



הכינוס הישראלי להנדסת חומרים
ISRAEL MATERIALS ENGINEERING CONFERENCE



האגודה הישראלית
לפולימרים ופלסטיקה

הכינוס הישראלי התשיעי להנדסת חומרים
The 9th Israel Materials Engineering Conference - IMEC-9

הכינוס השנתי ה-28 של האגודה הישראלית
לפולימרים ופלסטיקה
The 28th Annual Conference of the Israeli
Polymers & Plastics Society

בשיתוף עם האגודה הישראלית לחומרים ותהליכים
In Conjunction with the Israel Materials & Processes Society

December 6 - 7, 1999

Technion - Israel Institute of Technology, Haifa, Israel



Program and Abstracts

www.technion.ac.il/technion/materials
imec9@tx.technion.ac.il

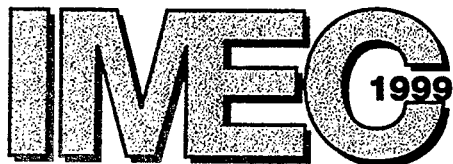
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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE 6-7 December 1999	3. REPORT TYPE AND DATES COVERED Conference Proceedings	
4. TITLE AND SUBTITLE IMEC-9 : The 9th Israel Materials Engineering Conference. Program & Abstracts			5. FUNDING NUMBERS F61775-00-WF002	
6. AUTHOR(S) Conference Committee				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Technion - Israel Institute of Technology Technion City Haifa 32000 Israel			8. PERFORMING ORGANIZATION REPORT NUMBER N/A	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) EOARD PSC 802 BOX 14 FPO 09499-0200			10. SPONSORING/MONITORING AGENCY REPORT NUMBER CSP 00-5002	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE A	
13. ABSTRACT (Maximum 200 words) The Final Proceedings for IMEC-9 : The 9th Israel Materials Engineering Conference, 6 December 1999 - 7 December 1999 Metallurgy, Ceramics, composite materials, polymers, electronic materials, characterization methods of materials, modelling, corrosion, energy-related materials, functional materials, bio-materials, high temperature materials, MEMS, Reliability, self assembly, superconductive materials, quasicrystals.				
14. SUBJECT TERMS EOARD, Materials			15. NUMBER OF PAGES 260	
			16. PRICE CODE N/A	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	



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Program and Abstracts

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Dear Colleague,

The aim of IMEC-9 is to provide an overview of the recent developments in all areas of Materials Science and Engineering, and to strengthen the interaction between Israeli research and development and Israeli Industry.

This year IMEC will be held in parallel with the Annual Conference of the Israeli Polymers and Plastics Society (IPPS) and in conjunction with the Israel Materials and Processes Society. The program includes 10 plenary and 22 invited lectures by internationally reputable scientists. Each day 8 parallel sessions, one poster session and a professional exhibition will be held.

The conference organizers hope that you enjoy the conference.

**Prof. Rachman Chaim – Chairman
IMEC-9 Organizing Committee**

Dear Colleague,

Traditionally, the Annual Conference of the Israeli Polymers and Plastics Society is the central stage for interaction between Israeli academia and industry at which the latest developments in the fields of polymers and plastics are presented.

This year, the Conference will be held in parallel with the 9'th Materials Engineering Conference and its theme will be "Materials in the Third Millennium". The program includes invited lecturers of international repute, parallel sessions, a poster session and a professional exhibition.

**Dr. Hanna Dodiuk-Kenig
President, Israeli Polymers and Plastics Society**

עמית יקר,

מטרת הכינוס הישראלי ה-9 להנדסת חומרים היא לסקור את ההתפתחויות האחרונות, בארץ ובעולם, בכל תחומי המדע והנדסת החומרים, ולחזק את הקשר בין המחקר והפיתוח במוסדות הוראה ומחקר לבין התעשייה בארץ.

השנה יערך הכינוס יחד עם הכינוס השנתי של האגודה הישראלית לפולימרים ופולסטיקה ובשיתוף עם האגודה הישראלית לחומרים ותהליכים. תכנית הכינוס כוללת 10 הרצאות מפתח ו-22 הרצאות מוזמנות של מדענים בעלי מוניטין בינלאומי. בכל יום יתקיימו 8 מושבים מקבילים, מושב פוסטרים ותערוכה מקצועית.

מארגני הכינוס מאחלים לך כינוס מועיל ופורה.

בברכה,

**פרופ' רחמן חיים
יו"ר הועדה המארגנת**

עמית יקר,

מסורת היא כי הכינוס השנתי של האגודה הישראלית לפולימרים ופולסטיקה מהווה את הבמה המרכזית למפגש בין האקדמיה, והתעשייה הישראלית, בו מוצגות ההתפתחויות האחרונות בתחומים של הפולימרים והפולסטיקה.

השנה יערך הכינוס השנתי של האגודה הישראלית לפולימרים ופולסטיקה יחד עם הכינוס הישראלי התשיעי להנדסת חומרים בסימן של "החומרים באלף השלישי". תכנית הכינוס כוללת מרצים מוזמנים בעלי מוניטין בינלאומי, מושבים מקבילים, תצוגת פוסטרים ותערוכה מקצועית.

