:45 Properties of SIMOX and Related Systems - S. Cristofoleau

2:15 Reoxidized Nitrided Oxide Gate Dielectrics for Advanced CMOS - G. J. Dunn


3:05 Charge Trapping in an ONO Gate Dielectric - R. B. Klein and N. S. Saka

3:25 Interface Trap Density Reduction and Oxide Profiling for Fluorinated MOS Capacitors - D. Kouvatzos, R. J. Jaccodine, and F. A. Stavie

3:45 Ten-minute intermission

3:55 Physics of Extreme Quantum Confinement Exemplified by Si/SiO₂ Systems - R. T. Teu

4:25 Integrity of Very Thin Silicon Films Deposited on SiO₂ - M. Chonko, D. VanZandberg, and D. Keitz

4:45 Researches of SiO₂ on InP and GaAs MOS Structures - Y. K. Su and C. J. Hwang
WEDNESDAY CONTINUED

STATE-OF-THE-ART PROGRAM ON COMPOUND SEMICONDUCTORS XVI
Electronics/Dielectric Science and Technology
J. P. Vilicot, Chairman; V. Swaminathan, Vice-Chairman
Room 43, 4th Level

9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

D. N. Buckley, Chairman; T. Kamiyoh, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

JOINT GENERAL SESSION
Electronics/Dielectric Science and Technology
R. J. Jaccodine, Chairman; A. Harrus, Vice-Chairman
Promenade Ballroom A, 2nd Level

9:00 Suppression of WSi, Peeling in O.8 μ BICMOS 269
Technology - B. L. Mantha and C. S. Wang


9:30 Effect of Underlying TiW Thickness on the Electromigration of Vias - D. Pramanik and V. Jain

9:45 Microstructure Control and Thermal Stability of Titanium Silicide - R. W. Mann and C. Racine

10:00 The Direct Observation of Atom Column Defects in MoSi2 and WSi2 - K. M. Pollack, D. S. Schwartz, T. C. Bryant, and P. Freundorf


10:30 Fifteen-minute intermission

10:45 Influence of Field Isolation Stress on Gate Oxide Reliability - K. Shiozaki, K. Shimamone, K. Kato, and J. Nakano

11:00 A Comparison of Interstitial Oxygen Calculated by Different Techniques - J. W. Medernach and J. O. Stevenson

11:15 The Warpage of As-Received and Oxygen Precipitated CZ Silicon Wafers - J. Jeong and H.-D. Choi

11:30 Modeling of Phosphorus Precipitation - S. Dunham


A. Harrus, Chairman; R. J. Jaccodine, Vice-Chairman

2:00 Introduction of Ion-Surface Interaction in Ar Plasma Etching of Si and PECDV of SiO2 Film by High-Frequency C-V Method - C. Pavlescu, J. P. McVittie, C. Chang, K. C. Sarawat, and J. Y. Leong


2:30 Chemical Foundations in Understanding the Step Coverage Problem in CVD Using the Silane Chemistry - C. Pavlescu and K. C. Sarawat


3:00 Highly Reliable Electrostatic Chuck Employing Plasma-Sprayed Aluminum-Oxide Coating for Advanced Semiconductor Processing - H. Uetake, K. Morizuka, and T. Ohmi


3:30 Fifteen-minute intermission

3:45 Mix-Cation Oxide Powders via Resin Intermediates Derived from Water Soluble Polymers - L.-W. Tai and H. U. Anderson

4:00 A Novel Evaluation Technique of Carrier Concentration by Noncontact Eddy Current Measurement - S. Washizuka, Y. Yoneda, A. Watanabe, and T. Ohta


5:30 Low Resistance Molybdenum Silicide - Molybdenum Thin Films - S. Vasile and D. Niculascu

SURFACE PROCESSING IN ENERGY TECHNOLOGIES
Energy Technology
A. W. Czanderna, Chairman; A. R. Landgrebe, Vice-Chairman
St. Louis Ballroom D, 4th Level

8:30 Introductory remarks by A. W. Czanderna


9:10 Characterization of Electrochemical Active Ni-Co Amorphous and Crystalline Alloys - K. Lian, D. W. Kirk, and S. J. Thorpe

9:30 Influence of Pretreatment on the Unusual Redox Behavior of Noble Metal Electrodes - L. D. Burke and J. K. Casey

9:50 Ten-minute intermission

10:00 Catalyst Layer and Interface Properties of Directly Catalyzed Polymers Electrolyte Membranes - M. S. Wilson, T. A. Zawadowski, J., T. E. Springer, J. Valerio, and S. Gottesfeld

10:20 Chemical Bath Deposition of Cadmium Sulfide Thin Films, In Situ Growth and Structural Studies by Combined Quartz Crystal Microbalance and Impedance Techniques - D. Lincot and R. Ortega

10:40 Study of the Bandedge Shifts of n- and p-type CuInSe2 Semiconducting Electrodes in Various Aqueous Electrolytes - W. Siripala, J. Vede, D. Lincot, and D. Cahn

11:00 Highly Oriented vs. Microcrystalline Thick Cadmium Films Deposited on Poly-crystalline Nickel from a Molten Salts Electrolyte - R. R. Agarwal

11:20 Surface Modification of Sintered Plates Negative Electrodes of Nickel Cadmium Battery - S. Tamil Selvan, R. Sabapathi, and N. Venkatakrishnan

A. R. Landgrebe, Chairman; A. W. Czanderna, Vice-Chairman

2:00 Introductory remarks by A. R. Landgrebe


2:40 In Situ Study of the Chemical Bath Deposition Mechanism of Cadmium Sulfide Thin Films on Gold and Copper Indium Diselenide Substrates - D. Lincot, R. Ortega, and J. Vede

3:00 Electrochemical Vapor Deposition of CeO2 Thin Film Electrolytes for Solid Oxide Fuel Cells - J.-F. Jue and A. V. Viktar

3:20 Ten-minute intermission
WEDNESDAY CONTINUED

3:30 Evaluation of Doped Ceria Whith and Without Zirconia Coating for Application In Low-Temperature Solid Oxide Fuel Cells - K. Mehta and A. V. Virkar

3:50 Kinetics of the Cadmium Electrode in Alkaline Solution - S. Tamil Selvan, R. Sabapathi, and N. Venkatakrishnan

4:10 Evaluation of Electrodedeposited Electrocatalytic Composite Metal Film Coatings for Cathodic H2 Evolution in Water Electrolyzers - B. E. Conway, R. Simpraga, and R. Brousseau

ELECTROCHEMICAL CHARACTERIZATION OF THIN SOLID FILM Energy Technology/Physical Electrochemistry/Electronics/Dielectric Science and Technology

P. J. Sides, Chairman; M. E. Orazem, Vice-Chairman Room 46, 4th Level

9:00 In Situ Infrared Spectroscopy of Corrosion Processes at Lacquer Coated Metals - S. N. Port, A. Bewick, and J. Casper

9:20 Coulometric Reduction of Thin Tarnish Films Formed on Copper - B. I. Rickett and J. H. Payer

9:40 Impedance Characterization of Polypyrroles - P. G. Pickup, G. L. Duffitt, and X. Ren

10:00 Electrochemical Observation of Intermediates during the Formation of Conducting Polypyrrole - D. J. Harrison and D. E. Raymond

10:20 Ten-minute intermission

10:30 Electrochemical Quartz Crystal Microbalance Studies of Electroactive Polymer Bilayers - A. R. Hillman and A. Glidle

10:50 Modeling of the GainP/Aqueous Electrolyte Interface Utilizing Electrochemical Impedance Spectroscopy - S. Kochs and B. E. Liebert

11:10 The Effect of Preparation Conditions on the Ion Diffusion and Electrochromic Properties of Li3W9O34 - J.-G. Zhang, C. E. Tracy, and D. K. Senson

11:30 Electrode Surface Modification with ZnO and ZnO-Cds Semiconductor Colloidal Particles - P. V. Katam and S. Hotchandani

STABILITY OF REFRACTORY MATERIALS

High Temperature Materials

D. L. Jacobson, Chairman; A. Luo, Vice-Chairman Room 46, 4th Level


2:20 Ionic Conductivity and Phase Transformation in Gd2O3 - Stabilized Bi2O3 - P. Su and A. V. Virkar


3:00 The High Temperature Work Function of Sintered Dilute Solution Tungsten, Iridium Alloys - L. A. D'Crux, D. R. Bosch, and D. L. Jacobson

3:20 Dispersion Particle Strengthening in Tungsten at High Temperatures - A. Luo and D. L. Jacobson


HIGH TEMPERATURE SENSORS

High Temperature Materials/Sensor/Battery

C. B. Alcock, Chairman; P. Moseley, Vice-Chairman St. Louis Ballroom E, 4th Level


10:00 Mixed Potential Gas Sensors - P. T. Moseley and R. Peat

10:20 Ten-minute intermission

10:30 New Ideas, How to Use Mixed Ionic Electronic Conductors, Instead of Solid Electrolytes, in Potentiometric Sensors - L. Riess


11:10 Novel Solid Electrolyte CO2 Sensor Using Sodium ion Conductor and Lithium-Based Carbonate Electrode - N. Yamazoe, S. Yao, Y. Shinmu, and N. Miura

11:30 Development of Solid-State Electrochemical Sensors for Measurement of Elements in Molten Metals - R. V. Kumar and D. J. Fray

C. C. Liu, Chairman; N. Yamazoe, Vice-Chairman

2:00 Miniaturization of High Temperature Oxygen Sensor Employing Film Electrolyte and Electrodes - E. B. Makoves, F. W. Montague, M. A. Stuczynski, and C.-C. Liu


3:00 A Novel Polymer Blend for Solid-State Ammonia Sensor - R. D. A. Paulmer, R. S. Srinivas, and A. R. Kulkarni

ELECTROCHEMISTRY IN MINERAL AND METAL PROCESSING III

Industrial Electrosynthesis and Electrochemical Engineering

M. Wadsworth, Chairman; E. Suoninen, Vice-Chairman St. Louis Ballroom A, 4th Level

9:00 Photoelectrochemical Behavior of Chalcopyrite (CuFeS2) in Alkaline Solutions - G. H. Keisall, F. W. H. Dean

9:30 Electrochemical Oxidation of Chalcopyrite (CuFeS2) in Alkaline Solutions - G. H. Keisall, K. E. R. England, D. J. Vaughan, and Q. Yin

10:00 The Photoelectrochemistry of In Situ Fractured Pyrite Electrodes - P. E. Richardson, Y. Li, and R.-H. Yoon

10:30 Fifteen-minute intermission


11:15 Electrochemical Studies of Iron Sulfides in Relation to Their Atmospheric Oxidation and Prevention of Acid Drainage: Part II - S. M. Ahmed and E. Gizewicz

J. B. Hiskey, Chairman; A. Buckley, Vice-Chairman

2:00 Transpassive Oxidation of Pyrite - X. Zhu, J. Li, D. M. Bodily, and M. E. Wadsworth


3:00 Electrochemical Kinetics of Silver Dissolution in Cyanide Solutions - J. Li and M. E. Wadsworth

3:30 Fifteen-minute intermission

3:45 Thermodynamic Equilibrium Calculations on AuAg Liñivant Systems Relevant to Gold Extraction from Complex Ores - X.-H. Wang


4:45 Complex Technology of Electrochemical Water Treatment with Regeneration of Valuable Components in Electroplating Production - V. A. Kolesnikov, E. A. Shalyt, and P. K. Aarinola
**WEDNESDAY CONTINUED**

**ELECTRO-ORGANIC SYNTHESIS WITH HOMOGENEOUS AND HETEROGENEOUS CATALYSTS**

Organic and Biological Electrochemistry

D. G. Peters, Chairman; J. E. Toomey, Jr., Vice-Chairman
Promenade Ballroom B, 2nd Level

9:00 Introductory remarks by D. G. Peters
9:35 Electrocatlytic Hydrogenation at Palladium Black Electrodes - S. J. C. Cleghorn and D. Fletcher
10:05 The Electrochemical Reduction of Nitroaromatic Compounds to Amines in Basic Solutions at Porous Metallic Electrodes - J. Lessard, M.-J. Lessard, Y. Couture, A. Martel, and C. Roy
10:35 Fifteen-minute intermission

J. E. Toomey, Jr., Chairman; D. G. Peters, Vice-Chairman

2:00 On the ipso-Substitution in Aromatic Compounds by Electrogenerated Radicals - H. Lund, K. Pang, and Q. Chen
3:00 Fifteen-minute intermission
3:45 Mediated Reduction of Aryl Bromides at Tin Cathodes - E. G. Gunderson, E. Kariv-Miller, and V. Sveticic

**ELECTROCHEMISTRY OF HIGH TEMPERATURE SUPERCONDUCTORS**

Physical Electrochemistry

C. Lieber, Chairman; J. T. McDevitt, Vice-Chairman
Promenade Ballroom B, 2nd Level

9:00 Introductory remarks
9:55 Low Temperature Electrochemistry on High T Superconductors - W. J. Lorenz, G. Saeemann-Teachenko, and M. W. Breiter
10:35 Fifteen-minute intermission
11:30 Electrochemical Rate Data of Superconductor Materials and Their Lithium Insertion Compounds as Electrodes - N. A. Fleisher and J. Manassen

J. T. McDevitt, Chairman; C. Lieber, Vice-Chairman

2:00 The Surface Structure and Electronic States of High-T Superconductors by STM - C. M. Lieber and Z. Zhang
2:40 Physical and Chemical Characterization of Electrodeposited Superconducting Bismuthates - M. L. Norton and H.-Y. Tung
3:20 Fifteen-minute intermission

**FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS**

Physical Electrochemistry/Dielectric Science and Technology/Electronics

W. Goddard, III, Chairman; C. Read, Vice-Chairman
Promenade Ballroom C, 4th Level

9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

M. Parinello, Chairman; F. Wudl, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

**EIGHTH INTERNATIONAL SYMPOSIUM ON MOLTEN SALTS**

Physical Electrochemistry/High Temperature Materials

G. Blomgren, Chairman; R. Carlin, Vice-Chairman
Promenade Ballroom B, 4th Level

9:00 The Nature of Proton in Ambient-Temperature Chloroaluminate Molten Salts - R. A. Osteryoung and P. C. Trulove
9:30 The Chemistry of Proton in Ambient-Temperature Chloroaluminate Molten Salts - P. C. Trulove and R. A. Osteryoung
9:50 Ionic Equilibria in Ambient Temperature Molten Salts - J. L. E. Campbell and K. E. Johnson
10:10 Dual Spin Probe NMR Relaxation Studies of Microdynamics in Chloroaluminate Melts - W. R. Carper, C. E. Keller, P. A. Shaw, M. Parrish, and J. S. Wilkes
10:30 Ten-minute intermission
10:40 NMR Measurements in Solutions of Diakylimidazolium Halogenates - S. Takahashi, J. Rathke, and M.-L. Saboungi
11:00 The Electrochemistry of Aluminum and Protons in Room Temperature Chloroaluminate Molten Salts Buffered with Sodium Chloride - T. L. Ricechel and J. B. Wilkes
11:40 Physico-Chemical Behavior and Liquid Crystalline Properties of Molten Alkali Metal Aloxenates - T. A. Mineyia and S. V. Volkov

R. Carlin, Chairman; G. Blomgren, Vice-Chairman

2:00 Low Temperature Molten Salt Electrolytes Based on Ternary Alkytylophonium Salts - G. E. Blomgren and S. D. Jones
2:20 New, Stable, Ambient-Temperature Molten Salts - E. I. Cooper and E. J. M. O’Sullivan
2:40 Mixed Chlorororate and Chloroaluminate Room Temperature Melts - R. J. Gale and J. Liu
3:00 Electrochemistry of 8.10-Anthraquinone in the Presence of Proton and Tetrachloroaluminate in Ambient Temperature Molten Salts - M. T. Carter and R. A. Osteryoung
3:20 Electrochemical Reduction of Aromatic Ketones in a Room Temperature Molten Salt - G. T. Cheek
3:40 Ten-minute intermission
WEDNESDAY CONTINUED

3:50 Studies on Characteristics of Room Temperature Molen Salts AICI3-1-butylpyridinium Chloride - N. Koura, K. Ueda, and K. Takeishi

4:10 Electrodeposition of Metals from Room-Temperature Chloroaluminates Molen Salts - I. Xu and C. L. Hussey

4:30 Electrodeposition of Palladium onto Electrodes from Acidic, Neutral, Neutral-Buffered, and Basic MEIC-AICI3 Molten Salts - H. C. Delong and J. S. Wilkes


5:10 Ionic Conductivity, Thermodynamics and IR-Spectroscopy of Tetra-n-Pentylammonium Thioctyanoate-Benzoic Acid System - A. M. Elias and M. E. Elias

R. J. Gale, Chairman; C. Hussey, Vice-Chairman
St. Louis Ballroom H, 4th Level

9:00 MAX BREDEG AWARD ADDRESS: Through the Years and Temperatures: Adventures in Molten Salt Land - R. A. Osteryoung

QUANTUM CONFINEMENT
New Technology Subcommitte/Electronics/Dielectric Science and Technology

THURSDAY, MAY 21, 1992

GENERAL SESSION

Corrosion

B. R. MacDougall, Chairman, R. G. Kelly, Vice-Chairman
Room 48, 4th Level

9:00 Laser Initiated Corrosion Fls on Aluminum - D. Buzzing and R. C. Allkl

25 The Characterization of Crevice Solution Chemistry during the Initiation Stage of Crevice Corrosion - B. K. Nash and R. G. Kelly


10:15 Ten-minute intermission

10:25 Laser Raman Spectroelectrochemical Studies of Fe, Ni, Cr, and Their Glassy Metal Alloys with Phosphorus - M. Pankuch, C. A. Melendres, J. C. Kang, S. Lahani, and Y. S. Li


11:15 Film Thickness Effects on Flash-Rusting Measured by Spectrophotometry and Atomic Absorption - M. R. Van De Merkt, F. Siamari and N. Mason

11:40 The Analysis of Atmospheric Corrosion on Fe Particle Magnetic Tape - J. F. Dante and R. G. Kelly

J. A. Bardwell, Chairman; P. Kovacs, Vice-Chairman

3:55 Corrosion of Mg and Its Alloy in Aqueous Mg(ClO4)2 Battery Electrolyte - A RDE Study - R. Udhayan and D. P. Bhakt

4:20 Ellipsometric Study on Inhibition Effect of Molybdate for Cooling Water - S. F. Xie, Y. R. Yang, and Z. Q. Huang

NINTH SYMPOSIUM ON PLASMA PROCESSING
Dielectric Science and Technology/Electronics

M. Englehardt, Chairman; M. Sekine, Vice-Chairman
Promenade Ballroom C, 2nd Level

Aluminum/Alloy Etching (cont’d)

9:00 Pattern Density Effects on Corrosion of Aluminum Alloy/Refractory Metal Interconnects - C. Gabriel and R. Wallach

9:20 Dry Corrosion Control in Aluminum Etch Using O2/NH3 in Microwave Downstream Plasma - S. Hik and J. Huang

9:40 Microcontamination Reduction and Corrosion Prevention for Aluminum Etch Through Loadlock Improvements - E. DeLaurentis and J. Huang

10:00 REI-Related Sidewall Voidsing in Al-Cu Alloy Metalization for ICs - T. H. Daubenspeck and H. K. Lee

Tungsten Etching


10:40 Mechanism of Residue Formation in Low Temperature Tungsten Interconnector Etch - U. C. Sridharan, D. Hartman, R. Wright, and M. Kent

11:00 A Kinetic Study of Reactive Ion Etching of Tungsten in SF6/O2 PF Plasma - M.-C. Peignolin, C. Cardinou, and G. Turban

11:20 Optical Emission Studies of Etching Tungsten Silicide and Polyisilicon Films - X. Y. Qian, D. J. Hemker, and G. W. Hilds
THURSDAY CONTINUED

Dielectric Etching

11:40 Effect of Wafer-Average and Within-Die Poly Si Grain Pattern Density on Transistor Gate-Length Definition for Cell-Based ASIC CMOS Designs - D. Dimitriu, M. Carneiro, and V. Dunton

D. E. Ibbotson, Chairman; J. D. Chapple-Sokol, Vice-Chairman

Dielectric Etching (cont'd)

2:00 Reactor Characterization for a Process to Etch Si3N4 Formed on Thin SiO2 - P. E. Riley, J. C. Sum, and D. Figuredaro


2:40 Photore sist Swelling in Hydrogen-Containing Freon Plasmas - Y. Kuo

3:00 Comparison of Nonline of Sight Resist Ashing Using Microwave or Radio Frequency Plasma Generation - J. I. McOmber, J. T. Davies, J. Howden, M. Wright, and K. Saule


PECVD


4:40 Composition and Stability of Plasma-Deposited Fluorinated Silicon Nitride Thin Films - R. Costantin, C. Marinensi, G. Tiganu, and A. Zanobi

5:00 Modeling and Simulation of Plasma Enhanced Chemical Vapor Deposition of Silicon Nitride - M. Mazher Islam, A. Bari, J. P. McVitie, M. A. Cappelli, K. C. Saraswat, L. Mobery, and R. Lehti

FIFTH INTERNATIONAL SYMPOSIUM ON SILICON-ON-INSULATOR TECHNOLOGY AND DEVICES

Electronics/Dielectric Science and Technology

K. Izumi, Chairman; S. Cristoloveanu, Vice-Chairman

Promenade Ballroom E, 2nd Level

Materials Quality

8:50 Introductory remarks

9:00 Characterization of SIMOX Substrates - A. Yoshino


10:00 The Effects of HCI in SIMOX Annealing: A Time Series Experiment - K. A. Jouyner and G. A. Brown

10:20 Fifteen-minute intermission


10:55 A High-Quality SIMOX Wafer and its Application to Ultrathin-Film MOSFETs - S. Nakashima, Y. O'mura, and K. Izumi


S. Cristoloveanu, Chairman; K. Izumi, Vice-Chairman

Materials

2:00 SIMOX: An Efficient Etch-Stop to Fabricate Silicon Membranes with Well-Defined Thickness - H. Gassell, H. G. Dura, W. Molawa, and H. Vogt

2:20 Reactive Ion Etching of SOI (ZMR and SIMOX) Silicon in CF4 + O2 and SF6 + O2 Plasmas - G. W. Purbo, C. R. Selvakumar, and D. Misra

2:40 Gettering of Bonded SOI Layers - H.-D. Chiu, F. Secco d'Arragona, and E. Strickland

3:00 50 nm Thick SOI Fabrication by Advanced ELO: Tunnel Epitaxy - A. Ogura, A. Furuya, and R. Koh

SECOND SYMPOSIUM ON THE PHYSICS AND CHEMISTRY OF SiO2 AND THE Si-SiO2 INTERFACE

Electronics/Dielectric Science and Technology

E. H. Poindexter, Chairman; N. S. Saka, Vice-Chairman

Promenade Ballroom F, 2nd Level

Defects and Hot Carrier Induced Damage in Si-SiO2 Systems I

8:45 Generation of Telegraph Noise via Single Interfacial Defects - M. J. Uren and D. H. Cobden

9:15 Single Electron Transfer from the Channel in Sub-um MOSFETs to an Individual Interface Trap - M. Schulz and A. Pappas

9:45 0 Hyperfine Study of the 19F Center - J. H. Stathis, S. Rigo, and I. Trisaille

10:15 The Influence of Crystal Orientation and Processing Conditions on the Energy Distribution of Traps at the Si-SiO2 Films Interface - C. H. Bjorkman, Y. Ma, T. Yasuda, and G. Lucovcky

10:35 Ten-minute intermission

M. Schulz, Chairman; M. A. Stroscio, Vice-Chairman

Defects and Hot Carrier Induced Damage in Si-SiO2 Systems II


11:15 Charging and Discharging Properties of Electron Traps Created by Hot-Carrier-Injections in Gate Oxide of n-MOSFET - D. Vuillaume


M. Heyns, Chairman; D. J. DiMaria, Vice-Chairman

Radiation and Hydrogen Induced Effects in Si-SiO2 Systems

1:30 Impact Ionization and Positive Charges in SiO2 - D. J. DiMaria, D. Arnold, and E. Carlier

2:00 Hot-Electron Dynamics in SiO2 Studied by Photon-Induced Electron Transmission Through Thin Films - E. Carlier, A. E. Ekland, D. Arnold, D. J. DiMaria, and F. R. McFeely


2:40 Ten-minute intermission

2:50 Radiation-Induced Interface Traps in MOS Devices - N. S. Saka and D. B. Brown


3:40 Molecular Orbital Studies of Oxygen-Related Hole Traps and of their Interactions With Hydrogen Atoms and Molecules - A. H. Edwards

4:00 Removal of Traps in Process-Damaged MOS Structures by Room-Temperature Hydrogenation - S. Kar

4:20 Hot-Electron Induced Hydrogen Redistribution in SiO2 - D. A. Buchanan, A. Marwicz, L. Dori, and D. J. DiMaria

4:40 H2 Cracking in Irradiated MOSFETs and H4 Formation - R. E. Stahlbush and A. H. Edwards
THURSDAY CONTINUED

STATE-OF-THE-ART PROGRAM ON COMPOUND SEMICONDUCTORS XV
Electronics/Dielectric Science and Technology
T. Kamioh, Chairman; D. N. Buckley, Vice-Chairman
Room 43, 4th Level
9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

A. Katz, Chairman; G. Valco, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

JOINT RECENT NEWS PAPER SESSION
Electronics/Dielectric Science and Technology
L. K. White, Chairman; D. Bailey, Vice-Chairman
Promenade Ballroom A, 2nd Level
9:00 The Joint Recent News Paper Program will be sent to all members of the Electronics and Dielectric Science and Technology Divisions in April. Copies of the Recent News Paper abstracts will be available at the Session.

D. Bailey, Chairman; L. K. White, Vice-Chairman

2:00 The Joint Recent News Paper Program will be sent to all members of the Electronics and Dielectric Science and Technology Divisions in April. Copies of the Recent News Paper abstracts will be available at the Session.

ELECTROCHEMISTRY IN MINERAL AND METAL PROCESSING III
Industrial Electrolysis and Electrochemical Engineering
P. E. Richardson, Chairman; T. J. O'Keefe, Vice-Chairman
St. Louis Ballroom A, 4th Level
8:45 A Comparative EIS Study on Cermet and Platinum Anodes for the Electrolytic Production of Aluminum - C. F. Windisch, Jr.
9:15 Solid Metal Reductive Stripping of Cerium from Tri-n-Butyl Phosphate - T. J. O'Keeffe and J. Dillon III
9:45 The Electrochemical Behavior of Group V Elements during Copper Deposition - J. B. Hiskey and Y. Maeda
10:15 Fifteen-minute intermission
10:30 Mass Transport in High-Current-Density Zinc Electrowinning - K. J. Cathro
11:00 Full-Scale Hydrogen Diffusion Anodes for Immersed Tank Electrowinning and Electroplating - P. C. Foller, R. J. Allen, and R. Vora
11:30 Cathodic Reduction of Hg(II)-Cl(II) Complex on Ag-Hg Electrode - Q. Yin

ELECTRO-ORGANIC SYNTHESIS WITH HOMOGENEOUS AND HETEROGENEOUS CATALYSTS
Organic and Biological Electrochemistry
D. G. Peters, Chairman; J. E. Teooney, Jr., Vice-Chairman
Promenade Ballroom B, 2nd Level
9:00 Use of [2,2'-Ethylenbis(nitromethylidene)]diphenolato[tricarboxylato] as a Homogeneous and Polymer-Based Catalyst for Reduction of Organic Halides - D. G. Peters, C. E. Dahm, and M. S. Mubarak
9:30 Long Life Anion Radicals from AOX or RX Type Compounds: Mediators, or Ar and R Providing Species? Case of Olivo-Blei(allylsulphuryl) Benzenes - J. Simonet
10:00 Conversion of -isooctene to Piperonal Using Electrolytically Recycled Higher Oxides of Manganese - J. Grimshaw and C. Hua
10:30 Fifteen-minute intermission
11:15 Electrochemistry of 9,9'-Spirobifluoren Derivatives - D. J. Rampazzo

F. M. Hawkridge, Chairman; D. G. Peters, Vice-Chairman

2:00 A Study of Substitutedカテchols as Electron Transfer Mediators in Electrocatalytic Enzyme Reactions - T. J. Moore and L. A. Coury
2:30 Electrocatalytic Cleavage of the C-Cl Bond of 9-Chlorofluorenes - F. Maran and E. Vianello
3:00 Fifteen-minute intermission
3:15 Electrochemical Behavior of Cytochrome c, Hildenborough on a Gold Electrode - B. A. Gorecka and G. S. Wilson

ELECTROCHEMISTRY OF HIGH TEMPERATURE SUPERCONDUCTORS
Physical Electrochemistry
M. Norton, Chairman; J. T. McDevitt, Vice-Chairman
Room 26, 2nd Level
9:00 Reactivity of Compound Superconductors: Cuprates, Bismuthates, Fullerides - B. Miller and J. M. Rosamilia
9:40 Controlled Room Temperature Formation of Weak Link or Josephson Junction in Thin Film YBa$_2$Cu$_3$O$_y$ - D. Cahen and Y. Socolnik
10:20 Fifteen-minute intermission
10:55 Electrochemical Response of YBa$_2$Cu$_3$O$_y$ as a Function of Oxygen Content - D. R. Riley and J. T. McDevitt
11:15 Application of High Temperature Electrochemical Techniques to YBa$_2$Cu$_3$O$_y$ - R. V. Kumar, D. J. Fray, J. E. Etvets, H. W. Williams, and A. Misson

FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS
Physical Electrochemistry/Dielectric Science and Technology/Electronics
L. Chiang, Chairman; D. Bethune, Vice-Chairman
St. Louis Ballroom C, 4th Level
9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

S. Iijima, Chairman; C. Lieber, Vice-Chairman

2:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

K. M. Kadish, Chairman; R. S. Ruoff, Vice-Chairman

7:00 Open Discussion/Late News
Abstracts

JOINT GENERAL SESSION

Battery/Energy Technology

1 Experimental Determination of the Transport Number of Water in Nafion 1170 Membrane: T. F. Fuller and J. Newman, Lawrence Berkeley Laboratory, and Dept. of Chemical Engineering, University of California, Berkeley, California 94720

The transport number of water in Nafion® 117 membrane over a wide range of water contents is determined experimentally using a new technique. The transport number of water in Nafion® 117 at high hydration is close to 0.5, which is consistent with the assumption that water molecules are transported in the membrane. At lower water contents, the transport number decreases, indicating a change in the water transport mechanism. These results have implications for the design and optimization of fuel cell systems that use Nafion® 117 as a membrane.


A new electrochemical catalysis (ECC) technique is used to prepare high utilization carbon supported platinum electrocatalysts for proton exchange membrane fuel cells (PEMFCs). The electrocatalysts are prepared by sputtering Pt on a carbon substrate and then activating. The resulting electrocatalysts have high activity and selectivity for the oxygen reduction reaction (ORR) and are suitable for use in PEMFCs. This technique offers a promising approach to improving the utilization of Pt catalysts in PEMFCs and other fuel cell applications.

3 Temperature and Pressure Dependence of the Electrode Kinetics of Oxygen Reduction at the Platinum Microelectrode/Nafion Interface: M. Parhasarathy, Dept. of Chemistry, Colorado State University, Fort Collins, CO 80523

The dependence of the oxygen reduction reaction on temperature and pressure at the platinum microelectrode/Nafion interface is investigated. The reaction rate is found to be dependent on both temperature and pressure, with the activation energy and pre-exponential factor changing with pressure. This information is crucial for the design and optimization of fuel cells that use Nafion® as a membrane.


It has been established that hydrous oxides can form spontaneously on platinum surfaces. These oxides have been found to improve the catalytic activity of platinum for the oxygen reduction reaction. The formation of these oxides is strongly influenced by factors such as temperature, pressure, and gas composition. Understanding the formation and properties of these oxides is crucial for the development of more efficient fuel cell technologies.

5 Effect of Platinum Loading on Proton Exchange Membrane Fuel Cell Performance: A. C. Ferrera, S. Sirimuthan, and A. J. Appleby, Center for Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-3402

The effect of platinum loading on the performance of PEM fuel cells is investigated. The results show that the performance of PEM fuel cells increases with increasing platinum loading up to a certain point, after which further increases in platinum loading do not significantly improve performance. This information is crucial for the design and optimization of fuel cells that use platinum as a catalyst.

6 The Hydrogen Diffusion Porous Electrode Catalyzed by Platinum: Z. E. Z., A. P. Huag, T. G. Zheng, and G.-N. Fang, Dept. of Chemistry, East China Normal University, Shanghai, China

The hydrogen diffusion porous electrode (HDPE) catalyzed by platinum is studied. The HDPE is composed of a porous platinum film and a solid platinum core. The diffusion of hydrogen through the porous film is facilitated by the presence of the solid platinum core. This results in an improved performance of the HDPE as a hydrogen diffusion porous electrode. The HDPE is a promising candidate for use in fuel cells that require high hydrogen diffusion rates.

7 Optimization of Electrode Structure to Further Minimize the Platinum Loading in Proton Exchange Membrane Fuel Cells: A. C. Ferrera, S. Sirimuthan, and A. J. Appleby, Center for Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-3402

One of the promising approaches to develop and commercialize proton exchange membrane fuel cells is to minimize the platinum loading. The present investigation shows that the electrode structure is an important factor in determining the platinum loading. Improvements in the electrode structure can lead to a significant reduction in the platinum loading, which is crucial for the development of more cost-effective fuel cells.


A thin film solid oxide fuel cell (SOFC) is prepared using reactive DC magnetron sputtering. The sputtering process is used to deposit a thin film of platinum onto the SOFC. The platinum layer is used as an electrode for the SOFC. The results show that the SOFC prepared using reactive DC magnetron sputtering has high performance and is a promising candidate for use in fuel cell systems.

9 Effects of a Sputtered Film of Pt on Performance of Proton Exchange Membrane Fuel Cells: Electrode Kinetic and Morphological Characterizations: S. M. Kenney, S. Sirimuthan, and A. J. Appleby, Center for Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-3402

The effects of sputtered platinum films on the performance of PEM fuel cells are investigated. The results show that the performance of PEM fuel cells is improved by the presence of sputtered platinum films. The improvement in performance is attributed to the changes in the morphology and structure of the platinum films.

10 Mechanism of Mixed Fuel/Oxidant Solid-State Thin Film Fuel Cells: D. M. Coffey, T. O. Fiehler, and A. J. Appleby, Dept. of Materials Science, Johns Hopkins University, Baltimore, MD 21218

The fabrication and characterization of solid-state thin film fuel cells is investigated. The cells are fabricated using a thin film of platinum as the catalyst. The results show that the performance of the thin film fuel cells is comparable to that of conventional PEM fuel cells. The thin film fuel cells are promising candidates for use in fuel cell systems that require high performance at low cost.


For solar energy systems, long term energy storage is required. Hydrazine is a suitable energy carrier and its generation is a well-established technology. Electrolyzers based on proton exchange membranes are known for their high energy density, high efficiency, and long term performance. The present investigation shows that the performance of these electrolyzers is improved by the use of a solid electrolyte. The electrolyzers are promising candidates for use in solar energy systems.

12 Calorimetric Concentration and Mass Flow Monitor for Gas Phase Oxides: P. C. Foller, 2000 Commonwealth Ave., Unit 1110, Boston, MA 02125

An inexpensive calorimetric ozone mass flow and concentration sensor has been developed using catalyzed platinum electrodes. The sensor is sensitive to a wide range of gas phase oxides and is capable of determining concentration and mass flow rates. The sensor is promising for use in monitoring gas phase oxides in industrial and environmental applications.
THURSDAY CONTINUED

EIGHTH INTERNATIONAL SYMPOSIUM ON MOLTEN SALTS
Physical Electrochemistry/High Temperature Materials
H. Kojima, Chairman; J. R. Selman, Vice-Chairman
St. Louis Ballroom B, 4th Level

9:00 Synthesis of Molybdenum-Doped Alkaline Metal Vanadium Bronzes by Molten Salts Electrolysis - Y. Kaneko, H. Ishikawa, and H. Kojima
9:20 Electrodeposition of Molybdenum and Molybdenum Carbide Coatings from Oxide-Based Molten Salts - J. R. Selman, B. Aladjov, and D. Topor
9:40 A Novel Pulsating Current Technique Used in the Study of Platinum and Molybdenum Electrodes in Molten Silicates, Borates, and Phosphates - J. K. Higgins
10:20 Ten-minute intermission
10:30 Electrochemical Formation of Thin Carbon Film from Molten Chloride System - Y. Ito, T. Shimada, and H. Kawamura
10:50 Melting Behavior for Powder/Hydrated Meth(CaCl2,nH2O n=6.00, 7.38) Coexisting Systems - S. Deiki, M. Mizuhata, A. Kajinami, and Y. Kanaji
11:10 Transfer Numbers in Molten Fluorides by an Operationally Defined EMF-Method - T. Forland, S. K. Ratlje, and H. Rajabu
11:30 Thermodynamics of Li2O-LiF-CaF2 Melts - S. T. G. Sampath Kumar, A. Narayan, and R. G. Reddy

J. R. Selman, Chairman; H. Kojima, Vice-Chairman

FRIDAY, MAY 22, 1992

NINTH SYMPOSIUM ON PLASMA PROCESSING
Dielectric Science and Technology/Electronics
J. P. McVittie, Chairman; G. S. Mathad, Vice-Chairman
Promenade Ballroom C, 2nd Level
PECVD (cont'd)

9:00 Characterization of PECVD Nitride Films Used in VLSI Applications - A. K. Stamper, S. L. Pennington, and G. Bazan
9:20 Deposition of Silicon Nitride Films by ECR-Enhanced CVD - J. D. Chapple-Sokol and D. E. Kotecki
9:40 Plasma Deposition and Characterization of Fluorinated Silicon Nitride - S. V. Nguyen, D. Dobuzinsky, R. Gleason, and M. Gibson
10:00 Modeling of PECVD TEOS Oxide Step Coverage Using an Overhang Structure - C. Chang, J. P. McVittie, and K. C. Saraswat
10:20 Low Temperature Deposition of SiO2 by DECR-PECVD - B. Agius
10:40 Effects of RF Frequencies and Deposition Rates on the Moisture Resistance of PECVD TEOS-Based Oxide Films - S. A. Robles, M. Galliano, and B. C. Nguyen
11:00 Formation of High Quality Tantalum Thin Films on SiO2 by Dual-Frequency-Excitation Plasma Process - H. Wakamatsu, S. Aoyama, J. Watanabe, N. Konishii, and T. Ohno

J. P. McVittie, Chairman; G. S. Mathad, Vice-Chairman

11:20 Control of Stress, Stability, and Mechanical Properties of PECVD Dielectric Films for GaAs and Si Applications - T. H. Wu, L. A. Schneggenburger, B. van Schravendijk, B. Sparks, A. S. Harrus, and D. G. Hammes

FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS
Physical Electrochemistry/Dielectric Science and Technology/Electronics
S. Saito, Chairman; D. Lorents, Vice-Chairman
St. Louis Ballroom C, 4th Level

9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.

M. Schluter, Chairman; H. N. Shinohara, Vice-Chairman

11:20 Control of Stress, Stability, and Mechanical Properties of PECVD Dielectric Films for GaAs and Si Applications - T. H. Wu, L. A. Schneggenburger, B. van Schravendijk, B. Sparks, A. S. Harrus, and D. G. Hammes

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9:00 The program and abstracts will be published in the April issue of the Journal and included in the program booklet distributed at the Meeting.
137C


terminated via the resistance of the catalyzed PRT. Use is forecast in multiple remote sensing reporting to a central display, a task not totally accomplished with conventional ultraviolet spectrophotometric technology.

12 SPECIFIC Development at the Centre for Electrochemical and Energy Research, SPIC Science Foundation: S. Parthasarathy, Contribution from the Centre for Electrochemical & Energy Research Foundation, Madras 600 032, India

SPECIFIC development has been initiated at SPIC Science Foundation, as SPECIFIC is an ideal candidate for transportation for a number of reasons. This paper describes the work in progress since 1989 in collaboration with CESSHR, Texas A&M University. Considerable work has been done in reducing platinum load and optimizing the process for fabrication of electrodes. The results of characterization of electrodes using cyclic voltammetry and mass spectrometry are assessed. Platinum utilization is determined and the effect of the electrolyte in the separator should be included in the mathematical analysis.

15 The Rechargeable Li-TIS/LiAlCl(Li, CoO, Solid-State Electrode: K. W. Brehl and E. J. Plutchik, U.S. Army Electronics Technology and Devices Laboratory, Fort Monmouth, NJ 07703-5801, D. Veitch, H. S. W. Wang, and D. M. Schlesich, Polytechnic University, Brooklyn, NY 11201

An all-solid-state cell employing lithium tetrachloraluminate (LiAlCl) as the lithium ion-conducting material and lithium intercalating compounds, titanium disulfide (TiS), and lithium cobalt (III) oxide (LiCoO), as the electroactive materials in a "rocking chair" configuration is described. The cell was operated at 180°C and exhibited an open circuit potential of 2.1 V in the discharged state. The cell showed significant discharge characteristics at current densities up to 0.1 mA/cc and also showed no significant loss of capacity over one hundred charge-discharge cycles.


Polymerization and characterization of polyaniline and substituted derivatives are described. The thickness of the polyaniline studied in relation to the polymerization conditions. The charge capacity from the current-potential curves of each polymer is found to be different in different solutions and shows linear relationship to the thickness of the polymer over a wide range of thicknesses. The chemical structure of these polymers has been determined from XPS-data.

17 Investigations on a Novel Zn/KOH/DDH Primary Cell System: R. Udhayan, D. P. Bhat, and P. B. Mathur, Central Electrochemical Research Institute, Karaikudi-623 006, India

The work described in this paper relates to the studies on a novel zinc primary cell involving NaF, 1NaF, 1NiS, and alkylhydantoin (DDH) as a cathodic depolarizer and the commercial zinc as an anodic material. The novelty of the system lies in the fact that the organic cathodic material employed in this investigation possesses non-toxic property and the zinc cell system, reported here, in conjunction with DDH shows high energy density of 212 W h/kg and 360 W h/l (1) at the higher operating voltage of ~2.0 V. Workable current density of this system is reported up to the figure of 13 mA cm⁻².

18 Low Temperature Removal of Hydrogen Sulfide from Sour Gas and Its Utilization for Hydrogen and Sulfur Production: M. Petrov, I. Sucharski, and A. J. Appleby, Central Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-4002

This work is concerned with removal of H₂S from sour gas by the partial oxidation. The mixture of H₂S and air is fed into a reaction vessel where a stream of O₂ is blown through the mixture. The oxygen then oxidizes the H₂S to SO₂. The SO₂, which is produced, is then scrubbed with water to produce sulfuric acid. This process can be made continuous.

DIRECT ELECTROCHEMICAL OXIDATION OF METHANOL AND SMALL MOLECULES

19 Direct Electro-Oxidation of Methanol in Acid Medium at Modified Electrodes with Low Precious Metal Loadings: J. M. Leger, G. Meli, and C. Laboratoire de Electrochimie et Interactions, URA CNRS No. 350, Universite de Poitiers, 86022 Poitiers, France

The direct electro-oxidation of methanol in acid medium is an attractive way to develop fuel cells. For practical applications, the metal loading must be decreased and the electrocatalytic activity increased. Modified electrodes using non-precious metals with low platinum loadings, display much less poisoning than bulk electrode. By adding a second metal (ruthenium or tin), the potential of methanol oxidation is shifted negatively. PMO is highly enhanced by feeding methanol from the gaseous phase.


Pt supported carbon black catalysts have been investigated by surface adsorption measurements and cyclic voltammetry in sulfuric acid and methanol. The maximum in reaction rate in acid electrolytes showed a volcano-shaped correlation as a function of the pHm with a maximum at about pHm = 6. The methanol oxidation process showed higher overpotential for catalysts with extreme pHm values. This behavior may be interpreted in terms of different metal-support interactions.


We are investigating carbon supported platinum alloy electrocatalysts for methanol oxidation. The alloy electrocatalysts were selected from the binary systems Pt-Au and Pt-Ru and from the ternary system Pt-Au-Ru. We are preparing the electrocatalysts using both chemical and electrochemical techniques and are characterizing the alloys using TEM and XRD. We report the electrochemical activity for methanol oxidation and also the effect of alloy composition and chemical vs. electrochemical preparation.

22 Methanol Oxidation on NiTi: R. Mochanfar and J. B. Goodenough, Center for Materials Science and Engineering, University of Texas, Austin, TX 78712-1084

The 50-50 NiTi ordered alloy, in acid, was chosen as an initial candidate in a search for platinum-free alloy electrocatalysts for the methanol oxidation reaction (MOR). We report observation of the MOR on this alloy, showing Tafel slopes with an initial Tafel slope in the passivating layer. The effect of specific adsorption of anions and cations on the kinetics of methanol electro-oxidation on NiTi single crystal surfaces: N. Ross and N. Markovic, Materials Sciences Div., Lawrence Berkeley Laboratory, University of California, Berkeley, CA 94720

The kinetics of the electrochemical oxidation of methanol on Pt(111) and Pt(100) single crystal electrodes were studied in acid and alkaline electrolytes containing small quantities of various ions, both cations and anions, which interact to varying extent with the Pt surface. The Pt surfaces were prepared in open hydrogen-air flame and in UHV, and characterized by LEED and AES before transfer to the electrochemical cell via a differentially pumped manifold. Specifically adsorbing anions like bisulfate and chloride and cation like cesium have a strongly negative effect on the kinetics. The maximum in reaction rate in acid electrolytes (NHE) is shown to be due to the formation of a critical coverage of adsorbed anions, and not due to the formation of a "Pd-like" monolayer as previously supposed. The maximum in reaction rate in the alkaline electrolyte containing cesium is observed in the region where Cs⁺ is incorporated in the "Pt OH" layer which, surprisingly, has an inhibiting rather than a catalytic effect on methanol oxidation.

24 Oxidation of Methanol on Single Crystal Platinum Electrodes in Sodium Hydroxide and Sodium Carbonate Solutions: R. Adic, N. Markovic, V. Tripkovski, and N. Markovic, Institute of Electrochemistry, University of Belgrade, Njegoeva 12, Belgrade, Yugoslavia

Oxidation of methanol on platinum in sodium hydroxide and sodium carbonate solutions has a pronounced dependence on the crystallographic orientation of the electrode surface, contrary to some reports in the literature. The rates of reaction are considerably higher than in acid solutions when the activated Pt electrode is mixed with the solution obtained by hydrogen sulfide scrubbing and returned to the electrolytic cell. Hydrogen and sulfur with high purity are produced at low cell potential (E = 1 V) and at a high current density (t = 300 mA/cm²). This investigation demonstrates that the process can be made continuous.
tion of methanol to more negative potentials, but decrease of the current peak. Vicinals of Pt(110) cause increase of the current peak, without effect on the onset of the reaction.


A new method for the investigation of electrocatalytic reaction on high-surface area electrodes is described. Variable angle reflectance spectroscopy (VARS) was used as a tool for the identification of various attenuation and scattering components associated with the reflection of infrared light from the electrode under potential control. The potential dependent distribution of the products of the direct oxidation of methanol on Pt-carbon electrodes is measured, in situ, using this technique.


In a study of the effects of electrode macrostructure on the performance of certain gas sensors, L. D. Burke, D. T. Buckley, and J. K. Casey, Chemistry Dept., University College Cork, Cork, Ireland.

The oxidation of metal oxide to low potentials on platinum in aqueous media is inhibited due to formation of deactivating species, e.g., CO$_2$. The improved performance of certain alloys, e.g., 30% Pt/Ru, is due to the production of the second component in the mixture) that promote conversion of CO$_2$ to CO. Evidence for the formation of active oxides on platinum at unusually low potentials, and their role in electrocatalytic oxidations, are presented.

38 A Methanol–Aqueous Carbonate Fuel Cell: J. A. Koozek, S. Sarangapani, and J. Giner, Giner, Inc., Waltham, MA 02154
Methanol adsorption on Pt, measured by H₂ displacement in 1 M K₂CO₃ over a broad concentration range of 0.01–10 M, yielded a bell-shaped potential dependence. Tafel plots show that methanol oxidation in carbonate has better kinetics than in an acid electrolyte. Complete methanol–oxygen–aurous carbonate fuel cell testing yielded Ir-correlated performance of up to 570 mV at 150 mA/cm². The absence of any long-term poisoning effects was demonstrated.

39 Direct Electrochemical Oxidation of Methanol Vapor on PEM Electrode: A. S. Lin and W. E. O’Grady, Naval Research Laboratory, Washington, DC 20375
The study of gold as a catalyst on proton exchange membrane has been performed for direct electrochemical oxidation of methanol. A vapor deposited gold thin film on Nafion was used as electrode to study the methanol vapor reaction. An A/S model has been used in the gas fed configuration. Oxidation current occurs near the potential of Au oxide growth region. The result suggests that the active sites on gold surface are not deactivated by the strongly adsorbed intermediates.

40 Methanol Electro-Oxidation: Problems, Progress, and Prospects: S. M. Kulkarni, S. Srinivasan, and A. J. Appleby, Center for Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-3402
The development of high performance direct methanol air fuel cells for vehicles is of major importance for defense applications. In order to achieve the performance levels of indirect methanol fuel cells (power densities >200 mW/cm²), the challenging problem is to find electrocatalysts which have high electrocatalytic activities and which are not poisoned by the intermediates. Recently, significant advances have been made with bifunctional catalysts utilizing platinum or platinum–ruthenium alloys, with the latter as promoters for methanol oxidation. Furthermore, amorphous metallic alloys (Pt with Pd, Zr or Mo) have been found to be more stable than crystalline counterparts and exhibit higher catalytic activities. Here too the catalysts are bifunctional, by providing active O or OH sites for oxidation of intermediates during methanol oxidation. Progress for applications of these electrocatalysts at the electrode/solid polymer electrolyte interfaces are encouraging because perfluorosulfonic acids an increased activity for methanol oxidation has been demonstrated. An alternative approach is to use the fluorinated aids as additives to phosphoric acid and operate the cells at about 200°C.

41 Laser Initiated Corrosion Pits on Aluminum: D. Buzo and R. Alkire, Dept. of Chemical Engineering, University of Illinois, Urbana, IL 61801
A focused laser beam was used to initiate corrosion pits on 99.998% pure aluminum immersed in 1 M NaCl, pH 11 solution and held under potentiostatic control. The shape of the pits as measured by SEM and the close correlation with the electrochemical behavior of the induced anodic current were compared with the measured net anodic current. In order to investigate the shape evolution of a group of pits, the laser initiation technique was adapted to initiate ordered arrays of pits.

42 The Characterization of Crevice Solution Chemistry during the Initiation Stage of Crevice Corrosion: B. K. Nash and R. G. Kelly, Center for Electrochemical Science and Engineering, Dept. of Materials Science, University of Virginia, Charlottesville, VA 22903
This work is concerned with identifying the mechanism of crevice corrosion initiation in 304 stainless steel. It has been suggested that the acidification of the crevice solution by hydrolysis is not the mechanism responsible for initiation, but that exceeding a critical chloride and thiocyanate concentration is the condition leading to depassivation. Through the use of ion chromatography, this study will quantitatively determine the crevice solution concentrations of sulfur species and transition metals present in active areas in crevice solutions near the initiating sites. The findings of these experiments will allow an assessment of the critical crevice solution which develops prior to initiation of crevice corrosion in 304 stainless steel.

43 Stress Corrosion Behavior of TIG Welded 304 Stainless Steel: J. J. E. Teng, K. T. Cheng, and J. L. Yang, Materials Research Laboratories, Ching Hsing, Hsinchu, Taiwan, China
The sulfide stress corrosion cracking (SSC) of TIG welded 304 stainless steel was studied by stress corrosion cracking (SCC) test in NACE solutions. The microhardness test of weld specimens showed that a lower hardness region was obtained in the heat affected zone (HAZ) in comparison with those in fusion zone and base metal. The failure time of the welded specimen decreased with the increase of H₂S concentration. Failure was found at the border, which had a maximum hardness value of 190 Hv, of the affected zone and the fusion zone for all specimens studied.

44 Laser Raman Spectroelectrochemical Studies of Fe, Ni, Cr, and Their Glassy Metal Alloys with Phosphorus: M. Pankuch and C. A. Melendres, Argonne National Laboratory, Materials Science and Engineering, Argonne, IL 60439-4373, J. C. Kang and S. L. Lalvani, Dept. of Mechanical Engineering and Energy Processes, Southern Illinois University Carbondale, IL 62901, Y. S. Li, Dept. of Chemistry, Memphis State University, Memphis, TN 38152
The spectroelectrochemical behavior of Fe, Ni, and Cr was studied in 0.15M NACI solution with and without various glassy metal alloys with phosphorus. While a significant alteration in the corrosion and anodic dissolution behavior of the metals is observed on alloying, the composition of the surface film appears to be essentially the same. Fe(OH)₂, Fe₃O₄, and FeO(OH) were found on the Fe surface and Cr₂O₃ on the Cr. No evidence of phosphate incorporation into the film was observed.

45 Surface Analysis and Corrosion Studies of Iron-Based Metals in Para-Toluene Sulfonic Acid: M. Y. Teng and J. L. Yang, Materials Research Laboratories, Chuting, Hsinchu, Taiwan, China
Potentiodynamic and surface analysis techniques were performed to determine the surface analysis techniques were performed to determine the potential for ferrous substrates usually results in the formation of corrosion products, 2000 mW/cm². This approach entails further evaluation of spectral reflectance measurements as a new method of differentiation. The spectral method is compared to the former fuel cells (power densities >200 mW/cm²), the challenging problems are to find electrocatalysts which have high electrocatalytic activities. Here too the catalysts are bifunctional, by providing active O or OH sites for oxidation of intermediates during methanol oxidation. Progress for applications of these electrocatalysts at the electrode/solid polymer electrolyte interfaces are encouraging because perfluorosulfonic acids an increased activity for methanol oxidation has been demonstrated. An alternative approach is to use the fluorinated aids as additives to phosphoric acid and operate the cells at about 200°C.

46 Film Thickness Effects on Flash-Rusting Measured by Spectrophotometry and Atomic Absorption: M. R. Van De Mark, E. Sinmaurat, and L. E. Pounds, Dept. of Chemistry University of Missouri-Rolla, Rolla, MO 65401
The use of latex paint on ferrous substrates usually results in the formation of corrosion products, 2000 mW/cm². This approach entails further evaluation of spectral reflectance measurements as a new method of differentiation. The spectral method is compared to the former fuel cells (power densities >200 mW/cm²), the challenging problems are to find electrocatalysts which have high electrocatalytic activities. Here too the catalysts are bifunctional, by providing active O or OH sites for oxidation of intermediates during methanol oxidation. Progress for applications of these electrocatalysts at the electrode/solid polymer electrolyte interfaces are encouraging because perfluorosulfonic acids an increased activity for methanol oxidation has been demonstrated. An alternative approach is to use the fluorinated aids as additives to phosphoric acid and operate the cells at about 200°C.

47 The Analysis of Atmospheric Corrosion on Fe Particle Magnetic Tape: J. P. Dante and R. G. Kelly, Center for Electrochemical Sciences and Engineering, University of Virginia, Charlottesville, VA 22903
The degradation of metal particle magnetic recording tape during atmospheric exposure has been studied. Corrosion rates were measured using a quartz crystal microbalance (QCM). The composition of the adsorbed electrolyte layer has been analyzed using ion chromatography. The effects of relative humidity, temperature, pollutant gas type (SO₂, NOₓ, and Cl₂) and concentrations were investigated. The individual contributions of the degradation of the various components of the tape on the degradation of performance are presented and discussed.

48 Corrosion of Alloys in a Marine Environment under AC Conditions: M. A. Pagano, W.-W. Chu, and S. B. Lalvani, College of Engineering and Technology, Southern Illinois University Carbondale, IL 62901
Simosoidal alternating voltage (AV) was used to study the effects of periodic fields on the corrosion of 1018 carbon steel and 90/10 Cu/Ni in simulated seawater. Experimental data show that under the influence of AV, the corrosion rate of carbon steel increases with applied peak voltage then decreases in the range 100–600 mV. Increasing the voltage above 600 mV results in an exponential increase in corrosion rate. For 90/10 Cu/Ni, the corrosion rate increases monotonically with applied voltage (i–1500 mV). To help understand this phenomenon, series of experimental analysis were performed: (i) potentiodynamic polarisation, (ii) characterization of corroded specimens with SEM and EDX, and (iii) computer modeling of the corrosion system based on de polarization results. Additional effects of frequency and rectification of signals are also determined and discussed.

As the evaluation of electrochemical impedance spectra is model-dependent, there is a need for general models that can be applied to a variety of systems and have the most meaningful, too. Employing only circuit elements that can be related directly to single physical processes, a model is proposed in this paper. Several studies of applicability is demonstrated by a number of examples. The model, which takes into account the presence of diffusion zones with and without electrochemical reactions, seems to be very useful for the evaluation of the electrochemical impedance spectra of both active and passive metals and alloys. The interpretation of the results on extremely corrosion resistant materials like surgical implant alloys may also become less difficult, despite the more complex nature of the spectra.
50 Anodization of Copper in Ethylene Glycol-Water Mixture. A Study Using Microelectrode. J. Ostermeyer, and K. Wikel, Dept. of Chemistry, SUNY at Buffalo, Buffalo, NY 14214. Anodization of copper ultratrace electrodes in 35% ethylene glycol-water mixture in the presence of chloride ions was studied by means of microelectrode voltammetry, and constant potential step methods. Anodic behavior of copper in the presence of chloride ions in ethylene glycol-water solution does not basicly differ from behavior in pure chloride-ion-water solution. The product of anodic dissolution of copper, in solutions containing more than 0.05M chloride ion, is the soluble dichlorocomplex of monovalent CuCl and tetravalent CuCl₂. This dissolution potential overpotential is not very high and the potential is not applied to the electrode for a long time (milliseconds time scale). The analysis of the plot of potential vs. log chloride concentration gives the conditional number of copper dichlorocomplex as 2 and the stability constant of CuCl₂ complex in ethylene glycol-water mixture, as log β = 5.6.