בברכה,

**ד"ר חנה דודי-ק - קניג
נשיאת האגודה הישראלית
לפולימרים ופולסטיקה.**

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חברי הוועדה המארגנת

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ד. וגנר – מכון ויצמן למדע
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נ. טרביצקי – אוניברסיטת תל-אביב
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ר. פורת – ישקר בע"מ

The conference was supported by :

AG Associates
Ami-Oz Casting
Applied Materials
Ashot-Ashkelon
ASTeX
A.V.B.A. Engineers
Ben Gurion University of the Negev
Binyamina Winecellars
Daimler Benz
Development of Magnesium Technologies Consortium
Eisenberg Bros.
Electrochemical Industries (1952)
Electrotherm
Haifa Municipality
Iscar
Israel Aircraft Industries
Israel Electric Corporation
Israel Military Industries
I.B.M.
J.E.O.L.
KLA Tencor
MicroSwiss
Millenium Materials Technologies Fund
Ministry of Defense
Ministry of Science
Motorola Communication
Nof Hametaiel
Nova Measuring Instruments
Nuclear Research Center - Negev
Oramir
Ortal
Rafael
Siemens
Steag CVD Systems
Technion - Israel Institute of Technology
Teva Industries
Tel Aviv University
United States Air Force
United States Navy
Watairpoll

רשימת מציגים
(לפי סדר א'-ב')

אולטרה- מט בע"מ

ת.ד. 3011, אבן יהודה 40500, טלפון: 09-8826714, פקס: 09-8826715

איטק בע"מ ITEC

ת.ד. 10002, תל אביב 61100, טלפון: 03-6491202, פקס: 03-6497661

דע-מור ציוד רפואי ומדעי בע"מ

בארי 19 ת.ד. 5803, הרצליה 46456, טלפון: 03-9543616, פקס: 09-9588474

דר' אורי גוליק - מחשוב כימי

קיבוץ גלויות 34, תל אביב 66550, טלפון: 03-5187555, פקס: 03-5187575

ווטאירפול הנדסה בע"מ Watairpoll

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AVBA

ת.ד. 690, מגדל העמק 10500, טלפון: 06-6442575, פקס: 06-6442577

GENERAL INFORMATION

Registration & Information

The Registration Desk will operate on both days of the conference from 7:30 until 13:30 in the Churchill Building, and from 13:30 to 17:30 in the Amado Building.

Name Badge:

Your name badge is included in your personal envelope. Please wear it at all Conference sessions and events.

Poster Sessions:

The poster sessions will take place in the Amado Building foyer on Monday & Tuesday, December 6-7, 1999, between 11:30 and 13:30. Authors are requested to hang their posters by 10:30 on the board marked with their poster number. Please check your number in the program. Please remove posters at the end of the day.

Proceedings:

Authors are kindly requested to submit their papers (3 copies) at the Registration Desk.

Coffee and Lunch:

The morning coffee break will be in the Churchill Building. The afternoon coffee break will be in the Amado Building. Lunch will be served in the tent in front of the Churchill Building, upon presentation of the voucher included in your personal envelope.

Conference Dinner:

The Conference Dinner will take place at the Haifa Convention Center, on Monday, November 6, 1999, at 19:00. Additional tickets may be purchased from the Registration Desk until 13:00.

Exhibition:

An exhibition of scientific and industrial equipment will take place in the Amado Building. A list of firms exhibiting is included herewith.

מידע כללי

הרשמה ומודיעין:

דלפק הרישום יופעל ביומיים של הכינוס מ- 7:30 בבוקר עד 13:30 בבנין צ'רצ'יל, ומשעה 13:30 עד שעה 17:30 בבנין אמדו.

תגים שמייים:

תגן השמי נמצא במעטפת הרישום. בבקשה ענוד אותו לכל מושבי הכינוס ולארועים.

מושבי הפוסטרים:

מושבי הפוסטרים יתקיימו בבנין אמדו ברחבת הכניסה בימים שני ושלישי, 6-7 בדצמבר, 1999, בין השעות 11:30 ל- 13:30. המגישים מתבקשים לתלות את הפוסטרים עד השעה 10:30 על לוח עם מספר הפוסטר. בבקשה בדקו את מספר הפוסטר בתוכנית. יש להסיר את הפוסטר בסוף היום.

מאמרים:

מגישי מאמרים מתבקשים להגישם (3 העתקים) בדלפק הרישום.

קפה וארוחת צהרים:

בבוקר קפה יוגש בבנין צ'רצ'יל, ואחה"צ בבנין אמדו. ארוחת הצהרים תוגש באוהל בקדמת בנין צ'רצ'יל, כנגד הצגת שובר הנמצא במעטפת הרישום.

ארוחת ערב חגיגית:

תתקיים במרכז הקונגרסים, חיפה, ביום שני, 6 בנובמבר, 1999 בשעה 19:00 בערב. כרטיסים נוספים ניתן לרכוש בדלפק הרישום עד השעה 13:30.

תערוכה:

תערוכה מקצועית של ציוד מדעי ותעשייתי תתקיים בבנין אמדו. רשימת החברות המציגות מצורפת.

Lloyd-Instruments

Wallace



איטק ITEC

ציוד מדידה ובדיקה למעבדות הפלסטיקה, הגומי והמתכת

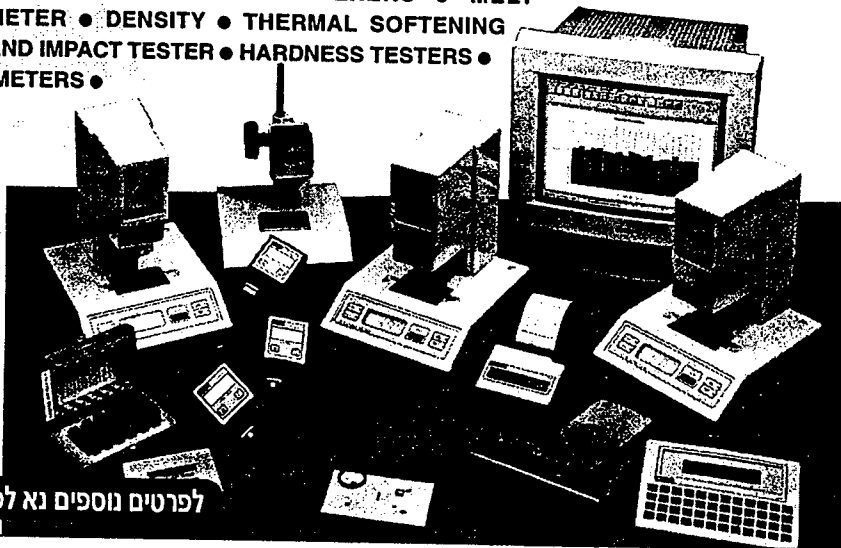


הרינו שמחים להודיע, כי החל מאוגוסט 1998, חברת איטק בע"מ הינה הנציגה הבלעדית בישראל של חברת WALLACE מאנגליה, אשר מתמחה בייצור של מכשור מדידה ובדיקה למעבדות הפלסטיקה והגומי.

- מגוון רחב ביותר של מדי קושי עם אופציה למיחשוב;
- כלים ותבניות לחיתוך דוגמאות;
- תנורים לבדיקות סביבתיות;
- מאזניים לבדיקת צפיפות ועוד.

כמו כן אנו ממשיכים לייצג נאמנה את חברת LLOYD-INSTRUMENTS אנגליה המתמחה בייצור מגוון רחב של מכונות לבדיקות תכונות חומרים לתעשיית המתכת, פלסטיקה, גומי, נייר, אלקטרוניקה מזון ועוד.

- TENSILE ● COMPRESSION ● PEELING ● FRICTION
- BENDING ● MELT FLOW INDEXERS ● MELT VISCOMETER ● DENSITY ● THERMAL SOFTENING POINT AND IMPACT TESTER ● HARDNESS TESTERS ● PLASTIMETERS ●



לפרטים נוספים נא לפנות ל:

מאורי גוטליב / דוריס שוורץ טל. 03-6491202 פקס. 03-6497661

PROGRAM

Monday, December 6

07:30 - 08:30 Registration, Churchill Auditorium

08:30 - 09:00 Welcome and Opening Address

09:00 - 10:00 Plenary Session Churchill Auditorium
Chair: M.S. Silverstein

09:00 - 09:30 **The Future of Equipment Development and Semiconductor Production**

D. Maydan, USA

09:30 - 10:00 **Mechanical Properties of Thin Film Polysilicon and their Relevance to MEMS Devices**

A.H. Heuer, USA

10:00 - 10:30 **Coffee Break** Churchill Auditorium

10:30 - 11:30 Plenary Session Churchill Auditorium
Chair: M. Bamberger,

10:30 - 11:00 **Applications of Quasi-Periodic Materials**
D. Shechtman, Israel

11:00 - 11:30 **Magnesium- an Old Metal with a Promising Future**
B.L. Mordike, Germany

11:30 - 13:30 Poster Session 1, Amado Building & Lunch, Tent

13:30 - 15:00 PARALLEL SESSIONS: Amado & Segó Buildings

Coatings and Surface Treatments I Hall A
Chair: L. Gal Or

13:30 - 14:00 **Interphase Control for Corrosion Protection in Coatings - Invited**
A.R. Marder, USA

-
- 14:00 - 14:20 **Surface Treatment of Tantalum to Improve Corrosion Resistance**
A. Rubinshtein, R. Shneck, A. Danon, J. Hayon, S. Nathan, A. Raveh, Israel
- 14:20 - 14:40 **Hydroxyapatite Coating of Dental Implants by HVOF Spraying**
O. Botstein, A. Shwartsman, O. Fromovich, Israel
- 14:40 - 15:00 **Electrodeposition of Metallic Multilayers by a Pulse Method**
T. Cohen, J. Yahalom, Israel

Advanced Silicon Device Processing **Hall B**
Chair: Y. Shacham

- 13:30 - 14:00 **Advanced Copper Wiring for Microelectronic Devices - Invited**
A.K. Sinha, USA
- 14:00 - 14:20 **Electroless Deposition of Cobalt Alloys for Diffusion Barriers in ULSI Cu Metallization**
A. Kohn, M. Eizenberg, Y. Shacham-Diamand, E. Sverdlov, B. Israel, Israel
- 14:20 - 14:40 **The Role of Surface and Gas Phase Kinetics in the Nucleation and Growth of Copper Using Chemical Vapor Deposition**
R. Kroger, M. Eizenberg, Israel, D. Cong, C. Marcandal, L. Chen, USA
- 14:40 - 15:00 **Effect of LDD Reactive Ion Etching on the Electrical Performance of Sub-Micron CMOS Devices**
T. Kohav, E. Shauly, Israel