51 Polarization Characteristics of Ni and Inconel 600 in Aggressive and Inhibitive Acidic Media. A. A. Abd El-Fat-tah, E. M. Mahrouk, H. E. Mogahid, and M. Ahd-Allah, Chemistry Dept., Faculty of Science, Zagazig University, Benha, Egypt. The pit corrosion potential of nickel and Inconel 600 varies with the concentration of Cl⁻ ions according to a semidual surface-controlled mechanism. This behavior is due to the existence of a film on the electrode surface. The potential shift of pitting corrosion of nickel and Inconel 600, varies with the concentration of Cl⁻ ions and also with the nature of the electrolyte. The potential of pitting corrosion of nickel and Inconel 600 is increased by the increase in the chloride ion concentration. The presence of chloride ions increases the passivation current of nickel and Inconel 600. The presence of chloride ions increases the passivation current of nickel and Inconel 600. The chloride ions shift the pitting potential of the electrode to lower values.

52 Corrosion of Mg and Its Alloys in Aqueous Mg(ClO₄)₂ Batter Electrolyte-A RDE Study. R. Udawant and D. P. Bhatt, Central Electrochemical Research Laboratory, Karaikudi-623 006, India. This paper reports the first results pertaining to the corrosion aspects of magnesium and its AZ31 alloy in aqueous Mg(ClO₄)₂ battery electrolyte employing a versatile A RDE technique. From the current-potential profiles, the pit corrosion potential, kinetic current, etc., have been determined, and the results have been discussed in terms of the film formation and the passive film controlled mechanisms. Interestingly, the mechanism of the dissolution process has been found uniform at all the chosen potentials and the inhibition mechanism is also discussed in detail.

53 Ellipsometric Study on Inhibition Effect of Molybdate for Cooling Water. S. F. Xie, Y. R. Yang, and Z. Q. Huang, Dept. of Applied Chemistry, Chongqing University, Chongqing, China. In consideration that it is difficult to obtain information about processes through the electrochemical methods, this paper describes the investigation of the inhibition effect of cooling water through the ellipsometric technique. The optical constant, thickness of film, and coverage are calculated from a. W measured. The inhibition mechanism is also discussed in detail.

CATHODIC PROTECTION SYSTEMS

54 Influence of Al,Fe Intermetallic Inclusions on Depassiva-

55 oxidation of Steel in Acidic 54 - with 55 tion of Cathodic Protection Systems: J. A. F. Santiago, J. C. F. Telles, W. J. Mansur, and L. Sathler, COPPE/Federal University of Rio de Janeiro, CEP 21945 Rio de Janeiro, Brazil. The present paper is concerned with the boundary element modeling of potential and current density distribution in cathodic protection systems. This phenomenon is governed by the Poisson equation, subjected to nonlinear time-varying boundary conditions, relating potential and current density, described by dynamic polarization curves determined from potentiostatic experiments. Two solution methods are studied in order to simulate the dynamic polarization curves as time advances, namely, fictitious time and fictitious potential.
and current flow to the buried region, are those found in the literature. With the purpose of ascertaining current distribution and electrochemical potential in the two regions, cathodic protection system. The x-rays were numerically analyzed through the PROCAT computer program, which is based on the boundary elements method.

**Ninth Symposium on Plasma Processing**

Dielectric Science and Technology/Electronics


This paper discusses the use of a profile simulator to help understand the mechanisms leading to experimentally observed profiles. The simulator is a special experimental test structure to extract needed parameters for anisotropic oxide etching processes. The focus is on developing correct models for the mechanisms which control wall slopes and RIE lag effects in CHF$_3$ based oxide etching.

63 Use of Overhang Test Structure to Understand RIE Lag in Oxide Etching: S.-D. Dow and J. P. McVittie, Center for Integrated Systems, Stanford University, Stanford, CA 94305-4070.

Overhang test structures which eliminate sidewall effects were used to study the mechanisms responsible for the RIE lag effect in CHF$_3$ based oxide etching. RIE lag was observed in these structures and is not restricted to sidewall mechanisms. Bottom etch widths and the linear dependence of the etch rate on aspect ratio suggest that neutral transport controls the lag in these test structures.

64 Modeling of Plasma Etching Reactors Including Wafer Heating Effects: Y. L. Wang and T. M. Niemczyk, Dept. of Chemical Engineering, University of Houston, Houston, TX 77204-4792, E. Aydil, AT&T Bell Laboratories, Murray Hill, NJ 07974.

A comprehensive model for chlorine etching of polysilicon has been developed including wafer heating effects. Spatiotemporal variations of atom density, etch rate, and wafer temperature were predicted. Wafer heating caused the etch rate to increase with time despite the fact that the etchant concentration decreased with time. Etch rate and uniformity were measured in real time using an novel multichannel optical emission technique. Measured and predicted etch rate transients were compared to model predictions. Results were found to be sensitive to wafer back-side cooling and the surface kinetics.


The nonuniform magnetic field gradient around the ECR position and the parabolic 875 G magnetic field profiles cause the inclination of plasma generation. The ion flight directions are disturbed by the nonuniform plasma. Conversion of the uniform magnetic field gradient and flat magnetic field distribution result in optimally uniform ECR plasma generation. The uniform ECR plasma prevents microloading effects in sub-half-micron pattern fabrication.


In various gas plasmas, variation of important plasma parameters such as ion energy, ion flux, and plasma potential has been evaluated by measurement of the RF waveform applied to the RF electrode, which is the substrate surface, without any contamination due to plasma to the process. We have demonstrated that high performance plasma process of low ion energy and high ion flux can be achieved by low bond energy gases excited plasma.

67 Dependencies of Negative Ions from Pulsed Radio-Frequency Discharge: T. J. Oorrent, L. Luo, and Y. Lin, University of Texas at Dallas, Richardson, TX 75083-6888.

The negative ion spectra from pulsed RF discharges through etching gases of density and direct ion mass spectrometry, and multiple negative ions were found in each gas mixture studied. The heavy mass negative ions in some discharges appear related to clustering with etching products. The signal intensities of small mass ions did not vary by a significant amount over long times, but some large mass negative ions exhibited an "onset" time dependence approximately 5 min. (This work was supported in part by the National Science Foundation ECS-9098662 and Texas Instruments Inc.)


In situ real-time ellipsometry coupled with comprehensive post-plasma surface analysis has been used to clarify selectivity and directionality mechanisms of dry etching processes of silicon-related materials. Examples of the techniques that have been employed are described. Combining reactive ion etching and ellipsometry also enables high-resolution depth profiling. The profiling of the superlattice structures and ultrathin oxide-nitride-oxide (ONO) films are described.

69 Modeling and Investigation of RF Electrical Signals from Nitride Etch: S. W. Butler and K. Branker, Texas Instruments Inc., Dallas, TX 75256.

RF current, voltage, and phase data were collected on a nitride etcher. Analysis of the data proved that delivered power was not only a function of power supply setpoint, but also a function of the wafer. Using data from the full process ranges, a simple model of the etch rate was formed. Based on comparison of the data with documented machine maintenance, it is apparent that phase and/or impedance at the harmonics can be used as a diagnostic tool.


In situ monochromatic and spectrophotometric ellipsometry is a very accurate and nonintrusive technique to study the modifications of a surface that is subject to plasma treatment. The paper gives three major applications: (i) measurement of surface temperatures by ellipsometry; (ii) depth profiling of optical parameters by RIE and HeNe ellipsometry; and (iii) surface of sample modifications by IR spectroscopic ellipsometry.


Emission spectra from 80 to 800 nm wavelength were used to identify species observed in a non-line of sight down silicon films as a function of the excitation plasma source, 13.56 MHz or 2.45 GHz. The emission spectra are used to explain the atomic, ionic, and molecular reactions and interactions in the plasma and the stream regions of interest to organic ashing. The excitation source has a major effect on the species generated, and thus the downstream ashing characteristics.


We report on a simple technique that enables estimation of process parameters and etch characteristics from optical emission spectroscopy measurements. Through principal component analysis, we observe that 99% of the variance in the more than 1100 optical and mass spectra channels are accounted for by very few principal components of each of the 50 etch experiments. Along with the measured dependent variables, are used to build empirical models for real-time monitoring of control of reactive ion etching. To this end, both neural networks and linear regression analysis and data fusion were employed. Examples that estimate chamber status, chamber contamination, etch rate, and leak detection are given.


We have exposed a series of hard anodized aluminum coupons to NF$_3$/Ar and CF$_3$/O$_2$ plasmas under a variety of conditions. The samples were transferred in vacuo for XPS analysis. After plasma exposure, the surface shows a decrease in the amount of aluminum oxide.
oxide type bonding with a corresponding increase in aluminum fluoride or oxy-fluoride bonding for both source gases. The surfaces of the NF/Ar and CF$_2$/O$_2$ plasma exposed coupons show the same diaphragm species present; the major difference is increased fluorination of the NF plasma exposed coupons.


Trench endpoint signal was achieved from small feature sizes on the wafer, 0.85 μm square in cross section and 8 μm deep. This paper talks about the interferometry and diffraction techniques, light dispersion and collection techniques, and signal processing techniques used to build the instrument. The analysis of the signals is shown and the theoretical and actual signals are compared. Then the experimental signals from the above features are shown, and the apparatus used to collect that signal is presented.


The plasma characteristics of an ECR plasma etching apparatus were measured by the Langmuir probe techniques for different discharge conditions. A new plasma parameter, plasma power, was defined as the ion transportation capability of a plasma. The properties of the ECR plasma were explained clearly employing this parameter. The plasma power at its maximum has obtained always near the ECR layer. The result of poly-Si etching was also discussed using this parameter.

77 Electrical Measurement of Etching Parameters in an Oxide RIE System: S. I. Dobma and J. P. McVittie, Center for Integrated Systems, Stanford University, Stanford, CA 94305-4077.

Etching parameters were measured electrically by simple diagnostic techniques in a SiO$_2$ RIE system and correlated with etching characteristics. The etch yield was found to be a linear function of the square root of the ion energy even in the conventional RIE chamber. This result indicates that more accurate control of etching can be achieved by available etching systems by making use of electrical discharge measurements.

78 Gate Oxide Break-down Phenomena in Magnetron Plasma: M. Sekine, K. Horioka, H. Okano, Y. Matsuno, T. Matsushita, K. Hishokha, and Y. Yoshida, ULSI Research Center (ZPG), Toshiba Corp., 1 Toshiba-cho, Saiwai-ku, Kawasaki 210, Japan.

Gate oxide breakdown phenomena in a magnetron plasma were investigated from two viewpoints: the field distribution, shown by the microwave field distribution measurement and the voltage waveform; and the voltage waveform, shown by the Langmuir probe measurements. New phenomena, such as the increase of flux lines parallel to the wafer, was verified to cause degradation in the gate oxide.

79 Gate Edge Effects on Oxide Damage during Polyide Etching: C. Gabriel, VLSI Technology, Inc., San Jose, CA 95131.

Gate oxide damage from plasma etching of silicon gates and interconnections was studied. Three commercial etchers were compared, including a microwave ECR, an RF tool, and an ARX tool. Oxide damage was measured on large capacitors with various conditions of gate area, gate edge, and isolation edge. From Deion density calculations, most of the oxide damage correlates with the gate edge for gate lengths below about five microns. The defects will affect device reliability more than yield.


Spectroscopic ellipsometry and Schottky barrier I-V measurements have been used to assess the lattice damage and residual layer formation produced by poly-Si etching. In this study, blanket oxides (1000 Å) were exposed to a CHF$_3$/O$_2$/contact etching chemistry in an ECR system under various magnetic field conditions. Samples were then analyzed for damage concentration effects both before and after UV ozone cleans. Our results show the dependence of the resulting residue thickness and damage layer thickness on magnetic field strength and on this surface treatment.

81 A Model for Thin Oxide Damage in Nonuniform Discharge Environments: J. P. McVittie, Center for Integrated Systems, Stanford University, Stanford, CA 94305-4076.

MOS gate oxides can be severely degraded by charge buildup during plasma exposures. In this paper, we present a new model to explain the role of discharge nonuniformity in this damage, and apply this model to SPICE circuit simulations, and probe and breakdown measurements to both uniform and nonuniform discharges. The model also explains the importance of device structures and the negligible dc bias on this damage.


The fluorinated, disordered silicon surface layer formed on the silicon substrate surface in a RF diode and/or electron cyclotron resonance (ECR) plasma fed with fluorine-based etching gases, CF$_4$, or SF$_6$, has been studied using real-time ellipsometry and x-ray induced photoemission spectroscopy after sample transfer in vacuum conditions. A thin (<6.5 nm) fluorinated layer is formed for conditions which minimize the ion bombardment of the Si substrate, e.g., ECR processing without additional ion landing. The thickness of the fluorinated layer is strongly with the sheath voltage at the substrate, but is relatively insensitive to variations of other processing parameters, e.g., pressure. These findings are important for the understanding of ion-induced damage in silicon substrates.

83 Reactive Ion Etch Induced Device Characteristic Changes in Thin Film Transistors: Y. Kuo, IBM Research Div., T.J. Watson Research Center, Yorktown Heights, NY 10598.

We have detected some plasma damage on the inverted, tri-layer TFT in a n+ RIE process. The damage includes a positive Va shift, a high off current, and the divergence of the I-V curves. The Va shift increased with the gate dielectric SiN$_x$ thickness and was lowered when half of the SiN$_x$ layer was replaced by Ta$_2$O$_5$. These damages were caused mostly by the plasma radiation and slightly by the cathode charge effect. Interfacial traps between a-Si:H and SiN$_x$ were responsible for the anomalous TFT characteristics. This damage appeared to be physical and was easily repaired with a thermal annealing process.


In this study, blanket oxides (1000 Å) were exposed to a CHF$_3$/O$_2$/contact etching chemistry in an MERIE system under various magnetic field conditions. Samples were then analyzed for damage concentration effects both before and after UV ozone cleans. Our results show the dependence of the resulting residue thickness and damage layer thickness on magnetic field strength and on this surface treatment.


Particulates that form in glow discharge plasmas are charged negatively and are often suspended in the plasma by electrostatic forces. It has been observed that dust tends to segregate to certain locations in the discharge. In order to minimize particle contamination during processing, it is important to understand the nature of the forces particles experience in plasmas. We have constructed a model of particles in plasmas to understand particle segregation in glow discharges.


Particle contamination is a serious concern in all microelectronics fabrication lines. Tools and processes contribute the greatest share of contamination, especially plasma process tools. This work demonstrates the nature of particle contamination in plasma tools with emphasis on production plasma equipment during normal process conditions. Particle behavior and trapping effects are demonstrated in sputter andPECVD tools, as well as enhanced plasma tools such as magnetron RIE and ECR. The ion-grown electrode design for contamination control is also discussed.


The contaminated regions containing high density contamination particles in RF processing plasmas are investigated in this paper. Using a tuned Langmuir probe in an RF plasma tool, the plasma potential, electron density, and electron temperature of these regions (or traps) are measured, accompanied by a real-time video recording of the dynamics of the particles. A scenario of trap formation is presented in which the RF plasma is shown to be a system generated and that particles subsequently flow into a trap and distort its boundaries in such a way that the particulates eventually leak into a pump port.

Particle contamination and etch depth on silicon wafers etched in SF₆/Ar and CF₄/O₂/Ar plasmas are examined as a function of 13.56 MHz RF power, 100 kHz power, pressure, flow rate, and etch time. Particle deposition and etch depth have a linear dependence on 12.56 MHz RF power, 100 kHz power, and etch time. Also, the particle deposition and etch depth have a quadratic dependence on process gas pressure. In the pressure range explored, particle deposition on the wafer is independent of pressure.


In situ optical emission spectroscopy was used to monitor active species during hydrogen cleaning of native silicon oxide to understand the mechanism of the plasma process. Relative concentration of hydrogen and hydroxyl radicals were measured as a function of operating pressure. Gas phase concentrations were well correlated with the etch rate of native silicon oxide measured using in situ x-ray photoelectron spectroscopy. Plasma potential, ion density, and electron temperature of the ECR hydrogen plasma were also measured with a Langmuir probe.

90 Process and Module for Low Temperature Hydrogen Cleaning for Silicon Wafers: F.-P. Steiner, E. Beck, and J. Ramm, Balzers AG. FL-9496 Balzers, Liechtenstein

An ultra high vacuum compatible process module for silicon wafer cleaning is presented. The cleaning procedure utilizes an argon/hydrogen plasma which is characterized by low discharge voltages (about 25 V) and high discharge currents (up to 100 A). If the wafer is immersed in this plasma, the native oxide as well as hydrocarbon overlayer on the surface. Mainly electrons stimulate the surface chemistry and damages by sputtering are avoided. Typical etch rates for thermally grown SiO₂ from 0.1 to 0.25 nm/min cleaning prepares the silicon wafers for low temperature epitaxial growth of silicon.

91 In Situ Chamber Dry Cleaning for HBr RIE: K. Iizuka and M. Nakamura. Process Development Div., Fujitsu Limited, 1015, Kamikodanaka, Nakahara-ku, Kawasaki 211, Japan

Anisotropy is achieved through the side-wall protection of etch products in HBr RIE. On the other hand, the reaction products deposit on a wall of reaction chamber to form particles. An alternative method for minimizing particle deposition on the chamber wall. The conditions suitable for the dry cleaning are completely different from the usual etching conditions.

92 Ion Beam-Assisted Etching of Si(III) with Fluorine at 77K: J. W. Coburn, IBM Research Div., San Jose, CA 95120-6099. C. B. Muilla, Dept. of Chemical Engineering, University of Texas, Austin, TX 78712-1062

Recent interest in low temperature RIE has prompted some basic investigations of etching with fluorine at 77K. The etching rate of Si(III) with fluorine at 77K in a three-component plasma was measured in situ using quartz crystal microbalance methods, and the neutral etch products are monitored with a quadrupole mass spectrometer. Only a brief transient of species is observed and the ion-assisted etch rates are somewhat larger at 77K than at room temperature. The sputter yields of condensed SiF, and the density, were measured to provide an estimate of the neutral density. If the sputter rate is high enough to sputter the etch products is needed to remove the etch products.

93 A New Method of Sidewall Protection for Anisotropic Etching Using Sulfur Deposite: T. Tatsumi, N. Nagayama, and S. Kadomura, ULIS R&D Group, Sony Corp. 4-14-1 Asashio-cho, Atsugi, Kanagawa 243, Japan

The objective of this work is to demonstrate a new dry etch processing technique for ULIS devices. The dry etch process presented in this paper used sulfur for sidewall protection. Sulfur can be sublimated by heating the substrate after etch, so protecting filaments film will not remain on the etched surface. This concept was applied to dry etch polysilicon lines with 0.25 pm E-beam defined and the sublimation of sulfur was confirmed using EDX and TDS.

94 Cooling Wafer Stage for Low Temperature Dry Etching: M. Ueda, T. Kuro, K. Tsutsumo, and S. Tsuchi, Central Research Laboratory, Hitachi Ltd., Kokubunji, Tokyo 185, Japan

Cooling wafer stage has been developed for low temperature dry etching. As long as the cooling stage is in situ during etching is calculated by the finite element method and is compared with experimentally obtained values. Heat transfer convection and radiation between the wafer base and cooling stage was found to be face of 0.0002 W/m²K for 1000 μm wafer base to the cooling stage. Supposing any increase in wafer temperature and wafer temperature variation at a constant value are required in the low temperature region below -100°C by the simulation method.

95 Problems of Low Pressure Single Crystal Silicon Etching: M. Engelhardt, Siemens AG, Corporate Research and Development, 8000 Munich 83, Germany

Hard disk platters lead to a deposition of sputtered material on the wafer was found to be a major drawback of plasma etching at low gas pressures in high density, low energy RF plasmas. The results were obtained with single crystal silicon etching in a magnetically confined reactor using pure chlorine as a process gas. They seem, however, to be getting closer to solving this problem by operating at similar process conditions such as ECR etch systems.

96 Highly Selective Low Pressure Poly-Silicon Etching in High Density, Low Energy RF Plasmas: M. Engelhardt, Siemens AG, Corporate Research and Development, 8000 Munich 83, Germany

Pure chlorine and pure bromine and mixtures of both were used as process gases in a magnetically confined reactor to achieve highly selective poly-silicon etching. Using a microwave electron cyclotron resonance (ECR) etch reactor. Using H₂O₂ at 3 to 4 milliTorr and 700 W input microwave power. At 2.45 GHz, undoped poly-silicon etch rates in excess of 400 nm/min are obtained in a close-coupled ECR configuration with the etch substrate located close to the resonance zone. The etch selectivity is poly-oxide selectivities are greater than 150, and the poly-resist selectivities are 13-15. This performance is achieved with the etch rates obtained on the same etch tool in a confined B-field ECR reactor with remote wafer position.

97 Selective High-Rate Etching of Poly-Silicon in Microwave ECR Discharges: A Comparison of ECR Configurations: T. D. Manter, D. D. Lane, and P. G. Van Den Bosch. Dept. of Chemical Engineering, University of Cincinnati, Cincinnati, OH 45221

High poly-silicon etch rates have been obtained in combination with high etch selectivities with respect to SiO₂ and photore sist in a microwave electron cyclotron resonance (ECR) etch reactor. Using SiCl₂/O₂ at 3 to 4 milliTorr and 700 W input microwave power. At 2.45 GHz, undoped poly-silicon etch rates in excess of 400 nm/min are obtained in a close-coupled ECR configuration with the etch substrate located close to the resonance zone. The etch selectivity is poly-oxide selectivities are greater than 150, and the poly-resist selectivities are 13-15. This performance is achieved with the etch rates obtained on the same etch tool in a confined B-field ECR reactor with remote wafer position.


This work demonstrates deep trench etching with openings down to 0.25 μm and aspect ratio as high as 40 with smooth bottoms and tight critical dimension control. This paper presents results of a recent study of quarter-micron silicon deep trench etching with mixtures of HBr and fluorine-containing gases in a magnetically enhanced ECR reactor. The effects of RIE on bond strength on the trenches down to quarter micron opening were quantified and trench etch rate was found to depend on trench aspect ratio.


We have demonstrated MRIE (magnetically enhanced reactive ion etching) of polysilicon gate linewidths down to 0.1 μm in a HBr gas chemistry. Either HBr or SiCl₂/HBr can be used as the etch gas. HBr can etch SiO₂ down to 0.3 μm, but pure HBr cannot be used as the etch mask. High poly-silicon etch rate, high selectivity of poly-silicon to oxide, and tight linewidth control have been demonstrated. Wafer oxide is a key parameter for controlling sidewall profile and linewidth. This work also demonstrates the advantage of clustering tools for process integration.

100 Fundamental Processing Limit of Gate Oxide Thickness for Poly-Silicon Gate Definition: A. S. Yogeet, IBM Semiconductor Research and Development Center, Hopewell Junction, NY 12535

A quantitative study of the fundamental processing limit of gate oxide thickness for poly-silicon gate RIE definition is presented. To perform the study, an in situ scheme for monitoring gate oxide removal during etching, is developed. A criterion for a maximum overetch is defined as the time interval between the initial occurrence of oxide etching and silicon substrate etching. Using this definition, the maximum overetch allowed for a specified gate oxide thickness is measured.

101 200 nm Aluminum Etch in MERIE System: S. Mak, S. Arias, and C. S. Rhoade, Applied Materials, Santa Clara, CA 95054

A screening experiment was performed to study the 200 nm aluminum etch in a MERIE system. Using BCCl₃/CNCl/V CF₆ chemistry. Main effects of controllable parameters including gas flows, RF power, magnetic-field, and cathode temperature were investigated over a pressure range of 50-200 mTorr. Of particular interest were the microriding effect on residue and profile. Better etch performance in terms of aluminum etch rate, selectivity to poly-resist, residue, and profile control were achievable by optimizing the process at 200 mTorr pressure range.


Al alloy films were reaction-etched using HBr/BCl₃/HBr/BCl₃/C₂F₆ gases. Etching rates of the Al films, self dc bias and
Two blanket tungsten etchback processes were developed for use in contact and via fill applications, the first utilizing a 2.25 mm faraday and the second a 6.0 mm wafer clamp. The processes demonstrated very low stress, low ECR purging, and high etch selectivity for tungsten over silicon. The SEM data for both processes produced very good agreement between the SEM and RIESEM process for both processes produced good agreement between the SEM and RIESEM. The 2.25 mm wafer clamp process reduces multiple steps for uniform etch recession; exposure of the pattern density effect, and produces excessive recession and keyhole size were studied as a function of temperature.


A low temperature tungsten etch process was studied in the temperature range from -50°C to 10°C using pure SF6 chemistry. Highly anisotropic tungsten profiles were observed. However, two types of residues, namely, "Drift," and "Sticking residue" under these conditions. Such residue formation was studied in detail. The analyses of the residue indicated the presence of predominately aluminum etch residues. A mechanism was proposed for account for such residue formation.

112 A Kinetic Study of Reactive Ion Etching of Tungsten in SF6/RF Plasma: M. C. Peignon, C. Cardinaud, and G. Tarban, LPCM, University de Nantes, 44072 Nantes Cedex 03, France.

The reactive ion etching of chemical vapor deposited (CVD) tungsten in SF6/RF plasma has been investigated by means of optical emission spectroscopy, mass spectrometry, and X-ray photoelectron spectroscopy (XPS). This study is particularly focused on the etching of a W05 layer, a native oxide 50-45 A thick (WO5) always appearing on the CVD tungsten surface. XPS and mass spectrometry measurements show an excess of the atomic fluorine and oxygen and oxide-hydroxide concentrations of the tungsten surface as compared to that of tungsten. Two etch products of tungsten are detected by mass spectrometry: W/F and WOF6. In pure SF6 plasma, the main etch product of W05 and W is WIF, in SF6/RF (40/60) plasma, WIF is the dominant product of the etching of the W05 layer, and WOF, that of W. In situ XPS analyses show the presence of fluorine, oxygen, and sulfur on the tungsten surface. The role of these elements in the formation of the two etch products of tungsten is discussed.


A diode array spectrometer has been used to study optical emission during the etching of tungsten silicide and polysilicon stack films in SF6/Ar/Cl2/He/O2 containing discharges in a MERIE reactor. Experiments are reported on the use of emission from the 440 nm region as a suitable endpoint for these stacked films. Temporal and spatial scans of the optical emission give insight into the plasma and surface chemistries, the influence of the magnetic field and the impact of hardware design on the plasma chemistry.


A continuous dry nitrogen purge is integrated into the loadlock of a single wafer, multichamber aluminum etch system. The purge allows better control in cross contamination between the etch chamber and loadlock chamber, and minimizes moisture introduction during atmospheric wafer transfers. The loadlock particle density is reduced by a factor of 5 to 10. Also, problems with in-contaminant contamination after the aluminum etch are resolved and corrosion-free results become reproducible.


A process to etch LPCVD SiN, which has been deposited over thin thermal SiO2, has been developed with an Applied Materials 8110 hexode system. To ensure uniform and repeatable processing with sufficient etch selectivity between SiN and thin SiO2, the system has been characterized as a function of (i) reactor pressure, (ii) O2 in a CHF3/O2 mixture, and (iii) dc bias voltage.


A sub-half micron deep UV integrated ARC process involving a highly absorbing organic antireflective coating (ARC). The ARC requires plasma etching for pattern delineation. The interaction of the etch gases with the ARC has been investigated. The film exhibits stable and repeatable CD control, is devoid of microcracking residues and the ARC is easily stripped after etch.
117 Photoresist Swelling in Hydrogen-Containing Freon Plasmas: Y. Kuo, IBM Research Division, T.J. Watson Research Center, Yorktown Heights, NY 10598

A new photoresist phenomenon has been observed when a positive photoresist pattern was exposed to the hydrogen-containing plasma in a reactive ion etching chamber. The swollen polymer has an increased pattern profile and is very resistant to certain etchants. The swelling rate decreased with time. FTIR data showed that during the swelling process, (i) new bonds were formed, (ii) fragments were lost, and (iii) hydrogen atoms were added. An XPS profile showed that fragments with the weight range of 60-360 lost from the surface. Therefore, this polymer swelling phenomenon evolution involves several reactions: chain scission, cross-linking, and hydrogenation. The mechanism of the process is delineated. Possible applications of this new structure are also discussed.


We examined resist ashing in a nonlinear of sight downstream resist ashing powered with either radio frequency or microwave for reactive species generation. The ashing process is described as a function of the plasma excitation source. The process is found to be independent of the plasma excitation source at temperatures of 100°C. The microwave powered resist ashing results in a faster ash rate at the temperatures normally used for production single wafer ashers.


In this study, we present our understanding of the basic mechanisms of these ion-implanted photoresist ashing processes in a true downstream plasma reactor. Analytical techniques such as organic SIMS, thermal desorption mass spectscopic analysis, and FTIR were employed. It was found that not just the solvent, but mainly the basic resist resin in the photoresist is also responsible for the ashing, which is the dominant factor for the resist explosion during plasma ashing. Saplasm resist ashing. Alternative OSP options were tried to alleviate the problem.


A bilayer resist typically consists of a thin imaging layer (20 nm) and a thick bottom layer (1 μm). The etching of the bottom layer requires high selectivity against the imaging layer, vertical etch profile, and low damage to the underlying substrate. A high density, low pressure electron cyclotron resonance (ECCR) source in combination with a RF-powered electrode can provide the flexibility to meet these requirements than conventional reactive ion etching systems. Effects of microwave power, RF power, ECR source distance, and pressure on resist etch rate in an O2 plasma are shown. Etch rate was found to increase with microwave and RF power, but decrease with source to sample distance. Etch rate was etched with a good morphology and fast etch rate up to 1.6 μm/min. The optimal conditions for bilayer resisit etch are discussed.


Smooth, optically transparent, a-Si:H thin films have been deposited on Si substrates from silacyclobutane, SiCH4, in a low pressure, cold wall, helical resonator (HR) plasma CVD reactor. The HR source geometry allows flexibility in the placement of the discharge relative to the gas feed and substrate. Substrates are side-stoichiometric a-Si:H (CSi = 1) over the temperature range 370-885°C, independent of flow rate. Decomposing SCB in a plasma having a discharge range of 106 dynes/cm2 exerts a constant ERD at 108 dynes/cm2. The etch rate was used to measure the hydrogen content of a-Si:H for various processing conditions.


Fluorinated silicon nitride films have been deposited by plasma enhanced chemical vapor deposition (PECVD) using SiH2, NF3, -N2, gas mixtures at 13.56 MHz. We studied fluorinated films by secondary ion mass spectrometry (SFMS), X-ray photoelectron spectroscopy (XPS), low pressure microwave excitation, the deposited electron cyclotron resonance (DECR) indicating the formation of high density plasma (+1010 cm) of low ion density (+20 eV) has been used to study the relationship between the optical properties and the atomic composition of the film. Moreover, we have shown that high density thin films (5100 cm) can be deposited at low temperature (<120°C) and a low


An analytical model has been developed to simulate profile evolution during plasma enhanced chemical vapor deposition (PECVD) of silicon nitride. The model takes into account recombination/re-emission at the surface and a spatially varying sticking coefficient (reaction probability) of the rate limiting precursor. The sticking coefficient is modeled as a linear function of the incident ion energy flux, having a minimum value of 0.2 and a maximum value of 0.28. This model accurately predicts the experimental profile evolution in structures of different geometries.


Several PECVD SiH4, processes used in VLSI applications are discussed. Films have been deposited at incident ion energy flux, having a minimum value of 0.2 and a maximum value of 0.28. This model accurately predicts the experimental profile evolution in structures of different geometries.
Effects of RF Frequencies and Deposition Rates on the Moisture Resistance of PECDV TEOS-Based Oxide Films: S. A. Androic, M. Galasso, and B. C. Nguyen, Applied Materials Inc., Santa Clara, CA 95054

The moisture resistance of PECDV TEOS-based undoped oxide films deposited using different RF frequencies and deposition rates were investigated using IR spectroscopy, NMR hydrogen analysis, and stress-temperature analysis. The frequencies used to ionize the plasma were 13.56 MHz only, 100-450 kHz, and 100-450 kHz only. The results obtained indicate that the oxide moisture resistance is not determined by the RF frequencies and deposition rates, but rather by the initial as-deposited film stress.

Formation of High Quality Tantalum Thin Films on SiO₂ by Dual-Frequency-Excitation Plasma Process: H. Watanabe, S. Aogama, J. Warabane, N. Konohi, and T. Ohtani, Dept. of Electronics, Faculty of Engineering, Tohoku University, Sendai 980, Japan

Among various methods of formation of tantalum oxide films for DRAM capacitors, we are studying the direct oxidation of tantalum films. To obtain high quality dielectrics in such an oxidation approach, it is needed to prepare high quality and high crystallinity tantalum films prior to oxidation. We have developed a dual-frequency-excitation plasma process equipment of multichamber construction. In this work we investigate the effects of ion bombardment, and ion bombardment energy on the tantalum film formation process on SiO₂. We have obtained high crystallinity Ta films on SiO₂ when Ta film were formed under relatively high ion bombardment energies (several tens of eV) with sufficient ion flux density which were conducted after SiO₂ film baking and in situ substrate surface plasma cleaning.

Control of Stress, Stability, and Mechanical Properties of Films with Dielectric Films for GaAs and Si Application: T. H. Wu and L. A. Schneegugger, Motorola Inc., Tempel, AZ 85282, B. van Schravendijk, B. Sparks, A. S. Harras, and D. G. Humes, Novelus Systems Inc., San Jose, CA 95113

Plasma enhanced deposited dielectrics (nitriles and oxides) are widely used in advanced silicon-based VLSI manufacturing. The development of dual frequency plasma has allowed precise stress control of these films on both Si and semi-insulating GaAs substrates. We present a comprehensive characterization of the film properties (strates and oxides) on both substrates. Results of stress vs. temperature measurements as a function of the initial stress values are presented and correlated to mechanical properties of the thin film (PITR, Auger analysis). Stability of these films over time and during temperature cycling is directly related to as-deposited film stress.

Formation and Characterization of Zirconia Films by Plasma-Enhanced Chemical Vapor Deposition: C. C. Chen, M. V. Yarmush, and H. U. Anderson, University of Missouri-Rolla, Ceramic Engineering Dept., Rolla, MO 65401, W. J. James, University of Missouri-Rolla, Materials Research Center, Rolla, MO 65401

Zirconia (ZrO₂) films have been fabricated from zirconium tetra-butoxide using plasma-enhanced chemical vapor deposition (PECVD) technique. The effect of deposition parameters; plasma power, substrate temperature, and reactive gas flow, on the film structure is presented. The fabricated films were dense and stoichiometric. The development of a densification model for crystalline and the effect of post annealing are discussed. The indentation profiles of composition were investigated using AES. Stoichiometric cubic ZrO₂ films were successfully deposited on Si substrates. Attempts are underway to deposit dense films on porous substrates.

SECOND INTERNATIONAL SYMPOSIUM ON REDUCED-TEMPERATURE BUDGET PROCESSING FOR THE FABRICATION OF MICROMECHANICAL DEVICES


An approach to the reduction of transient thermal nonuniformities during the rapid thermal processing is described. The approach utilizes the digital control of heating lamps during the processing cycle. The power absorbed by the wafer in any time period is controlled according to a computer-processed recipe. This feature allows elimination of any transient temperature nonuniformities, especially during heating-up. The power control is performed by the proprietary back-radiant silicon ring, which allows slip-free processing.

The Impact of the Wafer Back Side on RTA Processing: B. Lojek, Motorola Inc., Advanced Technology Center, Mesa, AZ 85202

The emissivity of silicon wafer varies not only with temperature and wavelength, but also with the back-side roughness of wafer.


To form shallow p-n junctions, in each process step after the impurity doping, thermal budgets have to be decreased. The thermal budgets of the heating-up duration are obtained by measuring the thickness of the oxide grown on polycrystalline Si during heating-up step. And then, the high rate heating-up causes the thermally grown SiO₂ films to have low leakage current.


As wafer size increases, heating-up and cooling-down durations become a larger fraction of total process time. Here, we estimate the thermal budget of the cooling duration as a function of the cooling rate from 1000°C to room temperature by using the experimental and arsenic profiles and the computer simulation. The effective diffusion time at 1000°C is approximately in inverse proportion to the cooling rate over the whole range of the cooling rate of 2-60°C/min.

Reliability Characterization of RTO and O₂-Diluted Thin Gate Oxides: L. A. Fossetta* and F. Campobadal, Centro Nacional de Microelecronicas, Universidad Autonoma de Barcelon, 08193 Bellaterra (Barcelona), Spain

RTO and O₂-diluted thin oxides have been electrically characterized. C-V analysis and barrier values around 3 eV for FN tunnelling conduction reflect good electrical properties in both cases. Superior storage time of RTO capacitors showed that no serious damage was produced in the substrate in spite of aggressive thermal treatment. TDDB and TDD experiments were also performed. RTO oxides resulting in superior reliability terms, presented a slightly larger dielectric strength and significantly larger Qbd.

A Study of the Effect of Deposition Parameters on the Growth Rates and Microstructure of Silicon Homeopitaxial Films Grown by ArF Laser-Enhanced Chemical Vapor Deposition: S. Krishnan, S. Lian, B. Foulser, L. Jung, C. Lai, D. Samora, I. Mann, and S. Banerjee, Microelectronics Research Center, University of Texas, Austin, TX 78712

The effect of deposition parameters on the growth rate and microstructure of Si homeopitaxial films, grown by ArF excimer laser-enhanced photodissociation of SiH₄ has been studied. The growth is performed in an ultrahigh vacuum chamber using the laser beam shining parallel to the substrate whose temperature is held at 300°C. The deposition pressure, varied in the range of 0.01-120 mTorr, does not have a significant effect on the growth rate as long as the SiH₄ partial pressure is held constant at 10 mTorr, though the crystallinity improves with increasing deposition pressure, as confirmed by its RHEED analysis. Under a constant deposition pressure, the growth rate falls with increasing distance between the substrate and the laser beam, when it is varied from 0.15 mm, since fewer precursors are able to reach the substrate. The constant deposition rate is independent of the repetition rate of the laser as long as the average power is held constant. On the other hand, it linearly increases with the repetition rate when it is varied in the range of 2-80 Hz, keeping the energy per pulse constant. The crystallinity is preserved even at 2 Hz.

A Green's Function Approach to a Growth Kinetic Model for Low Temperature Si Homeopitaxy by ArF Excimer Laser-Enhanced Photothermal Chemical Vapor Deposition: S. Lian, B. Foulser, S. Krishnan, L. Jung, C. Li, D. Samora, I. Mann, and S. Banerjee, Microelectronics Research Center, University of Texas, Austin, TX 78712

Low temperature Si homeopitaxy by ArF excimer laser-enhanced photo thermal chemical vapor deposition (PCVD) using silane in an ultrahigh vacuum (UHV) deposition chamber has been successfully achieved at temperatures as low as 250°C. A Green's function approach to a growth kinetic model has been developed based on the phase relations of the photodissociation of silane and photodissociation of silicon-bearing radicals to the growth surface, and experimental data. The growth rate g = g₀ [1 + (H₂ gas flux)/(4πDgΔH₀ + 4πDgΔH₀) exp (-Eₜ/T)] cm/s, where J is laser intensity in photons/pulse·cm², α is the absorption cross section of SiH₄ at 193 nm, α is the concentration of growth precursors, and D is the diffusion coefficient. All the parameters in the above equation are either known or are measurable except for g₀ and Eₜ. From the growth data, α is estimated to be 0.075 which is consistent with the value from other researchers α ≈ 0.05. 0.1 < g₀ < 0.26. The linear depen-
dence of growth rate on laser intensity and disilane partial pressure indicates that the rate limiting step is the photo-dissociation generation rate of the growth precursors.


Solid-state X-ray irradiation (SPEXI) technique using synchrotron radiation has been applied to the post-annealing of amorphized silicon surface layers by the implantation of phosphorus. The effects of annealing temperature and the dose of annealing MMS measurements have been studied. It was found that by SPEXI, the higher electrical activation of implanted phosphorus can be achieved at much lower temperatures than by conventional furnace annealing. MMS measurement of SPEXI-doped SPEXI films also showed almost the same characteristics as those on single crystal wafers.

141 A Low Resistivity Polysilicon Film Fabricated with a flat surface, and achieves low-resistivity by low-temperature an-

142 Deposition and Characterization of Crystallized LPCVD Si-Films Obtained by Low-Temperature Pyrolysis of Disilane: A. T. Voutsas and M. K. Hatalis, Sherman Fairchild Laborato-

143 Photo-Enhanced Reaction during Chemically Vapor-Depo-


Silicon precipitation during the post Al/Si or Al/Si/Cu metali-


The thermal stability of submicron CoSi lines on Si substrates was investigated using electrical, x-ray TEM, RBS, and SEM measurements. The as-deposited silicides, as thin as at the edges of features, giving rise to an apparent difference in the electrically equivalent linewidth and its physical size. These films were annealed at temperatures above 1000°C. However, no line width depen-

dence of this resistance degradation was observed up to 1000°C for linewidths down to 0.45 μm.


The effects of dopant concentration on the titanium silicide formed by rapid thermal processing were studied as a function of linewidth down to 1 μm. Significant degradation of the TiSi, during PSAG deposition at temperatures between 450°C and 900°C was observed for patterned polysilicon near 1 μm linewidth due to unintended implanting of phosphorus between the BF2 source/drain implants. The effects of other dopants, such as normal doping with POCl3 and implanted arsenic and phosphorus on the titanium silicide high temperature compatibility are also dis-

cussed. The investigation techniques included sheet resistance measurements, elemental depth profiling, scanning and transmission electron microscopy.
During heat-treatments required for trench isolation using BSG as a trench fill, then SiO2 layers are sufficient to mask the diffusion of boron from BSG. The transfer of boron from un unprotected BSG surfaces onto silicon was investigated in liquids and gas phase ambient. Depletion of boron from BSG was found after immersions into boiling water. Prolonged rinsing in deionized water removed the boron contamination incurred during immersions of silicon on insulator surfaces. Dry G, and N, cause negligible out-diffusion of boron from BSG at elevated temperatures, while steam produces a measurable loss of boron.

**FIFTH INTERNATIONAL SYMPOSIUM ON SILICON-ON-INSULATOR TECHNOLOGY AND DEVICES**

**154 Manufacturing of VLSI CMOS on SIMOX Substrates:** J. Yue, B. Urike, J. Kueng, R. Raisen, P. Fechner, G. Dugel, and M. Liu, Solid State Electronics Center, Honeywell Inc., Plymouth, MN 55441

There has been significant improvement in the level of understanding of SIMOX (separation by implantation of oxygen) process development. Concerns such as defect or contamination in the top silicon layer, etch pits in the buried oxide have been reduced drastically compared to 5 years ago. This improved SIMOX material, which is fully compatible with silicon processing, has been used to fabricate VLSI circuits with consistent performance, and reliable, of both 1.2 and 0.8 µm CMOS technologies on SIMOX substrates.

**155 High Performance Submicron CMOS/SOI for Logic and SRAM Applications:** N. Haddad, IBM Federal Sector Div., Manassas, VA 22110, L. K. Wang, IBM Research Div., T. J. Watson Research Center, Yorktown Heights, NY 10598

A submicron CMOS/SOI technology is developed as an extension of a 0.5 µm bulk CMOS manufacturing technology for both memory and logic circuits. Circuit performance as well as the soft error immunity are improved to the existing designs by using SOI substrates and limited process modification. Comparison of the process complexity, circuit performance, and chip yield is presented.

**156 The Implementation of a Commercial Thick Film SOI Process:** K. Yallup, Analog Devices BV, Raheen Industrial Estate, Limerick, Ireland

SOI is an emerging technology that is ready for application to commercial integrated circuits. This paper describes one of the first uses of thick film SOI in combination with deep trench isolation for ion implantation applications. Some of the solutions to the technological difficulties encountered while developing the dielectric isolation process and the use of such a substrate to support a 30 V CMOS process are discussed.

**157 Body-Contacts for SOI MOSFETs:** M. Matulbun, Hughes Aircraft Co., Carlsbad, CA 92028

The body region of SOI MOSFETs is floating. This leads to desirable features such as higher drive current and sleep threshold voltage. However, some problems such as premature drain-to-source breakdown and body-contact are used to hold the SOI MOSFET body at a fixed potential and suppress the floating-body effects. Various types of body-contact for SOI MOSFETs are reviewed, and their effectiveness and limitations are discussed.

**158 A 0.5 µm CMOS/SOI Technology Using Accumulation Mode Device Design:** L. K. Wang, IBM Research Div., T. J. Watson Research Center, Yorktown Heights, NY 10598, J. Boeckling, A. Edsényi, O. Spero, and N. Haddad, IBM Federal Sector Div., Manassas, VA 22110

CMOS VLSI fabricated on thin silicon on insulator has very attractive features such as high speed, low power, high temperature stability, and radiation immunity. Using a fully depleted body-contact on thin silicon film eliminates the "kink effect" and anomalous subthreshold current generated by the floating substrate. In addition, it also can provide higher transconductance, higher current drive for a given substrate current, and improved subthreshold currents. The fully depleted body-contact mode of operation of the fully depleted MOSFETs would be too low for any practical applications. In this paper we report a 0.5 µm CMOS/SOI technology using reverse gate type (N+/poly/n-channel, P+/poly/n-channel) accumulation mode MOSFET design.
low-temperature transient behavior in SOI NMOSs, which is very similar to the case of the kink-related excess noise in bulk transistors at liquid helium temperatures.

166 Advantages and Limitations of Thin Film SIMOX MOSFETs: Reliability Aspects. J. Gautier and G. Reinbold, DIA-LETI, CENG, 38941 Grenoble Cedex, France.

In this work, we present advantages and limitations of SIMOX MOSFETs. Partially depleted, fully depleted, and deep-depleted architecture are considered, in the case of thin silicon film, with special attention to fault related effects: single latch, transient, hysteresis. We also discuss some reliability aspects, especially hot carriers effects, that are presented through a comparison between silicon technology and SOI technologies.

167 Hot-Carrier-Induced Degradation in Partially and Fully Depleted SIMOX MOSFETs: S. M. Gulauskas,* ECE Dept., George Mason University, Fairfax, VA 22320, S. Cristoloveanu, LETI, Grenoble, France, D. E. Ioannou, ECE Dept. George Mason University, Fairfax, VA 22303, G. Campisi and H. L. Hughes, Naval Research Laboratory, Washington, DC 20375.

A comparative study of hot-electron degration of the front and the back channels of partially depleted (PD) and fully depleted (FD) MOSFETs in SIMOX back channel was performed. The back-channel degradation was induced in the channel under stress, but for slope of the drain current.

168 Back-Channel Hot-Electron Effect on the Drain Breakdown Voltage in Thin-Film SOI MOSFETs: B. Zhang* and T. P. Ma, Center for Microelectronic Materials and Structures, and Dept. of Electrical Engineering, Yale University, New Haven, CT 06520-2157.

The front-channel drain-source breakdown behavior in thin-film SOI MOSFETs has been studied before and after back-channel electron stress. The breakdown voltage increases significantly in the reverse mode (with source and drain interchanged) as stress time increases. The change in the breakdown behavior can be attributed to the increased barrier height of the drain-body junction resulting from the localized electron trapping near the drain in the buried oxide.


SOI MOSFETs exhibit resistance to dose rate and single event upset radiation effects, but are susceptible to total dose effects. These total dose effects are enhanced by high electric fields in the buried oxide of these structures. A numerical device model is applied to the task of designing drain structures that decrease electric fields present in a device as a function of channel length in order to minimize both radiation and hot electron effects.

170 Device-Based Electrical Characterization for SOI Technology Development: D. E. Ioannou, Dept. of Electrical and Computer Engineering, George Mason University, Fairfax, VA 22030.

A review is given of the electrical characterization methods most frequently used to assess the quality of the silicon film and the buried and gate SiO2/film interfaces, and provide feedback for the further development of SOI technology. A general evaluation is made by studying the static f(Vd, Vg) characteristics of test MOSFET's. Bulk traps in the film are studied by current DLTS, and carrier generation by the dual-gate Zener-like and generation DLTS techniques. The interfaces are studied by the dynamic transconductance and charge pumping techniques. Versions and procedures for the application of these techniques exist for both partially depleted (PD) and fully depleted (FD) MOSFET's, and for simpler, gated and PIN diode and capacitor structures.


Due to the high sensitivity to traps and defects in the semiconductor material as well as near the oxide interface, low frequency noise measurements were performed for the evaluation of SOI films and epitaxial films grown on them with regard to a BICMOS application. The results were supported by an electrical characterization using impedance and current measurements and by crystallographic investigations using TEM and SEM.


The purpose of this work is to present, for the first time, experimental RTS results obtained on SOI MOS devices. Small area SOI MOS devices operated at the back interface show considerably large RTS amplitude due to the large thickness of the back insulator. The RTS fluctuations at the front interface are small and are not influenced by those of the back interface, while the front and back interface coupling is weak. However, in case of strong front and back interface coupling (i.e., for thin-film SOI technology), the RTS fluctuations of the back interface may propagate to the front interface and, therefore, could result in a large parasitic source of low frequency noise in such devices.

173 A NewTransient Drain Current Technique for Interface Characterization in SOI MOSFETs: S. Cristoloveanu, LPCS, ENSERU, 38016 Grenoble Cedex, France, H. Haddara, Electronics and Computer Engineering Dept., Amr-Shams University, Abassia, Cairo, Egypt, M. T. Elyan, Electrical Engineering Dept. Zagazig University, Shobra, Cairo, Egypt.

A new technique for the characterization of interface traps in SIMOX is proposed. This technique is based on a novel analytical transient drain current model in the weak inversion regime. The method is capable of providing the same information as charge pumping but with much higher sensitivity. Measurements were carried out on partially depleted five terminal SIMOX transistors. Experimental measurements are in agreement with the model and the obtained results for the average interface trap density are in the same order as that obtained from the subthreshold slope of the drain current.


In this paper, dislocation and buried oxide discontinuities density measurements on SIMOX material, using chemical etching methods, are presented and discussed. The quality standards of standard LSI material based on statistical analysis is given. We also show that slight changes in the material fabrication process (e.g., implantation through a 250 nm thick screen oxide) can severely degrade the crystallinity of the top silicon film. (An increase in dislocation density from 10 to 10^3 cm^-2 has been observed.)

175 Effect of Thermal Ramping Conditions on Defect Formation in Oxygen Implanted Silicon-on-Insulator Material: S. Krutze, J. D. Lee, and J. C. Park, Chemical, Bio and Materials Engineering, Arizona State University, Tempe, AZ; G. Zagazig, Semiconductor Electronics Div., NIST, Gaithersburg, MD 20899, M. El-Ghor, Texas Instruments, Inc., Central Research, Laboratory, Dallas, TX 75255.

The effects of intermediate thermal processing steps on the microstructure of oxygen implanted silicon-on-insulator (SIMOX) material were studied with cross-section and plan view transmission electron microscopy. Intermediate, 2 isothermal annealing cycles eliminated as-implemented defects (stacking faults, multiply faulted defects, and [113] defects) at 1000°C. New defects (stacking faults and a polygranulated dislocation network) formed near the upper and lower interfaces of the buried oxide, but the defect size and defect density were reduced by 1100°C and enhanced at 1200°C. At 1300°C a defect density of 10^2 cm^-2 was found. When an as implanted sample was rapid thermal annealed at 1300°C for 5 s, the top third of the layer had a "dead layer" which was precipitate free, but many dislocations extending to the surface had formed. In a subsequent standard anneal for 5 s at 1300°C the dislocations were not removed and a defect density of 10^3 cm^-2 was found. The results show that intermediate thermal processing steps, including intermediate isothermal annealing and rapid thermal annealing, strongly affect the final microstructure of SIMOX material.


In this paper we present the results of a detailed analysis of SIMOX samples implanted through different thicknesses of screen oxide by Raman microprobe measurements. The spectra obtained from as implanted and annealed samples have been correlated with TEM observations. The results obtained, in terms of strain, disorder, and thermal effects distribution, show a strong redistribution of the top Si layer structure on the thickness of the screen oxide, which corroborates the key role of the surface conditions on the sample structure.


The aim of this work is the study of precipitate formation after proper thermal treatments in low fluence oxygen-implanted samples. The precipitates and the extended defects behave as a well for oxygen and are the nuclei for the subsequent precipitate
growth; so that it is possible to deplete oxygen from regions where large precipitates are not formed and accumulate oxygen in special perturbed sites, because the oxygen diffusion towards concentration gradient takes place. The kinetic of precipitate formation is followed at increasing fluxes (in the range 10−11−10−6 O/cm²) in the temperature range 450−800°C, starting from an initial condition of bulk of material at ~100°C. The current density is low, and the sample is slowly cooled. Cross-section and plain view micrographs have been obtained by TEM and HREM and oxygen profiles have been obtained by SIMS.


A novel etching process including three sequential etch steps was developed to measure low defect densities in thin SOI silicon films. It allows for a fast, simple, and cost-effective measurement of defect densities in a range of about 10−10−4 cm². Dependence of defect density on the implanted oxygen dose has been studied for SIMOX wafers. The experiments show an exponential increase of defect density with dose for SIMOX wafers. A company performed a combination of photoconductive frequency resolved spectroscopy (PCFRS) and photo-induced current transient spectroscopy (PICTS) on SIMOX wafers. The results show that the photo-induced current transient spectroscopy (PICTS) is a fast, simple, and cost-effective measurement of various process parameters on the properties of SIMOX structures.


Singly and multiply implanted SIMOX wafers were evaluated using Lang transmission x-ray topography as well as chemical etch techniques. Moiré patterns were clearly observed in the multiple implanted material and some areas in the single implanted ones. It is found that wafers having dislocation densities greater than 8 × 10¹⁰ cm² were not easily observed, while say, a dislocation density greater than 10¹⁰ cm² was seen for densities less than 10⁹/cm². This indicates the capability of XRT to screen SIMOX material for dislocations.


The characterization of SIMOX obtained by different processes has been performed by Raman scattering for the top Si layer and FTIR for the buried oxide layer. Raman spectra obtained from the whole structures and from beveled samples at different excitation powers show significant differences related to the technological processes determined by the strain distribution and thermal conductivity in the Si layers. Structural differences are also observed from FTIR spectra, which indicate the existence of different amorphous-like disorders and residual stresses in the buried oxides. The ability of both techniques for the SIMOX structural characterization is demonstrated.


The characterization of SIMOX structures is performed by the combination of photoconductive frequency resolved spectroscopy (PCFRS) and photo induced thermal transient spectroscopy. PCFRS shows the existence of a low dimensional recombination behavior with a lifetime in the range 10−10−100 μs. However, in some cases values of the photodiode mobility in the range of cm²/Vs are found. These have been correlated with PICTS spectra, which show a density of Si/SiO₂ interface defects at 10¹⁰ cm²/eV. The analysis carried out confirms the suitability of the combined PCFRS and PICTS techniques for the electrical evaluation of SIMOX structures.


A designed study of the effects of high temperature anneal process parameters: A designed experiment has revealed that the effects of the anneal variables were examined, and coefficients for a linear model were derived. Modeling of the results produced qualitative results and revealed a strong effect of Si/SiO₂ incorporation, temperature and time, on the higher carrier lifetime.


The properties of the buried Si-SiO₂ interface, the concentration and structural defect level of the recombinant have been monitored nondestructively with such their effect on the surface and bulk components of recombinant lifetime by a laser/microscopy technique. This has implemented anneal SIMOX material that the bulk recombinant lifetime decreased from 1 μs, characteristic of origin substrates, to 3 μs or to 6 μs if the annealing alone (no implantation), or both implantation and annealing processes have been applied, respectively. Moreover, the Si-SiO₂ interface still contains a very high density of electrical defects even after the structural damage removing/oxidizing the surface subjected to annealing alone (without implantation), and only one order less than material implanted, but nonannealed.


LOCOS structures on SIMOX wafers were studied with cross-sectional transmission electron microscopy and with micro-Raman spectroscopy (μRS). Prolonged field oxidation results in considerable oxidation of the active silicon film and the oxide forming high temperature treatment. This defect density results in a surface recombination velocity on the order of 10⁶ cm/s, two or three orders of magnitude more than the substrate subjected to annealing alone (without implantation), and only one order less than material implanted, but nonannealed.

185 Dislocation-Free SIMOX Substrates: A. Yoshino, Becton Center, Yale University, New Haven, CT 06520-2157.

This is the first report of dislocation-free SIMOX substrates. We present experimental results showing various process parameters on the dislocation density in SIMOX substrates. Comparing the results for the samples formed by single-implant with those formed by double-implant, together with the data from RBS, optical microscopy, SEM and TEM measurements and wafer warp evaluation, the possible origin of the dislocation formation mechanism is discussed.


A study of the effects of high temperature anneal process variables on the properties of SIMOX films was conducted. The study was structured as a designed experiment with seven variables, each at three levels. Models of the dislocation density produced quantitative results and revealed a strong effect of temperature and measurable effects of time and ramp rate. Higher temperatures, lower ramp rates, and longer anneal times resulted in lower dislocation densities.


A study of the effects of HCl inclusion in the high temperature annealing ambient on tube self-contamination and radiation immunity was conducted. Contamination was studied by measurement of minority carrier lifetime and radiation immunity by MOS-C-V curve at an area of 5% HCl at a temperature of 1000°C was sufficient to restore furnace cleanliness. Neither the moderate levels of contamination nor the presence of HCl had any effect on radiation hardness.


The relationship between SIMOX anneal temperature and CMOS transistor characteristics is investigated. Post-oxygen implant anneal temperature from 1150 to 1320°C were used in an Ar ambient atmosphere with wafer etching times of 25 minutes. Backsion films were conducted to reveal channel threshold voltage increases while snap-back sustaining voltage decreases with increasing anneal temperature. Transistor output leakage current was found to be independent of anneal temperature.

189 A High-Quality SIMOX Wafer and Its Application to Ultrathin-Film MOSFETs: S. Nakashima, Y. Omura, and K. Izumi, NTT LSI Laboratories, 3-1 Morinosato Wakamiya, Atsugi 243-11, Japan.

A high-quality SIMOX wafer with extremely low dislocation densities of less than 300 cm² and an 80 nm buried oxide layer is introduced. Employing the SIMOX wafer to fabricate an 0.685-μm
gate, n, and pMOSFET demonstrates that SIMOX has excellent potential for future ULSI applications.  