Mechanical Behavior I **Hall C**
Chair: D. Rittel

- 13:30 - 14:00 **Superplasticity and Joining of Ceramics - Invited**
A. Dominguez-Rodriguez, F. Gutierrez-Mora, Spain, J.L. Routbort, USA, R. Chaim, Israel
- 14:00 - 14:20 **Some Structural Effects of Plastic Deformation on Tungsten Heavy Metal Alloys**
R. Gero, L. Borukhin, I. Pikus, Israel
- 14:20 - 14:40 **Evaluating the Fracture Energy of Thin Ceramic Layers by Cleavage**
D. Sherman, Israel
- 14:40 - 15:00 **Effect of Proof Testing on Optical Fiber Strength**
D. Benderly, J. Sharon, S. Djerassi, Y. Margalit, D. Gorni, Israel

Nano-Structured Materials**Hall D***Chair: D. Gorni*

- 13:30 - 14:00 **Inorganic Nanotubes from Layered Compounds – Invited**
R. Tenne, Israel
- 14:00 - 14:20 **Fullerene Thin Films for Photovoltaics and Optoelectronics**
E.A. Katz, D. Faiman, S. Shtutina, A.I. Shames, Y. Cohen, S. Goren, Israel
- 14:20 - 14:40 **Sintering Study of Nanocrystalline Tungsten Carbide**
G.R. Goren-Muginstein, A. Rosen, S. Berger, Israel
- 14:40 - 15:00 **TiO₂ Colloids for Mesoporous Electrodes: The Effect of the Preparation Conditions on their Physical Properties**
A. Zaban, S.T. Aruna, S. Tirosh, S. Chappel, Israel
-

Magnesium Science and Technology I**Hall E***Chair: E. Aghion*

- 13:30 - 13:50 **Preparation and Solidification Features of AS21 Magnesium Alloy**
B. Bronfin, M. Katsir, E. Aghion, Israel
- 13:50 - 14:10 **The Influence of the Microstructure on the Creep Properties of AZ91 Magnesium Alloy**
M. Regev, O. Botstein, A. Rosen, Israel
- 14:10 - 14:30 **Galvanic Corrosion of Magnesium Alloys**
M. Starostin, A. Smorodin, L. Gal-Or, Sh. Tamir, Israel
- 14:30 - 14:50 **Microstructural Analysis of Gas Tungsten Arc Welded Magnesium AZ91 Alloy**
A. Stern, A. Munitz, U. Admon, M. Aizenshtein, Israel
-

Smart & Functionally Graded Materials I**Hall F***Chair: N. Frage*

- 13:30 - 14:00 **Functionally Graded Materials – Invited**
M.P. Dariel, Israel
- 14:00 - 14:20 **Diffusion Induced Compositional Gradient in TiAlN Thin Films**
M. Pinkas, N. Froumin, N. Frage, J. Pelleg, M.P. Dariel, Israel
- 14:20 - 14:40 **Thermal Testing of Functionally Graded Materials Using Laser Shock Method**
T. Elperin, G. Rudin, Israel
- 14:40 - 15:00 **Functionally Graded Materials Based on Nonstoichiometric TiC Infiltrated with a Fe-C Alloy**
S. Sabatello, N. Frage, M. P. Dariel, Israel

Composites I		Hall G
Chair: R. Albalak		
13:30 - 13:50	Synthesis of Dense Oxide-Based In Situ Composites via Thermal Explosion/SHS Under Uniaxial Applied Pressure <i>D. Horvitz, I. Gotman, E.Y. Gutmanas, Israel</i>	
13:50 - 14:10	Nano Particle Reinforced Polymer Composites <i>A. Ophir, F. Weiner, O. Shepelev, S. Kenig, Israel</i>	
14:10 - 14:30	Carbon Nanotubes-Based Polymer Composites <i>O. Lourie, J. Wood, E. Meurs, H.D. Wagner, Israel</i>	
14:30 - 14:50	Application of Fluoropolymers Based Composites in Electroluminescent Fibers <i>B. Gorelik, M. Voskobochnik, O. Berezin, D. Boumberg, M. Zaidman, Y. Dvir, Israel</i>	

Diffusion and Phase Transformations I		Hall H
Chair: R. Ghez		
13:30 - 14:00	Grain Boundary and Triple Junction Migration – Invited <i>L.S. Shvindlerman, Russia, G. Gottstein, Germany</i>	
14:00 - 14:20	Grain Boundary Grooving Phenomena in the Ni-Rich NiAl <i>E. Rabkin, L. Klinger, A. Berner, T. Izyumova, Israel</i>	
14:20 - 14:40	Investigation of Annealed Structures in Ternary U-Nb-W Alloys <i>E. Kahana, M. Talianker, A. Landau, Israel</i>	
14:40 - 15:00	The Observation of Non-Equilibrium Transformations and Metastable Structures in Ternary Titanium Aluminide <i>A. Tokar, A. Katzman, A. Berner, L.A. Levin, Israel</i>	

15:00 - 15:30 **Coffee Break** **Amado Building**

15:30 - 17:30 PARALLEL SESSIONS: **Amado & Segó Buildings**

Corrosion and Its Inhibition		Hall A
Chair: D. Eliezer		
15:30 - 16:00	Studies of Copper and Silver Deposition, Employing the EQCM – Invited <i>E. Gileadi, V. Tsionsky, Israel</i>	
16:00 - 16:20	The Effect of Composition and Microstructure on the Corrosion Behavior of Magnesium-Aluminium Alloys <i>P. Uzan, D. Eliezer, E. Aghion, Israel</i>	
16:20 - 16:40	Inhibition Ability of Some Organic Acids in Engine Coolants <i>M. Starostin, A. Smorodin, L. Gal-Or, Sh. Tamir, Israel</i>	
16:40 - 17:00	Corrosion of Sinterized Steel in Salty Water <i>A. Duta, R. Tica, D. Nanu. I. Dinescu, D. Ursutiu, Romania</i>	

17:00 - 17:20 **Analysis of Corrosion Failures in Refinery Equipment**
A. Groysman, Israel

Silicon Technology: Dielectrics, Packaging **Hall B**
Chair: R. Levin

15:30 - 16:00 **Multi-Layer Under Bump Metallurgy Flip Chip Bonding Technology – Invited**
A. Katz, P. Sferlazzo, USA

16:00 - 16:20 **The Interface of Fluorinated Amorphous Carbon with Copper Metallization**
N. Ariel, M. Eizenberg, Israel, Y. Wang, USA

16:20 - 16:40 **Effects of Silicon Concentration on Mechanical Characteristics of Silicon Containing Diamond-Like Carbon Films Deposited by PACVD**
I. Moskowitz, S.V. Babu, USA

16:40 - 17:00 **Silicon Nitride Deposition by RTCVD - An Optional Gate Dielectric Replacement in Device Fabrication**
H. Spielberg, E. Iskevitch, S. Levy, H. Gilboa, Israel

17:00 - 17:20 **Two-Dimensional Diffusion Characterization of Boron in Silicon Using Reverse Modeling**
E. Shauly, R. Ghez, Y. Komem, Israel

Bio-Materials **Hall C**
Chair: S. Cohen

15:30 - 16:00 **Biological Composites or All You Wanted to Know About Shells – Invited**
A.H. Heuer, USA

16:00 - 16:30 **Using Polymer Science to Influence Cell Biology – Invited**
M.S. Shoichet, X. Cao, P. Dalton, S. Saneinejad, Y. Tong, Canada

16:30 - 17:00 **Self Assembly of Nanometer Scale Electronics by Biotechnology – Invited**
U. Sivan, E. Braun, Y. Eichen, G. Ben Yoseph, Israel

17:00 - 17:20 **Titanium Alloy Grade 7 Under the Influence of Micro-Organisms in Diluted NaBr Solution**
D. Itzhak, T. Greenberg, Israel

Functional Ceramics **Hall D**
Chair: N. Travitzky

15:30 - 16:00 **Science and Serendipity in Functional Ceramics - Materials Integration and Materials Innovation – Invited**
N. Setter, Switzerland

16:00 - 16:30 **KLTN – A New Crystal for Photorefractive and Electrooptic Applications at the Paraelectric Phase – Invited**
A. J. Agranat, Israel

- 16:30 - 16:50 **Gas Sensing in Thin Films of TiO₂ at Low Temperatures (100-325°C)**
A. Rothshild, F. Edelman, Y. Komem, M.S. Silverstein, Israel, F. Cosandey, USA
- 16:50 - 17:10 **Growth and Characterization of Barium Titanate Based Materials for High Strain Applications**
Y. Avrahami, H.L. Tuller, USA
- 17:10 - 17:30 **Development of Piezoelectric Thin Film Deposition Techniques for MEMS Applications**
Y. Yeshurun, H. Ashuach, Israel
- 17:30 - 17:50 **Base Metal Electrodes for Multilayer Capacitors**
Y. Tsur, C.A. Randall, USA

Magnesium Science and Technology II **Hall E**
Chair: G. B. Barkay

- 15:30 - 15:50 **Grain Refinement of Magnesium Alloys AZ31, ZK60 Towards Superplasticity Studies**
A. Ben-Artzy, A. Shtechman, A. Bussiba, S. Ifergan, Israel
- 15:50 - 16:10 **Mechanoelectrochemical Behavior and Creep Corrosion of Magnesium Alloys**
E.M. Gutman, A. Eliezer, E. Abramov, Ya. Unigovski, Israel
- 16:10 - 16:30 **Development of High Strength Magnesium Alloys**
I. Popov, D. Shechtman, Israel
- 16:30 - 16:50 **Mechanical Properties and Microstructure of Gas Tungsten Arc Welded 4mm Thick Magnesium AZ91D Plates**
A. Munitz, C. Cotler, A. Stern, G. Kohn, Israel

Energy-Related Materials **Hall F**
Chair: S. Berger

- 15:30 - 15:50 **Molten Salt Electrolytes for High Power Thermal Batteries**
D.R. Machado, Israel
- 15:50 - 16:10 **Electrochemical Deposition of Organic Semiconductors for Solar Cells**
A. Zaban, Y. Diamant, Israel
- 16:10 - 16:30 **Dielectric Properties of Tungsten-Silica Nanocomposites**
T. Tepper, S. Berger, Israel
- 16:30 - 16:50 **Thermodynamic Characterization and Statistical Thermodynamics of the TiCrMn-H₂(D₂) System**
O. Beer, D. Cohen, Z. Gavra, Israel, J.R. Johnson, USA, M.H. Mintz, Israel