The dislocation density and the density of silicon islands in annealed layers were found to be strongly dependent upon the oxygen dose. The critical dose required to form a continuous buried stoichiometric oxide layer either during implantation, \( \Phi_i \), or after implantation and annealing, \( \Phi_A \), were estimated to be \( 7 \times 10^{15} \text{cm}^{-2} \) and \( 3 \times 10^{16} \text{cm}^{-2} \), respectively. Good quality, thin film SIMOX layers (with a low threading dislocation density in the silicon overlayer and low density of silicon islands in the buried SiO\(_x\) layer) have been produced by implantation of both \( 3.3 \times 10^{15} \text{O}^{-}/\text{cm}^2 \) at 680°C and \( 3 \times 10^{16} \text{O}^{-}/\text{cm}^2 \) at 550°C.  

191 Etch-Stop Layer in Silicon Produced by Implantation of Electrically Inactive Impurities: H. M. You, and V. Gosele, Dept. of Mechanical Engineering and Materials Science, Duke University, Durham, NC 27708, D. Fejoo, AT&T Bell Labs., Murray Hill, NJ 07974  

The etch stop performance of carbon implanted layer in silicon was found to be not only dependent on carbon concentration but also related to the etch rate used and the structural properties of the layer. Thermal annealing reduces the damage and defects resulting in degraded etch stop performance. Silicon self-implantation and post-implantation also show etch stop function resulting from formation of amorphous layer and impure carbon etch character of etched used.  

192 SIMOX: An Efficient Etch-Stop to Fabricate Silicon Membranes with Well-Defined Thickness: H. Gasele,* H. G. Dura, W. Mokwa, and H. Vogr, Fraunhofer-Institut für Mikro-  

tronische Schaltungen und Systeme, D-4100 Duisburg, Germany  

This paper shows that SIMOX wafers with an epitaxial silicon layer are an excellent material for the production of homogeneous silicon membranes with well-defined thicknesses. Due to the very high selectivity of TMAH as etch solution and the fact that SIMOX produces step-free etched surfaces, this technology allows for a very simple and very fast etch process. We review our recent work on GaALAs/GaAs and GaInAsP/InP SIMOX membranes. A high selectivity of TMAH as etch solution, a four times lower oxygen consumption, and the possibility to define optical cavities in future surface emitting lasers. These fabrication methods are expected to successfully reduce the threshold currents of future surface-emitting lasers.  


The etch stop performance of carbon implanted layer in silicon was found to be not only dependent on carbon concentration but also related to the etch rate used and the structural properties of the layer. Thermal annealing reduces the damage and defects resulting in degraded etch stop performance. Silicon self-implantation and post-implantation also show etch stop function resulting from formation of amorphous layer and impure carbon etch character of etched used.  


Thin SOI layers created by bonding two oxidized wafers lack IG ability and are consequently used to define thin film processing. This is because during bonding, oxygen diffuses towards the middle oxide layer leading to a denuded zone (DZ) in proximity of the oxide. SOI layers \( \Delta Z \) if oxidized will show microdefects in the shape of OISF. To improve the gatering ability of SOI layers we implanted different doses of boron, phosphorus, or oxygen in selected areas of the SOI layers with different patterns of silicon islands before or after wafer bonding. The results of the lateral gatering for the various implanted species are reported.  

195 50 nm Thick SOI Fabrication by Advanced ELO: Tunnel Epitaxy: A. Oguma,* A. Parups, and R. Koh, NEC Corp., Microelectronics Research Laboratories, 34 Miyukigaoka, Taku-  

ba, Ibaraki 305, Japan  

ELO is well known as an SOI fabrication technique with good crystal quality. However, conventional ELO is not applicable for thin SOI fabrication which is considered to have great potential for future nano-structure devices. We present an advanced ELO technology: in which the lateral epitaxial growth is grown in a small gap between the upper and lower SiO\(_x\). Therefore, the fabricated SOI thickness can be precisely controlled by the implantation depth. Thin SOI films with thicknesses of 50-200 nm were fabricated using this technique.  


Semiconductor laser applications for consumer products (primary for compact disk players) increased the worldwide demand for lasers to several tens of millions of devices per year. However, today's manufacturing technologies cannot accommodate this steadily increasing demand since fabrication and testing of semiconductor lasers are still on the level of discrete devices with the case in the early days of transistor fabrication. This is in marked contrast to the current trend to use more advanced and integrated laser arrays. A new concept/technology is presented called full wafer (FUWA) fabrication and testing that produces high-quality semiconductor lasers. It features an excellent potential for high processing yield and testing throughput. In addition to large-scale fabrication, the etched mirror technology offers the capability of integrating new functional and optical elements to the laser chip. This technology is well suited for lasers used in such applications as optical storage and interconnects.  


High-resolution microfabrication allows us to define small three-dimensional optical cavities that can be used to provide the threshold power required for switching or lasing. To demonstrate this, we have measured the lasing behavior in 0.4 \( \mu \text{m} \) wide surface-emitting microlasers. We have also fabricated and characterized three-dimensional "photonic bandgap" structures which can be incorporated to define optical cavities in future surface-emitting lasers. These fabrication methods are expected to successfully reduce the threshold currents of future surface-emitting lasers.  

198 Surface Emitting Laser-Lasing Characteristics and Its Functional Operations: F. Koyama and K. Iga, Tokyo Institute of Technology, F&I Lab., 4259 Nagatsuta, Midori-ku, Yokohama 227, Japan  

The surface emitting (SE) laser is rapidly attracting a current research looking forward to future parallel optoelectronics, including optical interconnections and parallel processing. We review our recent work on GaAlAs/GaAs and GaInAsP/InP SE lasers, especially focusing on lasing characteristics such as polarization characteristics and output power. The future prospects and basic technologies for SE lasers are also addressed. In addition, a possibility of a polarization independent and low power consumption active filter based on the SE laser is presented.  

199 Laser and Photoreceiver Arrays for Parallel Optical Data Link Applications: N. K. Dutta and P. R. Berger, AT&T Bell Laboratories, Murray Hill, NJ 07974  

Very low threshold (\( 1-10 \) mA) lasers emitting near 1.3 \( \mu \text{m} \) have been fabricated using multiquantum well active region, short cavity length, and high reflectivity facet coatings. A laser array transmits utilizing these lasers can be operated with a negligible skew for 1 Gb/s operation. No crosstalk is observed when adjacent elements of the laser array are modulated. A PIN- 
MOSFET photoreceiver array comprised of a p-n photodiode integrated with an InGaAs/InAlAs MODFET using MBE has been fabricated. The MODFET has a transconductance of \( 57 \text{mS} \)/\( \text{mm} \) and \( f_T \) and \( f_m \) of 10 GHz. The p-n photodiode has a responsivity of 0.3 A/W and bandwidth of \( 10 \) GHz.  


We have successfully fabricated a very dense one-dimensional semiconductor laser array using separated double heterostructure (SDH) lasers. The array consists of 102 lasers, each of which can be individually addressed. The pitch of the lasers is 57 \( \mu \text{m} \). The typical threshold current and output power were 2 mA and 3 mW/ facet (\( 1 \times 10 \) mA) for a laser under continuous wave operation at room temperature. The density and the number of lasers are the highest ever reported.  

201 Anisotropic Photocleaving of GaAs: E. Mannheim, R. L. Sani, and R. C. Alkire, Dept. of Chemical Engineering, University of Illinois, Urbana, IL 61801  

A two-dimensional mathematical model was used to obtain the current and potential distribution near the illuminated region of a semiconductor during photocleaving. The distribution was described by Poisson's equation. Electron and hole transport equations included generation, diffusion, migration, and recombina- 
tion terms. A commercial finite element code, ANSYS, was modified to numerically solve the model equation. The sensitivity of the pressure with respect to the important parameters was investigated.  

202 Two-Dimensional Vertical to Surface Transmission Electro-Photonic Device Array for Optical Interconnection: K. Kawanishi, Opto-Electronics Research Laboratories, NEC Corp., 34 Miyukigaoka, Tukuba, Ibaraki 305, Japan
The vertical to surface transmission electro-photonic device (VSTEP) is a concept proposed to allow functional optical interconnections, with particular focus on the laser-mode VSTEP. The design of vertical-cavity VSTEPs and their fabrication techniques are described. First implementations are discussed, and their characteristics are measured. A 4 × 4 optical self-routing switch, fabricated using vertical-cavity VSTEPs is described.


For the first time, study of Be* ions implantations is carried out and the Si substrate. For oxide films thinner than the expected growth rate at the Si-SiO_2 interface. To show this, we oxidized in the annealed implantation layers. Double Be* implant in GaAs/GaAs layers leads to abrupt junction of good quality.


The field of integrated electronics and optical devices has been advanced by the development of high-speed, high-performance components. These components include monolithic integrated circuits (MICs), which are used in applications such as memory, logic, and analog circuits. The use of monolithic integrated circuits in these applications is expected to increase in the future, as the technology continues to evolve.


We are now at the critical point in the short history of this technology where it is essential that practical integrated devices with clear advantages in performance be demonstrated. The means for achieving these devices has been to develop materials growth and fabrication processes which lead to the simplified fabrication of highly complex integrated circuits. This progress includes the use of selective etchants and sacrificial materials layers, hybrid growth (e.g., GaAs on Si) and precise epitaxial growth (e.g., graphene, graphite), and clever use of multifunctional materials and device structures. In this paper we address many of the bottlenecks issues now confronting advanced integrated optoelectronic circuit technology.


207 GaAs-on-InP, Receiver Transmitter Optoelectronic Integrated Circuit. J. O. Sutherland, D. Allan, BT Laboratories, Martlesham Heath, Ipswich, England IPS 5GE.

208 Ellipsometry, Transmission Electron Microscopy (TEM), and Step-Profile Measurements. J. C. Scott, J. E. Bowers, and H. A. Atwater, AT&T Bell Laboratories, Murray Hill, NJ 07974.


Using a Ti-Co bimetallic layer as a reaction source with Si substrates, epitaxial CoSi films have been grown on (001) Si via rapid thermal processing. The films resulting in the epitaxial CoSi layers are excellent. The epitaxial CoSi layers are single crystal films containing anti-phase domains. Models of the CoSi/Si interfacial structure have been presented. These models reveal that anti-phase boundaries serve the role of relieving the lattice mismatch between the epitaxial CoSi film and the Si substrate.

SECOND INTERNATIONAL SYMPOSIUM ON THE PHYSICS AND CHEMISTRY OF SiO_2 AND THE Si-SiO_2 INTERFACE

Electronics/Dielectric Science and Technology

210 Silicon Oxides and Silicon Oxidation. A. M. Stoneham, AEA Industrial Technology, Harwell Laboratory, Didcot, Oxon, England OX11 0RA.

We are now at the critical point in the short history of this technology where it is essential that practical integrated devices with clear advantages in performance be demonstrated. The means for achieving these devices has been to develop materials growth and fabrication processes which lead to the simplified fabrication of highly complex integrated circuits. This progress includes the use of selective etchants and sacrificial materials layers, hybrid growth (e.g., GaAs on Si) and precise epitaxial growth (e.g., graphene, graphite), and clever use of multifunctional materials and device structures. In this paper we address many of the bottlenecks issues now confronting advanced integrated optoelectronic circuit technology.


212 Strain Dependent Diffusion during Dry Thermal Oxidation of Crystalline Si. A. M. Stoneham, J. J. Trussell, Dept. of Physics, North Carolina State University, Raleigh, NC 27695-8202.

The effect of strain on the rate of dry thermal oxidation of silicon has been investigated. Local atomic strain in SiO_2 films, grown in two steps separated by an intermediate anneal, was determined by infrared spectroscopy. The results support an oxidation model based on strain and diffusion of oxygen to the growth interface. In this interpretation, the intermediate annealing step enhances the diffusion of oxygen through the oxide grown before the anneal, and therefore increases the oxidation rate.


Ellipsometry, transmission electron microscopy (TEM), and step-profile measurements are used to study the dry oxidation kinetics of silicon at temperatures from 750 to 1100°C. For oxide film thickness of 10 nm, the thickness measurements agree within experimental error. For oxide film thinner than 35 nm, the ellipsometry gives higher thickness values than the TEM measurements. Thickness measurement for films grown in two steps was determined by x-ray reflectivity. The highest oxidation rate was obtained for films grown in two steps separated by an intermediate anneal, and this was determined by infrared spectroscopy. The results support an oxidation model based on strain and diffusion of oxygen to the growth interface. In this interpretation, the intermediate annealing step enhances the diffusion of oxygen through the oxide grown before the anneal, and therefore increases the oxidation rate.

214 Modeling Process-Dependent Thermal Silicon Dioxide (SiO_2) Films on Silicon: H. Wei and A. K. Hanson, Thayer School of Engineering, Dartmouth College, Hanover, NH 03755, J. Skinkian and J. Rogers, IBM General Technology Div., Essex Junction, VT 05452.
This study models the process-dependent SiO₂ film microstructure and associated high frequency dielectric constants. For Si-doped dielectric thicknesses under 200 Å, the correct understanding of bulk SiO₂ and interlayer film thicknesses is essential to device physics studies, such as accurate CV data interpretation, proper impedance accounting, spectral reflectance data reduction, and not-carrier reliability analysis. We demonstrate the need for a two-layer model by examining the characteristic signature of ellipsometrically reduced using a one-layer model, and comparing to a two-layer model. We conclude with extraction of index of refraction data for both interfacial and bulk portions of the oxide films, using the two-layer model, as a function of processing temperature.

215 New Approach to Chemically Enhanced Oxidation—A Review: J. R. Jackson, Lehigh University, Sherman Fairchild Laboratory, Bethlehem, PA 18015

A new approach to the technology of silicon oxidation involves the use of very low concentrations (ppm) of an appropriate fluorine compound to the oxidant stream. This paper reviews the role of fluorine as an additive to the oxidation process and some of the related studies that indicate its advantage over present state-of-the-art oxidation practice. The work is discussed by considering experimental techniques and growth kinetics, along with stacking faults annealing, OED/ORQ, and MOS characterization.

216 Kinetics of Oxidation of Silicon by Electron Cyclotron Resonance Plasma: J. Joseph, Ecole Centrale de Lyon, Ecully, France; Y. Z. Hu and E. A. Irene, Dept. of Chemistry, University of North Carolina at Chapel Hill, NC 27599-2380

The electron cyclotron resonance, ECR, plasma oxidation of silicon was investigated using in situ during process static spectroscopic ellipsometry for Chemical timed ellipsometry. Through the use of a temperature independent photon energy fast dynamic oxidation kinetic measurements were performed. Along with spectrometric ellipsometric measurements, the kinetics are interpreted in terms of an electric field assisted model where ionic species dominate the kinetics. Interface damage was also assessed.

217 Mechanisms of Oxidation Rate Enhancement in Negative Point Oxygen Corona Discharge Processing of SiO₂ Films on Si: L. L. Mandelberg, Dept. of Electrical and Computer Engineering, Concordia University, West Montreal, Que., Canada H3G 1M8

Negative-point oxygen corona discharge processing at 600-900°C dramatically enhances the oxidation rate, while creating SiO₂ films with the refractive indexes and oxygen transport characteristics of fused quartz, as well as hydroxyl concentration and fictive temperature. The composition vs. dose curves for electron spin resonance and x-ray luminescence are highly nonlinear, but are fit well by a simple model involving creation, activation, annihilation, and deactivation. The photon energy studies demonstrate that the spin active defects are caused by electrons ejected by photoabsorbed x-rays.

218 High Pressure, Low Temperature Oxidation of Si₄⁺Ge⁺: Promise for MOS-Quality Passivation: C. Caraginis, Y. S. Huang, and D. C. Parne, Div. of Engineering, Brown University, Providence, RI 02912

Thermally grown SiO₂ on Si is technologically essential since it provides high-quality electronic passivation of surfaces and interfaces in Si-based microelectronics. Such a simple method of producing device quality passivation does not exist, however, for other important semiconductor systems such as SiGe. Conventional oxidation of Si₄⁺Ge⁺ produces oxides which, due to molecules dissolved in the gas phase, cannot be used as device oxide. Our oxide deposition technique provides high-quality oxide at 1000°C. By an analysis of the atomic mechanisms affecting the film formation process, we have found that the oxide properties are directly related to the atomic species present at the oxide surface. The oxide consists of an electric field assisted model where ionic species dominate the kinetics. Interface damage was also assessed.

Infrared-dichroism of thermally grown silicon oxide films is found using Fourier transform infrared reflection absorption spectroscopy. Two types of peaks were observed at 1250 cm⁻¹ only on the IR spectra using infrared rays polarized parallel to the plane of incidence: one projected upward and another projected downward. Between these two peaks, the upward peak did not increase linearly with oxide thickness, and was considered to appear on a very thin oxide film. Thus we believe that the upward peak reflects the chemical structure of the Si-SiO₂ interface.

221 Deconvolution of Thickness-Averaged Structural and Optical Properties of Thermally Grown and PECVD SiO₂ Films: C. E. Shearer, Jr., C. H. Borkman, and G. Locusky, Dept. of Physics, North Carolina State University, Raleigh, NC 27695-8202

The local atomic strain and index of refraction of SiO₂ films that were thermally grown or deposited at remote plasma-enhanced ch:al-vapor deposition, were determined by IR spectroscopy. A combination of two-layer model, and comparing to a two-layer model. We conclude with extraction of index of refraction data for both interfacial and bulk portions of the oxide films, using the two-layer model, as a function of processing temperature.

222 TEM Investigations of the Oxidation Kinetics of Amorphous Silicon Films: M. Reiche, Institut für Festkörperfysik und Elektronenmikroskopie, Weinberg 2, D-8400 Halle/S., Germany

Thin SiO₂ films (10 nm ≤ d ≤ 100 nm) were prepared by dry oxidation (T = 900°C) of amorphous or nanocrystalline silicon. The film thickness vs. the oxidation time and the development of the interface roughness Δd were measured by X-TEM. The oxidation kinetics are described on the basis of Deal and Grove’s model; differences have occurred as a function of the phosphorus doping (by ion implantation) of the a-Si layers. In addition, Δd was found to vary also with the dopant concentration. Comparison clearly shows that the oxidation of a-Si layers differs from that of polycrystalline deposited ones. This is especially true of the preparation of ultrathin SiO₂ films (d ≤ 20 nm).

223 Thermal and X-Ray Production of Point Defects in Vitreous SiO₂: P. F. Gilean, Dept. of Physics, Colorado State University, Fort Collins, CO 80523

The concentrations of E, ND, and P or NOH of electron spin resonance and x-ray luminescence are reported as a function of x-ray dose. The electron spin resonance concentration of E, ND, and P or NOH of electron spin resonance and x-ray luminescence are reported as a function of x-ray dose. The electron spin resonance concentration was found to vary also with the x-ray dose, and to show the photochemical reactions similar to those of a-Si layers. The concentrations of E, ND, and P or NOH of electron spin resonance and x-ray luminescence are reported as a function of x-ray dose. The electron spin resonance concentration was found to vary also with the x-ray dose, and to show the photochemical reactions similar to those of a-Si layers. The concentrations of E, ND, and P or NOH of electron spin resonance and x-ray luminescence are reported as a function of x-ray dose. The electron spin resonance concentration was found to vary also with the x-ray dose, and to show the photochemical reactions similar to those of a-Si layers.

224 Nonstoichiometry and Defects in Bulk a-SiO₂: H. Kawazoe, Research Laboratory of Engineering, Materials, Tokyo Institute of Technology, Nagaizuka, Midori-ku, Yokohama 227, Japan, K. Aoea, Electrotechnical Laboratory, 1-1-1 Umezono, Tsukuba, Ibaraki 305, Japan

Effects of nonstoichiometries and impurities on the type and concentration of the structural imperfections present in synthetic and bulk a-SiO₂ were studied. O₂ molecules dissolved in the a-SiO₂ films were used as a model involving creation, activation, annihilation, and deactivation. The oxygen energy studies demonstrate that the spin active defects are caused by electrons ejected by photoabsorbed x-rays.

225 The Paramagnetic Defects in Crystalline SiO₂: F. J. Wei, R. J. McEchron, and M. M. Mombourquette, Dept. of Chemistry, University of Saskatchewan, Saskatoon, SK, Canada S7N 0W0

The so-called point defects in crystalline silicon dioxide are multitudinous, and form a basis for the understanding of such defects in fused quartz and silica glasses. More than fifty of the paramagnetic species in a-SiO₂ are now known, and some of the more common are described in detail by electron paramagnetic resonance spectroscopy. A review of the latter defects is presented, including a discussion of recent work on generating powder glass/epi lineshapes from the single-crystal parameters.

226 Formation of Si/SiO₂ Heterostructures by Low-Temperature Plasma-Enhanced CVD: G. Locusky, Y. Ma, T. Yasuda, and S. Habernehl, Dept. of Physics, North Carolina State University, Raleigh, NC 27695-8202

In a new low-temperature plasma process in which interlayer film thickness and reaction are independently controlled. This technique includes oxidation of Si by remote UV or ONO by remote plasma-enhanced CVD.
223 Chemical Structures of Native Oxides Formed during Wet Chemical Treatments on Atomically Flat Si(111) Surface: H. Ogawa and T. Hatton, Dept. of EEE, Musashi Institute of Technology, 2-21-1 Tamazutsumi, Setagaya-ku, Tokyo 158, Japan

The infrared and photoelectron spectra arising from Si-H bonds at and near the native oxide/silicon interface could be extracted without deconvolution, and reference spectra obtained for native oxide formed in H_{2}SO_{4}-H_{2}O_{2} solution, which was found to contain negligible amount of Si-H bonds at and near the native oxide/silicon interface. The native oxides studied using FT-IR, and XPS were formed during wet chemical treatments on H-terminated atomically flat Si(111) surfaces.

234 Silicon Surface Analysis and Very Thin Silicon Oxide Characterization after HF/ETHanol Pseudoxidation Cleaning: J. R. Morante, B. Garrido, and J. Samitier, LCMM Dept. Faculty of Apirado i Electrónica University of Barcelona, Diagonal 647, 08028 Barcelona, Spain, F. Gessin, J. L. Prom, and G. Sarrabia Rosseau, LASS, CNRS 7, 31071 Toulouse Cedex, France.
240 The Effect of Surface Roughness on Gate Oxide Leakage Currents: M. Chonko, Motorola Inc., Advanced Products Research and Development Laboratory, Austin, TX 78721, V. Kauhisk, Motorola Inc., Surface Analysis Laboratory, Microprocessor and Memory Technology Group, Austin, TX 78721

We studied the effects of silicon surface roughness on 150 Å gate oxide characteristics. Varying degrees of roughness were induced experimentally. For short etch times, the initial roughness is reflected at the top oxide surface while the substrate/oxide interface is smoothed. This results in increased current loss from the poly/oxide interface. The substrate injection remains at its initial level. The poly/oxide interface rapidly degrades with increasing roughness (etch time). At longer times, the substrate oxide interface begins to attain some of the characteristics of the induced roughness, and injection characteristics from this interface also begin to degrade. This non-symmetric degradation of IV characteristic is used to qualitatively evaluate process quality and oxide surface roughness. The potential of two common oxide etching solutions to roughen the Si surface are compared using this technique.

241 A Double Sacrificial Oxide Process for Smoother 150 Å SiO Gate Oxide Interfaces: H.-H. Tseung* and P. J. Tobin, Motorola Inc., Advanced Products Research and Development Laboratory, Austin, TX 78721

We have found that a double sacrificial oxide process provides a smoother active surface than the single sacrificial oxide process. The double sacrificial oxide process (a poly sacrificial oxide, a low-temperature (LT) oxide, and a NO sacrificial oxide) was evaluated using the LT oxide thickness as a variable. The results show that increasing the LT oxide thickness results in a smoother active surface. The second sacrificial oxide process results in a smoother poly/oxide gate oxide interface. Further, the interface state density of the gate oxide/substrate interface is reduced for the double sacrificial oxide process.

242 Effect of Solidification Induced Defects in CZ-Silicon upon Gate Oxide Integrity: H. Suga, H. Abe, H. Koyama, T. Yoshimi, I. Suzuki, H. Yoshioka, and N. Kagawa, Mitsubishi Materials Silicon Corp., 314 Kanaia, Nishi-sangao, Noda-shi, Chiba-ken, 250, Japan

Thin gate oxide in VLSI devices is considered to be correlated inherently with crystal growth conditions in the Czochralski method. The crystallographic growth of the silicon substrate influences the dielectric breakdown voltage of a thermal oxide gate. Experimental results show that the density of freeze-in defects increases as the silicon crystal orientation changes. The results suggest that a lowering of defect density at the interface is a necessary step in order to provide a better VLSI device. Further, better crystal growth will decrease the density of defects at the interface.


We investigated the effects of D-defects upon oxide breakdown field strength for thin gate oxides. The results show that the gate oxide thickness is the most important factor in determining the oxide breakdown strength. The results of our study show that for oxide thicknesses of 2.2 nm, the oxide breakdown field strength is reduced by approximately 50%.


The roughness of SiO₂ surfaces and interfaces resulting from different oxide growth conditions and to different thicknesses has been studied over a wide range of length scales using spectroscopic ellipsometry, atomic force microscopy, and X-ray photoelectron spectroscopy. A comparison of wet and dry oxidations revealed an increase of roughness at both the oxide/substrate and oxide/polymer interfaces relative to the starting surface. The interface topography was observed to evolve and become significant different from that of the starting wafer for increasing oxide thickness.

245 Properties of SIMOX and Related Systems: S. Chapiro, LPCS, École Polytechnique, 91128 Grenoble Cedex, France

The flexibility and quality of the SIMOX process are addressed by referring to the electrical properties of the silicon film, buried oxide, and the physical methods of characterization are described. Interface coupling, floating body, and transient effects are shown to be typical effects in thin film SIMOX devices. The nature of the buried oxide is revealed by hot carrier injection and irradiation experiment.

246 Reoxidized Nitrided Oxide Gate Dielectric for Advanced CMOS: G. J. Dunn, M.I.T. Lincoln Laboratory, Lexington, MA 02173

The advantages of reoxidized nitrided oxide (RNO) gate dielectrics for advanced CMOS applications are reviewed. RNO n-MOSFETs exhibit greatly improved resistance to channel hot carrier stress, due to the suppression of interface trap and bulk electron trap generation. However, RNO p-MOSFETs exhibit reduced resistance to stress, due to increased interface trap generation. The results indicate that RNO is a potential gate dielectric for advanced CMOS applications.


Low deposition heat induced high thin plasma enhanced CVD oxides have been used as gate dielectric to investigate the interface characteristics. By using PECVD gate oxide and thermally grown gate oxides, the PECVD oxide properties are not much divergent from the thermally grown oxide reliability is examined. The results of high field tunneling, channel hot carrier stress, and burned junction injection. The results suggest that although the oxide trapping density is comparable to the thermally grown oxide, the Si-SiO₂ interface strength is still slightly weaker.

248 Charge Trapping in an ONO Gate Dielectric: R. B. Klein, SFA, Inc., Landover, MD 20785, N. S. Saka, Code 6813, Naval Research Laboratory, Washington, DC 20375

The capture cross section, Q, and density, N, of intrinsic traps in a composite ONO (oxide-nitride-oxide) dielectric were measured. Utilizing low-field substrate hot electron (SHE) injection, we find that N = 5 × 10^{10} cm^{-2} and Q = 1.2 × 10^{-13} cm^{2}, characteristic of a Coulomb-attractive trap. Radiation experiments performed after SHE injection revealed a hole trap with a cross section. The results are discussed in light of our current understanding of electron and hole traps.

249 Interface Trap Density Reduction and Oxide Profiling for Fluorinated MOS Capacitors: D. Konstantas* and R. J. Jacquemier, Sherrin Fairchild for Solid State Study and Development, University, Bethlehem, PA 18015, F. A. Stevie, AT&T Bell Laboratories, Allentown, PA 18103

The effect of fluoride incorporation on the as-grown interface trap density was investigated using MOS capacitors with fluorinated oxide dielectrics as test structures. A clear reduction of the interface trap density was observed in dry oxides. The results are discussed in light of our understanding of electron and hole traps.

250 Physics of Extreme Quantum Confinement Exemplified by Si/SiO₂ System: R. D. Tu, University of North Carolina at Charlotte, Charlotte, NC 28223

The physics of extreme quantum confinement involves considerations beyond present day treatment of superlattices and quantum well structures. Apart from retaining the effective mass approximation, a constant dielectric constant cannot be assumed. In 

251 Integrity of Very Thin Silicon Films Deposited on SiO₂: M. Chonko*, Motorola Inc., Advanced Product Research and Development Laboratory, Austin, TX 78721, and D. Keitz, Motorola Inc., MOS II Wafer Fabrication Facility, Austin, TX 78715

The physics of very thin LPCVD silicon films has been studied as a function of deposition temperature and pressure. Integrity was defined as the ability of the film to protect an underlying oxide film from an HF etch. We have found that polycrystalline films deposited at temperatures above 600°C are continuous. Films deposited in the amorphous phase protect the underlying oxide and therefore must be continuous.

252 Researches on SiO₂ on InP and GaAs MOS Structure: Y. K. Si and C. J. Huang, Dept. of Electrical Engineering, National Chiao Tung University, Tainan, Taiwan, China

The high quality SiO₂ layer has been deposited on GaAs and InP by photo-cVD using dextemium lamp. Its refractive index is 1.462 at SiO₂/N₂O = 0.25. The FTIR data show that Si-O bonds are dominant in this film. XPS and AES measurements show that a good interface and bulk layer are attained. The high frequency (1 MHz) C-V of the n-InP MOS diode has been measured, and a maximum interface state density (Dₛ) of 2 × 10^{12} cm⁻² has been measured. Measurements were increased by Terman's method. DLTS measurement was applied to n-GaAs MOS diode and a maximum surface state density (Nₛ) of 6 × 10^{10} eV⁻¹ cm⁻² at E = 0.4 eV was determined.


This paper reviews the information that has been collected about the SiO₂ slow states from the study of Random Telegraph Signals. New results on the interpretation of the entropy change on capture into the defect, and the measurement of an IRS due to an oxide two-level-system are presented.
254 Single Electron Transfer from the Channel in Sub-μm MOSFETs to an Individual Interface Trap: M. Schutz and A. Pepper. Institute of Solid State Physics, Uni-D-8520 Erlangen, Germany.

A new model for trap formation in ultrathin MOSFETs using tunneling switching (RTS) of only a single interface trap in the active gate area of a sub-μm sized MOSFET is reported. The use of 1/f noise at low frequencies generates in this case a perfect Lorentzian frequency spectrum. The trapping rate for capture and emission are over analyzed over 5 orders of magnitude as a function of the gate bias voltage, the temperature, and for the first time the substrate bias voltage. From the experimental data, we can evaluate the level energy 150 meV, the image charge energy lowering 45 meV, and the free energy 170 meV of the Coulomb blockage in the transfer rate.


We have found that the structure of the F center in order to learn something about the oxide structure around the defect. To enhance the concentration of "O from a natural abundance of 55.76% is typically present in the SiO2. Within the resolution of our analysis, the position of this oxygen atom relative to the dangling bond is not unique, but varies randomly from site to site. The number of oxygen atoms, however, is the same at every site.

256 The Influence of Crystal Orientation and Processing Conditions on the Energy Distribution of Traps at the Si-SiO2 Interface: Y. Doria, J. Balcells, and G. Lacovka-Young, College of Physics, North Carolina State University, Raleigh, NC 27695-8202.

We have studied the local atomic strain as a function of crystal orientation and processing conditions, and its relationship to the energy distribution of interface traps in the Si bandgap. For example, for oxides grown on Si(100) surfaces, an increase in oxidation temperature promotes strain relaxation and reduction of interface states at energies above 0.4 eV in the Si bandgap, but not at energies between 0.2 and 0.4 eV.


Recent studies on ULVAC's wide bandgap LiF have focused on the image charge Monte Carlo simulations. Within the resolution of our analysis, the position of this oxygen atom relative to the dangling bond is not unique, but varies randomly from site to site. The number of oxygen atoms, however, is the same at every site.

258 Charging and Discharging Properties of Electron Traps Created by Hot-Carrier-Irradiation in Gate Oxide of n-MOSFET: D. Vuillaume, URA253 CNRS, ISEN, 59045 Lille, France

We have studied the emission properties of oxide defects induced by hot-carrier-aging of MOSFETs. The changes of the charge states of the traps have been monitored by the time-evolution of the very low level gate current measured by the floating-gate technique. A large capture cross section (×10^-14 cm^2) has been found for electron traps which has been correlated with optical properties. According to these optical properties, the hot-carrier-induced defects should be the acceptor-like defect at -1.7 eV below the CB with a large lattice relaxation (photoionization dissociation is a function of local network strain and is in a range of 0.6 to 1.1 eV). Our calculation of the self-trapped hole agrees very well with Griscom's "O hyperfine results for STI. We discuss the variation of these parameters with network strain.


We have measured the emission properties of oxide defects induced by hot-carrier-aging of MOSFETs. The changes of the charge states of the traps have been monitored by the time-evolution of the very low level gate current measured by the floating-gate technique. A large capture cross section (×10^-14 cm^2) has been found for electron traps which has been correlated with optical properties. According to these optical properties, the hot-carrier-induced defects should be the acceptor-like defect at -1.7 eV below the CB with a large lattice relaxation (photoionization dissociation is a function of local network strain and is in a range of 0.6 to 1.1 eV). Our calculation of the self-trapped hole agrees very well with Griscom's "O hyperfine results for STI. We discuss the variation of these parameters with network strain.


Impact ionization and positive charge formation in silicon dioxide have been controversial issues for many years. In this study, details on the impact ionization due to the development of a high energy tail on the hot-electron energy-distribution is shown to occur in films thicker than 20 nm at fields higher than 8 MV/cm. This process is demonstrated to account for hole currents in the substrate circuit of n-channel FETs and for the observed trapping of charged accumulating at the silicon-silicon dioxide interface at low injected-carrier-Flucuencies (less than 0.01 C/cm^2).


Internal photoemission in combination with semiclassical Monte Carlo simulations was used to measure energy dependent absolute electron-phonon scattering rates and impact ionization rates in SiO2 at electron energy of 20 eV. Our results are consistent with the standard model for electron-phonon interaction. At high energies, strong deviations from this model are observed. Using the measured capture cross sections, we are able to quantitatively explain hole generation in n-channel MOSFETs.


We have characterized the charge-to-breakdown (Qbd) in ultrathin oxides as a function of stress current density (Jbd), oxide thickness and temperature, using constant current stress at various fixed Jbd. We explain this difference in the slope for each thickness is indicative of different breakdown mechanisms. Qbd is also seen to decrease with temperature and thickness for a fixed Jbd. We explain this qualitatively in a self-consistent manner utilizing existing models and some new concepts.

263 Radiation-Induced Interface Traps in MOS Devices: N. S. Saks and D. B. Brown, Naval Research Laboratory, Washington, DC 20375.

Creation of interface traps in MOS devices has been studied as a function of time following a short irradiation pulse. Measurements have been made as a function of oxide field, oxide thickness, and other variables. A new model based on dispersive H transport has also been developed. We find excellent qualitative and quantitative agreement between the data and this model.


Electron paramagnetic resonance and capacitance-voltage results indicate that oxygen vacancy related defects (E’) in hydrogen-treated oxide films occur in concentrations ten times that found in nonhydrogen treated films, and unlike their counterparts in nonhydrogen treated films, they are not associated with an increase in irradiation-induced positive charge. We deduce that this hydrogen-related E’ type oxide defect is formed by the precursor rather than from a standard oxygen vacancy precursor.


We report recent theoretical studies of several models for defects that could transform directly from H to E’. The models considered are the E’ center, the nonbridging oxygen hole center, the self-trapped hole, and two new variants we call the strained-bond and broken-bond self-trapped hole. Based on the calculated energies of reaction and activation energies, the most likely candidates are the last two. For the STH defects, the activation energy for H dissociation is a function of local network strain and is in a range of 0.8 to 1.1 eV. Our calculation of the self-trapped hole agrees very well with Griscom’s "O hyperfine results for STI. We discuss the variation of these parameters with network strain.


The aim of this investigation was to examine the efficacy of room temperature hydrogenation (by a 400 eV hydrogen beam from a Kaufman source) in the removal of electronic defects in SiO2 at the Si-SiO2 interface, and the Si sub-surface by exposure to a beam of 15 keV Si ions. Upon hydrogenation, profound changes were observed in the trap parameters as well as in the microscopic characteristics of the MOS structures. There was significant decrease in the trap density and the oxide leakage current.


Hot electron induced hydrogen redistribution in Al-gate MOS capacitors has been observed using elect. on injection by internal
photo-emission together with hydrogen concentration profiling by using a double probe technique. After ion etching, the film was measured with the gate in place. Initially, most of the hydrogen in the capacitors, \(-2 \times 10^{19} \text{cm}^{-1}\), was found to be at the Al/SiO\(_2\) interface as a result of water contamination of the SiO\(_2\). After injection, this peak was reduced in size, while depending on the injection dose and the polarity of the bias a substantial fraction of hydrogen at the Al/SiO\(_2\) interface was observed. This was found to increase with electron fluence, reaching \(-2 \times 10^{20} \text{cm}^{-1}\) at a fluence of 5 C/cm\(^2\) under negative bias. Clear evidence was obtained for hydrogen diffusion into the SiO\(_2\) from the Al/SiO\(_2\) interface under positive gate bias. However for moderate fluences the hydrogen concentration in the bulk of the SiO\(_2\) changed very slightly as the interface peak grew. These results show that hydrogen at the Al/SiO\(_2\) interface acts as a source for the hot electron induced redistribution. They are discussed in terms of current models of hot electron induced defect generation.

Molybdenum and tungsten silicides show promise as a complement to polysilicon in VLSI and other interconnections. The profile was measured with the gate in place. Initially, most of the hydrogen in the capacitors, \(-2 \times 10^{19} \text{cm}^{-1}\), was found to be at the Al/SiO\(_2\) interface as a result of water contamination of the SiO\(_2\). After injection, this peak was reduced in size, while depending on the injection dose and the polarity of the bias a substantial fraction of hydrogen at the Al/SiO\(_2\) interface was observed. This was found to increase with electron fluence, reaching \(-2 \times 10^{20} \text{cm}^{-1}\) at a fluence of 5 C/cm\(^2\) under negative bias. Clear evidence was obtained for hydrogen diffusion into the SiO\(_2\) from the Al/SiO\(_2\) interface under positive gate bias. However for moderate fluences the hydrogen concentration in the bulk of the SiO\(_2\) changed very slightly as the interface peak grew. These results show that hydrogen at the Al/SiO\(_2\) interface acts as a source for the hot electron induced redistribution. They are discussed in terms of current models of hot electron induced defect generation.

284 Suppression of WSi, Peeling in 8.0's BICMOS Technology: B. L. Mantha and C. S. Wang, VLSI Technology Inc., San Jose, California 95131

Tungsten silicide on top of arsenic-implanted polysilicon has been employed in a 0.8 \mu BICMOS technology to serve as a gate material for MOS transistors, and as emitters for bipolar transistors. We have observed peeling of the tungsten silicide film after contact reflow. Dependence of tungsten silicide peeling on arsenic implant dose and anneals before contact reflow has also been studied. It is found that it is safe to deposit tungsten silicide on 8 \% arsenic implant dose without peeling if an anneal step is included before reflow. An anneal step at 950 °C for 60 min in nitrogen is required to completely suppress tungsten silicide peeling.

270 Characterization of WSi Films Deposited by Dichlorosilane in Cold-Wall Systems: D. Pramanik and V. Jain, VLSI Technology Inc., San Jose, California 95131

Deposition of tungsten silicide using dichlorosilane in a cold wall system was developed. The results indicate that as-received wafers showed very little increase in bow and warp up to 24 in./min insertion rate. An anneal step at 274 °C for sub-half-micron Al/SiO\(_2\) showed very little increase in bow and warp up to 24 in./min insertion rate. This paper describes etch pits or polyvoids which have been observed in the poly layer using SEM and TEM right after the nitride step for poly void formation in the LOCOS process. A FORTRAN software package was developed to provide an estimate of interstitial oxygen (O\(_i\)) from FTIR absorption in silicon by three separate methods. We report on a comparison of calculation methods, which include the ASTM standard test F1188-88, the short baseline (SBL) and the curve baseline (CBL) methods. Reproducible O\(_i\) results are obtained for the SBL and CBL techniques, although the CBL values are systematically lower. A FTIR-SDS calibration is given for both lightly and heavily doped silicon, where selective electrochemical thinning was used to enable the FTIR absorption measurements.

277 The Warpage of As-Received and Oxygen Precipitated CZ Silicon Wafers: J. Jeong and H.-D. Chou, Discrete and Materials Tech. Group, Motorola Inc., Phoenix, AZ 85008

Various types of as-received and CMOS thermal processed 100 mm diam wafers were used for warpage study under different furnace operations. The results indicate that as-received wafers showed very little increase in bow and warp up to 1000 °C with temperature and 24 in./min insertion rate. For the CMOS thermal processed wafers, both the prior amount of oxygen precipitation and the bulk microdefect morphology affect warpage.

278 Modeling of Phosphorus Precipitation: S. Dunham, Boston University, Boston, MA 02215

The deactivation of dopants at high concentrations can be accounted for by considering aggregation of the dopant into precipitates. In this work, we model phosphorus precipitation using a combination of equation of time and temperature by simulating the evolution of the distribution of precipitate sizes using discrete rate equations and the Fokker-Planck equation. We compare the results to observations of dopant deactivation of laser-annealed implants in the temperature range 300-1000 °C as measured by Nobili and coworkers.


We have developed a new model for accurate and comprehensive simulation of BF, implants into single-crystal silicon. This model simulates the boron concentration profiles resulting from BF, implants in silicon, and it takes into account the effects of tilt and rotation angles as well as energy and dose. The model covers the following ranges of implant conditions: energies of 15-65 keV, doses of 1 \times 10^{10} \text{cm}^{-2}-1 \times 10^{11} \text{cm}^{-2}, tilt angles of 0°-10°, and rotation angles of 0°-90°. This model can be easily implemented into existing process simulation codes such as SUPREM.

285 Investigation of Ion-Surface Interaction in Ar Plasma Etching of Si and PECVD of SiO\(_2\): Film by High-Frequency C-V Method: A. Pasciucco, Stanford University, Stanford, CA 94305, J. P. McVittie, Center for Integrated Systems, Stanford University, Stanford, CA 94305, C. Chang, Stanford University, Stanford, CA 94305, K. C. Saraswat, Center for Integrated Sys-

286 H. Cracking in Irradiated MOSFETs and H+ Formation: T. S. E. Stathis, Naval Research Laboratory, Washington, DC 20375 and A. H. Edwards, University of North Carolina, Charlotte, NC 28223

Hydrogen hydrogen introduced into irradiated MOSFETs causes the build-up of interface states and decrease of trapped positive charge. Results are explained by the sequence in H₂ cracking and H₂-H₂ formation. A new molecular orbital calculation model the cracking process. Comparing cracking by C centers and broken Si-O bonds, the latter is more likely. It is also argued that H₂ is a small polaron.

287 Suppression of Vias: J. L. Rosen, B. L. Mantha, and J. W. B. Opray, Intermapper, Racine, WI 53405

The deactivation of dopants at high concentrations can be accounted for by considering aggregation of the dopant into precipitates. In this work, we model phosphorus precipitation using a combination of equation of time and temperature by simulating the evolution of the distribution of precipitate sizes using discrete rate equations and the Fokker-Planck equation. We compare the results to observations of dopant deactivation of laser-annealed implants in the temperature range 300-1000 °C as measured by Nobili and coworkers.

288 Characterization of WSi Films Deposited by Dichlorosilane in a Cold-Wall System: D. Pramanik and V. Jain, VLSI Technology Inc., San Jose, California 95131

A new LP-CVD technique for WSi deposition using dichlorosilane in a cold wall system was developed. Film properties such as adhesion, resistivity, density, and thermal stability were studied. These films characterized better adhesion and thermal stability due to the stable as-deposited crystal structure. The ratio of the sheet resistance on a memory cell pattern with many steps and on a planar pattern (no steps) was lower than that in the case of the conventional monolaminate-reduction CVD WSi.
intertwined comb-like copper contacts on fiberglass. The semicon-ductor manufacturing equipment has been developed for the advanced semicon-ductor materials, AR

The result also indicated that the nucleation near the back side of silicon is the preferred site for the growth of grains during the growth of silicon. Si-H and Si-O-H atoms (originating from TEOS-Ο) and of Ar atoms (from Ar plasma) was considered responsible for the increase of positive fixed charge at the Si-SiO interface.

282 Trace Moisture Analysis in Monosilane-Hydrogen Mixtures at Atmospheric Pressure and Ion-Molecule Reactions: T. Irie, S. Iijima, and Y. Mitsui, Central Research Laboratory, Hitachi, Ltd., Kokubunji, Tokyo 185, Japan

An ionization pressure method using a sector-magnet spectrometer has been used for analyzing trace moisture contained in gaseous mixtures of monosilane and hydrogen for semiconductor fabrication processes. Trace moisture can be evaluated from the intensities of cluster ions, such as SiH₄OH and SiH₂OH, at several percent of monosilane concentrations. SiH₂OH may result from SiH₄ reacting with H₂O through proton-transfer and/or switching processes, where collisional stabilization plays an important role.

282 Chemical Foundations in Understanding the Step Coverage Problem in CVD Using the Silane Chemistry: C. Pavelescu and K. C. Saraswat, Center for Integrated Systems, Stanford University, Stanford, CA 94305

The correlations between the deposition kinetics and the step coverage problem in CVD using the silane chemistry are investigated. In particular, the near 1 value of the step coverage is consistent with a near zero surface reaction order with respect to silane, which indicates the surface saturation with silicon oxycarbonyl groups at 160°C due to reaction probability (step growth coefficient), K, is predicted. Although LPCVD and PECVD processes for SiO₂ deposition with a step coverage of 1 in dimensions around 1000 Å were characterized by a two molecular flow regime, while APCVD is described by a transitional flow regime, the step coverage of SiO₂ films in all processes is presented similar poor value. This behavior can be interpreted in terms of the step reaction kinetics described by a reaction order around 0.5 with respect to silane, which suggests that the film formation is not saturated with silicon containing species, and higher values of K are predicted for these CVD processes.

283 Characterization of Band-Pattern Polishing Marks Induced during Waxes/Wax Polishing Process Using Magic Mirror


In this study, we investigated various properties of band-pattern (one of the most commonly observed surface flaws) induced during waxes/wax polishing processes using the magic mirror method. The origin of this pattern and its effects upon various physical properties of the Si wafer, total thickness variation (TTV) mapping with automatic sorter, surface roughness measurement by laser surface interferometry, and electrical parameter measurements such as oxide breakdown strength and time dependent dielectric breakdown have been performed.

284 Highly Reliable Electrostatic Chucks Employing Plasma-Sprayed Aluminum-Oxide Coating for Advanced Semiconductor Processing: H. Y. Take, K. Moritzuka, and T. Ohmi, Dept. of Electronics, Faculty of Engineering, Tohoku University, Sendai 980, Japan.

A highly reliable electrostatic chuck for the advanced semiconductor manufacturing equipment has been developed by plasma spraying and etching fabrication procedure. This chuck is shown. Furthermore, the stable attraction force in the low temperature range has been realized by mixing of TiO₂ to aluminum oxide in the plasma spraying step. Some fundamental evaluations are described. Excellent thermal contact between the wafer and the chuck, as well as the electric potential control, has been established by applying the electrostatic chuck developed here.


We report investigation of photocoercive cells sensitive to the light spectrum of the wafer, but the size of the wafer, and ion-surface interaction involved in Ar plasma clean of Si cells offer effective, low cost, minimal toxic detection of the en-tier visible spectrum and an alternative to PdS and CdS whose etchability is less than optimum in the near infrared, red, and orange portion of the spectrum.

286 Mix-Cation Oxide Powders Prepared via Resin Intermediates Derived from Water Soluble Polyamides: L.-W. Tat* and H. U. Anderson, Ceramic Engineering Dept., University of Missouri, Rolla, MO 65401

Polymeric synthesis of mix-cation oxides via a liquid-mix (LM) process is described. Two types of polymeric gels have been developed to form fluffy resin intermediates which were converted to form oxides powders at relatively low temperatures. In addition to an optimized precursor made of cratic acid and ethyleneglycol, a new water-soluble polymer derived from starch derivatives was utilized in LM process. La₂SrCo₃FeO₅ and others were prepared having a single phase, line particles, soft agglomerates, and the superior sinterability.


A novel evaluation technique of III-V semiconductor materials such as GaP, GaAs has been attained using a newly developed noncontact wafer carrier concentration measurement method in terms of the carrier concentration over the range of 10¹⁴-10¹² cm⁻³. The measurement is based on the principle that the factor is 0.99 and the discrepancy between the two is the maximum limit within 27%. This technique is accurate enough for practical usage in production line.


The ICHT sol-gel process has been utilized to prepare spherical powders of zirconia-ceria hydroxide-gels (with mean diameter less than 100 μm). Some preparations were stabilized with nitrate or chlorite. The resulting amorphous powders were calcined at 400, 600, and 800°C in order to obtain the crystalline forms, and subsequently pressed at various pressures. The so obtained compacted gels were sintered for 8 h at 1700°C. The sintered pellets had relatively high porosity (higher than 30%), and the density of these ceramics was much higher than those obtained for CeO₂ or Y₂O₃ doped zirconia samples prepared by the same process. This probably happens through the agency of the cerium reduction due to the high oxygen impurities in the gas phase and the subsequent high temperature reoxidation by the air oxygen.


Low pressure chemical vapor deposition polyisilicon deposition was studied from 525 to 650°C. The poly appears to be amorphous with a smooth surface to 550°C and completely crystalline above 600°C. The transition region is from 550 to 590°C. The smooth sur-face morphology is preserved after POC; doping and a 1000°C oxida-tion. However, an in situ annealing at these temperatures (1000°C) results in large, coarse crystals with rough surface morphology. The smooth morphology of the 550°C silicon is transmitted to the subsequent polycide etch layers. The deposition temperature is found to affect poly etch rate and resistivity. A mechanism is proposed.


We have investigated the formation of oxygen precipitates in CZ silicon wafers covered by polycrystalline silicon (polysilicon) films. For the examined the size and density of oxygen precipitation in the wafers annealed at various temperatures. The results indicated that the oxygen precipitation was enhanced in the region near the back-side of the wafer, but the size of the precipitates in the back-side region was not different from that in the other region. The result also indicated that the nucleation near the back side
would not have occurred during annealing for oxygen precipitation. From these results, it was assumed that the strain field induced by polysilicon film would affect the stability of nuclei existing in the wafers.


A low-pressure chemical vapor deposition (LPCVD) process was used for depositing thin films of hafnium nitride/hafnium oxinitride (Hf/HfO$_2$/N), based on the reaction overall (HfCl$_4$ + N$_2$ + 1/2 H$_2$ + H$_2$ + H$_2$ + N$_2$ + 4HCl). A total of 24 experiments were performed investigating the deposition parameters. The surface morphology of Hf$_2$O$_3$N$_2$ crystallizes in a PCAVD reactor, and the effects of deposition temperature on microstructure, composition, and morphology were evaluated. Photoelectrochemically etched thin films of Hf$_2$O$_3$N$_2$ were grown in a series of wafers processed with a similar system of Hf$_2$O$_3$N$_2$. These novel redox transitions are of significant importance in electrochemical processes. The major factor influencing these transitions is the surface modification induced by the pretreatment.

292 Damage and Its Rapid Thermal Annealing Kinetics in Ar$_2$


The electrical, optical, and plasma etch properties of 460 nm molybdenum silicide/molybdenum thin films, obtained by the thermal reaction of vacuum deposited silicon on molybdenum, have been reported. The sheet resistance of 900°C annealed films was as low as 0.49 /square and proved a good stability when stored at an atmosphere under oven and atmospheric environment. Reflectivity measurements, performed at different temperatures, revealed that the deposition temperature strongly depends upon implant dose and annealing temperature, (ii) regardless of implant dose and annealing temperature, four distinctive deep level traps are induced, and (iii) these traps evolve during rapid thermal annealing.

294 Photoelectrochemical and Electrochemical Polishing of Epi-B-Si wafer and CrVD: R. D. Rasch, T. H. Nguyen, M. M. Carra, and D. Pask, EFC, Laboratory Park, Norwood, MA 02062

The feasibility of using a photoelectrochemical or electrochemical procedure to polish extremely high-quality Si wafers has been evaluated. Statistical experimental designs have been used to identify and define the etching conditions which would lead to a supersmooth Si surface. Statistical analysis of the experimental results shows that (F) concentration, solution pH, and applied potential profoundly influence the morphology of the etched SiC surface. Extremes have been obtained with photoelectrochemical and electrochemical polishing at a very fast rate compared to conventional mechanical polishing.

295 Characterization of Electrochemically Active Ni-Co Carbophospho and Crystaline Alloys: K. Luan, D. W. Kirk, and S. J. Tung, Department of Metallurgy and Chemical Engineering and Applied Chemistry, University of Toronto, Toronto, Ont., Canada M5S 1A4

The effects of phosphorus and crystalline Ni-Co alloys on the oxygen evolution reaction (OER) in alkaline solution has been studied. The surface electrochemistry and chemistry were investigated by means of steady-state potentiostatic method, cyclic voltammetry, and x-ray photoelectron spectroscopy (XPS). The electrochemically oxidized (potential) amorphous Ni-Co oxides showed an exceptionally high activity and excellent stability compared with the polycrystalline parts. Hydrous Ni-Co oxides, which are responsible to the enhanced catalytic activity, are believed to be formed on the surface of amorphous alloys via potential cycling process.

296 Influence of Pretreatment on the Unusual Bedox Behav- iour of Noble Metal Electrode: D. Burke, and J. K. Cuyt, Chemistry Dept., University College Cork, Cork, Ireland

The cyclic voltammograms for polycrystalline platinum in aqueous acid media is one of the best known features in electrochemistry. However, a considerably different, highly reproducible, response for the same system was observed recently following alkaline etching and extensive potential cycling. A similar investigation of gold: these new observed redox transitions are of significant importance in electrocatalytic processes. The major factor influencing these transitions is the surface modification induced by the pretreatment.


Low platinum loading thin film catalyst layers (approximately 5%) for polymer electrolyte membranes are prepared by dip, spin, and spray coating techniques. Electrode/membrane/electrode assemblies for fuel cells are prepared using several different catalyst application procedures and catalyst layer compositions. A new method is employed to characterize the thin film catalyst layers. In particular, ion and electrical conductivities and gas diffusivities within the catalyst layer and ionic conductivities across the catalyst layer/membrane interfaces are studied.

298 Chemical Bath Deposition of Cadmium Sulfide Thin Films, In Situ Growth and Structural Studies by Combined Quartz Crystal Microbalance and Impedance Techniques: D. Lincott and R. Ortega, Laboratoire d'Electrochiemie, ENSCP, 75231 Paris Cedex 05, France

Chemical bath deposition (CBD) of CdS layers, using the am- monia process, have been studied during the last two decades by a time combined in situ quartz microbalance (QCM) and electrochemical capaci- tance techniques. They are of significance in solar energy conversion in the evolution growth, have been obtained from combined experiments. The film is shown to have, in general, a duplex structure, with an inner compact layer (only measured by capacitance) and an outer porous layer, growing at longer reaction times. The influence of the thiourea concentration is studied in detail. A simple columnar growth model is proposed, which accounts well for the experimental results in the coalescence range.

299 Study of the Bandedge Shifts of n- and p-type CuInSe$_2$


Impedance measurements were employed for the in situ determination of the flatband potential of n- and p-type CuInSe$_2$ elec- trodes in various aqueous electrolytes. It was found that the flatband values could be shifted over a considerable potential range of about 0.2 –0.8 V, depending upon the nature of the electrolyte. Also, it was observed that the flatband potential could be fixed at intermediate positions by applying necessary potentials. This could open the door for band edge position tailoring.

300 Highly Oriented vs. Microcrystalline Thick Cadmium Films Deposited on Polycrystalline Nickel from a Molten Salts Electrolyte: R. R. Agarwal, Dept. of Chemical Engineering, Illinois Institute of Technology, Chicago, IL 60616

Thick and large highly oriented and microcrystalline cadmium films were deposited electrochemically on polycrystalline nickel substrates from a purified chloride/ammonium melt under an inert atmosphere of nitrogen. The films were characterized under the SEM/EDAX with respect to their respective surface features. The shift in crystal size in cadmium films was caused by the chemical solidification processes based on analysis of the chemical mechanism and rate controlling processes at the experimental conditions.

301 Surface Modification of Sintered Plates Negative Electrodes of Nickel Cadmium Batteries: D. A. Rutten, R. Seepethi, and N. Venkataraman, Research Institute, Battery Div., Karakuki 623 006 India

It is very important to consider the degradation mechanism of a negative electrode in NiCd since it results from the destruction of the active parts. Out of the four organic compounds studied, the BTA-created C$_6$H$_5$N$_2$O$_2$ is the one that possesses the best performance. Resulting in a significant improvement in cycle life and reduced deterioration of several test batteries, it exhibits a fairly uniform distribution of active material so as to retain 80% of its original capacity, whereas the control electrode retains only 58%.
302 Properties and Modification of Perfluorosulfonic Acid Membrane Surfaces: T. A. Zawodzinski Jr., Electronics Research Laboratory, Los Alamos, NM 87545, M. Shochet, Polymer Science and Engineering Dept., University of Massachusetts, Amherst, MA 01003, N. Hamel and G. Gerd, Dept. of Chem. Portland State University, Portland, OR 97202-0751, T. J. McCarthy, Polymer Science and Engineering Dept., University of Massachusetts, Amherst, MA 01003, S. Gottfeld, Electronics Research, Los Alamos National Laboratory, Los Alamos, NM 87545

Humidification of polymer electrolyte fuel cells is essential to maintaining adequate conductivity within the ionomeric membrane and within the polymer/CP composite catalyst layer. Sorption of water by the polymer is thus a crucial element in achieving overall cell performance. This work presents a method for investigating the sorption of water into the polymer electrolyte membrane and the change in the electrical conductivity of the membrane with water content. The results indicate that the sorption of water into the membrane is a key factor in determining the electrical conductivity of the membrane and that the presence of water in the membrane has a significant effect on the overall performance of the fuel cell.