The Israeli Materials Industry		Hall G
<i>Chair: O. Gafri</i>		
15:30 - 16:00	Participating Successfully in the Fifth Framework Programme - <i>Invited</i> <i>N. Soffer, Israel</i>	
16:00 - 16:20	New Family of Laminar-Band Ceramic Metal Composites <i>A. Gorodnev, Israel</i>	
16:20 - 16:40	Non Destructive Strength Evaluation of Steel Wires and Cables <i>G. Kohn, M. Melamud, E. Kochavi, Y. Danon, H. Etedgui, Israel</i>	
16:40 - 17:00	Recent Developments with Sapphire - A Missiles Best Friend <i>A. Horowitz, G. Hayoun, S. Biderman, Y. Einav, D. Gazit, G. Ben-Amar, A. Venkert, D. Sherman, Israel</i>	
17:00 - 17:20	Millenium Materials Technologies Fund L.P. - (MMT) <i>N. Belzer, Israel</i>	

Laser Processing of Materials		Hall H
<i>Chair: E. Rabkin</i>		
15:30 - 15:50	Improving Tribological Performance of Mechanical Components by Laser Surface Texturing <i>I. Etsion, G. Halperin, G. Ryk, Israel</i>	
15:50 - 16:10	Graded Coatings on High Speed Steel Produced by Laser Alloying with Tungsten Carbide <i>M. Riabkina-Fishman, E. Rabkin, N. Frage, M.P. Dariel, Israel, A. Weisheit, R. Galun, B.L. Mordike, Germany</i>	
16:10 - 16:30	Laser Nitriding of Iron and Stainless Steel <i>P. Schaaf, F. Landry, K.-P. Lieb, Germany, M. Bamberger, Israel</i>	
16:30 - 16:50	Optimum Laser Surface Treatment of Fatigue Damaged Ti-6Al-4V Alloy <i>E. Altus, E. Konstantino, Israel</i>	
16:50 - 17:10	Novel Laser - Based Processing of Sensor and Electronic Devices and Subsystems <i>D.B. Chrisey, A. Pique, J. Fitz-Gerald, R.C.Y. Auyeung, R.A. McGill, H.D. Wu, USA, J. Greenblatt, Israel, M. Duignan, USA</i>	

19:00 **Conference Dinner**

List of Posters - Poster Session 1

- P1-1 **A Study of Properties of Oxides Ceramic Coatings by Microarc Oxidation**
G. Xiaohui, W. Xujun, G. Yu, China
- P1-2 **Experimental Results Concerning Hardening by Nitrogen Diffusion in Titanium Alloys**
C. Badina, L. Druga, Romania
- P1-3 **Chemistry of a Novel Day/Night Solar Cell GaAs/Si with In-Situ Metal Hydride Storage**
B. Wang, S. Licht, Israel
- P1-4 **Effect of Oxygen on Wetting of Alumina by Liquid Aluminium**
G. Levi, W.D. Kaplan, Israel
- P1-5 **Capillarity Phenomena in the TiC/(Cu, Al) System**
N. Froumin, N. Frage, M. Polak, M.P. Dariel, Israel
- P1-6 **Processing of Nano-Ni Reinforced Al₂O₃**
M. Lieberthal, W.D. Kaplan, Israel
- P1-7 **Alloyed Silumins, Casted and Heat Treated in Magnetic Field**
I. Giacomelli, R. Florea, Romania
- P1-8 **Direct Experimental Supervision of Outstripping Diffusion on Triple Junctions in Al**
B. Bokstein, V. Ivanov, O. Oreshina, A. Peteline, S. Peteline, Russia
- P1-9 **Ferrography As a Tool in Tribological Study**
Z. Horowitz, I. Ronn, Israel
- P1-10 **Morphological Structures of an Extruded AZ-81 Magnesium Alloy**
G.A. Frank, G. Kimmel, D. Dayan, E. David, D. Dadon, Israel
- P1-11 **Properties of Reversible A+B to C Reaction Diffusion Process**
M. Sinder, J. Pelleg, Israel
- P1-12 **Effect of Electric Field on Protection of Si Wafers from Metallic Contamination During Wafer Processing Technology**
M. Beregovsky, B. Brudnik, V. Gorodokin, A. Klyuch, I. Naot, I. Rapoport, Y. Raskin, A. Sergienko, S. Zaidman, Y. Zinman, Israel

- P1-13 **RF Heating of a Conductor Film on Silicon Substrate for Thin Film Formation**
M. Sinder, J. Pelleg, L. Meerovich, V. Sokolovsky, Israel
- P1-14 **The Effect of 1% Prior Creep Deformation on the Resultant Resistance to Fatigue Crack Growth (FCG) of Precipitation-Strengthened Superalloy Type Udimet-700**
O. Golan, A. Arbel, D. Eliezer, Israel
- P1-15 **Plastic Deformation in Impure Nanocrystalline Ceramics**
R. Chaim, Israel
- P1-16 **Alumina Ceramics for Ballistic Protection**
E. Medvedovski, D. D. Marchant, Canada
- P1-17 **The Microstructure of Laser Treated α -Al₂O₃**
L. Shepeleva, B. Medres, W.D. Kaplan, M. Bamberger, Israel, M.H. McCay, T.D. McCay, C.M. Sharp, USA
- P1-18 **New Type of Glass-Ceramic Zircon Beads**
R. Khodakovskaya, I. Maiman, S. Weissberger, A. Schon, M. Yancu, L. Simkin, Israel
- P1-19 **Some Interesting Structural Aspects of Nanocrystalline Zirconia**
R. Ramamoorthy, Israel
- P1-20 **Experimental Study of the Packing Behaviour of Binary Mixture of Powders Intended for Powder Injection Molding**
L. Dihoru, R. Orban, Romania
- P1-21 **Determination of Pore Fractal Dimensions and Porosity of Porous Materials from the Dielectric Response at Percolation**
E. Axelrod, A. Gutina, Y. Feldman, Israel
- P1-22 **Solidification of Ternary Quasiperiodic Phases in Mg-Zn-Y System**
I. Popov, D. Shechtman, Israel
- P1-23 **Correlation of Viscoelastic Properties of Die-Cast Magnesium Alloy with Processing Conditions**
E.M. Gutman, Ya.B. Unigovski, Israel
- P1-24 **Mathematical Modeling of Laser Alloying for Graded Surface Layers Fabrication**
P. Levin, N. Frage, M.P. Dariel, M. Riabkina-Fishman, E. Rabkin, Israel

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- P1-25 **Modeling of Reactive Synthesis in B₄C-Ti Powder Blends Applied to Thermal Explosion in the Conditions of Enhanced Heat Transfer**
D. Horvitz, L. Klinger, I. Gotman, Israel
- P1-26 **The Theoretical Base of Software for Continuous Casters Technological Line Design and Casting Characteristics Verification**
P. Levin, Israel
- P1-27 **Modeling in General Dimensional Analysis**
C. Samoila, D. Ursutiu, Romania
- P1-28 **Corrosion of Mg-Zn-Y-Zr Alloys in Chloride Containing Water**
I. Popov, D. Starosvetsky, D. Shechtman, Israel
- P1-29 **Research Regarding The Achievement of the Total HIP Prosthesis**
R. Teodorescu, L. Druga, Romania
- P1-30 **Small-Sized Filter Device for Comprehensive Drinking Water Treatment**
A. Veprinsky, A. Gor, Israel
- P1-31 **Production and Characterisation of Hydroxyapatite Derived from Australian Corals for Artificial Eyes**
J. Hu, J. Russell, Australia, R. Vago, Israel, A. Rubel, B. Ben-Nissan, Australia
- P1-32 **Computer Aided Monitoring of the Heat and Thermomechanical Treatments**
D. Codescu, L. Adomnica, Romania
- P1-33 **Development of Nanocrystalline Films of PBS with High IR Sensitivity**
U. Abarbanel, R. Z. Shneck, Z. Dashevsky, S. Rotman, Israel

Tuesday, December 7

07:30 - 09:00 Registration, Churchill Auditorium

09:00 - 10:00 Plenary Session Churchill Auditorium
Chair: R. Chaim

09:00 - 09:30 **The Meaning of Strategic Research as Exemplified by Our Work on the Aromatic Copolyesters**
J. Economy, USA

09:30 - 10:00 **Tailoring of Interface in Polymer Composite Materials**
A.T. DiBenedetto, USA

10:00 - 10:30 **Coffee Break** Churchill Auditorium

10:30 - 11:30 Parallel Plenaries – IMEC-9 Churchill Auditorium
Chair: M. Talianker

10:30 - 11:00 **Materials for the Electronic Industry**
R.D. Isaac, USA

11:00 - 11:30 **Instruction in Materials Science and Engineering: From Blackboard and Chalk to Modern Technology**
W. Brostow, USA

10:30 - 11:30 Parallel Plenaries – IPPS Hall E
Chair: H. Dodiuk-Kenig

10:30 - 11:00 **Welcome & Opening Address**
Honorary Member Award to Prof. M. Narkis, Technion

11:00 - 11:30 **Structured Electrically Conductive Polyaniline/Polymer Blends – Award Address**
M. Narkis, Israel

11:30 - 12:00 **Exploring Technical Properties on the Molecular Level - Modules for the Design of Smart Materials in the Next Millenium**
K. Reihls, Germany

12:00 - 12:10 **Annual Financial Report**

11:30 - 13:30 Poster Session 2, Amado Building & Lunch, Tent

13:30 - 15:00 PARALLEL SESSIONS: Amado & Segó Buildings

Coating and Surface Treatments II **Hall A**
Chair: E. Gutmanas

- 13:30 - 13:50 **Microstructure and Mechanical Properties of Hard TiN-Based Coatings on Surgical Titanium Alloys**
A. Shenhar, I. Gotman, E.Y. Gutmanas, Israel, S. Radin, P. Ducheyne, USA
- 13:50 - 14:10 **Protective Coating Behavior on C-C Material**
M. Boas, D. Gorni, Israel
- 14:10 - 14:30 **Electrophoretic Deposition: The Technology & Its Applications**
L. Gal-Or, D. Brandon, Israel
- 14:30 - 14:50 **Electrodeposited Nano-Composite Coatings**
P. Ari-Gur, J. Easwaran, USA
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Semiconductors: Growth, Characterization **Hall B**
Chair: Y. Komem

- 13:30 - 14:00 **GeSi Self-Assembled Dots - Invited**
L. Vescan, Germany
- 14:00 - 14:20 **Microstructure and Electrical Properties of CoSi₂ Contacts on Si_{1-y}C_y**
Y. Roichman, A. Berner, R. Brener, C. Cytermann, D. Shilo, E. Zolotoyabko, M. Eizenberg, Israel, H.J. Osten, Germany
- 14:20 - 14:40 **Surface Photovoltage Spectroscopy of Two Dimensional Structures and Devices**
N. Ashkenasy, S. Leibovitch, I. Halkon, M. Leibovitch, Y. Rosenwaks, Y. Shapira, Israel
- 14:40 - 15:00 **Nanoscale Characterization of Semiconductor Electronic Properties Using Kelvin Probe Force Microscopy**
R. Shikler, T. Meoded, N. Fried, Y. Rosenwaks, Israel
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Mechanical Behavior II **Hall C**
Chair: E. Altus