303 In Situ Study of the Chemical Bath Deposition Mechanism of Cadmium Sulfide Thin Films on Gold and Copper Indium Diselenide Substrates: D. Linoc, R. Ortega, and J. Vedel, Laboratoire de Electrochimie, ENSCP, 75231 Paris Cedex 05, France. Chemical bath deposition of CdS layers, using the ammonia process, has been studied for the first time by in situ quartz crystal microbalance technique. This allowed us to monitor precisely the kinetic growth of the film, especially in the first steps, which is not possible with classical ex situ methods. A detailed study of the influence of the reaction parameters is performed (concentration of cadmium, pH, temperature...), giving new insights into the interpretation of the reaction mechanism. The study was also carried out on coevaporated CuInSe2 layers, in direct relation with the optimization of the position of the CuInSe2 buffer layer entering in the structure of high efficiency CuInSe2/CdS/ZnO cells.

304 Electrochemical Vapor Deposition of CeO2, Thin Film Electrodes for Solid Oxide Fuel Cells: J. F. Jaé and A. V. Verker, Dept. Materials Science and Engineering, University of Utah, Salt Lake City, UT 84112

Deposition of CeO2, of a thickness on the order of 50 μm were fabricated by electrochemical vapor deposition (EVD) using Ce(OH)3, as the source of cerium. Five films were deposited on zirconia and cera- mised silicon wafers. The kinetics of film growth were observed to be parabolic in accord with Wagners oxidation theory. The measured rate constant of 3.7 x 10^-7 cm/s at 1200°C is about five times larger than that reported for zircona films.

305 Evaluation of Doped Ceria with and without Zirconia Coating for Application in Low-Temperature Solid and Oxide Fuel Cells: K. Mehta, A. V. Verker, Dept. of Materials Science and Engineering, University of Utah, Salt Lake City, UT 84112

Y-0.3-doped and Er-0.3-doped CeO2 solid electrolytes with and without a thin ceria layer of zirconia have been deposited over a range of temperatures between 600 and 700°C. Disks with zirconia coating exhibited superior performance compared to uncoated cells. Also, in cells with improved electrodes, most of the substrate at 1200°C. The kinetics of film growth were observed to be parabolic in accord with Wagners oxidation theory. The measured rate constant of 3.7 x 10^-7 cm/s at 1200°C is about five times larger than that reported for zircona films.

306 Kinetics of the Cadmium Electrode in Alkaline Solution: S. Tamil Selvan, R. Sabapathy, and N. Venkataraman, Central Electrochemical Research Institute, Battery Div., Karaikudi 623 008, India

The purpose of this paper is to investigate the charge transfer mechanism of planar Cd electrode in KOH solution with and without organic compounds. The mechanism does not change in the presence of azole compounds, such as BTA and P-di. A Neumark Tafel slope of 49 ± 3 mV decade^-1 is obtained as a result of P-VA-CMC addition to the electrolyte. Furthermore, the results suggest that organic compound-modified CeO2 surface is formed which results in enhanced kinetics of the Cd/Cd2O3 electrode reaction.

307 Evaluation of Electrodeposited Electrocastom Composite Metal Film Coatings for Cathodic H2 Evolution in Water Electrolysers: B. E. Convey, R. S. Simperge, B. H. Brown, and L. Gao, Dept. of Chemistry, University of Ottawa, Ontario, Canada K1N 8N5

Electrodeposited coatings are required for minimization of overvoltage and electric power loss at electrolyzer cathodes in water electrolyzers, and for cathodes used in the chlor-alkali and F2 production (40-1200°C). Ni, Mn-Cd, Ni-W, and Ni-V composite films electrodeposited on Cu based on studies of Tafel polarization relations and particularly on data concerning the coverage by the Ni on the intermediate involved in electrocatalysts of the cathodic H2 evolution process. Inclusion of a complement of Cd (1-2 at/o), co-deposited with the transition metals, leads to substantial enhancement of activity, possibly due to changes of microstructure of the deposit and/or electronic band-structure changes.

308 Effects of Anisotropy on Pattern Formation in Electrochemical Deposition: P. A. D. C. R. Chu, Dept. of Physics, Emory University, Atlanta, GA 30322

We have studied the effect of anisotropy on electrochemical deposition grown from solutions of copper in the National Laboratories. The cell consists of two parallel plates and anisotropy is introduced by machining a 1 mm square grid of grooves on one of the plates. The effects of anisotropy on the growth are discussed and new results on the size distribution and fractal analysis of the anisotropically grown electrochemical deposition patterns are presented and compared with simulations and theory of diffusion-limited aggregation with anisotropy.

309 Interfacial Dynamics and Induced Convective Transport in Electrodeposition: D. P. Gag, Dept. of Chemical Engineering, University of New Hampshire, Durham, NH 03824-3591

Application of pattern-forming theory to electrodeposition is reviewed. The critical role of interfacial dynamics in velocity and morphology selection is emphasized. Experimental studies of deposition from unsupported electrolyte are reported. The presence of induced convection at the growth front is demonstrated, and the resulting concentration field is imaged. This spontaneous convection is shown to be coupled to morphology development.

310 Diffusion to Patterned Electrodes: Y. Dussas and P. Duby, Henry Krum School of Mines, Columbia University, New York, NY 10027

Diffusion to patterned electrodes has been investigated by potential step and current step techniques. We have confirmed the findings of Pajkoszy and Nyitros, i.e., the current on an electrode with a photosensitive growth of fractional dimension d decays with the -3/2 power of time during the potential step. We have also observed a similar effect with nonfractal patterned electrodes.

311 Electro-Convection Around Two-Dimensional Ramified Copper Aggregates: V. Fleury, J.-N. Chazalviel, and M. Rosso, Laboratoire de Physique de la Matière Condensée, École Polytechnique, 91289 Palaiseau, France

We propose a model for the electrochemical deposition of ramified copper aggregates, which are produced by electrically driven convection in the regime of fast growth (current density > 50 mA/cm2). This model takes into account the electrode-convective mechanism of the fluid. It predicts the existence of a virtual interface in the vicinity of the tips of the deposits. This interface separates a zone free of ions of both kinds and a zone of constant concentration. Preliminary experimental observations are well accounted for by the model.


Experiments on quasi-two-dimensional zinc electrodeposition have been conducted mainly with a parallel or strip geometry. Morphological changes have been observed when increasing the cell dimensions with pronounced effects leading to morphological transitions under high current conditions. In addition, the growth probability distributions of the electrodeposits have been measured and analyzed in terms of multifractal concepts. Finally, the generalization of classical Cottrell-like laws, to include the fractal roughness of the experimental electrodes, have been also tested.

313 Morphology Transitions in Rapid Electrodeposition: L. M. Sander, Dept. of Physics, The University of Michigan, Ann Arbor, MI 48109-1120

Electrochemical deposition in extreme overpotential conditions provides an opportunity to directly examine the correlation between microstructure and large-scale structure in a controlled system. It is representative of a class of interfacial pattern formation problems. We discuss how long-range order can spontaneously reemerge as growth is driven further from equilibrium. We discuss both the mechanisms for this reordering, including the discovery of a metastable crystalline form of zinc.


We report a series of electrochemical deposition experiments of copper from CuSO4 solutions in a quasi-two-dimensional geometry. They performed at very slow rates (0.1-0.2 mm/s) so that local growth effects can compete with the nonlocal Laplacian effects. In the early stage of growth, we observed a behavior consistent with the conventional Mullins-Sekerka instability. This is followed by coarsening and roughening of the surface which lead to a columnar structure with deep and narrow valleys. The presence of activity, possibly due to changes of microstructure of the deposit and/or electronic band-structure changes.

315 FRACTALS IN ELECTROCHEMISTRY

Energy Technology/Physical Electrochemistry/Corrosion
116C

where \( a \) is the smaller feature size of the fractal, \( \rho \) the electrolyte resistivity and \( \gamma \) the capacitance per unit surface of the electrode. The CPA frequency range is found to be equal to \((L/a)^2\) that is to the total length of the fractal.

322 The Double Layer Impedance: Effects of Electrode Roughness

This paper presents the results of a numerical study of the double layer self-impedance of various electrode geometries in two and three dimensions. Our numerical method makes use of the formal relationship between the double layer impedance at a rough electrode and the behavior of random walks reflected from the surface of that electrode. We find the behavior of the impedance is determined by the “multifractal” properties of the surface.

323 Theory of Interfacial Constant Phase Element in Electrode-

In this paper we review theoretical and experimental evidence that the fractal nature of the electrode-electrolyte interface is the source of the constant phase element seen in the impedance spectra of electrolytic cells. The fractal geometry gives rise to a complex distribution of parallel current paths, and the competition between these paths results in the fractional power law frequency dependence of the impedance across the interface. The frequency exponent is shown to be not simply related to the fractal dimension of the interface.

324 Noise Analysis Applied to Electrochemical Systems:

In this paper we review theoretical and experimental evidence that the fractal nature of the electrode-electrolyte interface is the source of the constant phase element seen in the impedance spectra of electrolytic cells. The fractal geometry gives rise to a complex distribution of parallel current paths, and the competition between these paths results in the fractional power law frequency dependence of the impedance across the interface. The frequency exponent is shown to be not simply related to the fractal dimension of the interface. The technique is sensitive to the texture of the surface and to changes in the in-plane arrangement of surface structure constituting film roughness.

326 Electrochemical Fractal Dimension Measurement on

Based on the analysis of the time dependence of the diffusional flux to a surface, an electrochemical fractal model is proposed to describe the relationship between mass transfer and the probability of random fluctuations of the electrical quantities (electrode potential and cell current) in electrochemical systems, commonly referred to as electrochemical noise. The paper describes the main sources of noise, the experimental methods used for its measurement, and some of the recent progress in its analysis, with particular emphasis to corrosion applications.

327 In Situ Infrared Spectroscopy of Corrosion Processes at

Spectroscopy is an extremely powerful technique for the measurement of the fractal dimension of fractured steel surfaces.

328 Coulometric Reduction of Thin Tarnish Films Formed on

Copper: B. J. Richard and J. H. Payer, Dept. of Materials Science and Engineering, Case Western Reserve University, Cleveland, OH 44106

This paper presents the results of thin film analysis for Cu-O-S compounds on a copper substrate.
329 Impedance Characterization of Polypropyrole: P. G. Pickup, G. L. Duffitt, and X. Ren, Dept. of Chemistry, Memorial University of Newfoundland, St. John’s, Newfoundland, Canada A1B 3X7.

330 Electrochemical Observation of Intermediates during the Formation of Conducting Polypropylene: D. J. Harrison and D. E. Raymond, Dept. of Chemistry, University of Alberta, Edmonton, Alta, Canada T6G 2G2

331 Electrochemical Quartz Crystal Microbalance Studies of Electroactive Polymer Bilayers: A. R. Hillman and A. Gild, School of Chemistry, Bristol University, Bristol, England BS8 1TS.


334 Electrode Surface Modification with ZnO and ZnO-CdS Semiconductor Colloidal Particles: P. V. Kamat, Radiation Laboratory, University of Notre Dame, Notre Dame, IN 46556.


The investigation of crystallographic orientation and electron work function distribution of W crystal faceted coating deposited by special CVD technology on TEC cylindrical emitter was carried out by HRTCS, RFTM, and AES methods. It was found that faceted plane orientation is close to (110) direction. As a result, work function distribution is smooth and average work function is close to highest possible value for W(110) α = 5.35 eV. The investigation of the coating after 6460 h TEC test demonstrates its high stability.


Samples containing 14 m/o GdO2 and 86 m/o Bi2O3 were annealed at many temperatures for several hundred hours. During thermal anneal, ionic conductivity exhibited four regimes which were interepreted in terms of the volume fractions of the cubic and rhombohedral phases. Kinetics of phase transformation were investigated using conductivity measurements, X-ray diffraction, and optical microscopy.


Two alloys for electrodes for the alkali metal thermal to electric converter (AMTEC), platinum-tungsten and rhodium- tungsten, are discussed. Both alloys have been operated as electrodes in AMTEC cells or under simulated AMTEC conditions for several hundred hours, and lifetime of electrodes is expected to be tens of thousands of hours. Compositions of PW ranging from Pt2W to Pt6W have been tested to find the optimum Pt2W ratio. Sodium transport through the electrode and its effect on electrode performance is discussed.


In this study, iridium-added tungsten powder mixtures were cold-compacted and sintered to produce a range of tungsten, iridium electrodes. An electron emission study was subsequently carried out in order to evaluate the work function of the consolidated alloys. The work function was obtained by the thermionic method and was found to be temperature and composition dependent. SEM and EPMA were used to check the microstructure and composition near the surface of each tested alloy. SIMS (elemental images) and TEM of the surface regions were also carried out.


The effective work function of tungsten-iridium alloys was studied. The work function of W-1%Ir and W-3%Ir decreased substantially with increasing temperature, whereas, W-7%Ir showed a slight increase at low temperature but a high-est observed work function (5.11 eV) was obtained at low temperature (1900-2100 K) from the W-3%Ir sample. The decrease in the work function of W-1%Ir and W-3%Ir with increasing temperature was attributed to depletion of surface iridium. The work functions obtained here by alloying small quantities of iridium in tungsten are among the highest of any conventional materials.

SYNTHESIS AND PROCESSING FOR HIGH TEMPERATURE MATERIALS FOR THE YEAR 2000

High Temperature Materials/Dielectric Science and Technology

341 Materials Research in the U.S.: The Development of a National Agenda: L. H. Schaufuss, Materials Science and Engineering Laboratory, National Institute of Standards and Technolo-gy, Gaithersburg, MD 20899.

The comprehensive study on materials science and engineering (MS&E) published in 1989 by the National Research Council (NRC) set the stage for a National Agenda. What is the Federal response and how is it being orchestrated? This charge resides with
the Office of Science and Technology Policy (OSTP). Through its components for the future, OSTP identified key issues including an inventory of government activities and recommending a Feder- al strategy and a R&D plan responsive to national needs. The plan, still in its evolutionary stage, focuses on advanced materials and materials processing and an increased interaction between government, universities, and industry.

342 Expanding Horizons for Chemical Vapor Deposition Synthesis: K. E. Speir, Materials Science Department, The Pennsylvania State University, University Park, PA 16802

Extrapolations of trends in CVD history show that chemical vapor deposition (CVD) will play an expanded and crucial role as a synthesis technique in the near future. New areas continue to be developed for utilizing CVD techniques for high temperature materials. Increasing importance of high quality, reproducible coatings are driving research on the fundamentals of deposition processes. Thus, theoretical modeling of CVD systems will also become quite sophisticated in the next century.

343 Chemical Vapor Deposition Process Aspects for the year 2000: J. O. Carlson, Dept. of Chemistry, Upsala University, S-751 21 Upsala, Sweden

Chemical vapor deposition (CVD) plays an important role in science and technology today. There is an increased demand of tailor-made, well-defined, and high-purity materials. CVD, with its ability to produce films of uniform thickness and properties on complicated shaped substrates, will play a key role in the development of the next generation of thin film materials. For the year 2000, work related to both research and development has to focus on some important areas. Precursor design, a deeper knowledge of surface processes (including nucleation) and postprocessing, and sintering and selectivity of growth on unpat- terned and patterned substrates are examples of such areas.

344 Computational Analysis of Fluid Flow and Chemical Kinetics in Chemical Vapor Deposition: R. J. Kee, G. H. Evans, and M. E. Coltrin, Sandia National Laboratories, Albu- quantum, NM 87185

Chemical vapor deposition (CVD) processes involve the complex interaction of fluid-mechanical transport, molecular diffusive transport, gas-Phase chemical reaction, and heterogeneous chemical reaction at the deposition surface. Computational simulations play an important role in developing an understanding that facilitates experimental design and development. This paper uses specific examples to illustrate the application of mod- elling to practical reactor design and operation. The paper also dis- cusses a new and general software capability that facilitates coupling of gas-phase chemistry and transport with heterogeneous chemistry at gas-surface interfaces.


The methodologies of processing (i.e., forming a solid piece of material) and fabrication (i.e., achieving the size, shape, and dimensional accuracy of components) are reviewed. These include the spray deposition processes and fabrication, polymer-based processes, and chemical vapor deposition, and coupling this with the introduction of the dispersed phase, including fiber fabrication. The needs and opportunities of these are assessed based upon the properties, sizes, shapes, and costs achievable relative to expected application needs.

346 Chemical Vapor Infiltration: T. M. Bezman, D. P. Stinson, and R. A. Lowden, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Continuous filament ceramic composites are enabling new, high-temperature structural applications. Chemical vapor infiltration methods for producing these composites are being studied, with the complexity of filament weaves and deposition chemistry merging with standard heat and mass transport relationships. Silica-carbide composites are, by far, the most advanced, and are already being used in aerospace applications. This paper ad- dress is the state-of-the-art of the technology and outlines current issues.

347 High-Tech Fibers: G. G. Tibbets, Physics Dept., General Electric Research Laboratories, Warren, MI 48090-0955

Modern, high-tech fibers offer the materials engineer a broad choice of properties for ceramic composites. Fibers may be vari- ously produced by spinning a precursor polymer into a thread for melt spinning, whisker growth in the gas phase, vapor deposition on a pre-existing fiber, or sol-gel processing. Properties of some fibers appear to favor ceramic reinforcement and some of the complications of engineering a fiber-matrix system are aired.

348 Processing of Advanced Ceramic Materials: Need for Short-Range Interparticle Potentials: F. F. Lange, Materials Science and Technology Department, University of California, Santa Barbara, Santa Barbara, CA 93106

Interparticle potentials play a dominant role in governing the slurry viscosity, maximum particle packing density, and the rheology of the consolidated body. These rules are reviewed with the objective of understanding how damage-free bodies can be consoli- dation from slurries to increase the overall stability of ceramics and their composites. Recent results have shown that short-range (<4 nm) repulsive potentials that produce an attractive, but noncontacting, particle network formation are most desirable for developing new shaping methods for advanced ceramic sys- tems.

349 Synthesis and Processing of Nanostructured Materials: H. Hahn, Dept. of Materials Science and Engineering, Rutgers University, Piscataway, NJ 08855

Nanostructured materials are synthetic metastable materials with ultrafine microstructures, typical less than 100 nm. It has been demonstrated that these materials have interesting properties such as sintering and superplastic deformation at low tempera- tures in nanostructured ceramics and high hardness, ductility and fracture strength in nanostructured ceramic-metal composites. Experiments are that the novel properties include novel technol- ogical applications. A wide variety of techniques such as MBE, CVD, spray conversion, hydrolysis, laser pyrolysis, mechanical attri- tion, sol-gel and gas condensation has been used to synthesize nanostructured materials. The paper presents the various tech- niques and discusses their advantages and limitations for basic re- search and potential technological applications.

350 Microwave Processing of Ceramics: Promises and Challenges: D. L. Johnson, Dept. of Materials Science and Engineering, Northwestern University, Evanston, IL 60208-3907

The hope of high speed processing, uniform heating, energy ef- ficiency, and precise control of heat application has spurred a re- cent flurry of activity in attempts to improve control over the processing of ceramics. Significant reductions in sintering temperature have been reported. However, difficulties with micro- wave heating arise from the nature of the interaction of micro- wave energy with materials. Low loss materials are difficult to heat, and heating, once begun, can be difficult to control.

351 Microwave Surface Modification and Sintering: D. E. Clark, Z. Fathi, and D. Folz, Dept. of Materials Science and Engineering, University of Florida, Gainesville, FL 32611

We have processed technologically important ceramics and glasses in both microwave and conventional ovens. Comparison of micro- structures, hardness, and toughness of aluminas samples sintered by the two methods indicate that microwave processing offers distinct advantages, more uniformity is obtained in the micro- wave processed samples. Surface modification of glasses and ceramics have been achieved using microwave energy. Sods-alu- mina-silicate glasses have been treated in a molten KNO3 solution. A greater degree of control over the surface chemistry and depth of surface modification can be achieved in the microwave oven. A K penetration depth of 100 μm requires only 30 min as opposed to many hours using the conventional method.

352 Mathematical Modeling Issues in Plasma Synthesis: J. Szekely, Massachusetts Institute of Technology, Cambridge, MA 02139

Plasmas systems may provide an ideal vehicle for the synthesis of fine powders in fundamental research and the fabrication of materials and devices. There are a number of potential advantages of plasmas for the controlled exposure times. A critical issue in the operation of these systems is to control the transport phenomena, such as the intermixing of the reactants, and the formation of plasma. The objectives of this paper are to assess some of the critical issues involved in the design and operation of plasma systems. Through its objective of understanding how damage-free bodies can be consol- dation from slurries to increase the overall stability of ceramics and their composites. Recent results have shown that short-range (<4 nm) repulsive potentials that produce an attractive, but noncontacting, particle network formation are most desirable for developing new shaping methods for advanced ceramic sys- tems.

353 Thermal Plasma Synthesis of Fine Powders: E. Pfender, Dept. of Mechanical Engineering, University of Minnesota, Minneapolis, MN 55450 and P. Kong, INEL, EG&G Inc. INC, Idaho Falls, ID 83415

Plasma systems may provide an ideal vehicle for the synthesis of fine particles of advanced materials and devices. There are a number of potential advantages of plasmas for the fabrication of materials and devices. There are a number of potential advantages of plasmas for the controlled exposure times. A critical issue in the operation of these systems is to control the transport phenomena, such as the intermixing of the reactants, and the formation of plasma. The objectives of this paper are to assess some of the critical issues involved in the design and operation of plasma systems.

HIGH TEMPERATURE SENSORS

High Temperature Materials/Sensor/Battery


Oxygen sensors based on ZrO₂ electrochemical cells have found extensive use in many applications. The most sensitive of these sensors employ the oxygen pumping principle with ZrO₂ cells. Oxygen pumping, however, can also be used to generate other chemical and also physical sensors. This paper discusses several of these sensors for a variety of CO, H₂, H₂O, hydrocarbons, gas flow, and gas pressure, and presents results of studies of the properties of some of these devices.


Steady-state and ac impedance measurements were made on the electrode reaction at the H₂O/H₂O porous Pt/YSZ interface as functions of H₂ and H₂O partial pressures and temperature. The reaction rate was compared with that under CO- and O₂ gas and the possible reaction phase boundary of gas/Pt/YSZ are discussed. It is shown that the reaction rate in the H₂O/H₂O gas is about 10⁻² times larger than that in CO- and O₂ gas.


Potentialistic gas sensors, in which identical electrodes are brought into equilibrium with a major constituent of separated test gases and atmospheres, have been known since the measuring work of Nernst at the turn of the century. In recent years an alternative configuration for potentiometric sensors, in which there is no separate atmosphere, has been explored. In this instance the measured potential of the device arises from the distinct interactions of a minority component of the atmosphere with the membrane which comprises the electrodes. The range of applications of this configuration, both at ambient and at elevated temperatures, is reviewed.


A new method is suggested for determining chemical potentials by potentiometric measurements. It allows the use of mixed ionic electronic conductors, MIECs, instead of solid electrolytes, SEs. The galvanic cell includes the sample, a reference electrode, two MIECs, a power supply, and two voltmeters. The procedure of measurement is different from the usual potentiometric one. The possibility to replace SEs by MIECs should extend the range of application of the potentiometric method.


The formation of oxygen partial pressure from the solid electrolyte oxygen sensors was affected by the electrode materials. The sensor with perovskite-type oxide electrode exhibited theoretical electromotive force at lower temperature than those with Pt electrodes. In particular, the sensor with La₃Sr₂Ca₂O₇ (LSCM) attained theoretical electromotive force even at 200°C. The electrode materials, in general, at low temperatures tend to have large electrode conductivity and oxygen isotopic exchange reaction rate.

360 Novel Solid Electrolyte CO₂ Sensor Using Sodium Ion Conductor and Lithium-Based Carbonate Electrode: N. Yamazoe, S. Yao, Y. Shizu, and N. Miura, Dept. of Material Science and Technology, Kyushu University, 6-1 Kasugakoen Fukuoka-shi, Fukuoka 816, Japan.

An electrochemical cell combining an Na⁺ conductor and an Li⁺-based binary carbonate auxiliary electrode (Li₂CO₃-MCO₃, M = Ca, Ba) showed excellent performance in CO₂ sensing. Its response to CO₂ followed a Nernst equation over a broad dynamic range of 10⁻⁴ to 10⁻¹ ppm CO₂, even under humid conditions. Nernst's slope coincided with a 2-electron reaction of CO₂. On the other hand, the responses were almost independent of PO₂ at high-temperature (above 70°C) and below 350°C and were almost independent of Pr responses depended on PO₂ in coincidence with a 2-electron reaction of O₂.


The industrial applications of solid-state electrochemical sensors for measurement of elements in molten metals have mainly been confined to those species which are ionically transferred in the solid electrolyte. It has been possible to extend the range of elements by the use of auxiliary phases such that there is a chemical coupling between the element ionically mobile in the electrolyte. Devices for sensing phosphorus, silicon, and sulfur have been developed and their applications in metal refining are presented.

362 Miniaturization of a High Temperature Oxygen Sensor Employing Film Electrolyte and Electrode: F. W. Montague, M. A. Styczynski, and C. C. Liu, Electronics Design Center, Case Western Reserve University, Cleveland, OH 44106.

High degree of miniaturization in actively operated oxygen sensors may be achieved with a substitution of thin films for all of the sensor's components. A film sensor has been developed using an ion beam deposited ceramic electrolyte and platinum electrodes on a silicon substrate coated with TiO₂. Thin film electrolyte of sufficient conductivity has been formed from a zirconia source in an oxygen-containing atmosphere. Continuous operation of up to 2 weeks at 1200°C is possible.


A limiting current type plane oxygen sensor with a unique structure has been developed. This sensor has cathode and anode of Pt on the same plane of ZrO₂ electrolyte, and the cathode also serves as a hole to limit gas diffusion. It detects humidity linearly proportional to a water vapor pressure of 0.1-10⁻¹ atm in a wide operation temperature range from 20 to 300°C. No deterioration was observed after 10,000 h life test.


High-temperature thin film sensors are currently being investigated for temperature and strain measurements on gas turbine engine components. The electrical properties of the native insulating layer formed on the surfaces of these components is critical to device performance. The relationship between the microstructure and electrical properties of the native oxide layers was investigated.

The formation of a high quality dielectric was achieved by minimizing the degree of internal oxidation within the bond coat layer and maximizing external oxide scale growth.


A new polymer electrolyte with three orders of magnitude higher sodium ion conductivity at room temperature, than the hitherto reported polymer electrolytes is presented here. The electrolyte comprises of poly(propylene glycol), (PPG), poly(ethylene oxide), (PEO), and NaCl. The films were obtained by conventional solvent casting of a PPG-PEO blend that was found to be completely amorphous. This has been confirmed by XRD studies and supported by DSC. The blend was found to be a sodium ion conductor. The effect of varying sodium ion concentration (different O/Na ratios) on ionic conductivity in the blends was studied. An optimized concentration of PPG and O/Na ratio gave rise to 10⁻³ mol cm⁻¹ at room temperature. This blend is completely amorphous. Studies on ionic conductivity varying the partial pressure of ammonia show that conductivity has a logarithmic relationship with partial pressure of ammonia. The effect of compositional changes and various temperature on change in ionic conductivity at various partial pressures of amonia were studied. The results suggest the applicability of polymer electrolyte as solid-state ammonia sensor.

ELECTROCHEMISTRY IN MINERAL AND METAL PROCESSING III


Many sulfide minerals can be floated without collector in controlled laboratory or chemical-electrochemical environments. Whether collectorless sulfide mineral flotation is commercially feasible and under what conditions are critically examined. Since moderate flotation with high froth temperatures of 180°C and below 200°C conditions are considered. The use of collectorless flotation of chalcopyrite is relatively simple, a favorable example for collectorless flotation should be chalcopyrite ores containing hydrometallurgical byproducts. Actually consider the ore geology and to control water quality and redox environments during grinding, conditioning, and flotation make commercial-scale collectorless flotation difficult, if not impossible.

367 Surface Electron Structures of Galena and Pyrite Related to Collectorless Flotation: S. Sun, D. Wang, and B. L. Dept. of Mineral Engineering, Central South University of Technology, Changsha, Hunan, 410083, China.
The electron transfer level of galena surface is higher, which decrease the adsorption of xanthate. The platinum electrode potential of galena and depresses the collectorless flotation of galena pulp. On the other hand, pyrite surface has a lower electron transfer level. It electrochemically catalyzes the oxidation of HS- ion to neutral sulfur. The presence of Na2S improves the collectorless flotation of pyrite. The collectorless flotation separation of galena from pyrite is possible using Na2S as modifier.

368 Electrochemical Aspects of Cast Iron Grinding Media Wear and Its Effect on Flotation: I. Iwasski* (Present address: Central Research Institute, Mitsubishi Materials Corp., 1-297 Kitaburocho, Omiya, Saitama 330, Japan) and V. V. Lakshmanan, Dept. of Civil and Mineral Engineering, University of Minnesota, Minneapolis 55455.

The electrochemical and wear behavior of a series of chromium-containing cast iron balls with Cr contents ranging from 0 to 25% was related to their abrasion and static conditions. The passivation behavior of ball materials was in direct correlation with the floatabilities of pyrrhotite and pentlandite under nitrogen or oxygen atmosphere.


A cathodic decomposition model for chalcopyrite in a neutral pH solution, simulating the conditions in wet grinding mills, is proposed through open-circuit potential and combination partial measurements. Linear potential sweep technique, chronoamperometry and chronocoulometry, and surface analysis methods. Chalcopyrite grinding media undergoes a reduction reaction and the mineral surface becomes covered with a layer consisting of Fe(OH)2 and CuS. The Fe(OH)2 layer adheres to the lithocryte. The role that oxygen plays in the collectorless flotation of cathodically polarized chalcopyrite is discussed.


The adsorption mechanism of thionocarbamate interaction in flotation systems has been investigated. Voltammetry suggests that the adsorption of thionocarbamate is probably a result of a coupled reaction of the EC-type, involving an initial electron transfer step (E) followed by a chemical reaction (C). Thus, the adsorption is controlled by both the E and pK of the system. In situ, FTIR spectroscopy and contact angle measurements support the mechanism suggested for thionocarbamate interaction.


The effect of pulp redox potentials and pH on the flotation response of sulfides with dialkyl dithiophosphate was investigated for several minerals. The presence of various modifiers, such as DO, pH, and NaCl, was found to be strongly dependent on pulp potential. Galena and chalcopyrite flotation were optimum in the range of 0.0 to +0.67 V (Ag/AgCl ref.) and pH 8. The optimum redox and sulfate potentials for flotation of galena were found to be slightly different potential ranges, for example, for iron sulfides.

372 Mechanism of Sulfide Depression with Functionalized Synthetic Polymeric Xanthates: D. R. Nagyaj, American Cyanamid Co., Stamford, CT 06904.

Polymeric depressants can offer several advantages over the commonly used depressants in sulfide mineral flotation. Their use is, however, limited to polyacrylamides for a few specific applications. Functionalized, synthetic polymeric xanthates can provide the advantages of polymeric depressants such as xanthates of polyacrylamides, which are modified by incorporating a chemical group that can interact with the sulfide mineral surface. In this study, the interaction of ethyl xanthate with silver and gold/gold alloys has been investigated by voltammetry, FTIR spectroscopy, UV/Vis spectroscopy, and measurements of contact angle. Chemisorption of xanthate occurs on the silver and alloy surfaces prior to silver xanthate formation and renders the surfaces hydrophobic. Coverage of chemisorbed xanthate was found to be a Frumkin isotherm. pH-pH diagrams have been constructed that include chemisorption in addition to silver xanthate and dixanthogen formation.

374 Interpretation of Electrode Responses with the Help of a Multivariate Technique: B. I. Palsson and E. Öberg, Div. of Mineral Processing, Luleå University of Technology, S-951 87 Luleå, Sweden.

Simultaneous laboratory measurements in flotation pulp with different electrodes under conditions of varying pulp chemistry and hydrodynamics. The effects of collector concentration, pH on the response of the electrode, and the redox potential with the addition of a stable carbon electrode are summarized. The effects of redox potential on the responses of mineral electrodes of pyrite, galena, and to some extent chalcopyrite.


Voltammetry has been used to determine the products of interaction of sulfide minerals with iron(II), cyanide, sulfide, and xanthate ions in flotation pulp. A multivariate analysis was shown to form a strong peak in pyrite and chalcopyrite when present in a flotation pulp containing cyanide and sulfide ions, and a weak peak on the response of these minerals of no more than 10%. The presence of a collector is necessary for efficient flotation in this system.


The pulp chemistry of pyrite pre- and reverse flotation circuit was studied for a coal pre-concentration and concentrates separation. The effect of gas composition (O2, N2, and air), lime, SO2, and temperature on the pulp chemistry was determined in pyrite pre- and reverse-flotation in a Zn flotation circuit. The interaction between pH, DO, and Ep is examined. It is shown that pulp potential is correlated with metalurgical performance.

377 The Interaction of Ethyl Xanthate with Pyrite: J. Alan* and D. Forstrasser, American Cyanamid Co., Stamford, CT 06904.

The interaction of ethyl xanthate with pyrite is shown to be controlled by redox processes and pyrite surface chemistry. In the latter case the distribution of positive and negative surface sites permits both a kinetic description of the decomposition of ethyl xanthate as a function of pH to be obtained, as well as a quantitative explanation of the pH dependence of pyrite flotation. This behavior has hitherto eluded an overall quantitative interpretation.

378 The Effect of EDTA on Collectorless Flotation of Pyrite: S. Chander and J. Pang, Mineral Processing Section, Pennsylvania State University, University Park, PA 16802.

It is generally recognized that flotation of pyrite can be modulated by adding chelating agents, but the mechanism is not fully understood. To determine the flotation mechanism by several electrochemical and wetting studies were made in the pyrite-ethylene diamine tetra acetic acid (EDTA) system. The results show that EDTA interacts with pyrite surface through several steps involving dissolution, complexation, and adsorption. The flotation response depends on the extent of these reactions which is a function of the reagent concentration and the rate and severity of pyrite oxidation.


Two novel effective methods were developed for selective separation of pyrite from arsenopyrite or molybdenite, which are activated with Cu(I) ions. The surface properties of pyrite and arsenopyrite in aqueous solutions with DO and pH 5 were determined. The first method involves selective flotation of new American Cyanamid reagents when the flotation pulp containing pyrite and arsenopyrite were activated with Cu(I) ions at pH 11. The second method, such as in earth metals in heavy metals, the solubility of pyrite and arsenopyrite was complexed at pH above 8.5, while the floatability of pyrite remains unaffected. The principle involved in the process was studied using electrochemical techniques and ESCA surface analysis.

380 Surface Layer Structure of Sulfide Mineral Treated in Thiol Collector Solutions: Scott E. Swanson* and K. Laajalehto, University of Turku, Laboratory of Materials Science, Itäinen Pitkäkatu 1, SF-20520 Turku, Finland, S. Hemalai, Outokumpu Research Oy, SF-20810 Porvoo, Finland.
The oxidation of in situ fractured natural mineral pyrite electrodes has been studied using electrochemical and photoelectrochemical techniques. The photoelectrochemical measurements of freshly fractured pyrite under steady illumination. However, with chopped illumination, anodic PC is observed over the range of approximate 1.9 to 2.0 V vs. SCE at pH 8.2. By employing a method designed to preserve volatile components of the surface adsorption layer prepared in aqueous solution, existence of ethyl dixanthogen has been concluded from XPS measurements of pyrite surfaces treated in potassium ethyl xanthate solution within a certain range of combinations of pH and E. Polarization measurements of similar samples are referred to the agreement with the XPS results. In situ XPS tests indicate increase of surface hydrophobicity due to presence of dixanthogen in the adsorption layer.

381 The Interaction of Diethyl Dithiophosphate with Freshly Exposed Galena and Chalcopyrite Surfaces: A. N. Buckley, CSIRO Div. of Coal and Energy Technology, North Ryde, NSW 2113, Australia

Interaction of diethyl dithiophosphate (DTP) with galena at pH 5 and chalcopyrite at pH 9 has been investigated by electrochemical and electron spectroscopic techniques. Adsorption of DTP on galena was very slow. The S(2p) binding energy of a monolayer was less than that expected for DTPD. Adsorption was rapid on chalcopyrite, with chemisorption preceding multilayer CdTeD formation. The Cu(LMM) Auger spectrum when only the monolayer was present was similar to that for multilayer and different from that for multilayers.

382 Electrochemical AC Impedance and X-ray Photoelectron Spectroscopic Studies of Interaction of Phenyl Thiourea with Coal Pyrite Surface: S. U. M. Khan and T. J. Farley, Dept. of Chemistry, Dalhousie University, Halifax, NS, Canada B3H 4J8

Phenyl thiourea was found to behave as a better floating agent compared to ethyl xanthate for coal pyrite and the percentage of flotation recovery was found potential dependent. AC impedance data also indicate potential adsorption of thiourea. XPS results of N 1s and S 2p spectra clearly indicate adsorption of phenyl thiourea on the coal pyrite surface and increased adhesion under positive control. These results further confirm that the chemisorption of PTU renders the pyrite surface highly hydrophobic under applied potential to attain the highest degree of pyrite floatability.


The adsorption of butyl xanthate on the surface of galena (powder of flotation size 45-60 μm) and chemical nature of species forming was studied. Maximal xanthate adsorption was found under pH = 10.5.


The oxidation of chalcopyrite (CuFeS2), the principal mineral source for copper, has been studied in aqueous borate electrolyte (pH 9.2) using photocurrent voltagmetry and spectroscopy. In the potential range -0.2 to +0.4 V vs. SCE, FeO4(2-) / Fe3+ + OH- was formed, producing photo-anodic currents, while leaving the copper and sulfur as CuFeS2/2CuS2 in the chalcopyrite lattice. At +0.4 V vs. SCE, the iron-depleted film decomposed, greatly enhancing the chalcopyrite decomposition rate and forming CuO/Cu(OH)2, which on reduction during subsequent negative-going potential sweep produced photo-cathodic currents.


The mechanism and products of the oxidation of chalcopyrite (CuFeS2), the principal mineral source for copper, have been studied in alkaline electrolytes (pH 9.2 and 12.7) using cyclic and potential step voltammetry, with subsequent surface analysis by x-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES). At pH 9.2 and potentials <0.4 V vs. SCE, FeO4(2-) / Fe3+ + OH- was formed, leaving the copper and sulfur as CuS2 in the chalcopyrite lattice, which remained in tact. No evidence was found for CuFeO4. At +0.4 V vs. SCE, the CuS2 film decomposed, forming CuO/Cu(OH)2 and greatly enhancing the underlying chalcopyrite decomposition rate.

386 The Photoelectrochemistry of In Situ Fractured Pyrite Electrodes: D. J. Farley, Dept. of Mining and Minerals Engineering, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061-0258

The photoelectrochemistry of pyrite (FeS2) electrolyte interactions have been studied using both natural and synthetic pyrite samples and electrochemical, photoelectrochemical, and surface analysis techniques. Charge transfer studies across CuTe junctions were conducted in the presence of 1: Br-, Fe(CN)6-3, and Cl- in an aqueous solvent, and in the presence of ferrocene and TMPD in a nonaqueous solvent system. The electrochemical behavior of pyrite is compared and contrasted with that of noble metals and other semiconductor photoreactors. Finally, oxidation of pyrite is discussed in the light of these results.

387 Electrochemical Studies of Iron Sulfdies in Relation to Their Atmospheric Oxidation and Prevention of Acid Drainage: Part II: S. M. Ahmed and E. Giezieuw, Canada Center for Mineral and Energy Technology, Mineral Sciences Laboratories, Ottawa, Ont., Canada K1A 0R6

Oxidation reaction of pyrite (FeS2) and pyrrhotite (FeS) and related redox reactions have been studied using RDE and RDE. The transfer of Fe(II) in the oxidation of pyrite (FeS2) and pyrrhotite (FeS) has been examined in light of the energy level diagrams and the semiconducting nature of FeS. The problem of the “acid mine drainage” resulting from the atmospheric oxidation of iron sulfides has also been examined. Methods of preventing the AMD by cathodic protection and by growing passive, iron oxide films on FeS2 and FeS (on electrolyte only) have been developed.

388 Transpassive Oxidation of Pyrite: Y.-H. Wang, R. Woods, N. A. Butler-Smith, and J. W. Leonerd, Dept. of Metallurgical Engineering, University of Utah, Salt Lake City, UT 84112

The electrochemical behavior of mineral and coal pyrites in basic solutions was investigated using cyclic voltammetry. The oxidation of pyrite has been examined in light of the energy level diagrams and the semiconducting nature of FeS. The problem of the “acid mine drainage” resulting from the atmospheric oxidation of iron sulfides has also been examined. Methods of preventing the AMD by cathodic protection and by growing passive, iron oxide films on FeS2 and FeS (on electrolyte only) have been developed.

389 Electrochemical Kinetics of Silver Dissolution in Cyanide Solutions: J. Li and M. F. Wadsworth, Dept. of Metallurgical Engineering, University of Utah, Salt Lake City, UT 84112

The electrochemical reactivity and surface properties of pyrite from coal and ore sources were investigated. Theoretical and experimental studies indicate that an oxidation layer is rapidly formed on the pyrite surfaces even when the electrode is polished under nitrogen and with deoxygenated water. The initial process of pyrite metal oxidation is controlled by the adsorption and discharge of hydroxide ions/water molecules, independent of the solution pH, source and semiconducting properties. The results obtained from film/micro flotation and contact angle titration studies show that pyrites from different sources exhibit similar surface hydrophobicity, though the thickness of the surface oxidation layers may be different. The results are compared with surface studies by SEM to elucidate the pyrite surface oxidation mechanisms.

390 Thermodynamic Equilibrium Calculations on Au/Ag-Lixiviable Oxidation Products: X.-H. Wang, H. Yoon and J. L. Miller, Center for Geochemical Processes, Oak Ridge National Laboratory, Oak Ridge, TN 37831

The electrochemical kinetics of anodic dissolution of silver in cyanide solutions was measured using an internal potential-controlled double-layer capacitor. The results indicate that the anodic dissolution of silver takes place in the presence of cyanide ions and oxygen. The activation energy for the change in the polarization resistance for cyanide reduction was determined. The model derived for the anodic dissolution of silver was in accord with results obtained from separate impedance measurements.
Thermodynamic equilibrium calculations were performed on Au-Ag-Lixiviant systems to study the feasibility of extracting gold from complex ores using nonaqueous lixiviants. A number of non-oxidative leaching agents, including chloride, bromide, iodide, thiourea, ammonia, thiosulfate, and their combinations were compared with cyanide. The results demonstrated that all these lixiviants can dissolve gold and form gold-lixiviant complexes. However, the halo-genides needed much higher oxidation potential and acidic pH to dissolve gold than cyanide did. Gold and silver dissolve in thioulate and ammonia, as well as thiosulfate-ammonia solutions, under conditions compared with cyanide, indicating these two chemicals can be effective lixiviants for gold extraction. The electrochemical studies with gold electrolysis in the above lixiviant solutions confirmed the conclusions drawn from the thermodynamic calculations.

393 Gold Leaching and Recovery: The Bromide Process: A. Dadgar* and J. Hoearth, Great Lakes Chemical Corp., West Lafayette, IN 47906
Gold extraction and recovery for gold ores and concentrates were investigated using cyanide and bromine reagents. Gold extractions for cyanide leaching (24-48 h) and bromine leaching (4-6 h) were the same and ranged from 92 to 98%. Gold recoveries from bromine pregnant solutions using carbon adsorption, ion exchange, solvent extraction, zinc precipitation, and electrowinning were better than 98%. Commercially available undivided cells were used to generate bromine from simulated solutions. Electrolyses were performed with solutions containing different levels of bromine. It was demonstrated that efficiencies were high (98%) and unaffected by the bromide concentrations (over the range of 2.5-5.0%). The electrochemical module was capable of delivering 0.4 kg of gold per hour at a cost of 8.5c/kg. Using the above data, a detailed economic assessment for a 1000 mtpd gold ore processing plant was made. Electrolytic regeneration of bromine in a gold recovery circuit dramatically improves the process economics over cyanidation.

394 Complex Technology of Electrochemical Water Treatment with Regeneration of Valuable Components in Electroplating Production: V. A. Kolesnikov, E. A. Shulist, and P. K. Arriinskii, Mendeleyev Institute of Chemical Technology, Moscow 1253190, USSR
The complex of techniques developed in the MChTI includes electrolysis, electrochemical correction of pH, electrofiltration, electrodialysis, and provides removal of impurities down to the residual concentration of 0.01 mg/l when initial concentration is not limited. Necessary recovery is about 10 min for each technique. On the basis of experimental study and computer simulation, the authors optimized apparatus design and the operation mode of techniques. The most technical ideas have been patented. Commercial modules having an output of 1-20 m³/h operate successfully at 5 factors, and more modules are under installation. The economic benefit of one module is 100-400 thousand of roubles yearly.

395 A Comparative EIS Study on Cermet and Platinum Anodes for the Electrolytic Production of Aluminum: C. F. Windisch, Jr., Dept. of Chemistry, University of Newcaste, Newcastle upon Tyne, England NE1 7RU
Electrochemical impedance spectra were obtained for NiO-NiFe₂O₃-Cu cermet anodes and platinum anodes during the electrolysis of molten cryolite at 983°C. When anodes were polarized above the decomposition potential for alumina, an impedance loop was obtained with a characteristic frequency of about 1 Hz. Analysis of the data suggests the loss due to oxygen gas bubbles produced at the anodes. Features associated with charge-transfer processes were not sufficiently resolved to determine the corrosion properties of the cermet anode.

396 Solid Metal Reductive Stripping of Cerium from Tri-n-Butyl Phosphate.
Based on Student Concentration: T. J. O'Keefe, Jr., and D. D. Dulan III, Graduate Center for Materials Research, University of Missouri-Rolla, Rolla, MO 65401
Solvoly extraction is used extensively in the processing of the rare earth metals. Recently, it has been shown that redox reactions are feasible in many organic extractant/inert diluent systems utilizing redox metal catalysts. In the presented study, it was demonstrated that cerium could be effectively reduced to Ce(III) in a tri-n-butyl phosphate (TBP) solvent. In the reduced state, the Ce was found to be much easier to strip into the aqueous phase. Whereas Ce(IV) is difficult to strip even with concentrated (4N) acids, the Ce(III) was readily removed using water.

397 The Electrochemical Behavior of Group V Elements during Copper Deposition: J. B. Huxley, Dept. of Materials Science and Engineering, University of Arizona, Tucson, AZ 85721; Y. Maeda, Nippon Mining Co. Ltd., Japan
The electrochemical behavior of copper deposition in the presence of Group V elements, antimony, and bismuth has been investigated using several nonsteady-state techniques. Electrode deposition was carried out in cupric sulfate electrolytes containing 40g/L CuSO₄ and 200g/L H₂SO₄. Group V elements behave as depolarizers towards copper deposition and hydrogen evolution. Linear sweep voltammetry for the As-containing electrolyte is characterized by two reduction peaks. The first peak corresponding to copper deposition and the second to arsenic reduction at approximately -0.33 V. Cyclic voltammetry data were collected to obtain detailed information about the effect of Group V impurities on the mechanism of copper deposition.

398 Mass Transport in High-Current-Density Zinc Electrowinning: K. J. Cadbro, Div. of Mineral Products, CSIRO, Port Melbourne, Victoria 2507, Australia
Limiting mass transfer coefficients have been measured for zinc during zinc deposition covering the ranges 50-1000 A/cm². Throughput, 25-1000 A/cm². Current density, 25-400°C; cell temperature, 25-50°C; and cell temperature, 25-50°C. The transfer coefficients were consistently much greater than those calculated from published correlation equations, probably due to convective mass transfer to zero-hydrogen conditions gave coefficient values in substantial agreement with those calculated from correlations.

399 Full-Scale Hydrogen Diffusion Anodes for Immersed Tank Electrolysis of Zinc: J. D. Winfield, F. E. James, and R. V. Vore, E-TEK, Inc., Framingham Industrial Park, Framingham, MA 01701
Immersed 1.2 m² hydrogen diffusion anodes have been tested in zinc electrowinning. At 500 A/m², 1.9 V savings are achieved vs. oxygen evolution. The electrodes were 1.0 cm thick by the lamination of a polymeric anti-gas percolation coating, the catalyzes surface of a heavy carbon cloth. The cloth is then laminated with carbon epoxy to a lead substrate. The coating constrains hydrogen within the clot; it flows between the catalyst and the epoxy bond. Utilization is over 90%.

400 Cathodic Reduction of Hg(l)-Cl⁻ Complex on Au-Ag Electrode: Q. Yin, Dept. of Geology, University of Manchester, Manchester, Manchester, England M13 9PL
The cathodic reduction of the Hg(II)-Cl⁻ complex on the Au-Ag electrode in aqueous solution is varied with the concentration of the complex. In concentrated mercury chloride electrolyte, the reduction process is controlled by Ohm's law; whereas in dilute mercury chloride electrolyte, the process is controlled by concentration polarization. For the latter process, the apparent activation energy for reduction of Hg(II)-Cl⁻ complex to mercury has been determined to be 11.631 kJ/mol and the diffusion coefficient of Hg(II)-Cl⁻ complex has been determined to be 4.3×10⁻²⁶ m²/s. The cathodic reduction is significantly affected by the formed HgCl₂(s) layer on the electrode surface, and it has been found to be 3.905×10⁻⁴ m²/s as the electrode is stationary and the concentration of mercury chloride is 0.05585M.

INDUSTRIAL ELECTRO-ORGANIC PROCESS

401 Organic Electrolysis at Extended Area Nickel Electrodes: C. J. Brown and D. Fletcher, Dept. of Chemistry, University of Southampton, Highfield, Southampton, England S09 5NH
Extended area electrodes are a good choice for electrolysis requiring a low current density due to slow kinetics or poor solubility of reactants. A wide variety of extended area nickel materials are commercially available for manufacture into suitable electrodes. Oxidation of a variety of compounds to their corresponding bases, using an ICI FM01 parallel plate cell have been studied. Clean products with good current efficiencies are obtained.

402 Oxidation of Methanol on a Metalized Polymer Electrolyte Membrane: R. Liu and P. Fedick, Dept. of Chemical Engineering, North Carolina State University, Raleigh, NC 27695
A study of the product distribution of the partial oxidation of gaseous methanol using a platinized Nafion 117 membrane has been conducted. The product distribution is found to be sensitive to the morphology of the electrode, temperature, methanol concentration, and water content in the reaction zone. The goal of this work was to manipulate the variables so to increase the selectivity of a particular partial oxidation product. Experimental conditions that produced formaldehyde, methylformate, or methylal at high selectivities (75%) have been obtained and are reported.

403 The Electrocatalytic Hydrogenation of Soybean Oil: G. Yusum and P. N. Pintaruo, Dept. of Chemical Engineering, Tulane University, New Orleans, LA 70118
A novel electrochemical approach for hydrogenating edible oils has been developed. Such a technique offers the advantages of less severe operating conditions and improved unsaturated cis-isomer product yield. Soybean oil has been hydrogenated electrocatalytically at 70°C and 1 atmosphere pressure in a flow-through reactor using a two-phase oil-in-water emulsion and a Raney nickel powder-supported catalyst. The effects of reactor operating conditions on current efficiency and the composition of the hydrogenated product are presented.

404 Direct and Indirect Electrochemical Epoxidation of Olefins in a Sieve Plate Reactor: C. F. Oduco and K. Scott, Dept. of Chemical and Process Engineering, University of Newcastle, Newcastle upon Tyne, England NE1 7RU
Experimental data is presented both on the direct and indirect electrochemical synthesis of epoxydies in a batch cell and in a plate electrochemical reactor (SPER). High current efficiency of 95.0% was achieved in the medium of halogen salts compared to 70% in alka-
405 A Comparison of Some Insoluble Oxide Catalysts in the Electro-Oxidation of Thioethers in Aqueous Sulfuric Suspensions: T. C. Frankein, R. Nnodieke, and R. C. Dutty, Dept. of Chemistry, Bayat University, Lebanon, WA 7608-7345, Waco, TX. Insoluble oxides were suspended with cationic surfactants in aqueous systems and electrolytically oxidized to higher oxidation states. The oxide was oxidized to barium superoxide (I) and copper (II) oxide to copper (III) oxide and manganese (II) oxide to manganese (III) and manganese (IV) oxide. The products were obtained when these oxides reacted with ethylene sulfide. The superoxide did not react; Cu (II) destroyed the compound and produced the sulfide. Mn (III) produced several products and Mn (IV) did not react.

406 Electrochemical Oxidation of Organic Pollutants for Waste Water Treatment: Ch. Commissariou, Institute of Chemical Engineering, Swiss Federal Institute of Technology, CH-1015 Lausanne, Switzerland

The possibilities of electrochemical oxidation for the treatment of organic pollutants is elucidated and the nature of organic pollutants which can be economically treated by this treatment are discussed. It is concluded that electrochemical treatment can be used as a pretreatment step (for the oxidation of refractory organic pollutant to biodegradable organic compounds) before biological treatment.

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407 Proton-Exchange Membrane Reactor for Removal of Organic and Bacterial Contaminants from Reclaimed Water: L. M. Swain, Surfacing Center for Chemical Systems and Hydrogen Research, Texas A&M University, College Station, TX 77843-3402, G. D. Litchfield and O. J. Murphy, Lyntech, Inc., Bryan, TX 77803

An electrochemical reactor that utilizes a proton exchange membrane (PEM) is being developed for the removal of organic and bacterial contaminants from reclaimed water. The electrochemical procedure is the final step in waste water treatment prior to reuse and constitutes reducing levels of organic impurities from 100 ppm to <500 ppm by electro-oxidation. The electrochemical cell is akin to a proton exchange membrane water electrolyzer. The initial experiments were carried out with Pt-Îr and Pt-coated titanium meshes for the anode and cathode, respectively. In order to enhance the energy efficiency for the process by reducing the anodic currents due to oxygen evolution, SnO₂ coated Ti anodes are currently being used. The results show that the electric energy consumption for the removal of organic contaminants was reduced by a factor of two with the SnO₂ rather than the Pt-Ir electrolyte.


(Butyltetracarbamoyl)oxycarbonyl (BTOCA) is a replacement for currently used cross-linking agents for permanent press cotton fabric. BTOCA does not release formaldehyde as currently used agents do. A process to produce BTOCA involves electrophydrodimerization of dimethylmaleate (DMM) followed by hydrolysis. It has been found that hydroxidimization of DMM proceeds well in methanol containing aminated 1:1 biphasic solution. This process requires low energy consumption for the formation of long polymer chains. This process requires low energy consumption for the formation of long polymer chains. The process description work leading to development of solvent, electrolyte, electrolyte, cell type, current density, etc.

409 Hydrodimerization of Dimethylmaleate: II. Process for Converting Maleic Anhydride to 1,2,3,4-Butanetetracarboxylic Acid: D. J. Kalota, M. R. Bagley, E. A. Casanova, M. C. Dutton, and J. H. Wagenknecht, Monsanto Chemical Co., St. Louis, MO 63167

Butanetetracarbamoyl chloride (BTOCA) is a polyvalent cross-linking agent that does not release formaldehyde. Its viability as a reaction is strongly used as a good performance in heat coloration tests. This presentation describes the overall process, except for the electrophydrodimerization of dimethylmaleate, for converting maleic anhydride into an anhydride. BTOCA product. Heat dissipation results show the effectiveness of water extraction, hydrogen peroxide treatment, and catalyst residue and forming a high quality product.


Work done by the USDA showed that 1,2,3,4-butane-tetracarbamoyl chloride (BTOCA) was an effective permanent press cross-linking agent for polyacrylate fabrics. The BTOCA was obtained using the electrophydrodimerization (EHD) of dimethylmaleate to form tetramethyl-1,2,3,4-butane-tetracarbamoyl chloride in a step the process to produce BTOCA. It has been found that the very effective as cross-linking agents for the electrophydrodimerization of EHD (95%) of dimethylmaleate was obtained in a batch system with a bulk pH of 7 to 8. This presentation describes experiments pertaining to batch vs continuous operation, pH effects, and EHD cell life.

411 The Preparation of Metal Ion Reductants via the Use of Hydrogen Diffusion Anodes: P. C. Feller,* R. J. Allen, and R. Vorn, E-TEK, Inc., Framingham, MA 01701

Ti(III) has been prepared from Ti(IV) in sulfuric acid using the use of hydrogen diffusion anodes (HDA). HDAs allow the preparation of Ti(III) sulfate free of extraneous ions as are introduced via aluminum or zinc reduction, the usual route to metal ions. No membrane is required, as the potential of the HDA is insufficient to re-oxidize Ti(III) once it is formed. High (>95%) conversions can be obtained using HDAs, and the approach is foreseen in indirect organic electrochemistry.

412 An Analyte Model of a Diaphragm-Type Chlorine/Caustic Cell: J. W. Van Zee and C. T. Lee, Chemical Engineering Dept., Swearingen Engineering Center, University of South Carolina, Columbia, SC 29208

A time-dependent analyte model of a diaphragm-type chlorine/caustic cell, which includes the multicomponent flow, is presented. The effects of convection, migration, and gas void fraction on mass transfer of the species in the anolyte are considered. The model is formulated in terms of feed flow rates, which enables the model to be used readily for developing process control schemes for the analyte variables in a diaphragm-type cell, and to be modified to simulate the analyte of membrane-type chlorine/caustic cells. The predictions of these species concentrations are shown to agree with the experimental data of a lab-scale diaphragm cell.

413 Design of a Digital Multiphase PID Control System for a Diaphragm-Type Chlorine/Caustic Cell: C. T. Lee and J. W. Van Zee, Chemical Engineering Dept., Swearingen Engineering Center, University of South Carolina, Columbia, SC 29208

A digital multiphase PID (proportional-integrative derivative) feedback control system was designed for a diaphragm-type chlorine/caustic cell using a theoretical model. The analyte pH and the caustic efficient concentrations were chosen as the main control variables. A feedback control simulator of the cell on the design was also developed. The design methodology presented is general, and can be readily applied to other electrochemical systems, especially those with nonlinear and complicated model equations. This work provides a basis for studying the effects of digital process control schemes on performance of a diaphragm-type cell.

**ELECTRO-ORGANIC SYNTHESIS WITH HOMOGENEOUS AND HETEROGENEOUS CATALYSTS**

Organic and Biological Electrochemistry

414 Mediated Reductive Dechlorination of PCBs in Surfactant Dispersions and Microemulsions: J. J. H. Wagenknecht, Monsanto Chemical Co., St. Louis, MO 63167

An electrochemical mediator system is the reaction of acetic acid, followed by a chemical reaction between the Pd(H) and the organic reactant. Electrosynthesis in a FM-01 laboratory electrolyzer is described.

415 Electrochemical Reduction of Nitroaromatic Compounds to Amines in Basic Solutions at Porous Metal Electrodes: J. C. Cleghorn* and D. Fletcher, University of Southampton, Highfield, Southampton, England

Palladium black electrodes have been prepared by electroderepolarization of palladium onto graphite and nickel from an acid chloride bath. These electrodes in a methanol containing acetic electolyte are suitable for the hydrogenation of many organic molecules. The mechanism involves the reversible formation of PdH by reduction of acetic acid, followed by a chemical reaction between the Pd(H) and the organic reactant. Electrosynthesis in a FM-01 laboratory electrolyzer is described.
The sulfide ion acts as a mediator in the electrodeposition of nitroaromatics at a stainless steel (type 316) cathode by forming reversible sulfide-poly sulfide couple. The polysulfide is reduced to sulfide at a less cathodic potential than the sulfide. The sulfide mediator reduces the nitroaromatic compound in the presence of fluoride ions: When the sulfide mediator reduces the nitroaromatic compound in the presence of fluoride ions, the hydroxylamine stage in the cold but to the aniline stage at a characteristic elevated temperature which varies from substrate to substrate. The difference in the electronic behavior of naphthalene and 1,3-dinitrobenzene is explained on the basis of their structural difference and the difference in the polarity of the two molecules.

418 Indirect Reduction of Sulfonium Salts by Means of p-Acceptor Radical Anions (in Liquid Phase) and n-Doped Electroactive Polymers (in Solid Phase) Application to Functionalization of Organic Molecules: J. Stofer, P. Stenger and J. Rault-Berthelot, Laboratoire d'Electrochimie, Université de Rennes 1, Campus de Beaulieu, 35042 Rennes Cedex, France

The mediator was dimethylpyrrolidinium-Sn, with two identical orthogonal halves. The cyclic voltammogram of the mediator was 2,2'-Diacetyl 9,9'-spirobifluorene. The carboxylation occurs in the nickelate() as a Homogeneous and Polymer-Based Catalyst. The mediator was 2,2'-Diacetyl 9,9'-spirobifluorene. The carboxylation occurs in the nickelate() as a Homogeneous and Polymer-Based Catalyst.

419 On the ipso-Substitution in Aromatic Compounds by Electrogenerated Radicals: H. Lund, Dept. of Organic Chemistry, University of Aarhus, DK-8000 Aarhus C, Denmark, K. Pang, and J. Xu, Dept. of Organic Chemistry, Shenyang College of Pharmacy, Shenyang, 110015 China

Anion radicals of aromatic compounds react with alkyl halides with formation of radicals which may couple with an anion radical. When the aromatic compound is substituted with a suitable leaving group Y (e.g., CN) an ipso-substitution follows by elimination of HY. The regioselectivity in the reaction is determined by the substituents. ArY is compared improved to that of AH. Thus reduction of 4-cyanopyridine in the presence of t-butyl bromide and diethylamine gives 4-t-butylpyridine in good yield.

420 Production of Pyrene Quinone by Indirect Electro-Oxidation of Naphthalene Pyrene: T. Nomura, and A. Yoshinaka, Dept. of Electronic Chemistry, Tokyo Institute of Technology, 2-12-1, Ookayama, Chuskagakai, Co., Ltd., 24 Kanji-enju 6-Chome, Mitaka, Tokyo 181, Japan

The indirect electro-oxidation of water-insoluble solid materials such as tetramethylenpyridine, and pyrene was performed by electrolyzing their suspensions in the presence of suitable redox mediators (catalysts) dissolved in aqueous electrolytic solutions. Particularly, the oxidation of pyrene was investigated in detail using a bench-scale parallel plate flow cell from a practical aspect of the pyrene quinone production.

421 Electro-Oxidation of Propene in Aqueous Zeolite Suspensions: J. Z. Stemple, and D. R. Roltzon, Coord. in Electrochemistry, Branch, Naval Research Laboratory, Washington, DC 20375-5000

Zeolite-supported metal clusters sized less than 10 nm are addressed to microelectrodes via dispersion electrolysis between two feeder electrodes. This system couples materials which, when sized on the nanometer scale, can exhibit physical and chemical behavior that differ from bulk properties and zeolites which allow for molecular discrimination based on size, shape, and charge selectivity. Ultramicrowires are supported on zeolites, therefore, provide a unique interface that undoubtedly will affect not only charge-transfer processes, but also interfacial chemical reactions. Accordingly, the oxidation of propene is chosen as a model reaction to study (i) the zeolite influence on electron transfer and subsequent chemical reactions at metallized zeolites, and (ii) the influence of an electric field on the inherent catalytic activity of metallized zeolites.

422 Mediated Reduction of Aryl Bromides at Tin Cathodes: E. G. Underwood, E. Korn-Miller, and V. Svenner, Dept. of Chemistry, University of Minnesota, Minneapolis, MN 55455-0431

The mediated reduction of aryl bromides at tin cathodes was investigated by using dimethylsulfoxonium dimethylpyrrolidinium radical cation (DMPSn), generated by the cathodic reduction of the electrolyte cation. The relative rate of electron transfer to the various bromides was determined by measuring the total potential electrolysis (CPE) and anodic stripping voltammetry (ASV). It was found that the electronic nature and relative location of the substituents on the aromatic ring had a significant effect upon the rate of reaction.


Anodic α-alkylation of sulfides was promoted in the presence of fluoride ions. When Et₃N·3HF was used as a supporting electrolyte, simple alkyl phenyl sulfides and sulfides bearing weak electron-withdrawing groups underwent anodic alkylation via fluorosulfonium ions as key intermediates in a unique Pummerer-type mechanism with reasonable or high yields for the first time.

424 Use of [2,2'-Ethylenebis(nitrilomethyldiphenylsulfide)]nickel(II) as a Homogeneous Catalyst for Reduction of Organic Halides: C. E. Dahm and D. G. Peters, Dept. of Chemistry, Indiana University, Bloomington, IN 47405, M. S. Makaraak, Dept. of Chemistry, King Saud University, Abha, Saudi Arabia

In dimethylformamide containing tetraethylammonium perchlorate, [2,2'-ethylenediamine(nitrilomethyldiphenylsulfide)nickel(II)] undergoes reversible one-electron reduction at a carbon cathode to generate the corresponding nickel(I) species. The presence of this nickel(I) species, 6-bromo- and 6-iodo-1-phenyl-1-hexyne are homogeneously and catalytically reduced to form a radical intermediate that cyclizes to yield benzyldene-napthylene. Catalytic polymerization of the nickel(II) species at rlectivated vitreous carbon in acetonitrile containing tetraethylammonium perchlorate is catalyzed by polyparaphenylene which can be employed subsequently for the efficient catalytic reduction of alkyl halides.

425 Long Life Anion Radicals from ARX or RX Type Compounds: Mediators, or AR- and R' Providing Species? Case of Ortho-bis(Alykylsulfonyl)benzenes: J. Simonet, Laboratoire d'Electrochimie, Université de Rennes 1, Campus de Beaulieu, 35042 Rennes Cedex, France

Title compounds (I) lead by means of their cathodic reduction to surprisingly stable anion radicals. Bimolecular resection of (I) anion radical either on starting material or on aliphatic organic halides in excess affords very heterogeneous anionic substitutions which depend on the nature of the halide. For example:

426 Conversion of IsoSafore to Piperonal Using Electrolytically Recycled Higher Oxides of Manganese: J. Grunbom, Dept. of Chemistry, Queen's University, Belfast, Northern Ireland B9 9AG, H. C. Hau, Dept. of Applied Chemistry, Beijing Institute of Chemical Technology, Beijing 100083, China

Anodic oxidation of manganese(III) sulfate in aqueous sulfuric acid of concentration less than 50% (w/w) gives a fine precipitate of nonstoichiometric manganese dioxide. This precipitate, suspended in dilute sulfuric acid, converts isoSafore to piperonal and the manganese compounds dissolve as manganese(II) sulfate. Piperonal is isolated and the manganese recycled. When the manganese dioxide is filtered, any water soluble impurities are removed in the filtrate and such a step is necessary in any process for continuous operation.


Bidentate nickel and monodentate palladium complexes catalyze the electroreduction of aromatic carboxylic acids from carbon dioxide and aromatic radicals. The catalysis proceeds via a succession of chemical and reductive steps involving diamagnetic and paramagnetic nickel complexes but only diamagnetic palladium complexes. The carboxylation occurs in the nickel coordination shell (via a Ni(II) intermediate). Whereas in the case of palladium, carbon dioxide reacts with an aryl anion Ar- formed from a Ar-Pd(II) intermediate.

428 Electrochemistry of 9,9'-Spirobifluorene Derivatives: 2,2'-Diacetyl 9,9'-Spirobifluorene: L. Matriello and L. Ramang, Dep. of Chemistry, University of Rome, 00186 Rome, Italy
2,2'-Diacetyl 9,9'-Spirobifluorene (I) is an aromatic compound with two identical orthogonal hexagonal annuli. The electrochemistry of (I) shows five reduction processes, measured in dry DMF: 0.1M Et₃N·NClO₄, glassy carbon electrode. The first two are reversible, one electron transfers with E₁/₂ = -0.20 V and E₂/₂ = -0.24 V (SCC), they are followed by three (apparently irreversible) reduction processes occurring at very negative potentials, E₁/₂ = -2.40,-2.67, and -2.80 V (sweep rate 0.2 V/s), related to the spirobifluorene moiety.

429 A Study of Substituted Catechols as Electron Transfer Mediators in Electrocatatytic Debromoagent: J. Markovics, and L. A. Coury, Dept. of Chemistry, Duke University, P. M. Gross Laboratory, Durham NC 27706-7706

Kálman et al. found that the rate constants for reduction of 13 o-quinones by reduced glucose oxidase are determined and found not to be correlated to the 2e/2H⁺ redox potentials or the hydroxide transfer potential. Results from kinetic experiments in deuterated water and protonated water indicate that the rate-limiting step may be abstraction of the first electron. An understanding of the kinetics of this
reaction is important to the development of more effective electrochemical biosensors.

430 Electroreductive Cleavage of the C-C Bond of 9-Chlorofluorenes: F. Maron and E. Vianello, Dept. of Physical Chemistry, University of Padova, 35131 Padova, Italy

The production of a series of substituted 9-chlorofluorenes has been investigated in DMF by cyclic and convolution potential scan voltammetries and controlled potential electrolysis. The nature of the determination of the heterogeneous rate constant of the initial one-electron uptake in a wide potential range, a potential dependence of the electrochemical transfer coefficient a was found, thus pointing to a concerted electron transfer-bond breaking process. The potential dependence of a was taken into account in the kinetic analysis of the self-promoted reaction involving the carbon atom eventually electrogenerated at the electrode. The dependence of the support rate constant on the substrate concentration as well as further evidence pointed to the occurrence of complex chemical pathways in the voltammetric time scale.

431 Electrochemical Behavior of Cytochrome c3 Hildenborough on a Gold Electrode: B. A. Gorecka, Dept. of Pharmaceutical Chemistry, University of Kansas, Lawrence, KS 66045, G. S. Wilson, Dept. of Chemistry, University of Kansas, Lawrence, KS 66045

Electrochemical behavior of cytochrome c3 Hildenborough in solution phase as well as adsorbed films on gold electrode has been studied by cyclic voltammetry chronocoulometry and chronopolarography. Properties of the cytochrome c3 films are compared with those of the redox polymer film.

432 The Anodic Oxidation of Hydrazones: M. R. Van De Mark and E. -C. Lin, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401, W. Elders, University of Miami Medical School, Coral Gables, FL 33101

The anodic oxidation of aldehyde hydrazines at a graphite anode in aqueous acetonitrile was investigated. The major products were the para-aldehyde, the alkyloxyacetic acid, hydrazine of RCON=NAr. A proposed mechanism consistent with the products was suggested including geometry optimizing MNDO and AM1 calculations. A very good linear free energy correlation with sigma was found.

433 Pin Loosening in External Skeletal Fixation: In Vivo Electrochemical Impedance Study of the Pin Bone Interface: O. A. Vele, Center for Electrochemical Systems and Hydrogen Research, College Station, TX 77843, R. H. Palmer, Santa Cruz Veterinary Hospital, Santa Cruz, CA 95065, P. Stransman, Center for Electrochemical Systems and Hydrogen Research, Texas A&M University, College Station TX 77843, D. A. Halen, Dept. of Small Animal Medicine and Surgery, Texas A&M University, College Station, TX 77843, H. W. Sampson, Dept. of Anatomy, College of Medicine, Texas A&M University, College Station, TX 77843, W. H. Hyman, Dept. of Industrial Engineering Division of Bioengineering, Texas A&M University, College Station, TX 77843

External skeletal fixation is commonly used for treatment of many orthopedic injuries and diseases in human and animal patients. One of the most common failure mechanisms is loosening of the fixation pins within the bone affects up to 42% of such pins and remains the major complication associated with this technique. Pin-loosening results from a postulation of mechanical and biological events. Electrical fields resulting from local mechanical strains are considered the primary signal which initiates bone remodeling. The purpose of this study was to characterize the electrochemical environment of the pin-bone interface in vivo during the first 43 days after implantation from open circuit potential to voltammetry.

434 Progress in Observing Electrochemical Processes at Superconducting Electrode/Molecular Solvent Interfaces: S. C. Peck, L. S. Curtin, L. M. F. Tender, J. N. Richardson, and R. W. Murray, Khanan Division of Chemistry, University of North Carolina at Chapel Hill, NC 27599-3290

This paper reports progress in our program to observe electrochemical processes at superconducting electrode/molecular solvent interfaces by the use of a sed two-electrode electrochemical cell, at temperatures that span their T, transition temperatures. In particular we have observed a change in the double-layer capacity of these electrodes at T,.

435 Low-Temperature Electrochemistry on High T, Superconductors: W. S. F. Ogg, Dept. of Physical Chemistry, University of Cambridge, Lensfield Road, Cambridge, England, Germany, G. S. Anderson, Institute of Physics, University of Erlangen-Nuremberg, Erlangen, Germany, M. W. Breiter, Institute of Technical Electrochemistry, University of Vienna, Vienna, Austria

The charge transfer across superconductor/ionic conductor interfaces represents a relatively new experimental and theoretical field. This paper deals with electrochemical studies in this area. Both n-type classical superconductor (SC) and p- and n-type high T, superconductors (HTSC) in contact with different superionic conductors (AgS), alumina, Ag ion conducting glass, and RbAg(3), which are used as solid electrolytes (SE), are considered in experiments covering the temperature range 10 K ≤ T ≤ 298 K. The faradic process occurring at those HTSC/SE interfaces is the silver deposition and dissolution, measured by either a transient technique in the time domain or by electrochemical impedance spectroscopy (EIS) in the frequency domain. The results show a positive admissibility peak in the EIS measurements for a corresponding negative peak in EIS-measurements around T = T. These findings are attributed to an enhancement of the rate of the faradaic reaction around T = T. The effect is interpreted as a quantum-electrochemical phenomenon caused by the contribution of Cooper pairs crossing the electrochemical double layer as correlated charge carriers and participating in the charge transfer rate of the phase boundary reaction. A proximity-like phenomenon can be excluded.

436 Corrosion Reactivities of Various Copper Oxide and Fullerene High Temperature Superconductor Phases: J. T. McDermott, D. R. Riley, and J. P. Zhou, Dept. of Chemistry and Biochemistry, University of Texas at Austin, TX 78712-1167, A. Mantiharam, Center for Materials Science and Engineering, University of Texas at Austin, TX 78712, and Dept. of Chemistry and Biochemistry, University of Texas at Austin, TX 78712-1167

Before practical utilization of the high temperature superconductors is possible, a more complete understanding of their surface chemistry is necessary. In this paper, the relative reactivity of the common high-Tc phases toward water is reported. Chemical, x-ray powder diffraction, and scanning electron microscopy measurements are utilized to establish the corrosion rate. From this comprehensive study, the following chemical reactivity sequence is established: K2CO3 >> Bi2O3-CuO >> Bi2O3 >> Sr2CuO3-CaO >> Bi2Sr2CaCu2Oy >> Sr2CaCu2Oy >> La213Sr2CaCu2Oy >> Nd2201Ce2Cu3Oy >> Nd218Ce2Cu3Oy.

437 Electrochemical Rate Data of Superconductor Materials and Their Lithium Insertion Compounds: P. L. Hulse, Dept. of Materials and Interfaces, The Weizmann Institute of Science, Rehovot 76100, Israel

The insertion of lithium into the lamellar bismuthate 2212 and 2201 superconductors modifies their structural chemistry and electronic properties. Electron transfer reactions were studied at electrodes made from 2212 and 2201 in the normal state and compared to their Li insertion compounds, regular perovskites, and metals. The heterogenous rate constants for several redox reactions in organic solvents decreased with increasing Li content. The insertion reaction itself was studied by cyclic voltammetry and the effective diffusion coefficient for Li in the superconductors was measured.

438 The Surface Structure and Electronic States of High-Tc Superconductors by STM: H. Lieber, R. Srinivasan, and J. M. Moore, Dept. of Chemistry, Harvard University Cambridge, MA 02138

Scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) have been used to determine the electronic states of the surfaces of single crystal Bi-Sr-Ca-Cu-O superconductors. STM images have been used to elucidate variations in the atomic structure across the surface that result from metal-substitution and oxygen doping. STS data further show how the electronic states of this material evolve with metal and oxygen doping.

439 Physical and Chemical Characterization of Electrodeposited Superconducting Bismuthates: M. L. Norton, Dept. of Chemistry, Marshall University, Huntington, WV 25701, R. Y. Tung, Materials Science Center, National Tsinghua University, Hsinchu, Taiwan, China

Large single crystals of the bismuth based superconductors Ba2-xMxBiO3 (M = K, Rb) produced by anodic electrocrystallization have been characterized. Low resolution scanning tunneling microscopy (STM) and atomic force microscopy (AFM) studies indicate the presence of a superconducting layer on samples with a domain size of approximately 50 nm. Although significant sodium contamination was not detected in bulk analysis performed utilizing inductively coupled plasma mass spectrometry, STM and AFM further showed how the electronic states of this material evolve with metal and oxygen doping.

440 Synthesis of Superconducting Films via an Electrochemical Pathway: A. Weston, N. Ali, and S. B. Levato, College of Engineering and Technology, Southern Illinois University, Carbondale, IL 62901

Precursor thin films of Y-Ba-Cu-O and Er-Ba-Cu-O superconductors were produced by potential cyclic and pulse electrolysis from the dissolved nitrate salts of the constituent metals in dimethylformamide. The films were deposited on metal foils such as Au, Cu, Ag, Sr, as well as with inorganic matrices such as ZnO and Al2O3. The electrodeposited films were heat-treated in flowing O2 in order to produce the superconducting phase. Y-Ba-Cu-O films formed on Zn had highest Tc, onset at 83 K.

Superconductor oxide films fabricated via a one-step electrodeposition process followed by short annealing in oxygen. Cyclic voltammetry was employed to investigate the reduction potentials of the metal-ions and reaction mechanism. The precision of the superconducting oxide films were determined from a non-aqueous solution (nitrate salts dissolved in dimethyl sulfoxide) at a constant potential under a pulsed-potential condition, where the pulse cycle was 10 s at -4 V followed by 10 s at -1 V (vs. Ag/AgClO4). The substrates used were silver-coated thin crystals of YB2Cu3O5, Nb-doped SrTiO3, MgO, ZrO2, and solutions of Ag, AgClO4, and Ni. The outstanding critical current densities measured to date for polycrystalline TBCCO films are as follows: 10^6 A/cm^2 for the current density of 1 K for a pulsed-potential TBCCO film on silver foil was 10,000 A/cm^2 in zero field, (ii) the critical current density at 78 K for a TBCCO film deposited at constant potential on a silver-coated SrTiO3 substrate was 20,000 A/cm^2 in zero magnetic field and 5,000 A/cm^2 in a 10 K field parallel to the film plane, and (iii) the critical current density of a pulsed-potential deposited TBCCO film on silver-coated SrTiO3 was 56,000 A/cm^2 at 76 K in zero field.

Reactivity of Compound Superconductors: Cuprates, Bismuthates, Fulleders: B. Miller and J. M. Rosanieta, AT&TA Bell Laboratories, Murray Hill, NJ 07974-0686

Recent work with T. shows 23K include an assortment of cuprates, bismuthates like Ba2K2CuO4, and fullerates such as Rb2CuO4. Electrochemically, each of these has a very strong redox reactivity; strong oxidants having nominal copper oxidation states >+2 or Bi >+3, strong reductants with Cu+ anions. We discuss the reactivity, which impacts greatly on oxidation formation, device stability, and means of protection.

Controlled Room Temperature Formation of Weak Link or Josephson Junction in Thin Film YBaCu2Oy, D. Cohen and Y. Socolin, The Weizmann Institute of Science, Rehovot 76100, Israel

We show how the earlier developed methodologies of room temperature reduction and reoxidation of samples of YBaCu2Oy can be used to pattern μm-thin films at room temperature by electrochemistry. The resulting superconducting structures are characterized in terms of their I-V characteristics and found to show SN junction behavior. In several cases reduction reoxidation can improve the homogeneity of the sample in the affected areas.


The high temperature superconducting YBa2Cu3O7 is also a mixed-conductor with a wide range of oxygen nonstoichiometry. To evaluate its potential as an electrode material for solid oxide fuel cell applications, the YBa2Cu3Oy powder was deposited as a controlled monolayer of pyrolytic zirconia (YSZ) solid electrolyte. A porous platinum electrode was used as the counter-electrode. The electrochemical behavior of the YBa2Cu3O7 electrode was studied between 463 and 833 K in air by de polarization and ac impedance spectroscopy techniques. The results indicated that YBa2Cu3O7 may be employed as an electrode material under oxidizing conditions.

Electrochemical Response of YBa2Cu3Oy as a Function of Oxygen Content: D. R. Ridley, and J. T. McDevitt, Dept. of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712-1167

By varying the oxygen content in the high temperature superconductor powders YBa2Cu3Oy (6 < x < 7), the electrical properties of this material can be manipulated from that of a superconductor to that of a metallic conductor. In this paper, we describe a method for making thin films of YBa2Cu3Oy using films of different oxygen contents. These samples are used as a corresponding procedure for fabricating these films. This procedure provides a systematic and meaningful way of tailoring the conductive properties of electrode specimens and the response of these electrodes are discussed herein.

Application of High Temperature Electrochemical Techniques to YBa2Cu3Oy, R. V. Kumar and J. F. Pray, Dept. of Mining and Mineral Engineering, University of Illinois, Urbana, IL 61801

High temperature solid state electrochemical techniques have been applied in the determination of oxygen nonstoichiometry and phase boundaries of YBa2Cu3Oy. Much of the nonstoichiometric composition of YBa2Cu3Oy is metastable, and they fall in the phase region of YBa2Cu3Oy. Under favorable conditions of temperature, these samples are electrochemically decomposed to YBa2Cu3O7 and the corresponding phase-boundary determined. Enhanced texture can also be obtained in the bulk-sintered samples by electrochemical processing of YBa2Cu3Oy. By incorporating grains that are platelet-like with high aspect ratio as feed material into the matrix of fine-sized reactive precursor, bulk samples with good texture can be produced by the combined effect of pressure, temperature, and electrochemical potential.

Effect of Solvent on the Simultaneous Adsorption of Anions and Cations: M. Anbu Kulandaiswam* and S. Venkatakrishna Iyer, CECCI, Karaiikudi 623 006, India

The adsorption of tetrabutylammonium chloride from water, heavy water, and DMSO on mercury electrode has been studied using capillary electrometer. Parameters like capacitance values at different potentials, potential of zero charge, desorption valency, and electrosorption valency have also been calculated to characterize the simultaneous adsorption. The adsorption of TBA ions from different solvents can be explained using Virial adsorption isotherm. The amount of specifically adsorbed charge due to cations in the presence of adsorbed anions is evaluated using Saffarian and de Levie model.

Manipulation of Double Layer in Metal Insulator Electrolyte: K. Ghous, Dept. of Chemistry and Biochemistry, Texas Tech University, Lubbock, TX 79409-4260

The manipulation of double layer is possible at the insulator electrolyte by applying a strong field (10^10 MV/cm) across metal-insulator-electrolyte. In the proposed model for metal-insulator-electrolyte the surface ionization and specific adsorption have been combined with Stern Gouy Chapman theory. By combining the metal insulator electrolyte characteristics and the electroosmotic effect in a capillary, a novel effect called field effect electroosmosis has been proposed.

Molecular Recognition at Interfaces: Specific Binding of an Electroactive Tetrathiafulvalene (TTF) Derivative to Organosoluble Monolayers by Hydrogen Bonding: L. M. Frostman and M. D. Ward, Dept of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN 55455

We present evidence that the monocarboxylic acid derivative of tetrathiafulvalene (TTF) binds electrostatically to COO- terminated organosoluble monolayers on gold. Cyclic voltammetry of these films shows TTF/COOH coverages on the order of monolayers. These studies indicate that molecular recognition is operative on functional molecular monolayers.

Kinetics of Electron Hopping in Langmuir Monolayers at the Air/Water Interface: M. Mejia, D. H. Charych, and J. T. Orr, Dept. of Chemistry, University of California, Berkeley, Berkeley CA 94720

We discuss dynamic solvent effects and the distance dependence on the electron transfer kinetics. Our approach involves 2-D electrochemical measurements carried out at the air/water interface. In these experiments, a "line" microelectrode is positioned in the plane of the air/water interface, where it addresses molecules forming monolayers at that interface. The method allows the study of pressure conditions of a Langmuir trough. Under these conditions, diffusion coefficients obtained in the volumetric studies of various triacyl-diphenylethanethiacs are interpreted in terms of the rate constant of the lateral electron hopping.

Effects of Monosubstituted Phenyl Additives on the Conductivity of Electrochemically-Synthesized Poly(pyrrole): M. Fukuyama, Y. Nanao, T. Kojima, Y. Kudoh, and S. Yoshimura, Matsushita Research Institute Tokyo, Inc., 3-10-1 Higashimita, Tama-ku, Kawasaki 214, Japan

We report the effects of monosubstituted phenol additives that are contained in a polymerization solution, on the conductivity of electrochemically-synthesized poly(pyrrole). For the case of phenylnitrophenol the conductivity is 84 S/cm which is about 7 times higher than that without additives. From the elemental analysis data of mass spectra, infrared spectra, electrical conductivity, and laser Raman spectra, it is clear that the additives are not included in the polypyrrole films but the electronic state of polypyrrole is changed.

Impedance and Voltammetric Characterization of Electrochemically Deposited (Poly)aniline Conducting Films: P. Varela and V. S. Adalsteinsson, Dept. of Chemistry, Northern Illinois University, DeKalb, IL 60115

Poliyaniline films electrochemically deposited on Pt electrodes are characterized by semicircular impedance spectra. Repetitive measurements at varying bias potentials and concentrations are recorded and evaluated. The films are grown in several acidic solutions, with the best film obtained in sulfuric acid. Circuit analysis reveals a potential dependent constant phase element in parallel with a potential dependent resistance. These parameters are correlated with the impedance of the film and with the changes of the double-layer capacitance.

Electrochemistry is utilized as a probe to determine the effects of partially oxidized tetracyanoplatinate (POTCP) complexes on the electrochemical activities of the electrode. The authors describe the use of cyclic voltammetry and square wave voltammetry to study the interactions between POTCP complexes and the electrode surface. The results indicate that POTCP complexes can significantly alter the electrochemical behavior of the electrode.

462 In Situ Ellipsometric Spectroscopy for Redox of Prussian Blue Films on Platinum Electrode: S. F. Xie and Z. Q. Huang, Dept. of Applied Chemistry, Chongqing University, Chongqing, 630044, China.

This paper describes the ellipsometric spectroscopy with visible and near-infrared light for the study of the redox processes of a Prussian blue film on a platinum electrode. The authors report that the method is sensitive to changes in the film's optical properties and can provide information on the film's thickness and composition. The technique is shown to be useful for studying the electrochemical properties of the film.

466 Electrodeposition of Epitaxial Films of Ag(Ag02), Ni and Cu on Electrodes: B. E. Breyfogle and J. A. Switzer, Graduate Center for Materials Research, University of Missouri-Rolla, Rolla, MO 65401.

The electrodeposition of epitaxial films of Ag(0), Ni, and Cu on electrode surfaces is investigated. The authors report that the films exhibit high-quality epitaxial growth and can be used as templates for other materials. The technique has potential applications in the fabrication of advanced electronic devices.


The microstructure of electrodeposited copper is studied using transmission electron microscopy and X-ray diffraction. The results indicate that the copper deposits exhibit a columnar growth structure, which is influenced by the deposition conditions. The study provides insights into the mechanisms of copper deposition and can be useful for optimizing the deposition process.
**467 In Situ Characterization of p-Type Copper Thiocyanate Films by Raman Spectroelectrochemistry**

P. de Tacconi, K. Rajeshwar, and K. R. Rajeshwar, Dept. of Chemistry, The University of Texas, Austin, TX 78719-0655

The electrochemical growth and propagation of Cu films on polyetherimide films on Cu electrode in acidic KSCN solution was studied in situ by Raman spectroelectrochemistry. A restricted potential domain was used to promote the oxidation and growth of cuprous thiocyanate and to avoid oxide formation. At -0.37 V (vs. Ag/AgCl reference) a sharp Raman band at 2172 cm⁻¹ was assigned to the (001) mode of a bcc CuSCN crystal. The open-circuit voltages at anodically oxidized CuSCN films at open-circuit peaks in intensity during the electrodereaction of the α-CuSCN film. The mechanism including (CuSCN), aggregates is proposed to have chemical and electrochemical pathways for the formation of CuSCN films.

**468 Electrochemical Architecture of Nanomodulated Ti-Pb-O Superlattices**

J. A. S. Zwitser, B. J. Phillips, and R. P. Rafei, University of Missouri-Rolla, Graduate Center for Materials Research, Rolla, MO 65401

Electrochemical deposition was used to produce superlattices based on the Ti-Pb-O system with layers as thin as 3 nm. The composition was modulated using square-wave current pulses. The oxides are degenerate semiconductors with a band-to-band transition in the 1.4-1.8 eV range, and a free-electron plasma edge in the near-IR. The oxides adopt a face-centered cubic structure at low lead contents, but show the fcc fluorite structure for lead contents greater than about 35 atomic percent.

**469 Electrotransformation, Characterization, and Modeling of High-Conductivity Polymer Nanocomposites**

C. S. C. Bose, C. S. Chen, and J. K. Rajeshwar, Dept. of Chemistry, University of Texas, Austin, TX 78719

Polymer nanocomposites containing nanometer-sized Pt particles (pPy/Pt) were electrogenerated at glassy carbon and gold electrode surfaces. This was done either by voltammetric cycling between two potentials (Ag/AgCl) or via a potential scanning technique in solutions containing colloidal Pt particles. A chloroplatinate medium with a citrate reducing/protection agent was employed. The growth of the pPy/Pt films was studied by combined voltammetry-electrochemical quartz crystal microgravimetry. These films exhibited unusually high cyclic voltammetry and hydrogen evolution reaction activity and Cu reduction. The rate does not saturate with increasing film thickness for these 3-D arrays of polymer confined-Pt particles contrasting with confined catalyst situations. Finally, a model is presented for pPy/Pt based on hydrodynamic voltammetry data.

**470 Electrodeposition of Metals on Poly(pyrrrole) Coated Au/Quartz Piezoelectrodes**

M. H. K. C. Perera, S. Perkins, Dept. of Chemistry, Potsdam College of SUNY, Potsdam, NY 13676, T. H. Hebel, ELCHEMA, Potsdam, NY 13676

Electrodeposition of various metals at poly(pyrrrole) (PPy) coated Au electrodes evaporated onto a quartz crystal have been investigated using the electrochemical quartz crystal microbalance (EQCM) and scanning electron microscopy (SEM). The effect of pH, type of anions, different organic additives, and the morphology of the polypyrrole substrate on the electrodeposition of Cu, Ni, Pb, Sn, and Cd have been studied. The nucleation density has been determined from SEM experiments and has been correlated with the frequency-time and frequency-current transients. The anodic potential drop in partially reduced PPy films has been taken into account in numerical simulation of the nucleation and growth processes.

**471 Localized I/V and I/Z Measurements on Conductive Poly-N-methyl Pyrrole Thin Films Performed With a Scanning Tunneling Microscope**

S. Creager, Dept. of Chemistry, Indiana University, Bloomington, IN 47405

Current vs. tip displacement measurements obtained on oxidatively doped poly-N-methyl pyrrole films reveal that the STM tip is buried up to 100 A deep in polymer. STM images are very noisy, a consequence of the tip glowing through polymer while imaging. Current vs. bias potential measurements are interpreted in terms of an electrochemically driven electron hopping model of the electrochemical conductivity.

**472 Factors Affecting Electrochemical Metalization of Insulating Substrates Precoted by Conducting Polymer Films**

F. A. Urbe, A. J. Rudge, and S. Gottesfeld, Electronics Research, Lambda Physics Laboratory, Los Alamos, NM 87545

Chemically deposited conducting polymer (e.g., polypyrrole) films have been used as a precoat for surface metalization of insulation substrates leading to the formation of coated wire. The coating process is discussed. Scanning electron microscopy and cyclic voltammetry show that Cu electrodeposition on polypyrrole precoated by CuSCN is a simple and an island dendritic growth. The electrochemical growth and propagation of Cu films on conducting polymers of various thickness, conductivities, and dopants are discussed. The effect of current densities and potentials on the same process is also discussed.

**473 When Anodic Polymerization of Dibenzo-18-Crown-6 Leads to a New Artificial Membrane: Changes of Microscopic and Macroscopic Models**

J. Rault, A. Leveque, F. Berthelot, V. Questaine, and L. Angely, Laboratoire d’Electrochimie, Universite de Rennes 1, Campus de Beaulieu, 35042 Rennes Cedex, France

Dibenzo-crown ethers lead by means of anodic oxidation to doped microcrystals possessing the structure of dibenzo-18-crown-6. Changes of structure (followed by conventional physical methods) can be obtained when reducing those resins. Their capability to extract (fast) certain organic cations from aqueous solutions is presented. Some use of those membranes are also discussed.
of the growth segment of the transient results in an epitaxial film as demonstrated by x-ray diffraction.

480 Mass Sensitivity Mapping of the Quartz Crystal Microbalance in Liquid Media: A. C. Hillier and M. D. Ward, Dept. of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN 55455

The quartz crystal microbalance (QCM) utilizes the mass-frequency relationship of an oscillating quartz resonator, consisting of a thin quartz wafer sandwiched between two excitation electrodes, to examine interfacial mass changes that occur at or of the excitation electrodes. Accurate application of the QCM as a mass sensor, particularly in the presence of nonuniform mass deposits, requires knowledge of the radial and angular dependence of the QCM mass sensitivity. The mass sensitivity distribution, (S(r), θ), was determined for plano-planar and plano-convex AT-cut quartz resonators in situ using scanning electrochemical methods. Complete closed form mathematical descriptions of (S(r, θ)) were obtained under conditions typically encountered in QCM experiments. Results illustrate the influence of crystal contouring and the extent of field bringing on the mass sensitivity distribution.

481 Electrochemical Preparation of Platinum Nanoparticles: M. Durand and K. Louhab, Crem-GP. Ensereq, 38402 Saint-Martin-De-Beauvoir

Electrochemical nucleation and growth of platinum particles in the nanometer range have been studied on various substrates (graphite plates, carbon fibers, and powders) with short repetitive (or galvanostatic) pulses, from PtCl₂ and Pt(NH₃)₄Cl₂ solutions. The processes were characterized by TEM, STM, and transmission electron microscopy. The nucleation is progressive or instantaneous, depending on potentials and activations. We demonstrate that electrochemical deposition of nanoparticles inside preformed voids in electrodes is one of the most Pt-saving preparation of fuel cell electrodes.

EIGHTH INTERNATIONAL SYMPOSIUM ON MOLTEN SALTS

Physical Electrochemistry/High Temperature Materials

482 On Charging Palladium in an Al/LiCl-KCl Eutectic, Excess LiD/Pd Cell: B. Y. Lau and F.-P. Tao, Natural Energy Institute, School of Ocean Earth Science and Technology, University of Hawaii, Honolulu, HI 96822, S. E. J. Hill, Dept. of Mechanical Engineering, College of Engineering, University of Hawaii, Honolulu, HI 96822

An anomalous heat effect was found during high-current-density charging of an Al/LiCl-KCl eutectic with excess LiD/Pd cell at elevated temperatures. The principle regarding electrochemical and calorimetric behavior of this molten salt approach is presented. The thermochrometical aspects of the reactions at each charging stage are discussed to seek a possible explanation for the anomalous heat effect. We were unable to identify any conclusive chemical nature of the anomalous phenomenon. The phenomenon is quite irreproducible, because of several materials problems and the lack of understanding of the predominant electrochemical reactions during the excess power excursion.

483 Structure of Molten Iron Chloride: D. L. Price and M.-L. Saboungi, Materials Science Div. Argonne National Laboratory, Argonne, IL 60439, S. C. Moss, Dept. of Physics, University of Houston, Houston, TX 77004, S. Hashimoto, Institute for Materials Research, Tohoku University, Sendai 980, Japan

The structure of molten FeCl₃ at 320°C has been measured with neutrons from the intense pulsed neutron source at the National Institute of Standards and Technology. The structure factor exhibits a three-peak structure with a first sharp diffraction peak at wave vector Q = 1.0 A⁻¹. The first peak in the radial distribution function can be fitted by two gaussians centered at 2.17 and 3.2 Å, slightly larger than the two Fe-Cl distances observed for FeCl₃ molecules in the vapor. The results indicate that melting in FeCl₃, associated with a large (63%) volume change, is accompanied by a change in local structure from the octahedral environment of the Fe in the solid FeCl₃ molecular liquid. This work was performed under the auspices of the U. S. Department of Energy, Division of Materials Science, Office of Basic Energy Sciences, under Contract W-31-109-EN-383.

484 Electronic Conduction in Molten KBr·K Solutions: G. M. and M. O. Haacke, Laboratories of Industrial Electrochemistry, University of Trondheim, 7034 Trondheim, Norway, J. E. Jorgensen, Brookhaven National Laboratory, Upton, NY 11973

The electronic conductivity of molten KBr·K solutions was determined as a function of the activity of potassium at various temperatures by using the Wagner polarization technique. The diffusion coefficients obtained from potential step measurements. The concentration of defects in the salt was calculated as a function of the K activity by applying a thermodynamic model.

485 Electronic Polarizabilities of LiCl-CaCl₂, LiCl-KCl and LiCl-CaCl₂-Br: Y. Ikuta, T. Yamauchi, T. Hayami, N. Onoe, K. Senju, T. Shiga, and T. Tsuchiya, Institute of Technology, Faculty of Engineering, Tohoku University, Sendai 980, Japan

Refractive indexes and densities of LiCl-CaCl₂, LiCl-KCl, and LiCl-CaCl₂-Br binary melts have been measured. The electronic polarizabilities of the melts have been derived from the refractive indexes and densities by the Clausius-Mosotti equation. The correlation between the electronic polarizability and the structure of the melts has been examined. We demonstrated that the melts have the positive temperature dependence and those of the binary melts negatively deviate from the additive rule. Factors which affect the electronic polarizabilities of the melts have been investigated.

486 Computer Assisted Data Acquisition and Analysis of Brillouin Spectra of ZnCl₂, Single and ZnCl₂-NaCl Binary Melts: Z. Hongmin, Y. Sato, T. Yamamura, and K. Sugimoto, Dept. of Metallurgy, Faculty of Engineering, Tohoku University, Sendai, Japan

Brillouin scattering experiment has been carried out for single ZnCl₂ and ZnCl₂-NaCl binary melts. The sample was usually refined to avoid the spikes caused by inclusion. Brillouin spectra were obtained at scattering angles of 45°, 90°, and 140°, over full frequency range. The results were compared with those obtained by the molecular dynamics simulation method, indicating the presence of the relaxation of the sound wave propagation. The spectra obtained by a computer assisted retrieval system were analyzed using the basis of the relaxation theory.


A study of the Raman spectra of the soluble magnesium species in melts such as NaCl-KCl-CaCl₂ (41-41-18 mol%) has been carried out. Over the range of 0-25 m/o MgCl₂ in NaCl-KCl-CaCl₂ at 700°C, the intensity of the MgCl₂/MgCl₁/MgCl₀ system was found to vary linearly with MgCl₂ concentration. A new normalization procedure has been developed to generate these results. An all silica fiberoptic probe has also been developed for the in situ measurement of the samples. Measurements Details of these studies are discussed.

488 Development of Raman Spectroscopic Sensors for the Determination of Magnesium in a Molten Salt System: G. Moan and S. D. M. Miller, Department of Chemistry, University of Tennessee, Knoxville, TN 37996-1600, M. G. Began, Chemistry Div., Oak Ridge National Laboratory, Oak Ridge, TN 37831, J. P. Young, Analytical Chemistry Div., Oak Ridge National Laboratory, Oak Ridge, TN 37831, J. E. Coffee, Dept. of Chemistry, University of Tennessee, Knoxville, TN 37996-1600

A study of the Raman spectra of the soluble magnesium species in melts such as NaCl-KCl-CaCl₂ (41-41-18 mol%) has been carried out. Over the range of 0-25 m/o MgCl₂ in NaCl-KCl-CaCl₂ at 700°C, the intensity of the MgCl₂/MgCl₁/MgCl₀ system was found to vary linearly with MgCl₂ concentration. A new normalization procedure has been developed to generate these results. An all silica fiberoptic probe has also been developed for the in situ measurement of the samples. Details of these studies are discussed.

489 Structural Properties of Zinc Halide Melts: E. A. Papanatou and G. N. Papaefthodorou, Institute of Chemical Engineering and High Temperature Chemical Processes and Dept. of Chemical Engineering, University of Patras, GR 261 10 Patras, Greece

Vibrational Raman spectroscopy has been used to identify and determine the structure of species formed in ZnCl₂, ZnBr₂, and in the binary mixtures ZnCl₂-ZnBr₂, ZnCl₂-MgBr₂, and ZnCl₂-NaCl. The data indicate that the network-like structure of the zinc halide melts breaks up with the addition of alkali halide species and with increasing temperature. The tetrahedral structure around the Zn is preserved but one, two, three, or four "terminal" halogen atoms having A as nearest neighbors are formed.

490 Structural Investigation of Molten Lithium Bromide: Y. Ichinokawa, The Nishi-Tokyo University, Ueno, Tokyo, 214, Japan

The structural properties of molten LiBr were investigated by the molecular dynamics simulation method. The temperature dependence of the partial pair distribution functions, the distribution of the coordination numbers, the angular distribution functions were examined at 850, 1000, 1200, and 1500 K. On increasing the temperature, the first peak position and the second peak position of the g(r) of the Li and Br interaction shift to shorter distance and longer distance, respectively.

491 Use of Raman Spectroscopy for Determining the Corrosion Rates of Metals in Molten Fluorides: C. C. Kontoyannis and N. S. Tsakiris, Institute of Chemical Engineering and High Temperature Chemical Processes, GR 261 10 Patras, Greece

Laser Raman spectroscopy was employed in a semi-quantitative way in order to probe the changes caused in the crystal structure of partially and fully stabilized zirconia ceramics after their immersion in the abrasive environment of molten LiF, NaF, KF/FLiNAK eutectic for different periods of time. From the test...
ed ceramics, ZrO₂-8 m/o Y₂O₃ exhibits the best resistance to the corrosive influence of molten FLINAK.

492 Raman and Infrared Spectroscopic Studies of the Platinum Electrode-Molten Nitrate Interface: M. G. Gaphorun, Institute of Physical Chemistry, Academy of Sciences of the USSR, 387003, Makhabatur St., 1/29, Tashkent, USSR, and Inorganic Chemistry, 252601, Kiev, USSR

We present the results of the Raman and reflectance-absorptionspectroscopy (RAIRS) study of the molten lithium, sodium, and potassium nitrates-platinum electrode interface. The composition of the high temperature electrochemical cells which are used for obtain vibrational spectra of interfacial species are described. The variations of the nitrate ion NO₃⁻ internal vibrational species upon change in the electrode potential have been investigated.

493 Selective Precipitation of Oxide Superconductors from Molten Hydroxides and Molten Nitrate: A. M. Stacey, S. L. Stott, L. N. Marquez, and S. W. Keller, Dept. of Chemistry, University of California, Berkeley, CA 94720

Copper and bismuth oxide superconductors generally have been prepared by solid-state reactions at temperatures above 800°C. Unfortunately, there are many disadvantages with the use of solid-state reactions, several of which are especially detrimental to the performance of the oxide superconductors. Here we report the results of oxide superconductors by selective precipitation from molten salts at substantially lower temperatures than have been reported previously. Specifically, La₂CuO₄⁺δ (La₂O₃) and EuBa₂Cu₃O₆⁺δ (EBSCO) were precipitated from molten NaOH and/or KOH at 320 and 450°C, respectively, and Ba₂K₄BiO₀ decreased from molten alka metal nitrates at 260°C.

494 A New Series of Complex Metal Oxides: Crystallization of Al₂O₃ [M₂O₃ (M = Ba, Sr, Mg)] from Hydroxide Melts: V. A. Carlson and A. M. Stacey, Dept. of Chemistry, University of California, Berkeley, CA 94720

Molten hydroxides are excellent solvents for the synthesis of new complex metal oxides. Five new materials with the structure type Al₂(MgO₃) (M = Ba, Sr, Mg) have been crystallized from mixtures of alkali and alkali earth metal hydroxides at 350°C. In this paper we discuss the synthesis method, as well as the crystal structure and properties of these highly oxidized products.

495 Investigation of a Molten Salt Extraction/Electrolysis Process: Converting Uplumbium to a High Grade T0, Feedstock: K. J. Leary, E. J. du Pont de Nemours & Co., Inc., DuPont Chemicals, Iler Research Center, New Johnsonville, TN 37092

The feasibility of using a molten salt extraction/electrolysis process to convert Uplumbium ore to a high grade T0, feedstock is considered. Basic ideas are the recovery of KCl-KOH, LiBO₂, LiCl-KCl-LiBO₂, Na₂CO₃, and Li₂CO₃. It has been demonstrated that iron oxides can be extracted from ilmenite and converted to metallic iron at the cathode using various electrodes with different hole sizes and numbers.

496 New Principles for the Processing of Ceramics in the Domain of Molten Salt Hydrates: H. H. Emons, Central Institute for Inorganic Chemistry, Berlin-Adlershof, Germany

Based on the phase diagram of KCl-MgCl₂-H₂O (up to 250°C) and structural models of molten salt hydrates, new principles for the processing of ceramics are discussed. Basic ideas are the recovery of KCl and MgCl₂. The use of water as a carrier of complex mixtures. Despite the increased temperature range, all stages of the process are realized at pressures below or equal to 0.1 MPa.

497 Molten Carbonate Fuel Cell Reaction Mechanisms: K. Hemmes, R. C. Malatka, R. Wieser, and J. H. W. Wit, Delft University of Technology, Faculty of Chemical Technology and Materials Science, Laboratory of Materials Science, Div. of Corrosion Technology and Electrochemistry, 2823 AL Delft, The Netherlands

An overview is presented of our research on the reaction mechanisms in a molten carbonate fuel cell of the last four years. Chemical composition and impedances were measured on gold, Ni, and Cu flag electrodes in a half-cell setup and on porous anodes and cathodes in a small laboratory-scale fuel cell. The results showed that the composition was varied over a wide range to determine the reaction orders accurately. The research is described in detail in two theses.


The results of MCFC component development and a 70-cell integrated modular high efficiency (IMHEX) subcell stack test are reported. The performance confirmed “stackability” and the absence of carbonate migration in IMHEX® MCFC stacks. Specific power 1000 W/kg was obtained at current density 180 mA/cm² and 75% fuel utilization. The stack operated 1500 h with average power output of 5.5 kW. Maximum stack power exceeded 9.9 kW. A 20 kW full-area (1 m²) stack will be tested in the beginning of 1992.


Electrochemical properties of oxygen species have been studied in molten sodium-potassium-lithium carbonates between 500 and 750°C by means of voltammetry and other electrochemical techniques at gold electrodes. Relative stability of the reduced forms: peroxide and superoxide was examined as a function of the alkaline cations and of the oxygen fugacity. The reactions of alkaline oxides and peroxides was determined. Experimental values are compared with theoretical results calculated from thermodynamic data. The choice of suitable media for applications in catalytic oxidations is discussed.

500 High Temperature Corrosion of Nickel Coated with Molten Alkali Carbonates: A. M. Stacey, K. M. Namiya, Dept. of Energy Engineering, Yokohama National University, 156 Tokiwadai, Hodogaya-ku, Yokohama 240, Japan

The high temperature corrosion of CaO, MgO, and Ni elec- trodes have been investigated in a molten carbonate coating for high temperature corrosion resistant metals. The acceleration oxidation was observed at low pressures CO₂ at 1053°C. The corrosion products were affected by the solubility of NiO that was formed on the metal.

501 Galvanic Corrosion of Coupled Pt-Ni System in Molten Sodium Carbonate: M. Stacey, Dept. of Mechanical Engineering, Hokkaido University, Sapporo 060, Japan

An aluminum-chlorine fuel cell to be used in the production of high-purity aluminum from scrap aluminum is proposed. The output voltage drop due to a chlorine reaction of the fuel cell was investigated in a mixture of MgCl₂ 25 m/o NaCl 75 m/o in CO₂ atmosphere using various electrodes with different hole sizes and numbers. The reaction resistance decreased with the decrease in hole diameter and had minimum values when the hole size was about 3 mm.

502 Development of Cathodes for an Aluminum-Chlorine Fuel Cell in High Temperature Chloride Melt: T. Ishikawa, T. Sasaki, and S. Konda, Dept. of Metallurgical Engineering, Faculty of Engineering, Hokkaido University, Sapporo 060, Japan

A new aluminum-chlorine fuel cell to be used in the production of high-purity aluminum from scrap aluminum is proposed. In CO₂ atmosphere, there was little corrosion. In CO₂, a little mass loss of Ni with NiO formation was observed. Whereas O₂, massgain of Ni anode by oxide formation and severe corrosion of Pt cathode were observed.

503 Application of Molten Salts in Pyrochemical Processing of Reactive Metals: B. Mahr and D. L. Olson, Dept. of Metallurgical and Materials Engineering, Cornell University, Ithaca, NY 14853

The role of molten salts as a media for conducting pyrochemical processes in the production and purification of reactive metals. These processes generate a significant amount of contaminated waste that has to be treated for recycling or disposal. Molten calcium chlorides based salt systems have been used in this work to generate calcium metal for the in situ reduction of reactive metal oxides. The process of the reaction is characterized by the process efficiency to overcome back reactions in the electrolysis cell. However, it has been possible to potential combine the two processes of reduction and electrowinning. Theoretical treatment of the reaction rates has been presented to determine the temperature and current density for the combined process which must be maintained. To carry out the in situ reduction of metal oxides by the electrolysis calcium. The formation and behavior of double layers adjacent to the electrodes has also been analyzed.


Calcium chloride has been investigated as a media for the reduction of plutonium oxide to metal and for the molten salt extraction of americium from aged plutonium metal. To provide data for the optimization of plutonium oxide reduction, the solubility of plutonium metal has been measured in solutions comprised of varying amounts of calcium oxide in calcium chloride. Additionally, recent work has demonstrated the viability of calcium chloride as an electrolyte for plutonium electrowinning.
Voltammetric Study of a Planar Electrode with Super-

meminums Film in Molten Carbonate: G. L. Lee and J. R. Sel-

ogy, Chicago, IL 60616

Oxygen voltammetry with a variable scan window was applied to study the mechanism of oxygen reduction at a flat electrode in molten carbonate. To account for the effect of supermeminums film formation, the model electrode was modified with a side window. The current and linear potential scanning at a wire electrode. Characteristics peaks are observed in the -0.3 to -0.5 V range, which are due to a diffusion reaction (most likely the reduction of superoxide) than that producing CV peaks at -0.1 to -0.3 V.

Material-Deposition Processes in the Separator of Li-Al-


The deposition of Li and Si in the separator layer of Li-Al-

loy/Fe/Si, thermal cells during discharge was studied. The parameters having the most impact included: anode activity, catholyte treatment (fused, unfused, lithiated un lithiated), and ionic composition. The quenched cells were subjected to detailed postmortem examination by optical microscopy to measure the distribution and amount of deposition products in the separator layer. Complementary examination of select cells was also conducted using scanning electron microscopy and energy dispersive spectrometry.

Performance of a Sodium/Selenium(VI) Molten Chloroal-

ume for an Electric Vehicle: M. Matsunaga, K. Morimitsu, G. Mori, S. Obata, T. Kitazaki, and K. Hosokawa, Dept. of Applied Chemistry, Faculty of Engineering, Kyushu University, 1-1 Sensuicho, Tobata, Kitakyushu 804, Japan

The discharging behavior of Ni/Se/Alumina/Se(IV) in a basic AICI3 melt has been studied to develop the new battery for an electric vehicle. This cell shows the quick response to high current pulses even at 160°C. The reversibility of this cell was improved to be 82% at 120°C, 68% at 200°C. The current distribution in the positive current collector is also discussed.

The Modification of Flenmon Membranes for Use in Energy

Generating Devices: D. S. Neuman, Dept. of Chemistry, Bowling Green State University, Bowling Green, OH 43403. S. Li, Dept. of Chemical Engineering, University of Kentucky, Lexington, KY 40506, and J. S. Wilkes, T. Howard, Dept. of Chemistry, Morehead State University, Morehead, KY 40351

Flemion is a perfluorocarboxylated polymer, somewhat similar to Nafion, which allows Na+ ions to pass through it while excluding OH+ and Cl- ions. This membrane was modified by first converting the carboxylic ester to an acyl group and then acylating dibenz-18-crown-6 with the ambient temperature molten salt pyridinium heptachloroaluminate as the Friedel-Crafts catalyst. Crown ether containing Flemion was complexed with LICI forming an electrolyte with a Cl- ion transference number of 0.39 and a Li+ transference number of 0.61.

Molten Salt Regular Mixture Theory Applied to Ion Ex-

change Membranes: K. S. Fortland and S. K. Ratky, Div. of Physical Chemistry, Norwegian Institute of Technology, N-7034 Trondheim-NTN, Norway, T. Okada, Industrial Products Research Institute, 1-1-4 Higashi, Tsukuba, Ibaraki 305, Japan

We have developed a theory for the composition of a molten salt system. For molten salt systems, where the ions are chemically fixed, the ion exchange properties are determined by the ion transference number. Ion exchange membranes are systems where the ion transference number is controlled by the chemistry of the system. Membrane selection is based on the ion transference numbers of the ions present in the solution. The stability of the ion exchange membrane is determined by the ion transference numbers of the ions present in the solution.

Properties of Transported Entropies and Heat of Transfer in AgSO4-LiSO4, A. Grimstedti and S. K. Ratky, Div. of Physical Chemistry, Norwegian Institute of Technology, N-7034 Trondheim-NTN, Norway

New expressions for determination of thermoelectric powers from thermoplot measurements are presented. These expressions are given for the first time and are derived from a general method of determining the transport properties of materials. The expression for the heat of transfer has been derived from a general method of determining the transport properties of materials. The expression for the heat of transfer has been derived from a general method of determining the transport properties of materials.

Nature of Proton in Ambient-Temperature Chloroal-

ume Molten Salts: R. A. Grimstedti, Dept. of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214, and P. C. Trautwein, Frank J. Seiler Research Laboratory, U.S. Air Force Academy CO 80840

Proton is a ubiquitous contaminant in ambient-temperature molten salts composed of mixtures of aluminum chloride and 1-ethyl-3-methylimidazolium chloride (MEIC). FTIR, NMR, and electro-

chemistry have been used to study the nature of proton in these ionic liquids. In oxide-free basic melts (excess ImCl) there exist two forms of proton, HCl and HCl+, in equilibrium, while in acidic melts (excess AICI3) only HCl is present. Implications of this composi-

tion dependent proton speciation with respect to properties of inorganic materials and melt purification are discussed.

The Chemistry of Proton in Ambient-Temperature

Chloroaluminate Molten Salts: C. M. Trudel, Argonne


In basic molten salts composed of aluminum chloride and 1-ethyl-3-methylimidazolium chloride, proton is partitioned between HCl and HCl+. The equilibrium between these two species was studied using 1H and 2H NMR and values for the equilibrium constant at various temperatures and the heat of reaction was obtained. In acidic melts proton is a Bronsted superacid which exists primarily as HCl. 1H NMR and FT-IR were used to show that HCl in the acidic melts exchanges rapidly with the imidazolium cation ring hydrogens at the 4,5 position. These data have been evaluated with respect to changes in the acidic melt composition.

Ionic Equilibria in Ambient Temperature Molten Salts:

J. L. E. Campbell and K. E. Johnson, Dept. of Chemistry, University of Regina, Regina, Saskatchewan, Canada S4R 5B6

ImHCl (0.38 < XImHCl < 0.67) and ImAICI3 (0.33 < XImAICI3 < 0.67) ionic liquids (Im + 1-ethyl-3-methylimidazolium chloride) have been investigated by 200 MHz 1H and 13C NMR spectroscopy. It is believed that the protic species formed upon addition of HCl to these liquids is the hydrogen dichloroalumonic anion. Our investigations, however, indicate the presence of other protic species with different quantities. Molecular HCl does not contribute significantly to the total HCl content of these liquids. The probable identity of the protic species present in acidic chloroaluminate melts has been determined and the equilibrium constants governing the concentrations of protic species in ImHCl and ImAICI3 liquids have been evaluated.

Dual Spin Probe NMR Relaxation Studies of Microdyna-

mics in Chloroaluminate Melts: W. R. Carpenter*, C. E. Keller, and P. A. Shano, Dept. of Chemistry, Wichita State University, Wichita, Kansas 67260, M. Parratt and J. S. Wilkes, Frank S. Seiler Research Laboratory, USAF Academy, CO 80840-6528

The microdynamics of room temperature chloroaluminate melts consisting of 1-methyl-3-ethylimidazolium chloride (MEIC), AICI3, and EtAICI3 have been investigated by NMR relaxation methods over the temperature range of 0-70°C. These studies support the existence of AlCl3 species adsorbed to AICI3, and other complex ions. The dual spin probe method (DSP) has been developed to establish the existence of intersections between EIAICI3 and MEICI and to determine the liquid state quadrupolar coupling constant (QC) for EIAICI3 and AICI3 in various melt compositions. Determination of the QC as a function of melt composition provides information concerning the symmetry of these complex species.

NMR Measurements in Solutions of Dialkylimidazolium

Halogenoionates: S. Takakashi, J. Rathyke, and M.-L. Saboungi, Argonne National Laboratory, Argonne, IL 60439

Measurements of 1H and 13C NMR spectra were carried out on different chloroaluminate melts consisting of AICI3-EMIC solutions ranging from basic to acidic regime. The temperature was varied in order to improve the resolution of 1H spectra and to gain a better understanding of the kinetics of the exchange processes. For each of the 45, 50, 60, 67 m/o AICI3, only one 1H resonance was observed. At 50 m/o AICI3, mixture, only one 1H resonance was observed, while another 1H resonance appeared with increasing AICI3 for acidic melts.

The Electrochemistry of Aluminum and Protons in Room

Temperature Chloroaluminate Molten Salts Buffered with

Sodium Chloride: T. L. Ruchel and J. S. Wilkes, The Frank S. Seile-

r Research Laboratory, United States Air Force Academy, CO 80840-6528

Lewin neutral room temperature chloroaluminate molten salt made by mixing 1-methyl-3-ethylimidazolium chloride (MEIC) and aluminum chloride are being investigated as battery elec-

trolytes because of their wide voltage windows about 4 V. To maintain this voltage window, the electrolyte must be Lewis buffered by NaCl. In this paper we present a detailed investigation of the electrochemistry of NaCl-buffered MEIC/AICI3 melts at 75 and 95 electrodes. Based on the electrolysis and SEM examination of the electrolyte surfaces, it appears that when a melt is buffered with NaCl, it remains almost a single phase electrolyte, but may result in unwanted side reactions in an operating battery.

Reduction Potentials for Lithium and Sodium in Ambient-

Temperature Chloroaluminate Molten Salts: R. T. Carlin

and C. Scodilla-Kelley, Dept. of Chemistry, University of Alaba-

ma, Tuscaloosa, AL 35487

Reduction potentials for lithium and sodium in ambient-
temperature chloroaluminates melts were determined. The negative potentials were used to determine the reduction of sodium and lithium in these melts.
By adding protons to a LiCl buffered neutral AICI3-MEIC (1-methyl-3-ethylimidazolium chloride) molten salt, elemental lithium can be deposited and stripped as a metal. By measuring the open-circuit potential of lithium deposited from melts having different Li+ concentrations, the standard reduction potential for the Li/Li+ couple was found to be (±0.010) V (vs. Al(III)/Al). By employing proton-containing buffered neutral AICI3-DMPIC (1,2-dimethyl-3-propylimidazolium chloride) melts, both lithium and sodium can be deposited and stripped well within the electrochemical window. Lithium and sodium deposits in the DMPIC melt system exhibit open-circuit potentials of ~ +2.106 and -2.107 V, respectively. In situ optical observations confirm the deposition of elemental lithium and sodium which are stable in the melts for several minutes.

518 Physico-Chemical Behavior and Liquid Crystaline Properties of Melon Alkali Metal Alkanolates: T. M. Mirranda* and S. V. Volkov, Institute of General and Inorganic Chemistry, USSR Prospect Palladina, 252680 Kiev-142, Ukraine

The electrochemical behavior, ion liquid phase stability, and 1HNMR spectra of binary monoval alkali metal n-alkanol systems with common anion are discussed in connection with their phase diagrams. As increasing the difference in radii of uncommon cations of the melts it is found: (i) increasing of physico-chemical properties deviation from additivity; (ii) increase of tendency to ion associate formation; and (iii) enhancement of the reciprocal orientational ordering of n-alkanolate anions along their main axes of symmetry. Elongation of the n-alkanolate anion chain gives rise to association and ordering and promotes liquid crystal formation in the salt melts.

519 Low Temperature Molten Salt Electrolyses Based on Quaternary Alkylphosphonium Salts: G. E. Blomgren, and S. D. Jones, Eveready Battery Co., Inc., Technology Laboratory, West Haven, CT 06516.

A new room temperature molten salt comprised of a 1:2 mixture of quaternary alkylphosphonium chloride and aluminum chlorate has been discovered. Dissolution of a room temperature AlCl3 melt was unexpected due to the high melting point of tetraethylphosphonium chloride and the absence of an inversion vi-bration. The melt has good conductivity and also has higher stability toward electrochemical reduction than any previously studied room temperature AlCl3 melt. Aluminum metal can be reversibly plated and stripped from the melt.

520 New, Stable, Ambient-Temperature Molten Salts: E. J. Cooper and J. M. O. Sullivan, IBM, T.J. Watson Research Center, Yorktown Heights, NY 10598

The ambient-temperature organic molten salts are described. They are easily prepared (usually in one step) and consist of alkyl-substituted aromatic heterocyclic cations and triflate or methanesulfonate anions. For example, 1-ethyl-3-methylimidazolium trflate is a 1.0 N salt at -10°C, a 0.5 N electrolyte at 25°C, and loses <2 w/o at 350°C in N2. Interesting differences between triflate and methanesulfonates are discussed. The thermal and chemical stabilities of these materials are discussed and the electrochemical properties of the new salts make them promising electrolytes and antistats.

521 Mixed Chloroborate and Chloroaluminate Room Temperature Molten Salts: R. J. Gale and T. T. DeLong, Dep. of Chemistry, University of Mississippi, University, MS 38677.

Tetrachloroborate salts of n-butylpyridinium or methyl-3-ethylimidazolium chloride are known to be viscous melts at room temperature. It is of interest for battery electrolyte development to see if mixed chloroborate/chloroaluminate systems might be useful for reversible Al deposition, and if mixed complex species of type [AlCl4]3- form, since boron compounds would minimize electrolyte density. Our studies indicate, however, that addition of AlCl3 to a tetrachloroborate melt tends to displace volatilize BCl3, although an excess of the Lewis acid AlCl3 seemed to permit reduction of boron species. Aluminum generally bonds more strongly to chlorine then boron does, and thus Al species are likely to be the stronger Lewis acids.

522 Electrochemistry of 9,10-Anthraquinone in the Presence of Proton and Tetrachloroaluminate in Ambient Temperature Molten Salts: M. T. Carter and H. A. Ostermayer, Dep. of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214

The electrochemistry of 9,10-anthraquinone (AQ) in a basic room-temperature molten salt composed of AlCl3 mixed with 1-ethyl-3-methylimidazolium chloride (ImCl) is described. AQ is reduced via a quasi-reversible two electron transfer to AQ/2AQ? by the general reaction: 

\[ \text{AQ} + 2\text{H}^+ + 2\text{e}^- \rightarrow 2\text{AQ}^2- \]

Addition of proton as imidazolium tetra-n-pentylammonium thiocyanate (ImCI) is described.

523 Electrochemical Reduction of Aromatic Ketones in a Room-Temperature Molten Salt: G. T. Cheek, Dep. of Chemistry, United States Naval Academy, Annapolis, MD 21402-5026

The electrochemical behavior of fluorenol and benzophenone in the aluminum chlorides:1-methyl-3-ethylimidazolium chloride molten salt system has been investigated. The fluoroenol radical anion in the acidic melt produces an anion radical which couples to form the pinacolone. This latter species can exist in two conformations, the energy form undergoing oxygen:abstraction to produce fluoroenol (5-methyl) and fluorenol pinacoline. The benzophenone anion radical is much more stable under these conditions, and products analogous to those found for fluoroenol reduction have been identified.

524 Studies on Characteristics of Room Temperature Molten Salts AlCl3-1-butypyridinium Chloride: N. Koura, K. Ueda, and K. Tachibana, Dept. of Industrial Chemistry, Faculty of Science and Technology, Science University of Tokyo, 2641 Yama-mazaki, Noda 278, Japan

The changes of an Al plate surface, an Al/Polyaniline cell performance, and ion species in the bath with AlCl3 concentration and with temperature were investigate to study the characteristics of the room temperature AlCl3 bath. Reduction of fluorenol (FPe) at 30-30°C and at 42.0-66.7 m/o AlCl3 content. Vis- cosity, conductivity, and EMF of the bath changed at 50 m/o AlCl3.

525 Electrodeposition of Metals from Room-Temperature Chloroaluminate Molten Salts: X. Xu* and C. L. Hussy, Dep. of Chemistry, University of Mississippi, University, MS 38677.

The electrodeposition of lead, silver, and gold was studied in the aluminum chloride:1-methyl-3-ethylimidazolium chloride room temperature molten salt. Evidence for the unusual deposition of silver was found at polycrystalline gold in acidic melt. The electrochemical behavior of silver and glassy carbon in the molten salt examined. Reduction of fluorenol radical is much more stable under these conditions, and products analogous to those found for fluoroenol reduction have been identified.


The electrodeposition of palladium on various electrode surfaces was examined in AlCl3-MEIC molten salts at room temperature. The behavior of palladium electrodeposition was markedly dependent on the mol fraction of the AlCl3 in the molten salts. Shifting from basic to acidic melts, results in a 2.0 V shift in the potential of palladium. Mole fractions from 0.33 to 0.67 AlCl3 were examined. Large nucleation potentials were evident in the basic melts, but not the acidic and neutral melts.

527 Nucleation and Morphology Studies of Aluminum Deposited from an Ambient-Temperature Chloroaluminate Molten Salt: T. P. Carlin, W. Craford, and T. L. Dederer, Dep. of Chemistry, University of Alabama, Tuscaloosa, AL 35487. M. Berach, School of Mines and Energy Development, University of Alabama, Tuscaloosa, AL 35487

Aluminum deposition from AlCl3-MEIC (1-methyl-3-ethylimidazolium chloride) was studied employing an inverted optical microscope to perform in situ optical observations during the deposition process at a 250 μm tungsten microelectrode. Thin, continuous aluminum coatings were produced from a 1:1:1 AlCl3-MEIC molten salt using constant potential deposition at potentials -5 to -3V. At less negative potentials, the deposit displayed a graney appearance. Analysis of chronoanamperograms indicated that the deposition process consisted of instantaneous nucleation followed by diffusion-controlled growth of the three-dimensional centers. Calculated nucleation site densities and grain sizes were in agreement with the optical and electron microscope images.

528 Ionic Conductivity, Thermodynamics and IR-Spectroscopy of Tetra-n-Pentylammonium Thiocyanate-Inorganic Acid System: A. M. Elias and M. E. Elias, Dept. of Chemistry, Lisbon University, Lisbon, Portugal

Electrical conductivities were measured for the binary system tetra-n-pentylammonium thiocyanate (GnHSCN) with benzoic acid in the range of temperatures from 30 to 180°C and theoretical glass transition temperature. The ionic conductivity equation. Electrical conductivities decrease as the acid content increases in the mixtures and the opposite happens to Tg and Td. A partial phase diagram of the system is given. The glass transition melting point compound. The infrared study of the systems, particularly in the range 1025-1150 cm^-1 also gives support to the formation of that organic complex.
A retrospective of the author's tour through the molten salt vineyards is presented. Initial work was performed in molten BaCl₂-BaF₂-BaOCl₂; studies in KCl-LiCl and melt-aided metal salt solutions, at biasafier lower temperatures, were followed by forays into molten nitrates and alkali metal chloroaluminate solutions. At present, work has been extended out at ambient temperature in organochloroaluminates. The work in the chloroaluminate area has been supported by the Air Force Office of Scientific Research. To whom much gratitude is due for EMF measurement is proposed, using an operationally defined method. The procedure is applied to molten fluorides using literature data. We show that EMF measurements with NaF-AlF₃ are incorrect, with results with NaF-AlF₃ have no thermodynamic significance. Data for the system NaF-Al₂O₃-AlF₃ give only the movement of one ion with respect to the others, and t₁₂ is given with F⁻ as a reference.

537 Thermodynamics of Li₃O-Li₂F-CaF₂ Melts: R. G. Reddy, S. T. G. Sampath Kumar, and A. Narayan, Dept. of Chemical and Metallurgical Engineering, University of Nevada, Reno, NV 89557.

Processing of aluminum-lithium alloys by an electrochemical method was investigated. Thermodynamics of solubility of lithium oxide in Li₂O-LiF-CaF₂ melts was measured as a function of temperature and composition: and the solubility of Li₂O was found to increase from 16.6 weight percent (w/o) at 1058 K to 14.8 w/o at 1133 K. The liquidus temperature of 4Li₂F-CaF₂-Li₂O (sat.) melt was determined to be 1094.5 ± 2.5 K. The equilibrium phase and activity of Li₂O in the melts as a function of temperature was determined.

538 Predominance Area Diagram of Niobium Species in Molten LiCl + KCl Eutectic: G. S. Piccard* and P. Bocage, Laboratoire d’Electrochimie Analytique et Appliquée, Unité associée au C.N.R.S. (URA 218), Ecole Polytechnique Supérieure de Chimie de Paris, 75233 Paris Cedex 05, France.

Redox and acidic properties of dissolved niobium chlorides in the molten LiCl + KCl eutectic melt at 450°C have been investigated. Niobium is soluble in the eutectic melt under four oxidation states: 0, III, IV, and V in oxoacidic media. The standard potentials of the corresponding electrochemical systems have been determined vs. the standard chlorine-chloride reference electrode. A new oxobasic medium (lithium oxide containing melt), the niobium(III) melt, has been explored in this study. The results in acidic media leading to the formation of metallic niobium and niobium(III) in very basic media. NbO(s) disproportionates into niobium niobate, and niobium(III) is a product of oxides and oxychlorides have also been determined. An equilibrium potential-potassium dichromate diagram of niobium is given.

539 Thermogravimetric Study of the 800°C Reaction of Zirconia Stabilizing Oxides with SO₃-NaVO₃: R. J. Peterson, Code 6170, Naval Research Laboratory, Washington, DC 20375.

The reaction of the zirconia stabilizing oxides, Y₂O₃, MgO, SrO, and In₂O₃, with molten NaVO₃ at 800°C under SO₃ partial pressures of 10⁵ to 10⁸ atm was studied by thermogravimetry. The difference in tendency for reaction with SO₃-NaVO₃ for the individual oxides could be clearly distinguished. Certain information concerning activity coefficients and other thermodynamic data for the SO₃-Na₂O-V₂O₅ melt system was also revealed.


The mechanism of the reduction of K₂WCl₆ in molten LiCl-KCl eutectic was studied using cyclic voltammetry, chronopotentiometry, and controlled potential coulometry over a temperature range of 400-500°C and a WCl₆ concentration of 0.0005-0.005 mol/liter. A single irreversible wave attributed to the reduction of W(VI) to W(III) was observed. The experimenter indicated the process to be diffusion controlled with n = 1.3 and a ranging from 0.85 to 0.95. W(III) is soluble at 500°C and insoluble, but still electroactive, below 400°C.

541 Electrochemical Behavior of Tantalum in Halide Melts: E. Poljakova and L. Polykova, Institute of Chemistry KSC, Apatity, Murmansk Reg., 184200 USSR.

A comparative study of cathodic and anodic processes during the electrolysis of tantalum containing melts is presented. The voltammetric data indicate that the reduction of Ta(V) in CsCl-KCl-NaCl-TaCl₅ melt includes two steps: Ta(V) → Ta(IV) → Ta(0), whereas CsCl-KCl-NaCl-ZrCl₄ melt, the reduction of Ta(IV) ions. Gravimetric data as well as linear voltammetry demonstrate the appearance of Ta(IV) and Ta(V) chloride complexes during the anodic discharges and these results are confirmed by voltammograms, which distinguishes a chloride-fluoride melt from a chlorinated one is associated with the dissolution of tantalum in the form of fluoride complexes.

Two simple models for the dissolution of dispersed alumina in molten cryolite have been developed. The first model, where the rate of reaction depends on only the surface area of the alumina, the dissolving alumina is regarded as shrinking hard sphere. The second model, development of the first, where a competing reaction surface is supposed to take place which is dependent on the concentration of alumina in the cryolite bath.

543 The Effects of pH and Temperature on the Structure and Properties of Materials (Sn(II) Dicarboxylates): T. A. Bihade, Dept. of Chemical and Polymer Engineering, Lagos State University, P. M.B. 1087, Apapa, Nigeria

Sn(II) salts of dicarboxylic acids (i.e., suberic, sebacic, dodecaneic, isophthalic, propanoylpheric, and terephthalic) were synthesized from aqueous solution by the double decomposi-
tion reaction using various conditions of temperature (20, 50, and 90°C) and pH (6.4, 8.0, and 11.6) and then fused. The molten salts were characterized by the properties of polymeric materials. They exhibited relatively high shear rate and temperature dependent tensile strength and within the polymer/C/Pt composite catalyst layer. Dept. of Biotechnology, University of Lagos, P.M.B. 12254, Lagos, Nigeria.


A needle-type subcutaneous glucose sensor was developed and its performance was tested in diabetic rats. The experimental results in the first few hours of implantation and then remains essentially unchanged for at least 10 days. The sensor output follows glucose closely. A bioassay and histological studies were also employed to evaluate biocompatibility.

545 Permeability of Glucose and Other Neutral Molecules in the Artificial Skin: F. T. Austin and Z. Pan, Dept. of Chemistry, University of Toronto, 80 St. George St., Toronto, Ont., Canada, T1S 2G9.

The permeation of neutral species through Nafion is critical to its application as a protective, selective coating on electrodes in harsh sample environments. Nafion protects glucose electrodes in whole blood, however, both glucose and alcohol readings appeared unstable. To optimize these characteristics, we have compared simplicity, alcohol (PVA) membranes were successfully spin-coated onto the polymer is thus a crucial element in achieving optimal fuel cell performance. Qualitative observations suggest that the surface of these permeated membranes is hydrophobic. Characterization of the surface of several membranes by contact angle measurements and attempted chemical modifications for enhanced hydrophilicity are described.

546 Amperometric Glucose Sensors Based on Glucose Oxidase: A. A. McLeod, J. T. A. E. Davison, Jr., and J. H. Gottesfeld, Electronics Research, Los Alamos National Laboratory, Los Alamos, NM 87545, J. Rushpon, Dept. of Biotechnology, University of Tel Aviv, Tel Aviv, Israel.

Humidification of polymer electrolyte fuel cells is essential to optimal fuel cell performance. Qualitative observations suggest that the use of these transfused membranes is hydrophobic. Characterization of the surface of several membranes by contact angle measurements and attempted chemical modifications for enhanced hydrophilicity are described.


The measurement of alcohol consumption over long time periods is important for monitoring treatment outcome and for research applications. Giner, Inc. has developed a small wearable device that senses ethanol vapor at the skin site and stores several days of data recorded at 2 to 5 min intervals. The sensor is an electrochemical cell that generates a continuous current proportional to ethanol concentration. This paper describes a microchip of electrochemical transdermal ethanol measurement and presents some of the clinical data collected in support of the electrochemical sensor/ recorder development.


The partial pressure of oxygen in blood is measured routinely in medical laboratories. For some time this measurement has been made using variations of the amperometric Clark sensor. Over the years the requirements of commercial blood instruments have become increasingly stringent. The requirements for ease of use and design have increased, while the need for great accuracy and preci-
sion remains. The evolution of the oxygen sensor will be addressed.

549 Moving Sensors from the Journal to the Clinical Lab: Some Real World Considerations: R. W. Mason, Ciba Corning Diagnostics, Medfield, MA 02052.

There is much to be learned from today's many journal publications dealing with sensors. Unfortunately the tendency has been for authors to report some of the science, and not to consider the implementation of that science as successful products for the medical community. This paper presents some of the issues that must be considered in the translation of sensors from research articles to useful clinical laboratory tools.


Structurally firm electrolyte containing hydrophilic polysilain alcohol (PVA) membranes were successfully spin-coated onto p-type SiO, wafers using wet processing techniques. A coupling agent was applied to improve the adhesion between the silicon device and the electrolyte-carrying membrane. The performance of the novel polarographic solid-state oxygen sensors based on this technology is shown in this paper. From the preliminary study we have demonstrated the possibility of mass production of Clark-type oxygen electrode miniaturized sensors.

551 A Thin Platinum Island Film Glucose Sensor: B. Kasap, V. Bokuli, W. C. Hanly, and Y. Macley, Dept. of EECS and Microbiology and Immunology, The University of Illinois, Chicago, IL 60616.

A 25 Å platinum thin film glucose sensor has been developed. The island design of the metal film allowed the immobilization of the glucose oxidase on the SiO, surface in a thin layer adjacent to the Pt film. The glucose oxidase was measured over a frequency range of 100 Hz to 1 MHz. The series resistance component of the cell and equivalent circuit impedance changed by 50% at 100 Hz with 500 mM of glucose. The rate of change in the film impedance at 1 kHz with time exhibited a rapid response.


Light-addressable potentiometric sensors in microvolumes can be used as sensitive immunoassays, and for the rapid measurement of metabolic rates of small numbers of cells. The sensitivity and precision of detection depend on the properties of the pH sensing surface used. To optimize these characteristics, we have compared three types of insulators: Si,N, Ta, and NbO. The best pH response was obtained with TaO, although long-term drift appeared higher than with Si,N, NbO has the highest pH response, but suffers from some drift, presumably caused by current leakage through the insulator.

553 Design, Fabrication and Testing of Flexible Ion Microsen-

Sors for Cardiovascular Applications: R. P. Buck, E. Lind-

ner, and V. V. Conoyet, Dept. of Chemistry, University of North Carolina, Chapel Hill, NC 27599. R. P. Yu, Dept. of Orthodontics and Biomedical Engineering, University of North Carolina, Chapel Hill, NC 27599, T. A. Johnson, School of Medicine, Department of Cardiology, University of North Carolina, Chapel Hill, NC 27599, M. P. Neuman, Dept. of OB/GYN, MetroHealth Medical Center, Cleveland, OH 44109.

Flexible microsensor arrays on Kapton using aminated polysilain chloride) or high molecular weight neutral carriers prove to yield sensors rivaling glass pH sensors. Special redox inorganic reference electrodes prove to be superior to electrodes of the second kind. Results of both in vivo and in vitro testing, and biocompatibility studies show recent advances toward the goal of effective sensors for cardiology and perfusion measurements.

554 Measurement of Guinea-Pigs Heart Intracellular Potas-
i um Concentration with Potassium Microelectrodes: Y. M. Liu and Z. Q. Hung, Dept. of Applied Chemistry, Chongqing University, Chongqing 600044, China, J. S. Xue and S. Z. Yan, Dept. of Physiology, the Third Military Medical College, Chongqing 600044, China.

Potassium ion selective microelectrodes (K-ISE) were used to measure the K' content in myocardial cells of normal and early burn injured guinea-pigs. The results proved that during early burn injury, myocardial cell potassium is decreased, and the intracellular K' concentration also decreases, which suggests that during early burn injury, the decrease of myocardial cellular K' is the most important factors causing the functional reduction of heart-pump.

555 Carrier Based Optodes: K. Seiler and W. Simon, Swiss Fed-

eral Institute of Technology (ETH), Dept. of Organic Chem-

istry, CH-8092 Zurich, Switzerland
New optical chemical sensors (optodes) selective for many different analytes have been realized using plasticized poly(vinyl chloride) membranes. They incorporate specially designed chromionophores allowing the optical transduction together with conventional ion-selective or carrier. Such bulk optode membranes exhibit the theoretically expected response behavior. They have shown practical reliability for the determination of clinically relevant ions in diluted blood plasma samples, as well as several electrically neutral analytes in different media.

558 Towards Reversible Sensors Based on Photochemical-Electrochemical Switching: M. J. Preigh and S. G. Weber. Dept. of Chemistry, University of Pittsburgh, Pittsburgh, PA 15260

The reversible isomerization of photochromic spiropyran and spiro-oxazines potentially provides a means for remote modulation of sensor activity. The ability to switch a sensor photochemically and fix it electrochemically would solve many serious problems concerning sensor reversibility. We have performed the first investigation of spiropyran voltammetry and discuss the electrochemical mechanism as it relates to the photochemical isomerization of spiropyran and the development of reversible metal-ion sensors.


Electrochemical methods have been developed for ultrasensitive quantitation of the enzymes alkaline phosphatase (ALP) and horseradish peroxidase (HRP). The products of each enzyme-catalyzed reaction are coupled to redox mediators that are detected potentiometrically at an inert metal electrode. Quantitation of ALP at pH 10.0 is accomplished via the ALP-catalyzed hydrolysis of 5-bromo-4-chloro-iodophenol to the corresponding iodide; the iodide derivative is oxidized by a tetracazolium mediator, the reduction of which is detected potentiometrically. Quantitation of HRP at pH 5.5 is accomplished via HRP-catalyzed tetramethylbenzidine (TMB) oxidation by hydrogen peroxide; the oxidation product of TMB is reduced by ferrocyanide, the oxidation of which also is detected potentiometrically. In solid phase enzyme-linked immunosassays, enzyme bound to a solid phase may be quantitated with these electrochemical methods. In a small volume detection cell, the assays have extremely high sensitivity and precision. Detection limits of 12,000 molecules of ALP (1.5 attomoles) and 3.8 million molecules of HRP (620 attomoles) were observed. These electrochemical quantitation methods promise to have greater precision compared to optical detection methods; e.g., fluorescence or chemiluminescence, because of the absence of interferences from optically dense substances.

560 Biocatalytic Amperometric Sensors Using a Polymeric Electroactive Membrane: A. C. Michael and M. G. Gargiulo. Dept. of Chemistry. University of Pittsburgh, Pittsburgh, PA 15260

A biocatalytic amperometric sensor for choline can be prepared with a combination of choline oxidase and horseradish peroxidase immobilized onto an electrode surface in a redox polymer gel. Detection limits in the low micromolar range are obtained and the sensors exhibit subsecond response times. Microsensors based on this approach will be suitable for in vivo studies of cholinergic neurotransmission. Incorporation of acetylcholine esterase will allow detection of acetylcholine directly.


Two types of glutamate membrane have been constructed, one by the creation of a membrane sandwich using glutamate oxidase, the second by NMP-TCNQ mediated direct coupling of glutamate oxidase to a carbon electrode. The enzyme electrodes have been shown to be stable, linear up to 15 mM glutamate and capable of long term storage without significant loss of activity. The sandwich membranes have been used in experiments to monitor glutamate on the surface of the rabbit brain cortex after intravenous injection of glutamate.

562 Dehydrogenase-Modified Carbon Fiber Microelectrodes with Millisecond Response Times: W. G. Kuhl and P. Pantoliano. Dept. of Chemistry. University of California, Riverside, CA 92521

Recently we reported the construction of an enzyme-modified carbon-fiber microelectrode that has the size, speed, and sensitivity required for dynamic measurements of in vivo biochemical processes. The covalent attachment of the enzyme to the carbon surface through a hydrophilic tether employing biotin-avidin technology allows the surface coverage of enzyme to be closely controlled and subject to electrochemical and spectroscopic characterization. The biotin avidin "molecular sandwich" permits the enzyme to be closely attached to the carbon surface and the major virtue of the biotin-avidin coupling scheme is that it permits the selectivity of the electrochemical measurement to be easily changed. The possibility for fabricating a series of single-component-selective ultramicroelectrodes with millisecond response times is demonstrated here as the selectivity of the electrode ensemble is changed by incorporating glutamatedehydrogenase into the derivatization procedure.

563 Application of ω-Thiocarbonyl Acid Monolayers for the Detection of Dopamine in the Presence of High Concentration of Acetylcholine: D. Manulis and F. Ham, Dept. of Biophysical and Analytical Chemistry, The Hebrew University of Jerusalem, Jerusalem 91904, Israel

Self-assembled monolayers of ω-thiocarbonyl acids, HS-(CH₂)nCO₂H (n = 2, 5, 10) on gold electrodes were used as a means to induce electrochemical differentiation between a neurotransmitter, dopamine, and ascorbic acid. The optimum differentiation was found for n = 5, and it is attributed to a compromise between a well-organized system (requires to increase n) and a reasonable rate of electron transfer (requires to reduce n).

564 Controlled Binding and Electrorelease of Metal Ions, Drugs, and Neurotransmitters from Polymeric Films Studied by Piezoelectric Sensor Technique: M. Hepp* and L. Dentrone, Dept. of Chemistry. Potsdam College of SUNY, Potsdam, NY 13676

New composite polypyrrole films with cation gating properties containing Adenosine 5'-triphosphate (ATP) or flavin adenine dinucleotide (FAD) have been prepared. The use of the electrochemical polymerization process and interactions of these films with metal ions, drugs, and neurotransmitters. The uptake and release of metal ions, e.g., Ca²⁺, Mg²⁺, Zn²⁺, Cd²⁺, K⁺, Na⁺, neuroleptic drugs (e.g., chlorpromazine, thioridazine) and neurotransmitters (e.g., dopamine) from these composite polymer films have been investigated. We have found considerable differences in ion dynamics for different cations.

565 The Effect of Overoxidation on the Electrochemical Behavior of Poly(pyrrrole) Films Doped With Various Anionic Species: D. Belanger and F. Provencier, Dept. de chimie, Université du Québec à Montréal, Montréal, Québec, Canada H3C 3P9

Poly(pyrrrole) film electrodes doped with various anions and the enzyme glucose oxidase, GOD, were prepared on platinum electrode by oxidative electropolymerization of pyrrrole in an aqueous solution also containing the enzyme and one of the following anionic species: chloride, pentanulafonate, polyvinylsulfate or poly(4-styrenesulfonate). The cyclic voltammetry behavior of all these composite electrodes has been investigated in aqueous 0.1 M KCl and in the same aqueous solution containing electroactive redox species such as Fe(CN)₆³⁻ and hydroquinone. Following overoxidation of these composite electrodes at 1.2 V, the voltammetric responses changed dramatically. The electroactivity of poly(pyrrrole) was lost in all cases. The voltammetric responses of the overoxidized polymer electrodes in the presence of hydroquinone may suggest that the electrochemical oxidation of the latter occurs at a poly(pyrrrole) surface instead of the underlying platinum electrode.

566 Poly(pyrrrole)-Enzyme Film Electrode Mediated by Electropolymerized Catalytic Polymeric Film: H. Tachikawa, Z. Sun, and H. Ge, Dept. of Chemistry, Jackson State University, Jackson, MS 39217-0510

A bilayer thin-film conducting polymer has been prepared on a glassy carbon (GC) electrode by successive electrochemical polymerization of two different polymers: Catalytic polymeric and enzyme polymer. Either a polymethylphthalacidone (PMePC) or a polyelemaporphirn (PMePr) was used as a catalytic polymer and an enzyme (glucose oxidase) doped poly(pyrrrole) film (PPY-GOD) was used as an enzyme polymer. Both the GC/PMePC/PPY-GOD and GC/PMePr/PPY-GOD films show good catalytic behaviors which include a reduced electron potential and an enhanced amperometric response with a substrate.
INDEX TO AUTHORS

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aarnoud, P. K.</td>
<td>Bird, G. D.</td>
<td>Chang, L. N.</td>
</tr>
<tr>
<td>Averill, W. A.</td>
<td>Bisk, V.</td>
<td>Chang, W.</td>
</tr>
<tr>
<td>Abd Al-Aziz, M.</td>
<td>Bjorkman, C. H.</td>
<td>Chang, Y. H.</td>
</tr>
<tr>
<td>Abd El-Rashid, A. A.</td>
<td>Blaschoff, P.</td>
<td>Chapple-Sobol, J. D.</td>
</tr>
<tr>
<td>Abe, H.</td>
<td>Blaugher, R. D.</td>
<td>Charych, D. H.</td>
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<tr>
<td>Adio, R.</td>
<td>Blaustein, P. R.</td>
<td>Chater, R. J.</td>
</tr>
<tr>
<td>Agarwal, R.</td>
<td>Blomgren, O. E.</td>
<td>Chazalviel, J. N.</td>
</tr>
<tr>
<td>Aghazadeh, F.</td>
<td>Boocock, P.</td>
<td>Cheek, G. T.</td>
</tr>
<tr>
<td>Ahmed, S. M.</td>
<td>Boddy, D. M.</td>
<td>Chen, C. C.</td>
</tr>
<tr>
<td>Alattar, H.</td>
<td>Bona, G. L.</td>
<td>Chen, C. H.</td>
</tr>
<tr>
<td>Adriono, B.</td>
<td>Boote, G. R.</td>
<td>Chen, J.</td>
</tr>
<tr>
<td>Asli, N.</td>
<td>Bosch, D. R.</td>
<td>Chen, Q.</td>
</tr>
<tr>
<td>Anderson, E. B.</td>
<td>Bouse, L.</td>
<td>Cheng, T.-P.</td>
</tr>
<tr>
<td>Anderson, H. U.</td>
<td>Brady, F.</td>
<td>Cherne, R. D.</td>
</tr>
<tr>
<td>Angelo, D.</td>
<td>Brank, K.</td>
<td>Chiang, H.-Y.</td>
</tr>
<tr>
<td>Angel, L.</td>
<td>Breiter, M. W.</td>
<td>Cho, H.-C.</td>
</tr>
<tr>
<td>Antnucci, P. L.</td>
<td>Brelyshole, B. E.</td>
<td>Cho, K. H.</td>
</tr>
<tr>
<td>Antuuci, V.</td>
<td>Brina, J.</td>
<td>Cho, K.-C.</td>
</tr>
<tr>
<td>Aranu, S.</td>
<td>Brinkley, C.</td>
<td>Cho, S.-P.</td>
</tr>
<tr>
<td>Arany, A.</td>
<td>Brousseau, R.</td>
<td>Chonko, M.</td>
</tr>
<tr>
<td>Aplinsky, A. J.</td>
<td>Brown, C. J.</td>
<td>Chou, P. B.</td>
</tr>
<tr>
<td>Apert, D.</td>
<td>Brown, D. B.</td>
<td>Choy, A. J.</td>
</tr>
<tr>
<td>Arai, H.</td>
<td>Brown, G. A.</td>
<td>Chung, B. C.</td>
</tr>
<tr>
<td>Arce, T.</td>
<td>Brunet, T. R.</td>
<td>Chung, W.-J.</td>
</tr>
<tr>
<td>Arce, T.</td>
<td>Bryant, T. C.</td>
<td>Clancy, C.</td>
</tr>
<tr>
<td>Arico, A. S.</td>
<td>Buchanan, D. A.</td>
<td>Clarev, J.</td>
</tr>
<tr>
<td>Arrarong, O. A.</td>
<td>Buchanan, P.</td>
<td>Clark, D. E.</td>
</tr>
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<td>Arrald, D.</td>
<td>Buck, R. P.</td>
<td>Clark, L. C., Jr.</td>
</tr>
<tr>
<td>Atchison, S. N.</td>
<td>Buckley, A. N.</td>
<td>Clegern, J. S. C.</td>
</tr>
<tr>
<td>Augustynski, J.</td>
<td>Buckley, D. T.</td>
<td>Cowe, P. J.</td>
</tr>
<tr>
<td>Av, C. Y.</td>
<td>Buczekowski, A.</td>
<td>Coblenz, D. H.</td>
</tr>
<tr>
<td>Av, E.</td>
<td>Buitr, J. P.</td>
<td>Coburn, J. W.</td>
</tr>
<tr>
<td>Awazu, K.</td>
<td>Burke, L. D.</td>
<td>Coffey, B. M.</td>
</tr>
<tr>
<td>Ade, K. M.</td>
<td>Buzzo, D.</td>
<td>Coffield, J. E.</td>
</tr>
<tr>
<td>Aydil, E.</td>
<td>Cahu, D.</td>
<td>Collins, R. W.</td>
</tr>
<tr>
<td>Bai, Y. H.</td>
<td>Caheo, N.</td>
<td>Colmbrinck, M. E.</td>
</tr>
<tr>
<td>Bagley, M. R.</td>
<td>Casa, E. J.</td>
<td>Comminello, Ch.</td>
</tr>
<tr>
<td>Banerjee, S.</td>
<td>Campabadal, F.</td>
<td>Comway, B. E.</td>
</tr>
<tr>
<td>Bansch, R.</td>
<td>Campbell, J. E. E.</td>
<td>Cooper, E. I.</td>
</tr>
<tr>
<td>Baptista, W.</td>
<td>Cancio, G.</td>
<td>Cordis, B.</td>
</tr>
<tr>
<td>Barns, L. A.</td>
<td>Casado, C. A.</td>
<td>Cosseret, V. V.</td>
</tr>
<tr>
<td>Barley, D. P.</td>
<td>Canetti, R.</td>
<td>Costantino, R.</td>
</tr>
<tr>
<td>Brenner, G.</td>
<td>Cappelli, M. A.</td>
<td>Cote, D.</td>
</tr>
<tr>
<td>Barnett, S. A.</td>
<td>Caputo, E.</td>
<td>Couty, L. A.</td>
</tr>
<tr>
<td>Basioso, C. I.</td>
<td>Cardinal, C.</td>
<td>Couture, E. C.</td>
</tr>
<tr>
<td>Basioso, C. S.</td>
<td>Cerwinska, M.</td>
<td>Couture, Y.</td>
</tr>
<tr>
<td>Batan, G.</td>
<td>Cecile, R. N.</td>
<td>Cowan, D. O.</td>
</tr>
<tr>
<td>Beck, E.</td>
<td>Carelin, R. T.</td>
<td>Cox, J. N.</td>
</tr>
<tr>
<td>Becker, S. E.</td>
<td>Carlson, W. E.</td>
<td>Crawford, W.</td>
</tr>
<tr>
<td>Beke, E. D.</td>
<td>Carlson, W.</td>
<td>Creager, S.</td>
</tr>
<tr>
<td>Beggs, G. H.</td>
<td>Carter, J.</td>
<td>Crastoboavas, S.</td>
</tr>
<tr>
<td>Beil, W. K.</td>
<td>Cartwright, V.</td>
<td>Daubenspeck, T. H.</td>
</tr>
<tr>
<td>Beke, D.</td>
<td>Caruth, T. O.</td>
<td>Daughtery, J. E.</td>
</tr>
<tr>
<td>Beke, D.</td>
<td>Carb, M.</td>
<td>Davies, J. T.</td>
</tr>
<tr>
<td>Bellanger, D.</td>
<td>Carper, W. R.</td>
<td>Day, M. E.</td>
</tr>
<tr>
<td>Benedict, M. K.</td>
<td>Carrabba, M. M.</td>
<td>Dedeker, A.</td>
</tr>
<tr>
<td>Benson, D. K.</td>
<td>Carter, M. T.</td>
<td>Dahn, C. E.</td>
</tr>
<tr>
<td>Berger, P. R.</td>
<td>Carter, E.</td>
<td>Dalai, S.</td>
</tr>
<tr>
<td>Benich, M.</td>
<td>Case, 4, 36</td>
<td>Dane, D.</td>
</tr>
<tr>
<td>Bertocoti, U.</td>
<td>Case, 327</td>
<td>Danner, D. A.</td>
</tr>
<tr>
<td>Bertocotti, S.</td>
<td>Case, 499</td>
<td>Dante, J. P.</td>
</tr>
<tr>
<td>Besmano, T. M.</td>
<td>Case, 398</td>
<td>Dumas, V.</td>
</tr>
<tr>
<td>Bewick, A.</td>
<td>Cemini, G.</td>
<td>Dutwyler, K.</td>
</tr>
<tr>
<td>Beyer, K. D.</td>
<td>Che, O.</td>
<td>Daubenspeck, T. H.</td>
</tr>
<tr>
<td>Blatt, D. P.</td>
<td>Chakravorty, G. S.</td>
<td>Daughters, J. T.</td>
</tr>
<tr>
<td>Bhattacharyya, R. N.</td>
<td>Chan, K. C. B.</td>
<td>Davies, J. T.</td>
</tr>
<tr>
<td>Birag, M.</td>
<td>Chan, Y.-Y.</td>
<td>Day, M. E.</td>
</tr>
<tr>
<td>Bird, G. D.</td>
<td>Chang, 285, 474</td>
<td>Dedeker, A.</td>
</tr>
<tr>
<td>Bisk, V.</td>
<td>Chang, 247</td>
<td>Dahn, C. E.</td>
</tr>
<tr>
<td>Bjorkman, C. H.</td>
<td>Chang, 163</td>
<td>Dalai, S.</td>
</tr>
<tr>
<td>Blaschoff, P.</td>
<td>Chang, 125</td>
<td>Dante, J. P.</td>
</tr>
<tr>
<td>Blaugher, R. D.</td>
<td>Chang, 150</td>
<td>Dumas, V.</td>
</tr>
<tr>
<td>Blaustein, P. R.</td>
<td>Chang, 190</td>
<td>Dutwyler, K.</td>
</tr>
<tr>
<td>Blomgren, O. E.</td>
<td>Chang, 283</td>
<td>Daubenspeck, T. H.</td>
</tr>
<tr>
<td>Boocock, P.</td>
<td>Chang, 311</td>
<td>Daughters, J. T.</td>
</tr>
<tr>
<td>Boddy, D. M.</td>
<td>Chang, 523</td>
<td>Davies, J. T.</td>
</tr>
<tr>
<td>Bona, G. L.</td>
<td>Chen, 124</td>
<td>Day, M. E.</td>
</tr>
<tr>
<td>-------------</td>
<td>-------------</td>
<td>-------------</td>
</tr>
<tr>
<td>D'Cruz, L. A.</td>
<td>Panghaneh, T.</td>
<td>Giordanò, N.</td>
</tr>
<tr>
<td>Dean, P. W. H.</td>
<td>Fairley, T. J.</td>
<td>Giraudet, L.</td>
</tr>
<tr>
<td>Deki, S.</td>
<td>Farrell, R.</td>
<td>Giswizer, E.</td>
</tr>
<tr>
<td>Delarue, S. L.</td>
<td>Faith, Z.</td>
<td>Gleason, E.</td>
</tr>
<tr>
<td>Delaurier, E.</td>
<td>Fechner, P.</td>
<td>Gladie, A.</td>
</tr>
<tr>
<td>Delano, M.</td>
<td>Fedick, P.</td>
<td>Goodenough, J. B.</td>
</tr>
<tr>
<td>Delong, H. G.</td>
<td>Feijoo, D.</td>
<td>Gopal, J.</td>
</tr>
<tr>
<td>de Lourdes, M.</td>
<td>Feinman, D.</td>
<td>Gorrarea, B. A.</td>
</tr>
<tr>
<td>den Boer, J. W. H.</td>
<td>Felker, B. S.</td>
<td>Gorkens, A.</td>
</tr>
<tr>
<td>Deng, W.</td>
<td>Ferreia, A. C.</td>
<td>Gosele, U.</td>
</tr>
<tr>
<td>Destrade, L.</td>
<td>Figueredo, D.</td>
<td>Goto, K.</td>
</tr>
<tr>
<td>DePooter, G. L.</td>
<td>Finch, J. A.</td>
<td>Gottersfeld, S.</td>
</tr>
<tr>
<td>Depeula, A.</td>
<td>Fish, J.</td>
<td>Goukouk, J.</td>
</tr>
<tr>
<td>de Rooy, N. P.</td>
<td>Fisler, G. L.</td>
<td>Gratzel, M.</td>
</tr>
<tr>
<td>DeRuijter, W. J.</td>
<td>Fitzgibbon, G.</td>
<td>Graves, D. B.</td>
</tr>
<tr>
<td>de Taconi, V. R.</td>
<td>Fleischer, N. A.</td>
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<td>Donelon, R.</td>
<td>138, 139</td>
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<td>14</td>
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<td>123</td>
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<td>154</td>
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<td>278</td>
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<td>246</td>
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<td>114</td>
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<td>192</td>
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<td>481</td>
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<td>199</td>
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<td>Dutton, M. C.</td>
<td>408, 409, 410</td>
<td>Hash, M. C.</td>
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<td>405</td>
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<td>64</td>
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<td>158</td>
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<td>265, 268</td>
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<td>261</td>
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<td>175</td>
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<td>179, 186</td>
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<td>95, 96</td>
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<td>285</td>
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<td>308</td>
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<td>206</td>
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<td>254</td>
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<td>193</td>
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<td>73</td>
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<td>363</td>
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<td>218</td>
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<td>275</td>
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<td>135, 141</td>
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<td>302</td>
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<td>46</td>
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<tr>
<td>Simon, E.</td>
<td>161, 165</td>
<td>Tamil Selvan, S.</td>
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<td>555</td>
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<td>418, 425, 473</td>
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<td>146, 209</td>
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<td>552</td>
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<td>293</td>
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<td>299, 303</td>
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<td>301, 306</td>
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<td>304, 305, 336</td>
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<td>485, 496</td>
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<td>274</td>
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<td>45, 445</td>
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<td>Woods, R.</td>
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<td>Wright, M.</td>
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<td>Wu, T. H.</td>
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</tbody>
</table>

Xiao, J. S. ........................................ 122
Xie, S. F. ........................................ 34
Xu, J. ........................................... 34
Xu, M. ........................................... 34

Yablonovich, E. .................................... 197
FUTURE SOCIETY MEETINGS

182nd MEETING—TORONTO, ONTARIO, CANADA—
OCTOBER 11-16, 1992—HEADQUARTERS AT THE ROYAL YORK HOTEL

The final program will be published in the August 1992 issue of the Journal of The Electrochemical Society.


HONOLULU, HAWAII—MAY 16-21, 1993—HEADQUARTERS AT THE HILTON HAWAIIAN VILLAGE

183rd Meeting of The Electrochemical Society, Inc. Cosponsored by The Electrochemical Society of Japan and with the cooperation of The Japan Society of Applied Physics

The final program will be published in the March 1993 issue of the Journal of The Electrochemical Society.

EXTENDED DEADLINE SYMPOSIA

STATE-OF-THE-ART PROGRAM ON COMPOUND SEMICONDUCTORS XVI
• Sponsored by The Electronics/Dielectric Science and Technology Divisions
  Abstracts 565 SOA - 588 SOA

QUANTUM CONFINEMENT
• Sponsored by The New Technology Subcommittee/Electronics/Dielectric
  Science and Technology Divisions
  Abstracts 589 QUA - 595 QUA

MICROMACHINING AND MICROSTRUCTURES
• Sponsored by The New Technology Subcommittee/Sensor Group/Electronics/Dielectric
  Science and Technology Divisions
  Abstracts 596 MIC - 612 MIC

FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS
• Sponsored by The Physical Electrochemistry/Dielectric Science and
  Technology/Electronics Divisions
  Abstracts 613 FUL - 697 FUL

JOINT RECENT NEWS PAPER SESSION
• Sponsored by The Electronics/Dielectric Science and Technology Divisions
  Abstracts 698 RNP - 709 RNP
STATE-OF-THE-ART PROGRAM ON COMPOUND SEMICONDUCTORS
(SOTAPCONS XVI)
Electronics/Dielectric Science and Technology

TUESDAY, MAY 19, 1992
V. Swaminathan, Chairman; N. Buckley, Vice-Chairman
2:00 OEIC: Technological Compatibility: Some Examples for Photoreceiver Applications - D. Decoster
2:40 Technological Aspects of Prime Importance for Performances of Microwave Devices - M. Francois and J. Vanmaeckem
3:20 Novel InGaAs/InPAs Graded Superlattice Channels \(0.2 \leq y \leq 0.4\) for Pseudomorphic AlGaAs, InGaAs, As HET - J. Kraua, H. Meschede, O. Liu, W. Prost, and F. J. Tegude
3:40 Frequency Resolved Admittance Measurements on InAlAs/InGaAs/InAlAs Single Quantum Wells Applied to Determine the Conduction Band Offset and the Capture Time Constant - J. M. Lopez-Villegas, P. Roura, J. Bosch, J. R. Morante, A. Georgakilas, and K. Zekentes
565 SOA
566 SOA
557 SOA
558 SOA

WEDNESDAY, MAY 20, 1992
T. Kamiuji, Chairman; J. P. Vilcot, Vice-Chairman
9:00 Material and Process Considerations in the Manufacture of GaAs Electronic Devices - C. L. Reynolds
10:00 Atomic Layer Epitaxy Growth and Characterization of InP/GaAs - L. Lazzarini, D. Bertone, P. Franzosi, C. E. Norman, and G. Salvatii
10:20 Ten-minute intermission
10:30 Plasma Assisted Epitaxy of Compound Semiconductors - T. Haniu
11:10 Low Pressure MOVPE of GainP/GaAs Heterojunctions: Growth Intermittency Studies Using EDI and TEQ - K. Chou, B. Pathangey, and T. Anderson
11:30 The Effect of the Sulfur Partial Pressure on the Growth of CuInS, Single Crystals - M. L. Fearheiley, M. Kani, and S. Fiechter
11:50 Thermal Decomposition of Copper Acetylide Studied by Using Fourier Transform Infrared Spectroscopy - Y. Chang
569 SOA
570 SOA
571 SOA
572 SOA
573 SOA
574 SOA
575 SOA

WEDNESDAY, MAY 20, 1992
G. Valco, Chairman; A. Katz, Vice-Chairman
2:00 Gas-source MBE of (Ga,Al)As Using Only Gaseous Sources for HBT Applications - T. Fujii, H. Ando, A. Sandhu, N. Okamoto, S. Yamaura, T. Takahashi, and N. Yokoyama
3:20 Ten-minute intermission
3:30 Surface Modification and Stabilization in GaAs and InP - J. Shirafuji and T. Sugino
4:10 Achievement of the Theoretical Resolution of Electron Beam Lithography on Thick GaAs Substrates - S. A. Dickey, T. McCormick, and A. Majerfeld
4:30 Speculation on the Effects of the Interaction Between HCl and HNO, on the Etching of Illuminated n-GaP in HCl/HNO, - H. F. Hsieh and H. C. Shih
576 SOA
577 SOA
578 SOA
579 SOA
580 SOA
581 SOA

THURSDAY, MAY 21, 1992
C. L. Reynolds, Chairman; D. Decoster, Vice-Chairman
9:00 Wafer Level Characterization of InP and GaAs Based Devices - G. E. Carver and R. W. Heebner
10:00 Very High Quality GaAs/AlGaAs Multiple Quantum Well Structures Grown by Atmospheric Pressure MOVPE - E. Mao, Z. H. Lu, B. W. Kim, T. McCormick, E. G. Oh, and A. Majerfeld
10:20 Ten-minute intermission
10:30 AlGaAs/GaAs Lattice Parameter Measurement by High Resolution X-Ray Diffraction - C. Ferrari, C. Bocchi, P. Franzosi, A. Bosacchi, and S. Franchi
11:10 Structural Characterization by TEM of Strained InGaAs/InAlAs Quantum Well Structures - F. Piero, A. Cornet, J. R. Morante, A. Georgakilas, and G. Halkias
582 SOA
583 SOA
584 SOA
585 SOA
586 SOA
587 SOA
588 SOA

In this paper we discuss several technological aspects which are critical for the performance of field effect transistors and other microwave structures. We focus on the use of pseudomorphic heterostructures for the fabrication of GaAs field effect transistors (GFETs) and other microwave devices.

657 SOA Novel InXGa1-XAs Superlattice Channel FETs. S. Chaudhuri, C. W. Woo, and K. L. Chopra. AT&T Bell Labs, Murray Hill, NJ 07974.

We demonstrate the feasibility of a novel InGaAs superlattice channel field effect transistor. The superlattice structure is designed to enhance the mobility of the electron channel while suppressing surface roughness. Initial results indicate that the device exhibits high gain and high cutoff frequency.

658 SOA Frequency Resolved Admittance Measurements on InAlAs/InGaAs/AlGaAs Single Quantum Wells Applied to Determine the Conduction Band Offset and the Capture Time of the Donor States. A. V. Schubert, J. W. Schaff, and A. C. Gossard. AT&T Bell Labs, Murray Hill, NJ 07974.

The frequency resolved admittance measurements have been used to determine the conduction band offset and the capture time of the donor states in InAlAs/InGaAs/AlGaAs single quantum wells. The results indicate that the conduction band offset is strongly influenced by the thickness of the well.

* An asterisk by a name indicates which author will present the paper.
Fourier University.

troduction: 49a has been developed for low-temperature epitaxial growth of semiconductors including surface cleaning of substrates in view of its fundamental advantage that it provides an enhanced surface for enhanced migration over the growing surface can be both given to supplied species in plasma. Design consideration of growth mechanisms including optical emission spectroscopy to detect active species and the results of low-temperature epitaxial growth of compound semiconductors (III-V and II-VI) mainly on GaAs and Si are reviewed. Hydrogen plasma treatment has been found to be effective in removing native oxide at a lower temperature of GaAs (~270°C) and Si (~450°C). The successful epitaxial growth of AlGaInP on InP, GaAs/P, GaAs, and InGaAs has been achieved at lower temperatures than the growth without plasma. It is also shown that such growth conditions as RF power applied to plasma and supply ratio V/III or VI/II should be optimized because the electronic property and surface morphology depend much more critically upon these parameters. Under growth conditions resulting from the incorporation of content. Cross-section transmission electron microscopy revealed that one of the important advantages of PAE is the enhanced chemical reactivity of group V elements by plasma cracking of their molecules supplied by evaporation into excited atoms.


A wide range of nanostructures devices as optical lysis is possible with high speed capabilites are currently being grown by molecular beam epitaxy (MBE) and organometallic vapor phase epitaxy (OVPE). The performance of such devices depends on the quality of the interfaces between different compound semiconductors. Atomic scale structural disorder at the growth surface results in compositional variations that result in the poor device performance. This problem can be alleviated by scheduling growth interruptions to give interface stabilization for all devices. Growth compositions of GaInP/GaAs heterojunctions require simultaneously switching the arsenic, phosphorus and indium supply to the interface. Single and multiple quantum well structures have been achieved using both MBE and OVPE techniques. InGaAs/GaAs equal and variable well sizes are grown in a low pressure OVP/PE reactor. The influence of varying the duration of growth interruptions on the interfacial properties and composition was examined using the ex situ characterization techniques: high resolution x-ray diffraction (XRD), transmission electron microscopy (TEM), and energy dispersive x-ray spectroscopy (EDS). The growth conditions for each interruption were found to contain regions of altered growth compositions resulting from the incorporation of phosphorus and indium in GaAs layers. The optical thicknesses of interruptions in GaInP-to-GaAs and GaAs-to-GaInP interfaces have been found to vary with the growth temperatures as well as the ratio of the well layers.


The chalcogenide CuInS2 has an optimum bandgap for the direct conversion of solar energy. However, the optimization of this material has not progressed very rapidly. One reason is due to the fact that the growth from stoichiometric melts of large, high quality single crystals of CuInS2 have been inhibited by the presence of two solid-state phase transformations which induce cracking of the crystal. Currently we have developed a technique to produce single crystals of moderate size with the gradient freeze technique, but only under elevated pressures. To determine the optimum growth conditions for the growth of CuInS2, different vapor pressures of sulfur have been used with and without argon overpressures. We discuss the best conditions for single crystal growth of the material prepared by EDX, Hall measurements, and photoluminescence, with growth conditions is presented.

575 SOA Thermal Decomposition of Copper (acetylacetonate) Studied by Using Fourier Transform Infrared Spectroscopy. Y. Chang, Dept. of Chemical Engineering, Iowa State University, Ames, IA 50011

Thermal decomposition phenomena of copper (acetylacetonate) in the copper oxide metal-organic chemical vapor deposition (MOVCD)process was studied by gas phase transmission Fourier transform infrared spectroscopy (FTIR). differential scan-
More improvement of Schottky barrier contacts and insulator-doped semiconductor interface layers for bulk devices have been achieved. This paper describes effects of PH, (phosphine) plasma treatment on the surface properties of GaAs and InP, including partly H2 plasma and UV excited laser induced PL excited treatment effects. The PH, plasma treatment makes us expect the following various effects: (i) Atomic hydrogen removes native oxide of GaAs or InP surface. (ii) Phosphorus atom substitutes P native near the surface of GaAs. This may reduce elemental arsenic formation at the surface. (iii) Phosphorus atom fills As- and P-vacancies, if existing, near the surface of GaAs and InP, respectively. Preferential etching of phosphorus in the course of plasma processes can be suppressed in InP. (iv) Amorphous phosphorus layer which can be used as an interlayer or a passivation layer is deposited by adjusting the plasma temperature. Phosphidization of GaAs surface by PH3 plasma treatment reduces generation of arsenic oxide and free arsenic diffusion. (v) This method is suggested by the observation of the metal work function dependence of Schottky barrier height. Generation of EL2 defects which are induced during Ar or H2 plasma process is found to be suppressed in the case of PH3 plasma due to the existence of excess phosphorus. A visual inspection reveals reduced preferential etching and hole density in the PH3 plasma treatment in comparison with H2 or Ar plasma. The effective suppression of surface damage in InP during plasma process is demonstrated clearly by DLTS measurement of charged and neutral defects. (vi) Removal of phosphorus from the surface of GaAs and InP was found to be caused by the treatment, and Se vapor passivation shows a much improved dependence of the Schottky barrier height on the metal work function in InP.

580 SOA Achievement of the Theoretical Resolution of Electron Beam Lithography on Thick GaAs Substrates: S. A. Dickey, T. McCormick, and A. Mayerfeld, Dept. of Electrical and Computer Engineering, University of Colorado, Boulder, CO 80309

We present E-beam lithography experiments performed with an atomic resolution scanning transmission electron microscope (STEM) that show that the proposed resolution limit of 10 nm for PMMA resist on thick substrates (1) can be realized. This limit is independent of beam spot size or electron beam energy. We found that by aperturing experiments a spot size of 2.6 nm and beam energy of 200 keV were used. It is demonstrated that clean 11.5 nm lines can be defined in single-layer PMMA on thick GaAs substrates. We also observed that SEM imaging allowed accurate direct measurement of the written features in the resist at 200 keV. The dependence of feature size on dose was also established. We also observed that the spatial distributions of bulk defect density and localized radiation damage depend on the nature of the defect, the device design, and the location of the defects within the device. Wafer level maps in semi-insulating GaAs wafers have been spatially registered to regions designed to become the gates of FETs. Maps in quantum well InP wafers must be registered to areas that will form the mesas in semiconductor laser structures.

581 SOA Speculation on the Effects of the Interaction Between HCl and HNO3 on the Echting of Illuminated n-GaP in HCl/HNO3: H. F. Hsiao and H. C. Shih, Dept. of Materials Science and Engineering, National Tsing Hua University, Hsinchu, 30043, Taiwan, China

Some interesting phenomena were found in illuminated n-GaP in the mixture of concentrated HCl/HNO3 solutions. No trace of the peaks products has been detected by inductively coupled plasma emission spectrometry in the etching solution which was prepared by dissolving the most concentrated HCl and HNO3, in the adequate amount of pure water, followed by a uniform mixture. This method is based on the exact knowledge of the semiconductor laser parameters and on the application of Vegard's law. Unfortunately the composition measurement in the GaAs/GaAs system is difficult because of the small lattice parameter difference between AlAs and GaAs and the poor accuracy of the AlAs parameter values reported in the literature; some of them being obtained from powders. This work proposes a method, based on the exact knowledge of the semiconductor laser parameters and on the application of Vegard's law, for the determination of the composition of the GaAs/GaAs system.

582 SOA Wafer Level Characterization of InP and GaAs Based Devices: G. E. Cameron and R. W. Heebner, AT&T Bell Laboratories, Murray Hill, NJ 07974

Micron-sized defects in GaAs and InP wafers have been detected via spatially resolved photoluminescence (SRPL). Contrast levels in SRPL scans are due to local variations in electro-optical and/or recombination rates. Optical contrast is interpreted by analyzing the composition and structure of scanned areas with deep electronic techniques such as TEM, SIMS, and chemical etching. SRPL scans are displayed on a video monitor with a 250 by 250 micron field of view. This limited field size allows for the observation of defects in the bulk of the wafers. A very good fit between the experimental and simulated profiles could be obtained with an InAs laser parameter of 568203 Å and a Poisson ratio of 0.286. Broadening of the SRPL scans is due to the presence of dislocations, stacking faults, and other defects. The spatial distribution of bulk defect density and localized polishing damage. The impact of defects on device performance depends on the nature of the defects and InP necessitates the development of surface modification and stabilization techniques. Much interest is also paid on suppressing surface damage during various plasma processes such as reactive-ion etching. This paper describes effects of PH3 (phosphine) plasma treatment on the surface properties of GaAs and InP, including partly H2 plasma and UV excited laser induced PL excited treatment effects. The PH3 plasma treatment makes us expect the following various effects: (i) Atomic hydrogen removes native oxide of GaAs or InP surface. (ii) Phosphorus atom substitutes P native near the surface of GaAs. This may reduce elemental arsenic formation at the surface. (iii) Phosphorus atom fills As- and P-vacancies, if existing, near the surface of GaAs and InP, respectively. Preferential etching of phosphorus in the course of plasma processes can be suppressed in InP. (iv) Amorphous phosphorus layer which can be used as an interlayer or a passivation layer is deposited by adjusting the plasma temperature. Phosphidization of GaAs surface by PH3 plasma treatment reduces generation of arsenic oxide and free arsenic diffusion. (v) This method is suggested by the observation of the metal work function dependence of Schottky barrier height. Generation of EL2 defects which are induced during Ar or H2 plasma process is found to be suppressed in the case of PH3 plasma due to the existence of excess phosphorus. A visual inspection reveals reduced preferential etching and hole density in the PH3 plasma treatment in comparison with H2 or Ar plasma. The effective suppression of surface damage in InP during plasma process is demonstrated clearly by DLTS measurement of charged and neutral defects. (vi) Removal of phosphorus from the surface of GaAs and InP was found to be caused by the treatment, and Se vapor passivation shows a much improved dependence of the Schottky barrier height on the metal work function in InP.


We show an essentially nondestructive method of using low temperature photoluminescence (PL) spectroscopy for the evaluation of n-p heterojunction bipolar transistor (HBT) wafers. The hole density in the base of carbon doped HBT structures was determined from the PL emission peak associated with the p'-GaAs valence state temperature photoluminescence (PL) and intersubband infrared absorption data. This study proves that MQW structures with wells as thin as 14 Å can be grown by the MOVPE process having layer to layer thickness uniformity, inter-layer roughness and heterojunction abruptness of only one monolayer.

584 SOA Very High Quality AlAs/GaAs Multiple Quantum Well Structures Grown by Atmospheric Pressure MOVPE: S. Anderson, T. Miao, Z. H. Lu, B. W. Kim, T. G. Oh, and A. Mayerfeld, Dept. of Electrical and Computer Engineering, University of Colorado, Boulder, CO 80309

We demonstrate, for the first time, that AlAs/GaAs multiple quantum well (MQW) structures grown by the atmospheric pressure metalorganic vapor-phase epitaxy (MOVPE) process have state-of-the-art structural, electrical and optical properties. The 50-well MQW structures, with well thicknesses ranging from 14 to 90 Å, were grown at 720°C with trimethylgallium, trimethylaluminum, and arsine as sources. The structural properties were monitored by atomic resolution transmission electron microscopy (TEM), photoluminescence (PL), and deep level transient spectroscopy (DLTS) techniques. A theoretical model including both bound and virtual states in the MQWs was developed to correlate the TEM lattice imaging observations with the interband 10 K PL spectrum and interband infrared absorption data. This study proves that MQW structures with wells as thin as 14 Å can be grown by the MOVPE process having layer to layer thickness uniformity, interlayer roughness and heterojunction abruptness of only one monolayer.

585 SOA AlAs/GaAs Lattice Parameter Measurement by High Resolution X-Ray Diffraction: C. Ferrari, C. Bocchi, F. Franzoni, A. Rozacchi, and S. Franchi, C.N.R.-MASPEC Institute, Parma, Italy, and C.N.R.-MASPEC Institute, Parma, Italy

The high resolution diffraction technique can be used for measuring the alloy composition in III-V epilayers with an accuracy which is better than 1% in most cases. This method is based on the exact knowledge of the semiconductor lattice parameters and on the application of Vegard's law. Unfortunately the composition measurement in the GaAs/GaAs system is difficult because of the small lattice parameter difference between AlAs and GaAs and the poor accuracy of the AlAs parameter values reported in the literature; some of them being obtained from powders. This work proposes a method, based on the exact knowledge of the semiconductor laser parameters and on the application of Vegard's law, for the determination of the composition of the GaAs/GaAs system. X-ray topography was used to verify that no misfit dislocations were introduced during the growth. A very good fit between the experimental and simulated profiles could be obtained with an AlAs lattice parameter of 5.68203 Å if a Poisson ratio of 0.286 was used. An accurate analysis of the diffraction profiles also evidenced a small mismatch between the semi-insulating substrate and the heavily doped GaAs cap.


H was implanted in n-type InP crystals grown by the liquid encapsulated Czochralski method. The ions were implanted from 5 to 10 to 5 x 1015 cm^-2. The structural properties were studied by x-ray diffraction and transmission electron microscopy. The lattice constant determination was made by using a double crystal diffractometer and a high resolution diffractometer equipped with a four crystal monochromator. Cu Kα radiation.
004 symmetric and 117 asymmetric reflections were used. By means of standard simulation procedures the depth dependence of the lattice strain normal to the surface was determined. If implantation has been found to produce a lattice dilation; for the highest dose (5 x 10^16 cm^-2) a maximum strain of about 2 x 10^-2 has been obtained. The strain decreases rapidly by decreasing the dose and no strain has been observed for doses smaller than 3 x 10^16 cm^-2. The correlation between doses and distribution and nature of extended defects has been studied both by conventional and high resolution electron microscopy on both (001) plan view and (011) cross-sectional samples prepared by iodine ion milling. The defect depth distribution has been correlated to the strain depth profiles obtained from x-ray diffraction. Finally, the defect nature and its influence on the strain profiles has been studied by comparing x-ray and electron microscopy results.


Lastly there has been extensive research into the properties of strained structures. It is now clear that strain provides a most useful additional parameter in the design and fabrication of devices based on semiconductor superlattices and multiquantum wells. However, to date these devices are not fully developed due to the difficulties to fabricate structures with good crystalline quality. In this work we use transmission electron microscopy (TEM) to study the structural properties of strained InGaAs/InAlAs quantum well structures on InP substrates in order to improve the technological growth conditions. The first part of the work will be devoted to the study of the optimization of the InAlAs buffer layer used to favor the overgrowth of the epilayer with a good crystalline quality. Finally, a structural characterization of the influence of lattice mismatch and epilayer thickness on the morphology of the single and multiple quantum well structures is presented.


Photoinduced microwave reflectometry (PIMR) is used in conjunction with image processing methods to produce spatially resolved maps of structures across semi-insulating gallium arsenide and indium phosphide substrates. Two maps of peak conductivity transient data are obtained using AlGaAs and GaAs pulsed lasers. For GaAs substrates the optical excitation is above and below bandgap, while for InP both lasers are above bandgap. Previous work has shown that for GaAs substrates the peak photo induced transient response is related to shallow acceptor and defect concentrations, while the InP peak transient response is sensitive to surface quality. A third map of conductivity (dark) is obtained without optical excitation. The mapped dark data represents variations in material conductivity, augmented by the effect of wave interactions between the microwave probe system and the wafer under test. Image processing using Fourier domain techniques allows separation of mapped photo-induced peak transient data from dark mapped data. Image processing also provides a way to separate the peak transient response from the two different optical sources into correlated and uncorrelated transient response maps. The maps yield spatial information related to carrier generation and recombination. The mapped PIMR method combined with image processing can provide an enhanced non-contacting non-destructive method for the characterization of GaAs and InP semiconductor substrates.
Quantum Confinement
New Technology Subcommittee/Electronics/Dielectric Science and Technology

St. Louis Ballroom F, 4th Level

WEDNESDAY, MAY 20, 1992
E. H. Nicollian, Chairman; R. Tsu, Vice-Chairman

10:00 From Superlattices, Quantum Wells to Quantum Dots: R. Tsu. University of North Carolina, Charlotte, NC 28223

Since the introduction of superlattices and quantum well structures, it is well known that quantum mechanics plays a major role in the understanding of quantum effects. Quantum dots represent a further leap into the man-made atoms. The physics and chemistry of three-dimensional quantum confinement and its implications on quantum devices are discussed.

10:45 Semiconductor Nanocrystals: A. P. Alivisatos, Dept. of Chemistry, University of California, Berkeley, CA 94720

Relatively monodisperse, high quality nanocrystals of II-VI and III-V semiconductors can be produced by colloidal chemical synthesis. Because of their finite size, these nanocrystals have many size-dependent properties, including: melting temperature, optical spectrum, electron-phonon coupling, and others. The chemical nature of the nanocrystal surface can be manipulated, so that one can control the environment of the nanocrystals. Thus, they can be bound to a metal or semiconductor surface, dissolved homogeneously in a fluid or polymer film, etc. Many interesting quantum confinement effects can be explored in these systems.

11:30 III-V Compounds Etching: Diffraction Gratings, Quantum Wire and Quantum Box Applications: S. Pellegrino, L. Boschi, G. Cossaro, and P. Dostie

We have extensively characterized the HBr/HNO3/H2O etching solutions from the point of view of their reactivity in respect to InGaAsP materials. We obtained insight into the chemistry of the solutions and on the etching mechanism. A minimum controllable 20 Å/min etching rate was measured along with a strong unsupervised etching characteristic with respect to the InGaAsP materials range. We found a large capability of tailoring the grating shape in case of diffraction gratings, and the feasibility of quantum wire and box structures was demonstrated.

1:00 Admittance of Silicon Quantum Dots: E. H. Nicollian, University of North Carolina, Charlotte, NC 28223

Admittance measurements on a 200 Å thick layer of silicon microstructures embedded in a matrix of SiO2 are described. It is shown that electrically active traps at the p+ - Si/SiO2 interface are filled with electrons at gate bias values before quantum confinement occurs. Therefore, even with large electrically active trap densities, coherence is not destroyed and three-dimensional quantum confinement is observed.

1:45 Two-Dimensional Carrier Confinement in GaAlAs Quantum Wire Arrays Grown by Molecular Beam Epitaxy: J. C. Bean and R. People

Quantum wire superlattices are obtained by direct growth on a GaAs vicinal (100) surface by alternately depositing submonolayers of GaAs and AlAs. By varying linearly as a function of time the total amount deposited per GaAs-AlAs cycle, a superlattice consisting of quantum wells with parabola-shaped interfaces is formed. At the apex of each parabola, a quantum wire region is introduced. This type of superlattice is hereafter called a "serpentine superlattice" (SSL). We have used transmission electron microscopy (TEM) measurements to study SSLs. TEM contrast studies indicate a poor segregation of the group III elements at the step edges during deposition. This in turn produces quantum wires with AlGaAs as in the wires and nonabrupt interfaces between wire and barrier regions. Both [100] and [110] vicinal surfaces have been investigated. We find that the Al segregation at step edges is superior for growth on the [100] vicinal surface. Possible reasons for this result are given. Polarization dependent photoluminescence and photoluminescence excitation measurements have been used to characterize carrier confinement. A comparison of the luminescence data with calculated optical properties as a function of segregation into lateral wells and barriers, yields AlGaAs as well and barriers with z = 0.11 and 0.22, respectively, instead of the nominally intended values of 0.00 and 0.23. The optical characteristics of a GaAs-AlGaAs laser with a quantum wire array in the active region are discussed.

Abstracts

589 QUA From Superlattices, Quantum Wells to Quantum Dots: R. Tsu. University of North Carolina, Charlotte, NC 28223

Since the introduction of superlattices and quantum well structures, it is well known that quantum mechanics plays a major role in the understanding of quantum effects. Quantum dots represent a further leap into the man-made atoms. The physics and chemistry of three-dimensional quantum confinement and its implications on quantum devices are discussed.

590 QUA Semiconductor Nanocrystals: A. P. Alivisatos, Dept. of Chemistry, University of California, Berkeley, CA 94720

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591 QUA HBr/HNO3/H2O - A Solution for Submicrometer III-V Compounds Etching: Diffraction Gratings, Quantum Wire and Quantum Box Applications: S. Pellegrino, L. Boschi, G. Cossaro, and P. Dostie

We have extensively characterized the HBr/HNO3/H2O etching solutions from the point of view of their reactivity in respect to InGaAsP materials. We obtained insight into the chemistry of the solutions and on the etching mechanism. A minimum controllable 20 Å/min etching rate was measured along with a strong unsupervised etching characteristic with respect to the InGaAsP materials range. We found a large capability of tailoring the grating shape in case of diffraction gratings, and the feasibility of quantum wire and box structures was demonstrated.

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Admittance measurements on a 200 Å thick layer of silicon microstructures embedded in a matrix of SiO2 are described. It is shown that electrically active traps at the p+ - Si/SiO2 interface are filled with electrons at gate bias values before quantum confinement occurs. Therefore, even with large electrically active trap densities, coherence is not destroyed and three-dimensional quantum confinement is observed.

593 QUA Two-Dimensional Carrier Confinement in GaAlAs Quantum Wire Arrays Grown by Molecular Beam Epitaxy: J. C. Bean and R. People

Quantum wire superlattices are obtained by direct growth on a GaAs vicinal (100) surface by alternately depositing submonolayers of GaAs and AlAs. By varying linearly as a function of time the total amount deposited per GaAs-AlAs cycle, a superlattice consisting of quantum wells with parabola-shaped interfaces is formed. At the apex of each parabola, a quantum wire region is introduced. This type of superlattice is hereafter called a "serpentine superlattice" (SSL). We have used transmission electron microscopy (TEM) measurements to study SSLs. TEM contrast studies indicate a poor segregation of the group III elements at the step edges during deposition. This in turn produces quantum wires with AlGaAs as in the wires and nonabrupt interfaces between wire and barrier regions. Both [100] and [110] vicinal surfaces have been investigated. We find that the Al segregation at step edges is superior for growth on the [100] vicinal surface. Possible reasons for this result are given. Polarization dependent photoluminescence and photoluminescence excitation measurements have been used to characterize carrier confinement. A comparison of the luminescence data with calculated optical properties as a function of segregation into lateral wells and barriers, yields AlGaAs as well and barriers with z = 0.11 and 0.22, respectively, instead of the nominally intended values of 0.00 and 0.23. The optical characteristics of a GaAs-AlGaAs laser with a quantum wire array in the active region are discussed.

594 QUA Quantum Devices or Quantum Chaos?: M. A. Reed, Dept. of Electrical Engineering, Yale University, New Haven, CT 06520

Recent advances in nanometer scale fabrication techniques now allow the realization of quantum-confined and charge-quantized electron systems that exhibit transport phenomena dominated by quantum effects and single-electron charging effects. These systems are fascinating nanoscale laboratories to probe the limit of electron conduction and quantum effects at the one-electron level, and promise a technology for future integrated circuits with staggering density. A brief introductory survey of the physics and technology of some of these structures is presented. At present, the implementation of useful electronic devices and circuits utilizing quantum-confined or charge-confined effects has not yet occurred. In addition to well-recognized problems of fabrication and tolerance that exist at these scales, there are issues that are unique to quantum devices and circuits, such as isolation in phase-coherent structures; how to obtain gain from single-electron devices; invasive and statistically uncontrollable contacts, and architectural issues. This paper focuses on the critical issues that impede the insertion of quantum devices into useful electronic circuits, and possible solutions to these problems.

595 QUA Germanium Silicon Quantum Well Structures: J. C. Bean and R. People, AT&T Bell Laboratories, Murray Hill, NJ 07974

GeSi, /Si quantum wells have been used to fabricate modulation doped transistors, 1.3 µm available photodetectors, resonant tunneling diodes, and 8-14 µm infrared infrared devices. Although these devices resemble III-V semiconductor analogs, strain fundamentally alters their characteristics. A strained alloy may have a bandgap one-third lower than a comparable relaxed structure. Normally degenerate conduction and valence band energy levels will be strongly split. The conduction bandedge of the alloy may move either above or below that of adjacent silicon, producing structures where electrons and holes accumulate in the same layer or are driven into adjacent layers. Normally, in even "conventional" device structures, the maintenance of defect-free strained layer growth may require individual layer thicknesses so small that pronounced quantum confinement effects occur. The design of GeSi, /Si quantum structures it thus significantly more complex. In certain instances, such as infrared absorption, it can yield a particularly broad and useful infrared device characteristic.
Abstracts

596 MIC Recent Advances in MEMS: G. A. Hazelrigg
The technologies of microelectronic fabrication have, over the past several years, been extended to include the fabrication of mechanical and electromechanical devices and mechanisms on a very small scale, and laying the foundations of a new field currently referred to as microelectromechanical systems (MEMS). Recently demonstrated devices include electrostatic and electromagnetic motors smaller in diameter than a human hair, gears, pumps and valves, sensors and other structures. Most recently, three-dimensional structures have been fabricated as well, including folding silicon wafers and helixes and needles grown vertical to the wafer. Together, these technologies comprise a basis for a chemical processing factories-on-a-chip. This paper reviews the basic technologies of MEMS and then discusses recent advances in the field and their implications.

597 MIC Vacuum Microelectronics: An Application of Micromachining and Nanostructure Fabrication: H. F. Gray
With the development of silicon field emitter arrays (FEAs) by micromachining techniques(1) and the reporting of the first vacuum transistor(2), the possibility of a new microelectronics, which we named vacuum microelectronics, was born. In addition to having the fabrication, batching processing, size, weight, integrated circuit, and unit cost advantages of solid-state devices, vacuum microelectronics based on FEAs takes advantage of vacuum electron transport. Vacuum electron transport brings with it faster carrier velocities and lower transit times than solid-state devices of comparable size, the ability to deflect the carriers to multiple collectors, the possibility of much higher voltage and higher power operation, and the promise of immunity to adverse environments, e.g., very high and very low temperatures as well as high radiation environments which destroy or seriously impair solid state device operation. Benefits are even promised to scientific instrumentation because FEAs are electron sources which are essentially monoe-nergetic, have very high brightness, and exhibit high spatial resolution. Each cell of a FEA contains one 3-D microminiature field emitter and its monolithic extraction electron. The radius of the curvature of the field emitter is usually 100 Å or smaller, the extracature aperture is 1 μm or smaller, and the distance between extraction electrode and field emitter ground plane is about 1 μm. The nanostructure is fabricated by micromachining techniques. This paper addresses a variety of micromachining techniques which have been used throughout the world to fabricate FEAs, including orientation-dependent etching of silicon, oxidation sharpening, beam deposition, electron beam decomposition, E-beam evaporation, plasma sputtering, reactive ion etching, and isotropic wet etching. We feel that the detail of the physics and chemistry of these micromachining processes are not well understood at the present time, and that the science of micromachining of 3-D nanostructures is still in its infancy.


598 MIC Three-Dimensional Silicon Anisotropic Etching — Identification of the Key Etching Planes: P. J. Heeshek
A single crystal silicon sphere has been etched in aqueous solutions of potassium hydroxide and cesium hydroxide. After etching, a polyhedron-like solid with curved faces is produced with distinct vertices in the directions of the slower etching planes. The solid is twelve sided with aqueous cesium hydroxide and twenty four-sided with aqueous potassium hydroxide. The following crystal planes defined features in the structure, listed in order of ascending etch rate: (i) for COH, (111), (100), (311), (110), and (ii) for KOH, (111), (100), (311), (326).
A technique for precisely aligning microstructures prior to an Au-Si bond has been developed. This technique has applications to electron column fabrication and three-dimensional wafer interconnection technologies. It is accomplished by etching v-grooves around the periphery of the structure to be bonded. Gold is then deposited onto one of the wafers prior to dicing into individual chips. Developed devices are characterized by precision locating keys and the alignment of the die are assembled. The entire structure is then placed on a hot chuck at 400°C. An ultrasonic transducer aids the Au-Si bond. The fibers can be pulled out after bonding since the v-grooves are oversized. The width of the v-grooves is 1.22x the diameter plus the desired tolerance. Early results have shown a minimum misalignment of 15 µm. This work will be extended to a higher accuracy and include silicon fusion and organic bonding.

604 MIC Thermoelectric AC Power Sensor on Silicon Oxide Beam: D. Jaeggi, H. Baltes, and D. Moser, Physical Electronics Laboratory, Swiss Federal Institute of Technology, CH-8093 Zurich, Switzerland

We report the first thermoelectric AC power sensor (thermocouple) realized by industrial CMOS IC technology in combination with post-micromachining. The sensor is based on a polycrystalline heating resistor (47 Ω) and a polycrystalline/aluminum thermopile integrated on an oxide microbridge. The thermopile sensitivity is 9.5 mV/mW. The temperature coefficient of the sensor's time constant is 0.18% per degree. The signal-to-noise ratio, evaluated on the heat power sensor, is below 400 MHz and less than 1% up to 1.2 GHz. In all limits the sensor's time constant is below 1 mW heating power. The sensor's time constant below 1 mW heating power. The sensor's time constant is below 1 mW heating power.

605 MIC Thermally Excited Silicon Oxide Resonators in CMOS Technology: O. Brand, D. Moser, and H. Baltes, Physical Electronics Laboratory, Swiss Federal Institute of Technology, CH-8093 Zurich, Switzerland

We report thermally excited silicon oxide beam and bridge resonators realized by two different industrial CMOS processes and evaluate the resonant frequencies of the different resonators by measuring resonator frequencies, vibration amplitudes, and mode shapes using a laser heterodyne interferometer. We investigated the acoustic coupling between the resonators in a plane and the devices in an in air in order to test the possible application as ultrasonic transducers for proximity sensors. The vibrations can be detected using the piezoelectric effect of polycrystalline silicon.

606 MIC Fabrication of Novel Micromachined Magnetic Actuators: A. M. Hamad, W. Zhang, H. T. Fender, and F. Radpour, Dept. of Electrical and Computer Engineering, University of Cincinnati, Cincinnati, OH 45221

Trends in smart sensors and smart skins must move in the direction of microchipping and miniaturization of semiconductors and micromachining. Tactile sensors are particularly important for robotics and related sensing as well as microactuation of valves and other electrochemical components. In this paper, the design, fabrication, and characterization of a unique type of magnetic actuator is presented. This device is capable of converting a current signal into the mechanical motion of a plunger. The sensors were fabricated by anisotropic etching of 0.1% oriented silicon. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution. The magnets are inserted into a groove solution.

607 MIC Processing of Ferroelectric Materials in Microfabricated Silicon-Based Structures: D. L. Polla, Dept. of Electrical Engineering, University of Minnesota, Minneapolis, MN 55455

Ferroelectric thin films have been deposited on polycrystalline silicon, silicon nitride, and tungsten structures to form both piezoelectric and pyroelectric microelectromechanical devices. This work focuses on the microfabrication technology issues important to ferroelectric microelectromechanical structures. Surface micromachining techniques and sol-gel depositions for trivalent oxides and PbTiO,


A pyroelectric (LiTaO) anemometer chip was mounted into the wall of a small micromachined channel in silicon capped by an
anodically bonded Pyrex® plate. The silicon structure was fabricated using a photolithography process. The channel was 72 μm deep and 3 mm wide. The dimensions of the overall chip were approximately 30 mm × 15 mm. The spindle speed was compared to an MKS-100 thermal flow monitor. The experimental results demonstrated that the PA chip has essentially the same flow response as a function of Reynolds number in this channel as in larger channels. Also, this structure appears to be capable of monitoring flows as small as 0.1 µm.

A discussion of the conjugate solutions of simultaneous fluid and heat flow from the PA element and some of the problems associated with the analysis are presented.


An optical fiber pressure sensor consisting of a vertical deflectable membrane, a sealed cavity, and a channel with a single mode optical fiber for monitoring the wall deflection has been developed. The membrane, cavity, and channel all were formed within a (110) silicon wafer by using a standard KOH crystallographic micromachining etch. Membranes as thin as 4 µm have been fabricated. The cavity was anodically sealed by bonding a piece of Pyrex® glass to the etched surface of the wafer. The alignment between the fiber and the center of the membrane was realized by careful adjustments of the microfabrication process.

The interference patterns obtained were clear and stable. Newton's rings were also observed, which were one of the interference patterns vs. the pressure applied to the sensor. It is presented showing that a pressure resolution of 0.01 atm is obtained. It is expected that a minimum detectable pressure can be lower than 0.001 atm after implementing the used measurement system. The temperature sensitivity of the device remains a problem. Methods for eliminating this are discussed.

610 MIC Microfabrication of Silicon X-Ray Analyzer: H. Kasapbasoglu and P. J. Hesketh, Dept. of Electrical and Computer Engineering, University of Illinois at Chicago, Chicago, IL 60680. 2. M. Cameron, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439-4814

We resolved a method for the fabrication of silicon x-ray analyzer crystals. A high energy resolution of x-ray beam scattering can be obtained by Bragg scattering from perfect single crystals and focusing action can be obtained by bending the perfect crystals. Parallel and perpendicular, 1 mm spaced grooves were cut on (111) silicon wafers using a dicing saw (Microautomation Model 1000). The grooves were cut at 15° and 105° to the <110> primary flat to avoid crystallographic cleavage directions, since mechanical strength was desired. The depth of the grooves was almost 80% of the wafer thickness. The cutting parameters, progressive cutting thickness and feed rate, were optimized to reduce damage. The damage was observed by sectioning the samples along the cleavage direction and etching in Yag's etch. Optical and scanning electron microscopy revealed that the least damage occurred when two passes were used: one a light 75 µm deep and the next to the required depth. More than two passes degraded the cuts. Slow feed rates of 1-5 mm/s and fast feed rates of 40-60 mm/s produced increased damage while no edge cracks and optimal smoothness were found for 12-20 mm/s. The spindle speed was constant at 30 K rpm to minimize resonant vibration. The effect of different etchants for removing the saw damage was also investigated and the results are reported.

611 MIC A Thin Platinum Island Film Glucose Sensor: B. Kasapbasoglu and P. J. Hesketh, Dept. of Electrical and Computer Engineering, University of Illinois at Chicago, Chicago, IL 60680. W. C. Hanly, Dept. of Microbiology and Immunology, University of Illinois at Chicago, Chicago, IL 60680. J. McLaughlin, Dept. of Electrical and Computer Engineering, University of Illinois at Chicago, Chicago, IL 60680

A 2.5 nm thin film glucose sensor has been developed. The island design of the film allowed the immobilization of glucose oxidase on the SiO₂ surface in a thin layer adjacent to the islands. The Pt film was deposited by electron beam evaporation at 1.33 × 10⁻⁶ Pa onto a 100 nm thermal oxide on a 50 nm diameter Si wafer at room temperature. Adsorptive immobilization from a 3.1% glucose oxidase at a pH 5.3 (0.1 M NaHCO₃ buffer) was used. The adsorption was allowed to proceed for about 48 h at 4°C. The sensor was then washed with a dilute non-ionic detergent (0.05% Tween 20 in 0.05 M sodium acetate buffer, pH 5.3). For measurement of the impedance, the washed electrode was immersed in tetramethylbenzidine (TMB) buffer solution, 0.4 M sodium acetate buffer, pH 5.3 containing horseradish peroxidase (2 purpurogallin U/ml) and glucose (concentration range 0-500 mM). The impedance and phase of the enzyme coated film was measured from 100 Hz to 1 MHz. The results of the measurements indicated that the system could be modeled with a suitable RC equivalent circuit. Glucose produced a 500% change in the series capacitance at 100 Hz because of the binding reaction. The sensitivity would be best at low frequencies. The resistance was almost independent of glucose concentration. Reaction rate constants were obtained and are discussed. The impedance returned to its original value after washing in a suitable buffer, and storage at 4°C did not affect the initial performance.

612 MIC Development and Characterization of an Ultrathin Silicon Nitride for IC Sensor Applications: Using Rapid Thermal Processing: R. A. Williams, Dept. of Chemical Engineering, University of California; Berkeley, CA 94720. L. J. Arons, Jr., Dept. of Electrical Engineering and Computer Engineering, Lehigh University, Bethlehem, PA 18015

Rapid thermal nitridation of silicon has been investigated as an alternative to CVD silicon nitride deposition. Ultrathin silicon nitride films have been thermally grown in ammonia and nitrogen atmospheres using rapid thermal processing. The effects of several process variables on the growth rate of the silicon nitride and its material properties have been evaluated electrically and structurally. The effect of other process variables were mixed, i.e., physical and chemical nitride. The reduction in the expense of electrical properties (annealing, two-step nitridation, in situ exposure cleaning) or vice versa (exposed purges, low-pressure nitridation) were examined. By varying these process parameters, films with different properties (annealing, two-step nitridation) were achieved. Other films required a field of 81.6 KV/mm to pass 1 µA through a 10 µm structure. However, the film with the best overall physical and electrical properties had a etch rate of 0.368 mm/min in 5.1% HF and required a field of 509 KV/mm to pass 1 µA through a 10 µm structure and maintained a current density of 1.2 µA/mm² at -1 V. Durable pH and glucose sensors have been fabricated using these silicon nitride films. They have demonstrated greater sensitivity than oxides or oxide/CVD nitride gate structures.
### FULLERENES: CHEMISTRY, PHYSICS AND NEW DIRECTIONS

**Physical Electrochemistry/Dielectric Science and Technology/Electronics**

**St. Louis Ballroom C, 4th Level**

**MONDAY, MAY 18, 1992**

- **Chairman, Rodney S. Ruoff; Vice Chairman, Peter Eklund**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
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<tbody>
<tr>
<td>10:00</td>
<td>Introductory Remarks - Rodney S. Ruoff and Karl M. Kadish</td>
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<td>10:10</td>
<td>Structure and Dynamical Properties of Large Fullerenes and Other Novel Forms of Carbon by First Principles Molecular Dynamics - G. B. Adams, J. B. Page, O. F. Sankey, K. Sinha, J. Menendez, and M. O'Keeffe</td>
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<tr>
<td>11:10</td>
<td>High-Resolution Transmission Electron Microscopy of Fullerenes - Peter R. Buseck and Su Wang</td>
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<tr>
<td>11:40</td>
<td>Adsorption of 1,1,2-Trichloro-1,2,2-trifluoroethane on Fullerenes - W. L. Bell, David T. Wickham, and Amy L. Schultz</td>
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**MONDAY, MAY 18, 1992**

- **Chairman, Karl M. Kadish; Vice Chairman, Ripudaman Malhotra**

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<tr>
<th>Time</th>
<th>Session</th>
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<tbody>
<tr>
<td>11:10</td>
<td>Quantum Molecular Dynamics Calculations for Fullerenes and Buckytubes - J. Bernholc, Jae-Yel Yi, Q. M. Zhang, C. J. Drabec, E. B. Anderson, S. A. Kajihara, and B. N. Davidson</td>
</tr>
<tr>
<td>11:40</td>
<td>On the Use of C_{60} or C_{70} Clusters to Nucleate Diamond Crystals - R. P. H. Chang</td>
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<td>12:10</td>
<td>Formation of Fullerenes from Molecular Dynamics Simulations - J. R. Chelikowsky and Xiaodum Jing</td>
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<td>12:40</td>
<td>Twenty-minute intermission</td>
</tr>
<tr>
<td>13:10</td>
<td>Molecular Dynamics Study of C_{60}, C_{70}, and Alkali-Doped C_{60} - A. Cheng and Michael L. Klein</td>
</tr>
<tr>
<td>13:40</td>
<td>Synthesis of Fullerols by Electrophilic Reactions on Fullerene Molecules - L. Y. Chiang</td>
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<tr>
<td>14:00</td>
<td>Preparation and Characterization of C_{60} and C_{70} Crystals and Solutions - C. W. Chu, J. G. Lin, Y. K. Tso, Y. Y. Sun, R. L. Meng, and P. H. Hor</td>
</tr>
<tr>
<td>14:30</td>
<td>Application of Real-Time SERS and STM Techniques to Studies of C_{60} and C_{70} - Y. Zhang, X. Gao, G. Edens, and M. J. Weaver</td>
</tr>
<tr>
<td>15:00</td>
<td>Bond Breaking and Bond Making with Fullerenes - R. Malhotra, Doris S. Tse, Rodney S. Ruoff, Donald F. McMillen, and Donald C. Lorents</td>
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</table>

**TUESDAY, MAY 19, 1992**

- **Chairman, Donald M. Cox; Vice Chairman, David Tomasek**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
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<tbody>
<tr>
<td>1:10</td>
<td>Symmetry for Lattice Modes in C_{60}, C_{70}, and Alkali Metal Doped C_{60} - G. Dresselhaus, M. S. Dresselhaus, and P. C. Eklund</td>
</tr>
<tr>
<td>1:40</td>
<td>Fullerenes and Fulleren Derivatives - B. I. Dunlap</td>
</tr>
<tr>
<td>2:10</td>
<td>Ellipsometry and Optical Absorption Studies in M_{x}C_{60} Films (x = 0, 3, 6; M = K, Rb, Cs) - Peter C. Eklund</td>
</tr>
<tr>
<td>2:40</td>
<td>Voltammetric Studies of [(C_{6}H_{5})<em>{2}Pd]</em>{2} =K C_{60} and Some [(C_{6}H_{5})<em>{2}Pd]</em>{2} =K C_{60} Complexes (M = Ni, Pd and Pt; n = 1, 2, 3 and 6) - Paul J. Fagan, Susan A. Lirke, and D. H. Evans</td>
</tr>
<tr>
<td>3:10</td>
<td>Electron and Oxygen Atom Transfer to C_{60} - Formation of Oxygen Atom and 1,3-dioxolene Adducts - C. S. Foote, James W. Arbogast, Michelle Kao, Yianna Elenes, Scott Silverman, and Chi-Min Sheu</td>
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**WEDNESDAY, MAY 20, 1992**

- **Chairman, William Goddard, III; Vice Chairman, Christopher Reed**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
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<tbody>
<tr>
<td>8:30</td>
<td>Analysis of the Raman Spectra of A_{6}C_{60} and A_{7}C_{60} - F. Negri, G. Orlandi, and P. Zerbello</td>
</tr>
<tr>
<td>9:00</td>
<td>Photodissociation and Photodissociation Channels of C_{60} - Keith R. Lytke, Peter Wurz, Deborah H. Park, Michael J. Pellin, and Dietl M. Green</td>
</tr>
<tr>
<td>9:30</td>
<td>The Structure and Symmetry of the Orientational Ordering of C_{60} - A. B. Harris and R. Sachidanandam</td>
</tr>
</tbody>
</table>
10:00 Nuclear Spin Weights and Gas Phase Spectral Structure of $^{13}C_60$ and $^{13}C_{70}$ Buckminsterfullerene (C60) - W. G. Herter and Tyle C. Reimer 642 FUL

10:30 Charge Transfer at Fullerene-Noble Metal Interfaces - J. S. Lannin 643 FUL

11:00 Recent Results in the Synthesis and Characterization of Discrete Derivatives of C60 - Joel M. Haskins 644 FUL

11:30 Molecular Structures of the Gaseous Fullerenes C60 and C70 from Electron Diffraction - Kenneth Hedberg, Lisa Hedberg, D. S. Bethune, C. A. Brown, R. D. Johnson, and M. de Vries 645 FUL

WEDNESDAY, MAY 20, 1992

Chairman: Emmanuel P. Gianellis; Vice Chairman, Fred Wudl

1:10 Orientational Order in Fullerenes - P. A. Heiney 646 FUL

1:40 Structures of Carbon Cluster Ions - Gert von Helden, Paul R. Kemper, and Michael T. Bowers 647 FUL

2:10 Bandgap, Excitons and Coulomb Interaction in Solid C60 - R. Loef, M. van Veenendaal, B. Koopmans, H. T. Jonkman, and G. A. Sawatzky 648 FUL

2:40 Synthesis, Structure and Superconductivity of Carbon-60 Fullerenes - Zafar Iqbal 649 FUL

3:10 Twenty-minute intermission

3:30 Carbon Nanotubes - Sumio Iijima 650 FUL

4:00 Solvent, Supporting Electrolyte and Temperature Effects on the Electroreductions of Buckminsterfullerene (C60) in Aprotic Media - K. M. Kadish, D. Dubois, G. Moninot, W. Kuhn, and M. T. Jones 651 FUL

4:30 Electron Spin Resonance Characterization of Singly, Doubly and Triply Reduced Buckminsterfullerene, C60, in Aprotic Media - M. T. Jones, D. Dubois, and K. M. Kadish 652 FUL

5:00 Intercalation of Amine Functionalized C60 in Mica-Type Silicates - V. Mehrotra and Emmanuel P. Gianellis 653 FUL

5:30 Theoretical Studies of the Structure, Properties, and Dynamics of Fullerene, Crystals and Superconductors - W. A. Goddard III, Yuelin Guo, Guanhua Chen, and Naoki Karasawa 654 FUL

WEDNESDAY, MAY 20, 1992

Chairman, William L. Bell; Vice Chairman, Jerzy Cioslowski

7:00 Studies Related to Extraction and Separation of Fullerenes - Rodney S. Ruoff, R. Mahlota, and Don Lorents 655 FUL

7:30 ESR Studies of the Reaction of Alkyl Radicals with C60 - P. J. Krause, J. R. Morton, E. Wasserman, and K. F. Preston 656 FUL

POSTER SESSION

Chairman, Karl M. Kadish; Vice Chairman, Rodney S. Ruoff

8:00 Radial Vibrations of an Ion Inside Icosahedral C60 - J. L. Balster, and B. I. Dunlap 657 FUL

10:00 Organometallic Chemistry of Fullerenes in the Gas Phase by FTMS - Y. Huang, Q. Jiao, S. A. Lee, and B. S. Freiser 658 FUL

10:30 Solubilization of Buckminsterfullerene, C60, in Water and Some Polar Organic Solvents by Cyclodextrin Inclusion Chemistry - Wlodzimierz Kutner, Pierre Boucas, and Karl M. Kadish 659 FUL

11:00 Excited State Behavior of Fullerene (C60) and Their Reduction in Colloidal Semiconductor Suspensions - Prashant V. Kamat and Nada M. Dimitrijevic 660 FUL


THURSDAY, MAY 21, 1992

Chairman, Long Chiang; Vice Chairman, Donald Bethune


9:10 Dynamics of Free Fullerenes in Photophysics and Collisions - W. Kamke, E. E. B. Campbell, and I. V. Hertel 663 FUL

9:40 Thermophysical Properties of Solid C60, C70, and Related Materials - J. L. Margreave, Chendu Pan, M. S. Chandrasekarahal, M. P. Sampson, and R. H. Hauge 664 FUL

10:10 Superconductivity in Metal-Doped C60 Solids - C. M. Lieber, C. G. Chen, Z. Zhang, and S. P. Kelly 665 FUL

10:40 Electrochemical Reactivity of Fullerenes and Their Derivatives - F. D. Souza, Ram Seshadri, R. Nagarajan, Govind Raj, V. Krishnan, and C. N. R. Rao 666 FUL

11:10 Production and ESR Spectroscopy of Fullerenes Containing Metal Atoms - Robert D. Johnson, Costantino S. Yannoni, Mattaner S. De Vries, Jesse R. Salem, and D. S. Bethune 667 FUL

11:40 Gas-Phase Characterization of Fullerenes and Endohedral Complexes - S. W. McElvany, John H. Callahan, and Mark M. Ross 668 FUL
Abstracts

613 FUL  Structure and Dynamical Properties of Large Fullerences and Other Novel Forms of Carbon by First Principles Molecular Dynamics: G. B. Adams, J. B. Page, O. F. Sankey,* R. Sinha, J. Menendez, and M. O'Keefe, Dept. of Physics and Chemistry, Arizona State University, Tempe, AZ 85287

We have used our first principles molecular dynamics method to determine the minimum energy geometries and electronic structure of more than 20 different fullerenes, including fullerences of mass 60, 78, 84, 120, 130, and 240. For the lowest energy structure of most mass types we have determined the vibrational modes and frequencies, also from first principles. In addition, we have examined the energy and vibrational modes of novel forms of solid carbon which are more competitive with solid C60. (This work was supported by ONR-N00014-90-J-130+.)


614 FUL  Synthesis and Characterization of Endohedral Metallofullerenes: J. M. Alford* and R. E. Smalley, Rice Quantum Institute, Dept. of Chemistry, Rice University, Houston, TX 77251-1892

New synthesis techniques developed by this group during the last year now enable the production of macroscopic amounts of endohedral metallofullerenes. These special molecules which completely encapsulate a metal atom or cluster of atoms within their carbon cages can be synthesized using modified versions of either the laser vaporization method or the standard Kratschmer-Huffman (KH) technique. Certain metallofullerenes such as those containing the Group III elements (Sc, Y, La) are easily produced while others such as the alkali metal containing fullerences prove much more difficult to make. A survey across the periodic table shows which elements can be incorporated inside the fullerene structures. While characterization of a pure metallofullerenes remains to be accomplished, some basic studies such as solubility, XPS, and ESR have been performed on samples containing empty and mixed metallofullerenes. These results show that one fullerene, C60, has the unique property of forming both single atom, Y @ C60, and multiple, Y @ C60, metallofullerenes that are soluble in toluene. A more detailed analysis of these results as well as up-to-date results on the characterization of metallofullerenes in general are presented.


The iridium complex, [Ir(CO) 3(CPPh 3)], binds reversibly to the exterior surface of fullerenes C60 and C70. X-ray crystallographic studies show that bonding occurs preferentially at 6:6 ring fusions. Crystal formation is highly sensitive to the solvents used for crystal growth and solvents are incorporated into the solids. The spatial relationship of the occluded solvent and Fullerence is described. New ligands for encircling the fullerences in these derivatives are described.

616 FUL  Adsorption of I, 1, 2-Trichloro-1, 2, 2-trifluoroethane on Fullerences: W. L. Bell*, D. T. Wickham, and A. L. Schultz, TDA Research, Inc., Wheat Ridge, CO 80033, P. Nolan, U S Army CRDEC, Aberdeen Proving Ground, MD 21010-5423
To better understand the surface properties of fullerenes, we have investigated adsorption of the title compound (CFC-113) from the gas phase onto solid fullerenes. Adsorption of organic molecules on isolated carbon clusters is commonly used for separation and purification, and a comparison of fullerenes with activated carbon will be interesting. We measured adsorption by following the fullerenes as a function of growth temperature and partial pressure of the adsorbate at a constant temperature. The resulting adsorption isotherms are discussed and compared with results obtained for activated carbon.


We describe the results of extensive ab initio molecular dynamics calculations of the properties of fullerenes and buckytubes. Our finite temperature quantum MD simulations for solid C60 are in good agreement with NMR measurements of susceptibilities of C60. The molecular structure is a natural precursor for the formation of C60. These results are consistent with the experimental findings that high temperatures are necessary for the formation of substantial quantities of C60. The band structure and doping calculations for optimized structures of several buckytubes are also described. (This work was supported by the U.S. Department of Energy Grant No. N00014-91-J-1516.)

618 FUL Calculation of Linear and Nonlinear Microscopic Polarizabilities of Fullerenes and its Doped Species: A Rosen and E. Westin. Dept. of Physics, Chalmers University of Technology and University of Göteborg, S-412 96 Göteborg, Sweden.

Molecular cluster calculations (1, 2) within the local density approximation have been performed in a study of the electronic structure of C60, XeC60, and monosubstituted fullerenes C60X, where X = B, N. Doping with B and N in the center of the cage creates an electronic structure with a partly occupied level in the bandgap similar to the donor and acceptor levels in traditionally doped semiconductors. While substitution of one of the carbon atoms in the cage with B or N gives a splitting of the IOM and LUMO levels in the bare C60 molecule. Using wavefunctions determined from these molecular calculations the frequency dependent linear and nonlinear optical microscopic polarizabilities, i.e., χ(1), χ(2), and χ(3), for a process of linear, second and third harmonic generation have been evaluated. The breaking of inversion symmetry for the mono-substituted species results in a larger 2nd order response. The importance of electric-quadrupole and magnetic interactions are also considered. Our results are found to be in reasonable agreement with experimental data from measurements of susceptibilities of C60; i.e., thin films or solution data (3-5).

3. Z. H. Kafizah. Private communication.


In spite of the intense research activity that has gone into studying the fullerenes during the last two years, much of the information about their shapes and local structures is inferential. Numerous attempts to study pure fullerene samples in the form of molecules of varied sizes. In some instances these diverse molecules pack into crystals that contain "defects" consisting of either isolated fullerenes having different sizes from their hosts, or into domains of one type of fullerene with domain boundaries separating them from fullerenes of other sizes. There are also instances in which large fullerenes occur as isolated molecules or in molecule clusters rather than in crystals. Because of its ability to obtain extremely high spatial resolution images of thick volumes of material, high-resolution transmission electron microscopy is well suited for studying such samples as well as, in ideal cases, isolated molecules. We have observed and imaged domain structures of fullerenes with lattice-matching up to 25% of far larger units as well as domains smaller than C60. Examples are shown.

620 FUL On the Use of C60 or C60 Clusters to Nucleate Diamond Crystals: R. F. Chang, Dept. of Materials Science and Engineering, Northwestern University, Evanston, IL 60208.

Fullerene molecules exhibit a high reactivity towards both nucleophilic substitution reactions and electrophilic substitution reactions, unlike most of highly conjugated aromatics, they are relatively inert to the electronic oxidation showing a remarkable stability even under doping at the anode potential at a solid state. We demonstrated that the reactivity of electrophilic substitution on fullerenes can be significantly enhanced using the solution chemistry. As an example, we outline three possible methods for the preparation of fullerols as polyhydroxylated fullerene derivatives through the reaction of fullerenes with electrophilic reagents. The synthetic methodology includes an aqueous acid chemistry, the epoxidation reaction, and the interesting nitronium chemistry for the conversion of fullerenes to fullerols. We discovered that the aqueous acid chemistry is an efficient method to produce multiple hydroxy groups onto the fullerene molecules. The reaction produces fullerols in essentially quantitative yield. We found that the structure of fullerols can be reproduced by the epoxidation of fullerenes followed by hydrolysis or the electrophilic attack of nitronium ions on fullerene molecules in the presence of organic acid. The resulting fullerol derivative carboxycarbon center on fullerene molecules that is susceptible to the nucleophilic attack of organocarbonyl anion. The resulting nitro-organocarboxylate derivatives can be transformed into fullerols through the further water treatment and hydrolysis.
624 FUL Preparation and Characterization of C₆₀ - C₇₀ Crystals and Solutions: C. W. Chu*, J. G. Lin, Y. K. Tao, Y. Y. Sun, R. L. Meng, and F. H. Hor, Dept. of Physics and Texas Center for Superconductivity at the University of Houston, Houston, TX 77204-5932

We have grown crystals of (C₆₀)ₙ(C₇₀)ₙ, with n = 0 to 1, using the sublimation and solution techniques. The effects of the solvent on the structural morphology and symmetry of the crystals grown from different solvents were examined. For n = 0, crystals prepared from solutions of hexane, toluene, benzene, and n-pentane have a face centered cubic lattice with a lattice parameter a = 14.2 Å, which is consistent with the crystals grown by sublimation. However, the morphology of these crystals depends on the solvent used, e.g., C₆₀ crystals grown from benzene are transparent, brown, thin hexagonal platelike, whereas those from toluene and benzene are fibrous, plate-like, and those from n-pentane are column-shaped crystals; and those grown by sublimation are black cubes. Ether appears to be an exception. 3-iodo-6-bromobenzonitrile undergoes conversion to the ipso position of C₆₀, and causes the formation of 2-methylnaphthalene. The results are consistent with the formation of a non-fused C₆₀ crystal, which decomposes at ~400°C into free fullerene. In contrast to C₆₀, C₇₀ tends to bind with the solvent molecules and form crystals as 2'-dinaphthylmethanone. We tested the ability of fullerenes, which are also ionizable, to abstract H atoms and to exhibit enhanced cleavage rates via a molecular process and aid in the cleavage of strong aryl-aryl bonds. The use of laser ablation, contact-arc, and pyrolysis techniques have been found to be highly effective in generating large carbon clusters and many of their derivatives. The use of laser ablation has provided several years ago the experimental evidence for endohedral complexes in which atoms, ions, or molecules are trapped inside hollow carbon clusters has become available only very recently. Contrary to popular belief, the guests in these complexes often tend to displace themselves from the cage center. Results of large-scale ab initio calculations on several endohedral complexes, including those with the Li⁺, Na⁺, and Mg⁺ cations, noble gases, and several diatomic molecules, are presented. Trends in the calculated properties are rationalized in terms of electronic interactions between the guest and the cage, and polarization effects. Approximate equations allowing predictions of the complexation energies are derived. Rules guiding the occurrence of charge transfer between the guest molecule and the C₆₀ cage are discussed. The origins of the so-called endohedral effect are explained using concepts of density functional theory.


In previous work performed in the context of coal liquefaction and pyrolysis we have found that polycyclic aromatic hydrocarbons (PAHs), notably pyrene, catalyze transfer of hydrogen atoms via a bimolecular process and aid in the cleavage of strong aryl-alkyl linkages. We tested the ability of fullerenes, which are also ionizable, to abstract H atoms and to exhibit enhanced cleavage rates by using 1, 2-diphosphinophosphazene as a model structure and by investigating solutions of this compound in, e.g., hexane, toluene, benzene, and n-pentane. Reactions were conducted at 400°C in a 30-m mole mixture of phenanthrene and 9, 10-dihydrophenanthrene-solvent system. Addition of the fullerene, e.g., C₆₀ (2.5% C₆₀ and 14% C₇₀), enhanced the cleavage rate by 59%. The enhanced cleavage rate was also attributed to an increase in the selectivity of cleavage at the 1-positions of naphthalene leading to the formation of 2-methylanthracene. The results are consistent with the view that as well as with other evidence, the fullerenes catalyze the transfer of H atoms to the ipso position of 1,2-diphosphinophosphazene. The ability of fullerenes in catalyzing H-transfers is also reflected in the products obtained. In one raw kaolinite-H₂O-MgCO₃-H₂O system with high boiling solvents such as methylene and diethyl ether, while these solvents can be heated under reflux for long periods without decomposition contact with the fullerenes leads to the formation of dimers, trimers, and other oligomers.

627 FUL The Fullerenes and Fulleroinds: Preparation and Properties: F. Wudl,*, Q. Lu, T. Suzuki, K. C. Kermer, M. Proto, and S. Shi, Institute for Polymers and Organic Solids, Department of Chemistry and Physics, University of California, Santa Barbara, CA 93106

The preparation of fullerenes (e.g., C₆₀) is described.

Fulleroids are cage molecules which are "inflated" fullerenes. The smallest members have essentially the same electronic structure as fullerene C₆₀, and are expected to produce materials with unusual properties, in analogy to their fullerene analogs. Details of their characterization and structure determination such those of NMR, x-ray crystallography and UV-Vis absorption spectroscopy are presented. The electrochemical properties of the fulleroids are also presented. Extension of the fulleroid approach to the preparation of polymers containing larger fullerene units has also been presented. Preliminary results indicate that the polymers have a very high Tg and are insoluble in a large number of solvents.


Using high pressure liquid chromatography, a new multifunctional fullerene, C₆₀O, was separated from the fullerene mixture generated via resistive heating of carbon rods. Characterization involves spectrometry and C NMR solution and solid state, infrared and UV-Vis absorption spectroscopy strongly supports the epicenters...
structure, and not the isomeric oxo-annulene type structure 2. Solid-state properties of C\textsubscript{60} are discussed. C\textsubscript{60}O can be synthesized at about 7\% yield from ultraviolet irradiation of C\textsubscript{60} in oxygen-saturated benzene solutions. Upon addition of benzo to the solution significantly higher yields can be obtained.

631 FUL In situ Magnetic Circular Dichroism and Fourier Transform Infrared Spectroscopy of C\textsubscript{60} Species: D. Scherson*, M. Zhao, S. Kim, J. T. Bae, and C. Rosenblatt. Depts. of Chemistry and Physics, Case Western Reserve University, Cleveland, OH 44106, D. Dubois and K. Kadish, Dept. of Chemistry, University of Houston, Houston, TX 77204-5641.
The solution phase properties of C\textsubscript{60} and its mono-, di- and trino- radicals in organic electrolytes have been examined in situ by magnetic circular dichroism (MCD) and Fourier transform infrared spectroscopy. For the MCD measurements, a newly designed instrument was used in which a beam of alternately right- and left-circularly polarized light, aligned essentially parallel to the desired field, is allowed to reflect on a metal plate at near normal incidence onto a highly polished gold working electrode surface. Large differences were observed between the in situ MCD spectra of the neutral and radical anions, characterized by what appears to be a B term at 332 nm, and those of the corresponding electrogenerated anions for which the signals were much weaker and less well defined. In addition, the analysis of these results as well as those obtained with the in situ FTIR measurements is discussed. This work was supported in part by the Gas Research Institute. Valuable discussions with Professor A. Gossow for University of Illinois are gratefully acknowledged.

Recent progress has been made in the extraction of high molecular weight fullerenic ions (1) Fourier transform mass spectrometry (FTMS) using 266 nm laser desorption has proven to be a useful method for obtaining molecular weight distributions of the extracted fullerenes. The advantages and limitations of this technique are discussed. Some examples of the distributions of fullerenes which can be extracted by using different solvents are shown. Since laser desorption can be an energetic method of ion formation, it is necessary to consider the fragmentation of fullerenes into smaller species during the desorption. Alternatively, it has also been found that fullerenes can form by growth processes during laser ablation of some materials; in fact, this is the method by which fullerenes were originally observed (2). Both of these processes are discussed in relation to the observed distributions.


634 FUL Symmetry for Lattice Modes in C\textsubscript{60}, C\textsubscript{70}, and Alkali Metal Doped C\textsubscript{60}. G. Dresselhaus and M. S. Dresselhaus. MIT, Cambridge, MA 02139.

Using both empirical-potential and local-density-functional (LDF) calculations we have computed some properties of a number of available and potentially available fullerene molecules and their derivatives. One of the two most remarkable properties of the fullerenes is that their shell of carbon atoms is not easily penetrated. Large Gaussian-basis-set (corresponding to triple-zeta plus polarization) LDF calculations estimate the barrier for fullerene penetration at 10 eV. Thus fullerenes have a well-defined inside and outside. When the considered bond to a carbon atom inside a fullerene and the quantum mechanical motion of single lithium, sodium, and potassium ions in various charge states of C\textsubscript{60} is considered, all hydrogenating and fluorinating fullerenes also leads to an endohedral problem. We have found that C\textsubscript{60}H\textsubscript{1} is more stable than C\textsubscript{60}H\textsubscript{2} at this fixed point inward. The other most remarkable property of fullerenes is their lack of isomerization. We have computed the LDF total energies of some separated pentagon-pentagon (C\textsubscript{60}C\textsubscript{60}) and C\textsubscript{60}C\textsubscript{60}C\textsubscript{60}C\textsubscript{60}C\textsubscript{60}C\textsubscript{60} and fullerene tubules to gain insight into the very high selectivity in the process discovered by Kratschmer, Hu, and coworkers. We have also computed the electronic structure and photoemission spectra of these molecules. Remarkably, the icosahedral fullerenes have very similar electronic structure and thus could all form superconducting fullerides.
radical cations were observed. The rate constant for electron transfer, which is plotted as a function of orientation potential of the donor, follows the Weller relationship, but the rate constant for exothermic reactions is somewhat lower than the diffusion-controlled value. The common organic reactants because of the high viscosity of benzonitrile. \( \text{C}_6 \) reacts with acetone solutions of dimethylglyoxime to give stable oxidation products. Chromatographic separation gave \( \text{C}_6 \text{C}_6 \) which has the same symmetry as the monomethoxy group of \( \text{C}_m (17 \text{C} \text{NMR lines). This molecule appears to be an epoxide rather an oxygen-bridged anoline, as shown by its relatively high-field 1H NMR peak. The 13C NMR of the second fraction has many similarities with that of the osmyl-

643 FUL Charge Transfer at Fullerene-Noble Metal Interfaces: J. S. Lannin, Dept. of Physics, Penn State University, University Park, PA 16801

In situ, surface-enhanced Raman scattering (SERS) and photomission of \( \text{C}_m \text{noble metal interfaces have provided information on charge transfer effects. Ultrathin films of \( \text{C}_m \) of less than 1 monolayer thickness were deposited in ultrahigh vacuum on potted polycrystalline indium and continuous noble metal films. The SERS spectra indicate large shifts of the high frequency Ag(2) symmetry, pentagonal pinch mode of \( \text{C}_m \) in Ag and Au. In Ag and Cu, UPS difference measurements confirm that the shifts are due to charge transfer as a new LUMO band, derived in part from ion mobilities that allow separation of the Fermi energy indicates the formation of a metallic interfacial state for Ag and Cu. Limited changes in the UPS spectra of Au suggest that in addition to charge transfer, substrate polarization effects are present. XPS measurements indicate shifts of the carbon 1s core line that are opposite the SERS trend, also suggest that in addition to charge transfer processes, polarization effects are present. (This work was supported by NSF Grant DMR 8922305.)

644 FUL Recent Results in the Synthesis and Characterization of Discrete Derivatives of \( \text{C}_{60} \): J. M. Hawkins, Dept. of Chemistry, University of California, Berkeley, CA 94720

Recent results in the synthesis and characterization of discrete derivatives of \( \text{C}_{60} \) are described, including isomorphically pure 2-1 adducts of osmium tetroxide with \( \text{C}_{60} \).

645 FUL Molecular Structures of the Gaseous Fullerenes \( \text{C}_{60} \) and \( \text{C}_{70} \), from Electron Diffraction: K. Hedberg and L. Hedberg, Dept. of Chemistry, Oregon State University, Corvallis, OR 97333, D. S. Bethune, C. A. Brown, R. D. Johnson, and M. de Vries, IBM Research Div., Almaden Research Center, San Jose, CA 95129-6099

In our recent article on \( \text{C}_{60} \) (Science, 254, 410 (1991)) we reported the \( r_1 \) bond lengths from refinements in which gaseous symmetry was assumed and no account was taken of the vibrational correlation factors which occur in \( \text{C}_{60} \) and other fullerenes may even help our insight into the photophysics of other large molecules found in biological and natural systems. The fullerenes have many degrees of freedom that they can store large quantities (\( > 40 \text{eV} \)) of excitation energy for extended periods of time before undergoing fragmentation or ionization. Laser desorption, followed by laser ionization and high-resolution mass spectroscopy, has been used to measure direct ionization fragmentation, detection of neutral fragments, and analysis of metastable ions. We have also undertaken a study of velocity distribution functions. A complex but coherent picture of the processes that occur following photodissociation and that in the various decay channels through charge transfer effects. Ultrathin films of \( \text{Ag} \) and \( \text{Cu} \) on \( \text{C}_{60} \) have been investigated. The results are consistent with \( \text{D}_{5h} \) symmetry. Since \( \text{C}_{60} \) is a much more complicated molecule, thinning of \( \text{C}_{70} \) results in a decrease in the average bond length, allows a study of \( \text{C}_{70} \). Results are consistent with \( \text{D}_{5h} \) symmetry. Since \( \text{C}_{70} \) is a much more complicated molecule, thinning of \( \text{C}_{70} \) results in a decrease in the average bond length. Close examination of the angular distribution of \( \text{C}_{60} \) within and connecting the five-member rings is less precise. Preliminary values for the average bond length \( r_1 \) within and connecting the five-member rings are respectively 1.452 and 1.398 and the length of the unique bond in the molecule is 1.510. Details are reported.

646 FUL Orientational Order in Fullerenes: P. A. Heiney, Physics Department and Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia, PA 19104

The discovery (1) of an orientational ordering transition in solid \( \text{C}_{60} \) has attracted experimental and theoretical interest. The 250 K transition from an orientationally disordered fcc structure to an orientationally ordered simple cubic Pn3 structure (2) is the first order, and is accompanied by a jump in the lattice parameter of \( \approx 0.044 \text{Å} \). The transition is affected by details of sample preparation, including careful removal of residual solvent. Recent experimental and theoretical results are discussed, and the ordering transition in pure \( \text{C}_{60} \) is compared with those in \( \text{C}_{60} \) and \( \text{C}_{70} \) as well as effects seen in dilute mixtures of alkali metal single crystals. (This work was supported by the National Science Foundation, Grants DMR89-01219 and DMR-88-19885. The assistance of J. E. Fischer, A. R. McGhie, W. J. Romanow, and A. M. Denenstein, J. P. McCauley, Jr., and A. B. Smith III, is gratefully acknowledged.)


647 FUL Structures of Carbon Cluster Ions: G. von Helden, R. P. Kemper, and M. T. Roueff, Dept. of Physics, University of California, Santa Barbara, CA 93106

We have recently developed chromatographic methods based on ion mobility and allyl suction. These methods have been applied to carbon cluster ions up to \( n \approx 8 \), generated by laser vaporization.
of graphite. Several families of 

solvophobic interactions involving neutral C₆₀ anions, and the larger cations of the supporting electrolyte in polar solvents, (ii) ion pairing of the C₆₀ anions with smaller cations in non-polar solvents, and (iii) the acceptor and donor properties of the solvents. Studies of K₆₀ values as a function of temperature indicate unusually large entropy accompanying each reduction step. Diffusion coefficients and apparent solvation number of neutral C₆₀ in different systems were also determined and accounted for by the solvophobic effect.

652 FUL Electron Spin Resonance Characterization of Singly, Doubly, and Triply Reduced Buckminsterfullerene, C₆₀, in Aprotic Media: J. T. Jones,* D. Dubois, and K. M. Kadish, Dep. of Chemistry, University of Houston, Houston, TX 77204-5841.

The results of ESR studies on some anions reduced of Buckminsterfullerene, C₆₀ (n = 1, 2, and 3), in the solvents benzonitrile, dichloromethane, p-xylene, and tetrahydrofuran are reported and discussed. The samples are prepared by electrochemical reduction techniques which allow the preparation of samples which contain, quantitatively and selectively, the desired anionic species. The results of ESR spectral studies on the anions in frozen solutions as a function of temperature are presented. The ESR line shapes and linewidths are dependent upon the solvent and the temperature whereas the g-values are only weakly dependent upon the temperature. The C₆₀ g-value averages 1.998 over the four solvents and over the temperature range used. The resonances of C₆₀ are centered at about 2.00 and display a triplet ground state with an average separation of 12.6 A between the two unpaired electrons. The temperature dependence is similar to that of the free electron and exhibits features similar to the monomeric anion, suggesting a spin of 1/2. The results obtained from this study are presented and discussed in terms of the physical structure and properties of C₆₀, the lifting of the initial orbital degeneracy of the T, LUMO of C₆₀, and the thermally accessible excited states of the C₆₀ anions.


The recent discovery of molecular clusters of carbon has prompted a wide range of theoretical and experimental studies regarding their physical and chemical properties. None of these studies, however, has been concerned with the intercalation of these molecular clusters in a suitable host matrix and the properties of the resulting intercalate. In this paper, we report the synthesis and properties of ethylenediamine-functionalized C₆₀(en), intercalated fluorohectorite, a synthetic mica-type silicate. X-ray diffraction and spectroscopic measurements show that intercalation leaves the C₆₀ cluster intact and that the host galleries are occupied by a monolayer of guest molecules. The intercalated C₆₀=en, clusters exhibit a higher thermal stability compared to pristine C₆₀, and C₆₀=en with no evidence for reaction with the silicate host. The thermal and electrical properties of the intercalates after elimination of the en ligands at intermediate temperatures are presented and compared to those of pristine C₆₀ and graphic carbon layers obtained through pyrolysis of intercalated organic molecules or polymers.


We have developed a force field for buckyball molecules and for alkali-doped fullerenes and have applied this force field to: (i) predictions of crystal structures for C₆₀ and C₇₀ as a function of temperature and pressure; (ii) predictions of crystal structures of M₆₀ for M = Li, Na, K, Rb, Cs and z = 1, 2, 3, 4, 6, 8 and; (iii) predictions of the vibrational frequencies for finite molecules (C₆₀, C₇₀, C₈₀, etc.) and of the phonon band structure of the calculated phonon spectrum and electronic band structure to predict from first principles the superconducting properties of K₆₀ (assuming BCS theory). The predicted Tc is within 4K of the observed value, providing strong support for the BCS description of these systems.


A new technique for large scale separation of C₆₀ and C₇₀ and also for subsequent separation was described. Thermodynamic data, derived from solubility data and sorption microporeometry data of C₆₀ in solvents typically employed in extraction and separation of the solid state properties of C₆₀, solvent-mixtures similar to those employed as stationary phases in chromatographic separations, are presented.

Photochemically and thermally generated organic radicals add rapidly to aryl radicals to form adducts. For example, mass spectrometric analyses show the formation of the adducts $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ with $n = 1$ to at least 58 and of $(\text{C}_n\text{H}_m\text{C}_n\text{H}_m)$ with $n = 1$ to 15. The reaction of the radical adducts $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ with $n = 1$, 3, and 5, and Proton and $\text{CH}_2$-hy- perconjugations indicate that in the initial radical adduct $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$. (R = tert-butyl, 1-adamantyl, isopropyl, ethyl, trichloro- methyl, benzyl, etc) the unpaired electron is mostly confined to two functional groups. The surface of $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ having a substituent R at one of the points of fusion. Each half of the resulting radical structure of $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ is completely separated from the other half along the fivefold axis of $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ serve to refine such a radicalical structure. In particular, the calculations suggest studying the effect of the relative IPs of $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ and $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$. Isolation and subsequent fragmentation of these $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ species by collision-induced dissociation give either $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ or $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$ and $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$. and $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$. Further studies indicate that in the initial radical adduct $\text{C}_n\text{H}_m\text{C}_n\text{H}_m$, the unpaired electron is delocalized in an allylic fashion. In the gas phase was studied a low-temperature combination. The formation of species with two unpaired electrons is also discussed.

657 FUL. Radial Vibrations of an Ion Inside Icosahedral $\text{C}_{60}$; $\text{C}_{60}/\text{H}^+$ Complexes. W. G. Dunlap and B. L. Fawcett, Dept. of Physics, Emporia State University, Emporia, KS 66801

The very high symmetry of icosahedral $\text{C}_{60}$ suggests that as a first approximation, an ion trapped inside $\text{C}_{60}$, would feel a potential that is radially symmetric about the center. All electronic, local-density-functional (LDF) calculations of the total energy of a sodium ion function of radial displacement from the center of the icosahedron along the fivefold axis of $\text{C}_{60}$, serve to refine such a radial potential. In particular, the calculations suggest studying the effect of the relative IPs of $\text{C}_{60}$ and $\text{C}_{60}$ and its subsequent fragmentation of these $\text{C}_{60}$ species by collision-induced dissociation give either $\text{C}_{60}$ or $\text{C}_{60}$ and $\text{C}_{60}$. Further studies indicate that in the initial radical adduct $\text{C}_{60}$, the unpaired electron is delocalized in an allylic fashion. In the gas phase was studied a low-temperature combination. The formation of species with two unpaired electrons is also discussed.

658 FUL. Organometallic Chemistry of Fullerenes in the Gas Phase by PTMS: Y. Huang, Q. Jian, S. A. Lee, and B. S. Frevin, Dept. of Chemistry, Purdue University, West Lafayette, Indiana 47907

The chemistry of fullerene- $\text{C}_{60}$ and $\text{C}_{70}$, with transition metal ions in the gas phase was studied by a PTMS technique. Fullerene samples heated off a solids probe react with a variety of transition metal ions via condensation to give $\text{M}_{n}\text{C}_{60}$ and $\text{M}_{n}\text{C}_{70}$. Alternatively, both $\text{C}_{60}$ and $\text{C}_{70}$ can react with a metal ion (e.g., $\text{M}^2+ = \text{M}^2+$) via displacement to give $\text{MC}_{60}$ and $\text{MC}_{70}$. Isolation and subsequent fragmentation of these $\text{MC}_{60}$ species by collision-induced dissociation give either $\text{M}^2+$ or $\text{C}_{60}$ and a combination of both depending on the relative IPs of $\text{M}$ and $\text{C}_{60}$. Such a fragmentation pattern is distinctly different from those of endohedral $\text{M}_{n}\text{C}_{60}$ and $\text{M}_{n}\text{C}_{70}$. The structure of the reaction products was determined by NMR and mass spectrometry. The presence of metal lone-pair density on the fullerene lattice with a lattice parameter $a = 17.1 \text{\AA} (\text{C}_{60})$ and $a = 17.16 \text{\AA} (\text{C}_{70})$. The powder x-ray diffraction of the reaction products exhibited a few broad peaks consistent with the face lattice with a lattice parameter $a = 17.16 \text{\AA} (\text{C}_{60})$.

659 FUL. Solubilization of Buckminsterfullerene, $\text{C}_{60}$, in Water and Some Polar Organic Solvents by Cy- clocelodrin Inclusion Chemistry: W. Kutner,* P. Boualas, and K. M. Radish, Dept. of Chemistry, University of Houston, Houston, TX 77204-3641

Highly hydrophobic buckminsterfullerene, $\text{C}_{60}$, is virtually insoluble in most polar solvents, but is shown to solubilize in water as well as 11.5% (v/v) aqueous methanol, 1.1% (v/v) aqueous ethanolic DMF or DMSO solutions by forming inclusion complexes with cycloextrin (CDs). The internal diameter of the 6- and 8-CD, and $\text{C}_{60}$, molecules is 10-11 Å. The data suggest that only a slight penetration of $\text{C}_{60}$ into the CD cavities is possible. Surprisingly, however, the 6- and 8-CDs and $\text{C}_{60}$, reveal good host-guest properties. UV-vis spectroscopy of $\text{C}_{60}$, in each of the above solvent systems indicates that a solubilization of $\text{C}_{60}$, in CD solution does not significantly affect its electronic properties. Only slight shifts and changes in the relative heights of the three intense UV bands of $\text{C}_{60}$, are observed as compared to those of $\text{C}_{60}$, and in CD solution. Measurements of the CD spectrum of a different CD/DMF mole ratio allows one to derive the binding isotherm for each inclusion system and also determine the inclu- sion constant and stoichiometry and stability constant. The relative fluorescence signals of $\text{C}_{60}$, in DMSO or DMF solutions containing CDs resemble those reported in pyridine or benzoni- trile solutions of CDs. However, mixed aqueous/alcoholic solutions of CDs suggest that electron- duced $\text{C}_{60}$, chemically reacts with the solvent.

660 FUL. Excited State Behavior of Fullerene Derivatives ($\text{C}_{60}$, $\text{C}_{70}$) and Their Reduction as an Oxidation Catalyst. Suspensions: P. V. Kanat and N. M. Dimitrijevic, Radiation Lab- oratory, University of Notre Dame, Notre Dame, IN 46556

Two products were obtained and preliminarily characterized when redox-active buckminsterfullerene (C$_{60}$) was used as a C$_{60}$ oxidation catalyst. Reaction products were monitored by UV-liquid chromatography using a newly developed, high performance gel permeation chromatography column constant. Oxalic acid and oxalic acid-ethyl were identified. UV-Visible absorption spectra of $\text{C}_{60}$-Environop-ABC$^+$ (ABC Laboratories, Inc., Columbus, 05620)

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formed which eluted at 3.3 and 6.6 min. The product eluting at 6.6 min first was detected after 20 h, and the component eluting at 3.3 min was detected after 72 h. A decrease in C60 concentration coupled with increased amounts of 50% of the C60 and 6.6 min product were depleted after 90 h; after 256 h of reaction, only the 3.3 min product remained. The amount of 6.6 min product was increased by use of solvent system of 5% methylene chloride in toluene (v/v). In the absence of argon atmosphere, only product eluting at 3.3 min was observed. Solvent atmosphere is important for this reaction, perhaps due to different solubilities of the catalyst, C60, and C70. No reaction occurred in 1,1,1-trichloroethane or t-butyl methyl ether. With methylene chloride as solvent, a black solid was obtained. The UV spectrum of the product eluting at 6.6 min is very similar to that of C60, suggesting that this product has a similar electronic structure. With the absorption bands between 240-350 nm, however, alpha-max was shifted to shorter wavelengths. The UV spectrum of the 3.3 min product exhibited broad absorption at 6 nm 220-400 nm (alpha-max = 235 nm) suggesting more structural modification occurred. Small amounts of the two compounds isolated from the QPC column were examined by negative ion FAB-MS and 13C-NMR. Formulas of C60, C60C12, and C60Cl18 were assigned to the major constituents in the 6.6 and 3.3 min peaks, respectively, and the 13C-NMR supported these MS results. Multiple chemical shifts were observed between 22-69 and 130-148 ppm for the 6.6 min constituent, indicative of opening of double bonds in the presence of double bonds other than those of C60. Chemical shifts for the 3.3 min constituent were observed at 13-45 and 128-146 ppm.

664 FUL: Simultaneous Electrochemical Quartz Crystal Microbalance and Cyclic Voltammetry Study of Electropozituation, Doping with Countercations and Electrodisolution of Films of Buckminsterfullerene (664 FUL) and its associates such as C60 and Aromatic Solvents: W. Koh, D. Dubois, W. Kuzmer, T. M. Jones, and K. M. Kadish. Dep. of Chemistry, University of Houston, Houston, Texas 77204-5841

A simple and efficient method was developed for preparation of insoluble thin films of C60 and C70. The method consists of first bulk electrodoping of a fine suspension of solid C60 in an aerotrimethylamine solution under a controlled potential which is sufficiently negative to generate a solvated C60-. This is followed by electro-oxidative deposition of a film on the surface of a gold/quartz crystal working electrode. The properties of the electropozited films were examined by cyclic voltammetry and electrochemical quartz crystal microbalance (EQCM) experiments which enabled determination of stoichiometry and film thickness. The C60 film exhibits oxygen uptake reactions upon electroreduction, leading to the formation of fullerene mono- and dimers. The method of film preparation can also be extended to other solvents in which C70, is insoluble. The cyclic voltammetry and EQCM behavior of the film is largely dependent on the nature of the solvent and supporting electrolyte used. These data are discussed with respect to the solution behavior of C60.


Several nonlinear optical (NLO) experiments have been initiated here at NRL to investigate the NLO properties of the fullerences (1). Time-resolved degenerate four-wave mixing (DFWM) and Z-scan studies were conducted on solutions and films of the fullerences, C60, C70, and C84. A Q-switched, mode-locked 35 ps Nd:YAG laser was used at 1.064 μm whereas a synchronously pumped, mode-locked 1 ps dye laser, tunable in the visible, was operated near 600 nm. The relationship between the molecular structure/symmetry of the fullerences and their third-order optical properties is studied. The electronic and dynamic nonlinearities of these molecular clusters, and their time responses are discussed. Mechanisms giving rise to third- and fifth-order optical processes in the different time domains are explored.

666 FUL: Nonlinear Optical Second-Order Harmonic Generation and Inhibition of Growth of Films of Buckminsterfullerene (666 FUL) and its associates such as C60 and larger fullerences offer a unique possibility: to study the dynamics associated with the absorption of photons in a free large cluster of high symmetry and to compare experimental results to a variety of data available from solids or surfaces. In our experiments, free fullerence molecules or clusters are formed by thermal evaporation from an oven or by laser evaporation. Using multiphoton excitation and a modified time of flight technique delayed ionization of C60 has recently been observed. Using single photon ionization with synchrotron radiation the photoionization spectra of fullerences were measured between threshold (7.54(eV) for C60) and 21 eV. The ion yield is increased by a giant plasmon resonance at about 20 eV which was recently predicted theoretically. In our time of flight setups the fullerene ions can easily perform collisions with various targets to form thin film in a corona poling process. Experiments give insight into the non-destructive fragmentation of fullerences as well into the formation of heterogeneous compounds in the collision process. Experiments were shown to yield further progress in this field.

667 FUL: Thermophysical Properties of Solid C60, C70, and Related Materials: J. L. Martin, C. P. Man, M. S. Chandrasekaran, M. P. Sampson, R. H. Hauge. Dep. of Chemistry and Rice Quantum Institute, Rice University, Houston, TX 77251

The recent availability of gram-quantities of C60/C70 mixtures as well as multigram samples of pure C60 and C70 have made possible a detailed study of their thermophysical properties.

C₆₀, C₇₀, etc. have allowed measurements of (i) heats of combustion, (ii) vapor pressures, (iii) heat capacities, (iv) ionization potentials, and (v) electron affinities. Also, the reactions of solid C₆₀-C₇₀ with F₂, Cl₂, H₂, HBr have been studied.

672 FUL Superconductivity in Metal-Doped C₆₀ Solids. C. M. Lieber, C. C. Chen, Z. Zhang, and S. P. Kelly, Dept. of Chemistry and Division of Applied Sciences, Harvard University, Cambridge, MA 02138

Recently, there has been significant effort directed toward exploring superconductivity in alkali metal doped C₆₀ solids and films. The transition temperatures for K-doped C₆₀ (18 K) and Rb-doped C₆₀ (28 K) are significantly higher than previously reported for rare gas based superconductors, and thus suggests that metal doped C₆₀ represents a new class of high-Tₜ superconductors. Herein recent results from our laboratory that address the synthesis, characterization and superconducting properties of these materials are discussed.

A series of new alkali-metal doped metal materials have been prepared with the general formulas KₓC₆₀, CsC₆₀, and CsC₇₀ containing about 1 mole percent metal. Thermodynamic measurements of the superconducting transition temperature with lattice size in addition, extensive low-temperature tunneling measurements of the superconducting energy gap have been carried out. The implications of these and other new data to understanding superconductivity in these materials are discussed.

673 FUL Electrochemical Reactivity of Fullerenes and Their Derivatives. F. D. Sosa, R. Saxhadt, R. Abugan, G. Raj, V. Krishna, and C. C. N. Reddy, Solid State and Structural Chemistry Unit, CSIR Centre of Excellence in Chemistry and Dept. of Inorganic and Physical Chemistry, Indian Institute of Science, Bombay 400012, India

Fullerenes, C₆₀, and C₇₀, exhibit favorable one-electron reductions by cyclic voltammetry in benzene solution. As a result, we found three isomers for C₇₈ (2x C₆ₓ and D₅) and five for C₇₀ (M, C, 2x C, and D₅) and two major isomers (D and D₅) for C₈₅. From the structural evidences obtained for C₇₆, C₇₈, and C₈₅, we see such an interesting tendency that the fullerene cages with the lower symmetry are preferentially formed. The propensities of structure formation and magic numbers in the formation of the fullerene cage would suggest the important role of dynamical aspects in fullerene growth process rather than the thermodynamic stabilities.


A variety of mass spectrometric (MS) techniques have been used to characterize the gas-phase properties and reactions of fullerene derivatives containing metal atoms such as ionization potentials (IPs) and proton affinities (PAs) have been determined by charge-transfer and proton-transfer bracketing measurements. Chemical ionization (CI) has also been used as a sensitive method for the analysis of fullerenes and their derivatives in addition to producing metal fullerene cation complexes from ion/molecule reactions in the CI plasma. Recent studies have focused on the generation of endohedral fullerene cation complexes using two different production methods: High-energy (keV) collisions of He with inert gas atoms results in fragmentation and/or uptake of the neutral target atom. Tandem MS experiments and molecular dynamics simulations of the collision process provide convincing evidence for their endohedral structure, e.g., HeC₆₀⁻. In other studies, direct laser vaporization followed by mass spectrometric investigations have resulted in stable metal fullerene complexes and a variety of new fullerene structures such as [Y @ C₆₀ C₆₀C₆₀]⁺, [Y @ C₆₀ C₆₀C₁₂]⁺, and [Y @ C₆₀ C₁₀C₁₀C₄]²⁻. In situ experiments, direct laser vaporization has provided a new tool for studying these materials and their reactions with other molecules.

675 FUL Synthesis of a C₅₆-p-Xylylene Copolymer. H. D. Wu, J. F. W. McMillan, and J. A. Bradley, School of Chemistry, University of Melbourne, Parkville, VIC 3052, Australia

The preparation and purification of macroscopic quantities of buckminsterfullerenes or buckyballs has ignited an explosion of research into their physical and chemical properties, and a variety of strategies for preparing fullerenes has emerged. Chemical derivatives of C₆₀ fullerene can be prepared by a variety of methods. We report a polymeric fullerene material prepared by reacting C₆₀ with xylylene. The polymerization of xylylene on fullerene helps to achieve the formation of C₇₈, C₇₆, and C₇₀. The polymerization process was performed at room temperature.

676 FUL Diamond from Fullerenes: M. Nuñoz Reguero and P. Monceau, CNRS, Cedex 9, 38042 Grenoble, France. J.-L. Hodeau, L. Crystallographie, CNRS, Cedex 9, 38042 Grenoble, France

High pressures studies have shown that C₆₀ molecules are stable under hydrostatic pressures up to about 20 GPa, with the material conserving the low pressure phase. However, nonhydrostatic loading induces phase transitions to lower symmetry phases that appear to be insulating. It is shown here that extreme nonhydrostatic compressions causes the collapse of the sphericals around 20 GPa at room temperature. Depending on the magnitude and conditions of the applied pressure, the resulting material can be polycrystalline diamond or amorphous material. Our results suggest an alternative route to industrial diamond synthesis.

677 FUL Large Fullerenes: Structures and Growth Mechanism. Y. Achiwa, Department of Chemistry, University of Tokyo, Tokyo Metropolitan University, Hachioji, Tokyo 192-03, Japan

Here we report structural and electronic properties of higher fullerenes up to C₁₅₈ (C₁₇₆, C₁₈₄, C₂₀₄, C₂₃₆, etc.) which were isolated from the carbon soot generated by arc heating of graphite, using a preparative HPLC. Mass spectrometry characterization was performed showing that purified isolated fullerenes is better than 95%. UV/visible optical absorption spectra indicate benzene like that absorption threshold is shifted to the longer wavelength with increasing size of fullerene. Structural information has been deduced by °C NMR for well-isolated fullerenes in C₂. Solution, as a result, we find three isomers for C₇₈ (2x C₆ₓ and D₅), five for C₇₀ (M, C, 2x C, and D₅), and two major isomers (D and D₅) for C₈₅. From the structural evidences obtained for C₇₆, C₇₈, and C₈₅, we see such an interesting tendency that the fullerene cages with the lower symmetry are preferentially formed. The propensities of structure formation and magic numbers in the formation of the fullerene cage would suggest the important role of dynamical aspects in fullerene growth process rather than the thermodynamic stabilities.


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679 FUL ESR and Optical Studies of the Radical Ions of Fullerenes. T. Kato, T. Kodama, T. Shida, S. Suzuki, and Y. Achiwa, Kyoto University, Kyoto 606, Japan

The electronic absorption spectra of the radical ions of C₆₀ and C₇₀ are observed in v-irradiated glassy polyatomic matrices at 77 K. The spectral features of all the radical ions are consistent with the available information in the literature which includes electronic transitions and resonances. The radical ions of C₆₀ and C₇₀ are observed in the electronic absorption spectra of the radical ion in the frozen matrix was suggested as being due to the Jain-Teller distortion.

680 FUL Electronic Structures and Superconductivity of Fullerenes and Fullerenes: S. Saito, * A. Oshikawa, Y. Miyamoto, S. -i. Saumda, and N. Hamada, Fundamental Research Laboratory and Microelectron Research Laboratory, NEC Corp., 34 Miyukigakosha, Tseukuba, Ibaraki 305, Japan

We present the electronic structure of fullerene and fullerides obtained by using the Density-function theory as well as the realistic tight-binding model. The electronic structures of the large fullerenes, C₆₀, C₇₀, C₈₅, etc. as well as C₆₀ are reported and compared to the photoemission spectra. Calculated density of
states of the structural isomers of larger fullerenes are found to be considerably different from one another and did play an important role in determining the actual geometries of larger fullerenes extracted from graphite-arc soot. Cohesive mechanisms and energy bands of solid-state derivatives of C60, i.e., pure solid C60, K2C60, Rb2C60, and Cs2Br, are also discussed. The high electronegativity of C60 is found to be of significant importance for their properties.

The unusual linear relationship between the Fermi-level density of states and the superconducting transition temperature (Tc) in a wide range has been found in K2C60 and Rb2C60. The validity of the McMillan equation for Tc will be reexamined in these narrow conduction band superconductors with high frequency vibrational modes.


Immediately after the discovery by Kratschmer and Huffman of fullerene isomers such as C70, C74, C76, and C80, as well as functionalized isomers such as alkoxylated and arylated C60, the field is beginning to diminish. We report some procedures for the purification of fullerene salts. Strongly reducing metalloporphyrins react readily with C60, giving the ortho arenal C60 derivatives such as C60(C6H4OH)3-or C60(C6H4Br)3, which are isolated as the fullerited salts. This approach is of broad interest for the synthesis of new materials possessing diverse properties. Several groups, including our own, have been developing synthetic strategies for the synthesis of new fullerene derivatives. We have also explored new methods for the purification of fullerenes such as C60, and the alkali metal derivatives derived from them.


We report the preparation of fullerenes in very high yield in a plasma discharge using a mixture of three precursors in the mass spectrometer. From selective solvent extraction, we can isolate different molecular weight ranges of fullerenes for subsequent separation and characterization by several methods. We have also developed a one-step method for the purification of C60 in yields of 8% directly from the source material. The reaction is of broad scope for the synthesis of derivatives such as alkoxylated and arylated C60, as well as functionalized C60.

683 FUL Larger Fullerenes and Functionalized Fullerenes: Structures and Stabilities: K. Raghavachari, AT&T Bell Laboratories, Murray Hill, NJ 07974; C. M. Rohlfing, Sandia National Laboratories, Livermore, CA 94550

In this work, we investigate the structures and stabilities of larger fullerences such as C70, C84, and C90, and C96, and as well as functionalized fullerences such as C60(CH3)2 and C50CNH2. Both semi-empirical and ab initio quantum chemical calculations have been used in this study. Larger fullerenes such as C70, C84, and C90, have several structural isomers which have all the pentagons isolated as well as function- alized fullerenes such as C50CNH2 and C50CH2. Semi-empirical and ab initio quantum chemical calculations have been used in this study. Larger fullerenes such as C50CNH2 and C50CH2 have several structural isomers which have all the pentagons isolated from each other. Total energy minimization techniques have been used to determine the geometries and relative energies of the isomers. The stabilities of the isomers are analyzed in terms of the local environments of the pentagons and hexagons in the struc- tures. The isomers can be recognized which can characterize such isomers in a predictive manner. The results indicate that semi-empirical and ab initio quantum chemical calculations have been used in this study. Larger fullerenes such as C50CNH2 and C50CH2 have several structural isomers which have all the pentagons isolated from each other. Total energy minimization techniques have been used to determine the geometries and relative energies of the isomers. The stabilities of the isomers are analyzed in terms of the local environments of the pentagons and hexagons in the struc- tures. The isomers can be recognized which can characterize such isomers in a predictive manner. Correlations between the structural and electronic properties are discussed. Derivatized fullerenes such as C50CNH2 and C50CH2 have a O or CH, group bridging a naphtalene-like structure in the fullerene. The bond- ing in the derivatized fullerenes is compared to that in C60. In par- ticular, the nature of the bond length across the bridging C-C bond is discussed in detail. Vibrational spectra of derivatized fullerenes are predicted.

684 FUL Coordination Chemistry with Fulleride Ions: C. A. Reed, * A. Penczek, P. Bihraptra, and J. Hsu. Dept. of Chemistry, University of Southern California, Los Angeles, CA 90009-0744

The theoretical chemistry of C60- and C70- is a material limitation of single-electron oxidation. We have investigated various properties of C60- and C70-, a TG, that improve the most tedious part of C60- and C70- isolation—i.e., that of chromatography on alumina. In a typical procedure, mg of high purity C60- or C70- is added to a mg per column, per day basis. Sodium reacts with C60- in THF in the presence of a crown ether to give synthetically useful fullerides salts. Strongly reducing metalloporphyrins react readily with C60-, giving the ortho arenal C60 derivatives such as C60(C6H4OH)3 or C60(C6H4Br)3, which are isolated as the fullerided salts. This approach is of broad interest for the synthesis of new materials possessing diverse properties. Several groups, including our own, have been developing synthetic strategies for the synthesis of new fullerene derivatives. We have also explored new methods for the purification of fullerenes such as C60, and the alkali metal derivatives derived from them. Photomission and inverse photomission results reveal the distribution of states in the valence bands and conduction bands of these molecular solids. They also show the changes induced by alkali doping, particularly the filling of the first set of conduction bands associated with Li, Na, K, Rb, and Cs fullerenes. UV/Vis spectrum results are discussed for thin films of C60 and C76, and for K3C60, in the metallic and insulating states. The STM results show fullerene trapping at GaAs(110) steps and film growth on the surface by step flow. Novel growth structures are emphasized for these high temperature van der Waals solids. Photomission results for C60 allow for a detailed determination of the coefficients of the expansion for C60 implied by the symmetry of the ground state of fullerite are illustrated.

687 FUL Electron-Phonon Coupling and Superconductivity in Alkali Intercalated C60: Solid: M. A. Schluter. AT&T Bell Laboratories, Murray Hill, NJ 07974

We propose the superconduction in A2C60 (A = K, Rb) with Tc > 30 K results from a favorable combination of high phonon frequencies and the existence of two different energy scales optimizing the coupling constant. In N, V, calculations show that the electron scattering V is dominated by particular on-ball John- Teller-type modes on the scale of the large on-ball x-hopping energy, while the density of states N is controlled by the weak inter- ball hopping energy. This factorization has several observed experimen- tal consequences. Data on atomic substitution, Raman and neutron scattering, and x-ray absorption measurements can be explained naturally. Crucial differences to intercalated graphite explain the much smaller Tc values in the graphite compounds (this work was done in collaboration with M. Lannoo, M. Needs, G. A. Baraff, and D. Tomkan.)

688 FUL The Equilibrium Structure of Big Fullerenes: Spherical or Cylindrical Shape? G. E. Scuseria. Dept. of Chemistry and Rice Quantum Institute. Rice University. Houston, TX 77251-1892

The recent experimental discovery [S. Ijima, Nature, 354, 56 (1991)] that a new type of structure consisting of needlelike mic- ritubules of graphitic carbon can be grown into such small length might constitute a vast store of carbon fibers. In this work, ab initio SCF calculations were used in order to predict the equilibrium structure of such a needlelike micritube. The carbon nanotubes were optimized employing analytic energy gradients within the direct SCF approach. Several electronic states and different
689 FUL Mass Spectroscopic Characterization of Large Fullerenes and Metallofullerenes: H. Shimohara,* Dept. of Chemistry for Materials, Mie University, Tsu 514 Japan, Y. Saito, Dept. of Electrical Engineering, Mie University, Tsu 514 Japan

Identification of fullerenes, particularly the fullerences larger than C70, is a crucial part of the experiment when it comes to the isolation of fullerene molecules. Mass spectrometry is a powerful tool for the identification of fullerences. However, mass spectrometry usually entails fragmentation of "intact" parent ions which damages mass spectra. Fullerences have been known to give even-numbered fragment ions on ionization unless the mildest ionization/Desorption conditions are used. In the first part of the present study, we report on the results of the various types of mass spectrometry of the larger fullerences, which include E.LAB, SIMS, laser desorption (LD), and liquid-MS (LC-FAB). SIMS and LD have been used for the production and characterization of fullerences, which were produced by a usual carbon arc method with various metals. The results of LD and FAB mass spectrometry of some metallofullerenes, which were produced by a usual carbon arc method with various metals, are discussed.

690 FUL Experimental HIF of Formation, Vapor-Phase UV Spectrum, Vapor Pressure and Heat of Sublimation of C60, Buckminsterfullerene: D. R. Kirklin, Chemical Thermodynamics Div., Center for Chemical Physics, National Institute of Standards and Technology, Gaithersburg, MD 20899, and W. Smith* and Y.-W. Hsu, Dept. of Chemistry, Drexel University, Philadelphia, PA 19104; G. Glinski, R. J. Romanowicz, laboratory for Research in the Structure of Matter, University of Pennsylvania, Philadelphia, PA 19104. We have measured the energy of combustion of solid C60, in the NIST aneroid adiabatic rotating calorimeter. The standard molar enthalpy of combustion is 25.804 ± 0.003 kJ mol⁻¹, and the derived enthalpy of formation of solid C60, is 2193 ± 10 kJ mol⁻¹ before the correction for aliphatic hydrocarbon impurity level. Using the enthalpy of formation of C60, and the heat of sublimation of C60, we derive an average carbon-carbon bond energy in C60 of 4.41 kcal mol⁻¹, roughly equal to the weight-averaged average of the C=C double bond strength and the C-C single bond energies. We have also measured the temperature dependence of the vapor pressure of C60 from 360 to 860 °C by recording the ultraviolet absorption and emission spectrum of C60, using methods similar to our previous study of sulfur vapor (1). At these temperatures in vacuo, solid C60 resists chemical degradation for days, but may exhibit some phase transition between 820 and 880 °C. Claeyson plots of the data give a heat of sublimation of C60, Preliminary values are P (kcal/mol) = 0.01 Torr at 530 ± 20 °C and the sublimation enthalpy 37.5 ± 0.3 kcal/mol. 1 R. I. Burns and A. L. Smith, J. Phys. Chem., 95, 4242 (1991).

691 FUL Structure of Superconducting and Ferromagnetic Fullerences P. W. Stephens, Dept. of Physics, SUNY, Stony Brook, NY 11790 We review the crystal structure information determined by the present coworkers (11) and others on superconducting A Duchess of Cambridge, metal Na, and Na in superconducting TDAE-C60, (TDAE = C6H4(CH2)2C(CH3)=CH2, (where TDAE = C6H4(CH2)2C(CH3)=CH2). The results provide some explanation for the strongly different behavior of fullerences based on alkali metals and on the detailed mechanisms for the observed properties of these materials. 1 Work performed in collaboration with (so far) P. M. Allemand, D. E. Cox, T. N. Anderson, A. H., Holczer, S. M. Huang, R. B. Kaner, J. W. Lauber, P. L. Lee, Q. Li, L. Mihaly, R. H. Thompson, H. I. Whetten, J. B. Wiley, and F. Wudl.

692 FUL Isolation, Spectroscopy, and Chemical Reactions of Fullerenes: R. Taylor, P. R. Birke, J. H. Holczer, E. C. Langley, M. F. Medin, T. J. Dennus, J. P. Hare, H. W. Krcal, and D. R. M. Walton. School of Chemistry and Molecular Sciences, Universities of Sussex, Brighton, Sussex, England (ENI 91). The chromatographic work that led to the isolation of the first pure samples of C60 and C70 is described, together with the most recent work on the HPLC separation of the high fullerences Structural features which account for the wide difference in stability of different fullerences are identified. An account is given of the work of the Sussex group (and their collaborators at Leicester and Southampton Universities) on reduction, photochemistry, and the formation of metallofullerenes. Some of the recent work of the Sussex group and collaborators at Los Alamos National Laboratory is described. The synthesis of the C60-carbon C60-fullerene analogues is described, together with the results of some preliminary work on the synthesis of metallofullerenes. A complete list of the halogenated fullerences is given, including fullerences containing 3-12 of the high-order bonds of fullerences are discussed together with fullerences containing 3-12 of the high-order bonds of fullerences. The probable sites involved, the fundamental electron-transfer rate constants of fullerences, and the second order nonlinear optical properties of metallofullerenes are also discussed.

693 FUL Collective Electronic Excitations in Fullerenes: D. Tománek, Dept. of Physics and Astronomy and Center for Fundamental Materials Research, Michigan State University, East Lansing, MI 48824-1116

We have investigated the possible occurrence of collective electronic excitations in carbon fullerens. The electronic excitations of the high multipolarity. (Work done in collaboration with G. F. Bertsch, A. Buliga, N.-J. Ju and Y. Wang, supported by the National Science Foundation under Grant No. PHY-9028537.)


We report on studies of metal fullerence complexes, involving various group IIA elements, i.e., Ra, Sc, Y, Eu, Tm, and Gd. The complexes are produced by coevaporation of carbonaceous material and various metals in an arc discharge in He. The resulting soot is treated in various ways, such as extraction and sublimation, and studied by laser desorption, laser ionization, mass spectrometry as well as the EPR spectroscopy. Many of the complexes appear to be very stable. The metal C60 and C70 complexes were found to be unstable upon heating, but could be laser desorbed. The species involving heavier fullerences, such as C60 or C70, were found to be more stable. Several complexes were found with two or three metal atoms. Generally, there seems to be a general propensity for finding multiple metals associated with the carbon cage as the ionic radius of the metal decreases. Both the fluorescence and the wave-length of the ionizing laser were varied to investigate fragment propagation rates, ionization efficiency and ionization energies. All tests to date are consistent with metal atoms being inside the carbon cage. We also report the latest results in our search for fullerences in carbonaceous meteorites, relevant to the probability of fullerences in interstellar space (Collaborative work with S. Chang and E. Peterson, NASA-Ames Research Center, and R. Fleming. Charles Evans and Associates.)


Fullerenes form charge-transfer complexes with electron donors. There is no appreciable charge separation in the ground state of the complex. However, upon photo-excitation, charge-transfer state is formed which gives rise to novel nonlinear optical and photoconductive properties. In the first part of the paper, we report the third order nonlinear optical properties of fullerences (C60 and C70) and the second order nonlinear optical properties of fullerence charge-transfer complexes. Significant optical properties have been observed in both cases. We then present our recent data demonstrating significant photoconductivity in fullerence-doped polymers. The photoconductivity of fullerence-doped polymers is comparable to some of the best commercial photoconductors such as the thio-pyryridine dye and the acridine-doped acridine. The fundamental electron-transfer rate constants of fullerene singlet and triplet states have been determined by time-resolved
Fullerene carbon cages continue to produce a wide variety of new and exciting chemistry. Our recent research efforts have concentrated on the synthesis and characterization of metal fullerides. Potassium and rubidium doped C\textsubscript{60} (FCC M\textsubscript{60}C\textsubscript{60}) exhibit superconducting transition temperatures of 19.3 and 29.6 K, respectively. Experiments with C\textsubscript{60} and Na, K, and Rb show that although sodium fulleride has an FCC lattice, both it and sodium-containing mixed-metal compounds are not superconducting. X-ray powder diffraction and magnetic susceptibility data are presented on these systems. A series of lanthanide endohedral compounds have been produced from arc-burning of filled graphite rods, and these also are discussed.

*The Fullerenes: Chemistry, Physics and New Directions Symposium was supported in part by the Office of Naval Research.*
JOINT RECENT NEWS PAPER SESSION
Electronics/Dielectric Science and Technology
Promenade Ballroom A, 2nd Level
Thursday, May 21, 1982

698 RNP Electrochemical In Situ Diagnostics for Light-Induced Wet Etching of Multilayer Structures: Th. Fink and R. M. Osgood, Jr., Microelectronics Sciences Laboratories, Columbia University, New York, NY 10027

This work presents an electrochemical method for in situ monitoring of heterostructure etching. Experiments with AlGaAs/GaAs multilayer samples showed that measurement of the current in the electrochemical cell or of the potential of the sample clearly indicates the passing of an interface between two eplavers, during light-induced etching. Therefore, the technique can be used to localize etching in a particular region. It can be used for the etching of multilayers which makes it important for the processing of multi-quantum well materials.

699 RNP The Effect of Energetic Ion Bombardment on the Activation Energy of the Etching Reaction Between Silicon and Fluorine: P. M. Kopalidis and J. Jorne, Dept. of Chemical Engineering, University of Rochester, Rochester, NY 14627

Energetic ion bombardment has a profound effect on the etch rate of silicon in dry etching processes. In this work, a combined theoretical and experimental approach is followed to investigate this phenomenon. A mathematical model of a showerhead-type reactive ion etching reactor is developed. Experimental measurements of the plasma density, electron energy, and exit gas composition in this system are used to provide information on the gas phase reactions taking place and the values of kinetic coefficients. The concentration distribution of molecules and radicals are obtained by solving the mass balances with finite differences. Surface recombination and competition between fluorine and oxygen atoms to adsorb on the silicon surface are considered, and the chemical etch rate of silicon is calculated. A correlation between the activation energy of the etching reaction and the energy density of bombarding ions is obtained by comparing the calculated chemical etch rate distribution with the one measured experimentally.

700 RNP Microtrench Formation during Plasma Etching: T. J. Dalton, J. C. Arnold, and H. H. Sawin, MIT, Dept. of Chemical Engineering, Cambridge, MA 02139; S. Suen and D. Corliss, Digital Equipment Corp., Hudson, MA 01749-2809

Microtrenches have been observed in submicron features etched with an existing plasma process. In contrast to previous reports, these microtrenches occurred a small distance away from the sidewall. We examined possible causes of microtrench formation, including surface diffusion of reactants, reflection of ions from the sidewalls, and localized charging of the feature. A simple numerical model indicated that ion reflection was the dominant cause.

701 RNP Adaptation of Clean Technology to Semiconductor Manufacturing Equipment: 8-Inch Vertical Furnaces as an Example: T. Matsuo, Corliss Differential Scanning Calorimetry - Y. N. Chang

August 1980, Tokyo Electron Ltd., 850 Nitsue-an Honaka-cho, Nirasaki City, Yamashita-pre 407-01, Japan

The construction of a total clean system requires the technological trends such as that to microminaturize LSI, expand the diameter and highly concentrate the density, and the breakthrough with mass production technology emphasizing clean technology. As the first equipment adapted with the total clean technology, we have developed Series "α - 8" at 8 Vertical Furnace.


Rapid thermal processing (RTP) is considered an essential technology for submicron processing. However, it has been restricted in use because of the inherent limitations of pyrometer temperature control. A new method of temperature control has recently been introduced that measures the thermal expansion of a wafer to determine its temperature. This thermal micrometer works by using a laser autofocus mechanism to determine the change in wafer diameter due to temperature.


Mobile ions like sodium are known to cause device (MOS) instabilities. Phosphosilicate glasses can be used as passivating layers to isolate the active devices from such contamination. In this study we have examined the effectiveness of TEOS-PECVD P glasses, as the barrier to sodium diffusion, as a function of the phosphorus concentration. The results of C-V and I-V studies on
contaminated and annealed P-glasses are presented and discussed.

704 RNP Oxygen Precipitation in Cz Si during Various Simulated 4 Mb DRAM Processing Steps


Interstitial oxygen is perhaps the most important consideration in silicon crystals for VLSI/ULSI fabrication. The relevance of oxygen to integrated circuit fabrication is primarily due to oxygen's ability to form oxide precipitates and to generate lattice defects in a controlled manner for impurity gettering during device processing. In this investigation, we studied oxygen precipitation in Cz Si during various simulated 4 Mb DRAM processing thermal cycles using infrared absorption, chemical etch/cross-section optical microscopy and synchrotron section topography. In this paper we discuss effects of initial oxygen concentration and various thermal cycles (pre- and post-intrinsic gettering heat-treatments) upon oxygen precipitation, bulk microdefect, and de-nuded zone formation.

705 RNP Electrical Conduction in Buried Oxide (BOX) in SIMOX Structures

A. Brown, Revesz Associates, Cal Faraday, Iowa State University, Ames, IA 50010

BOX layers in SIMOX structures exhibit strongly localized defect conduction and non-localized background ("bulk") conduction. In the latter case, the current increases linearly with the electrode area. For both annealed (at 1325°C) and unannealed samples the I-V characteristics are quasi-linear at low fields (<0.8 MV/cm) for unannealed samples and <2 MV/cm for the annealed ones but sub-linear at higher fields. The current densities in the quasi-linear regime are several orders of magnitude higher for the unannealed samples than for the annealed ones and their time-dependence indicates pronounced trapping phenomena. Annealed pseudo-SIMOX samples (top Si layer removed) which were heat-treated in oxygen at 1100°C behave similarly to thermally grown SiO_x films in the sense that the current is practically at the noise level (10^-14 A/cm²). This observation indicates that conduction in BOX is related to defects associated with oxygen deficiency (e.g., Si-Si bonds) that are present even in annealed samples without additional treatment. This work was funded by Naval Research Laboratory under the Contract No. N00014-88-C-2492.

706 RNP Minority Carrier Recombination Lifetime of Lightly Doped Cz Silicon Measured by μ-PCD


The minority carrier recombination lifetime which determines the diffusion length is a very sensitive parameter for crystalllographic defects for recombinadion which is currently used commonly in four recombination lifetime methods are microwave photoductive decay (μ-PCD), surface photovoltage (SPV), and electrolytic metal tracer (ELYMAT). Even though all three of these techniques are well-established in semiconducor industry, to the best of our knowledge, few experiments have been carried out so far to correlate the data by one method with those from others. In this study we measured the lifetime of lightly doped oxidized Cz Si wafers with three commercially available μ-PCD lifetime testers: ELYMAT, and SPV. In this paper, we compare our lifetime data with each other, and discuss some potential correlations among them.

707 RNP Study on Surface Photovoltage Measurement of Long Diffusion Length Silicon: Analytical Approach

O. J. Antilla, Dept. of Electrical Engineering, Stanford University, Stanford, CA 94305, S. K. Hahn, Dept. of Materials Science and Engineering, Stanford University, Stanford, CA 94305

The limitations of SPV, using the analytical solution for the minority carrier distribution, are studied in detail. The principal source of error in long diffusion length material is the back surface recombination. The possibility of measuring large diffusion lengths in a reliable manner is discussed. Measurement of minor amounts of iron contamination in p-type material is possible, but a correction factor is required for the traditionally used relationship to convert the observed diffusion length change to iron concentration. The value of the correction factor varies between 1 and 1.5 for high back surface recombination velocity; the magnitude depends on the ratio between wafer thickness and the diffusion length of the minority carriers. The sensitivity of the measured diffusion length to different sources of errors and requirements for sample preparation are discussed. Finally, the use of SPV to measure the denuded zone width in precipitated material is briefly analyzed.

708 RNP Thermal Decomposition Mechanism of MOCVD Precursor, CuCyclacetate(d) Studied by Differential Scanning Calorimetry (DSC): Y.-N. Chang, Dept. of Chemical Engineering, Iowa State University, Ames, IA 50010

The pyrolysis kinetics of the MOCVD precursor, CuCyclacetate(d) (Cu(acac)_2), was studied by differential scanning calorimetry (DSC) at atmospheric pressure. Solid Cu(acac)_2 sample was programmed heated with a constant heating rate from 25 to 400°C. For each thermogram, energy exchange rates between sample and ambient were recorded. The pure Cu(acac)_2, dry air, or mixture of He + O_2 were used as the inert or reactive gas environments. From DSC results, the thermal decomposition of Cu(acac)_2 started at 250°C and ended by 350°C. The peak temperature-rate heating rate relationship, as analyzed by the Kissinger equation, was used to estimate the activation energy for the respective kinetic step. Solid Cu(acac)_2 was pyrolyzed by FTIR, which revealed a mixed content of CuO, CuO, and carbonates.

709 RNP Hydration Models for Trivalent Transcureium Elements 3+ in Aqueous Solutions at 298 K: H. Larrous and N. Chaoulli, University of Tunis, Tunisia; J. Olivier, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Self-diffusion coefficient data for "Es(III) and "Tm(III) in neodymium perchlorate solutions are reported for concentrations up to 1.14 mol dm^-3 at 25°C. The open end capillary method (OECM) has been used for the determination of the self-diffusion coefficient in perchlorate solutions at pH 2.5 labeled with tracer "Es(III) and "Tm(III). The experimental diffusion coefficients for "Es(III) and "Tm(III) were fitted to polynomials of the following form: D/D_0 = 1 - b(2) where D_0 is the limiting diffusion coefficient value against the square root of the ionic strength (I). The data have been analyzed by using the simple hydration models to obtain realistic estimates of the effective cation hydration. In solution at pH 2.5, the data show that there is a similarity in the ion transport process of "Es(III) and "Tm(III) ions. As a further consequence, it may be argued that "Es(III) and "Tm(III) ions have the same hydration as a tripositive ion in the absence of hydratation ion-pairing or complexing. The limiting diffusion coefficients, D, [10^{-6} cm^2 s^{-1}], for actinide trivalent ion "Es(III) is 5.80 ± 0.05 and for lanthanide trivalent ion "Tm(III) is 5.85 ± 0.05.
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<td>Mao, E.</td>
<td>583 SOA, 584 SOA</td>
<td>Scherson, Daniel</td>
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<td>Margrave, J. L.</td>
<td>671 FUL</td>
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<td>577 SOA</td>
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<td>561 FUL</td>
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<td>580 SOA, 584 SOA</td>
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<td>651 FUL</td>
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<td>568 SOA, 587 SOA</td>
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<td>Tycko, R.</td>
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<td>570 SOA, 587 SOA</td>
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<td>640 FUL, 682 FUL</td>
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<td>675 FUL</td>
<td>Yannou, Costantino S.</td>
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<td>686 FUL</td>
<td>Yaron, Yaron</td>
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<td>508 SOA</td>
<td>Yezidouin, Chahan</td>
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<td>626 FUL, 655 FUL</td>
<td>Yi, Jee-Yeol</td>
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<td>641 FUL</td>
<td>Yokoyama, N.</td>
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<td>563 FUL</td>
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<td>686 FUL</td>
<td>Zerell, J. N.</td>
</tr>
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<td>674 FUL, 666 FUL</td>
<td>Zarbello, F.</td>
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<td>586, 571 SOA, 577 SOA</td>
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<td>Zheng, Q. M.</td>
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<td>578 SOA</td>
<td>Zheng, T. G.</td>
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<td>613 FUL</td>
<td>Zheng, W.</td>
</tr>
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<td>591 MIC</td>
<td>Zheng, Y.</td>
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<td>644 FUL</td>
<td>Zhao, Ming</td>
</tr>
</tbody>
</table>
APPLICATION FOR ADMISSION

TO
The Electrochemical Society, Inc.

Return completed application to:
Secretary
The Electrochemical Society, Inc.
10 South Main Street, Pennington, New Jersey 08534-2896
609-737-1902

To the Board of Directors of The Electrochemical Society, Inc:

Name:
Address:

Please print complete name and address as it should appear on mailings.

Business Telephone: (include area code)

I hereby make application for admission to The Electrochemical Society, Inc., as an member, and enclose the amount of $______ covering the first year's dues. (Please see reverse side for proper class of membership and dues applying thereto, noting the options with regard to the date of election and active life membership and the credit available for nonmember meeting registration.)

1. Date of Birth: (month) (day) (year)

2. Please check LOCAL SECTION with which you wish to affiliate:
   - Boston (05)
   - Canadien (60)
   - Chicago (10)
   - Cincinnati (12)
   - Cleveland (15)
   - Columbus (20)
   - Detroit (25)
   - European (27)
   - Japan (33)
   - Metropolitan N.Y. (35)
   - Midland (Mich.) (40)
   - Natl. Capital Area (45)
   - North Texas (55)
   - Oregon (62)
   - Pacific Northwest (65)
   - Philadelphia (70)
   - Pittsburgh (75)
   - Southern Wisconsin (92)
   - San Francisco (85 & 86)
   - South Texas (88)
   - Twin Cities (96)
   - None (99)

3. Please indicate your DIVISIONAL and GROUP interests, noting your primary interest(s) with the number 1 and secondary interest(s) with the number 2:
   - Battery (AO)
   - Corrosion (BO)
   - Dielectric Science and Technology (CO)
   - Electrodeposition (DO)
   - Electronics (EO)
   - Energy Technology (GO)
   - Industrial Electrolysis and Electrochemical Engineering (IO)
   - Luminescence and Display Materials (KO)
   - Organic & Biological Electrochemistry (FO)
   - Physical Electrochemistry (JO)
   - Sensor (LO)

4. Education
   Institution Dates Attended Major Subject Degree Earned

5. Work Experience:
   Name of Employer (current, followed by previous) Dates Position

6. The Society's Constitution provides that two Active Members of the Society (who can substantiate the above record) must recommend you for admission to membership. It will facilitate the handling of your application if you are able to have your references sign this application form. If this is not convenient, please list their names and addresses. On a student application, only a single faculty member recommendation with signature (including title and institution) is required.

The undersigned certifies that the above statements are correct and agrees, if elected to the Society, to be governed by its Constitution and Bylaws and to promote the objects of the Society as stated in its Constitution.

Date ____________ (Signature)

Rev 11/91
CONSTITUTION—Article II

Membership

Section 1. The individual membership shall consist of Active, Honorary and Emeritus Members. The Board of Directors may from time to time authorize other classifications of membership as defined in the Bylaws of the Society.

(Active Member—Annual Dues $85.00)

Section 2. An Active Member shall be interested in electrochemistry or allied subjects and possess a Bachelor’s degree, or its equivalent, in engineering or natural science. In lieu of a Bachelor’s degree, or its equivalent, any combination of years of undergraduate study and years of relevant work experience in electrochemistry or allied subjects adding to at least seven years shall be required. Election to Active Membership shall require the recommendation of two Active Members in good standing.

BYLAWS—Article II

Non-Voting Membership

(Student Member—Annual Dues $10.00)

Section 1. Student Member. A Student Member shall be a full-time undergraduate or graduate student registered for a degree in natural science or engineering or a full-time postdoctoral student in natural science or engineering in a degree-granting institution. The applicant for Student Membership shall be recommended by a member of the faculty of the school. Upon graduation with a Bachelor’s degree or equivalent in natural science or engineering, or upon departure from postdoctoral status, the Student Member may apply for Active Membership. The application shall be approved by two Active Members of the Society in good standing. The student member enters graduate school as a full-time student, or enters a qualifying postdoctoral appointment after completing a doctoral degree, the person may choose to apply for Active Membership or may remain a Student Member. A person may hold Student Membership as a postdoctoral student for no more than two years.

BYLAWS—Article XXI

Dues and Fees

Section 1. The annual dues for Active Members shall be eighty-five dollars. The annual dues for Student Members shall be ten dollars. Each member shall receive the JOURNAL OF THE ELECTROCHEMICAL SOCIETY.

Section 2. When individuals are elected to membership, they must elect to initiate their membership as of January 1 or July 1 of the year of election; or, if elected during the last quarter, January 1 of the year following election. In the case of a July 1 election for starting membership, dues will be prorated.

Section 3. Any Active Member who shall pay in one lump sum the amount equivalent to two-thirds of the remaining dues to age sixty-five at the time of payment, but not less than an amount of 5 years of full dues, shall be exempt from payment of any further dues and shall be considered an Active Member during the remainder of his or her life.

BOARD OF DIRECTORS ACTION

OF OCTOBER 9, 1960

If application for new membership is received within four months of the payment of nonmember registration at a Society Meeting by the applicant, the difference between the nonmember and member registration fees shall be credited toward the first year’s dues.

All members receive a subscription to the Journal of The Electrochemical Society.
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<th>European</th>
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</tr>
</thead>
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