- 13:30 - 13:50 **On the Inelastic Response of Al₂O₃ Under Shock Loading**
H. Marom, D. Sherman, Z. Rosenberg, Israel
- 13:50 - 14:10 **Energy Absorber Materials for Impact and Crash Protection**
D. Moreno, S. Haroush, Israel
- 14:10 - 14:30 **Development of Adiabatic Shear Bands in Uranium Alloys**
B. Herrmann, A. Landau, D. Levi-Hevroni, N. Argaman, Z. Livne, Israel
- 14:30 - 14:50 **Nonlinear Dynamical (CHAOS) Analysis of the Crack Surface in Sapphire**
D. Sherman, I. Be'ery, Israel

Polymer and Plastic Surfaces**Hall D***Chair: J. Klein*

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- 13:30 - 13:50 **Direct Measurements of Forces Between Surfaces Bearing Polymer Brushes**
R. Tadmor, J. Janik, J. Klein, Israel, L.J. Fetters, USA
- 13:50 - 14:10 **Shear and Normal Forces between Smooth Solid Surfaces, in Solution of Adsorbing PEO in Toluene**
U. Raviv, R. Tadmor, J. Klein, Israel
- 14:10 - 14:30 **Brominated Flame-Retardant Blooming Prevention by Molecular Modeling**
D. Alperstein, N. Kornberg, Israel
- 14:30 - 14:50 **Discoloration of PP-Based Compounds Containing Magnesium Hydroxide**
S. Bron, Y. Keidar, G. Titelman, Y. Gonen, B. Masri, Israel

Polymer Blends I**Hall E***Chair: A. Siegmann*

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- 13:30 - 14:00 **Composites Based on Semiconducting and Light Emitting Polymers - Invited**
N. Tessler, Israel
- 14:00 - 14:20 **Microstructural Studies of Polystyrene/Polybutadiene/Organo-Bromine-FR Tertiary Systems**
M. Goldraich, M. Gottlieb, I. Finberg, Israel
- 14:20 - 14:40 **Segregated Structures in Carbon Black Containing Immiscible Polymer Blends**
O. Breuer, R. Tchoudakov, M. Narkis, A. Siegmann, Israel
- 14:40 - 15:00 **Structure and Properties of Ternary Immiscible Polymer Blends**
J. Zoldan, A. Siegmann, M. Narkis, Israel

Functional Materials**Hall F***Chair: M. Leibowitz*

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- 13:30 - 14:00 **Functional Polymeric Nanoparticles of Narrow Size Distribution: Design, Synthesis & Biomedical Applications - Invited**
S. Margel, Israel
- 14:00 - 14:20 **The Dielectric Relaxation in Complex Materials**
Y. Feldman, Israel
- 14:20 - 14:40 **Domain Engineering for Nonlinear Optical Devices**
G. Rosenman, P. Urenski, N. Angert, A. Skliar, M. Roth, M. Zeitlin, A. Zjarov, Israel
- 14:40 - 15:00 **Multifunctional Nonoxide Ceramic Laminates**
Z. Lences, P. Sajgalik, Slovakia, E. Roncari, Italy, M. Toriyama, Japan

High Temperature Materials**Hall G***Chair: N. Nir*

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- 13:30 - 14:00 **Materials Requirements for Life Steam Temperatures up to 700°C – Invited**
E. Tolksdorf, Germany
- 14:00 - 14:20 **The Mechanical Behavior of Ceramic/Metal Laminates Under Thermal Shock Loading**
D. Schlumm, D. Sherman, Israel
- 14:20 - 14:40 **Carbon/Carbon Composites with Randomly Oriented Reinforcement**
H. Weisshaus, M. Breslauer, A. Klauzner, M. Gerchcovich, I. Engelberg, Israel
- 14:40 - 15:00 **The Effect of the Stress Intensity Factor on the Morphology of the Fracture Surface During Fatigue-Crack-Growth (FCG) Studies of a Polycrystalline Superalloy**
O. Golan, A. Arbel, D. Eliezer, Israel

Diffusion and Phase Transformations II**Hall H***Chair: R. Shneck*

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- 13:30 - 13:50 **Solid State Reactions in BN-Ti System**
E. Faran, E.Y. Gutmanas, I. Gotman, Israel
- 13:50 - 14:10 **The SHS Reaction in Al-Ni Multilayers**
U. Admon, M. Amar, B. Yahav, G. Kimmel, I. Dahan, J. Sarel, D. Dayan, M. Santo, N. Frage, M.P. Dariel, Israel
- 14:10 - 14:30 **Grain Boundary Porosity in NiAl Induced by Cu Diffusion**
L. Klinger, E. Rabkin, Israel, V. Semenov, Russia, T. Izyumova, Israel

15:00 - 15:30 Coffee Break**Amado Building**

15:30 - 17:30 PARALLEL SESSIONS: Amado & Segó Buildings

Powder Metallurgy and Processing**Hall A***Chair: A. Layyous*

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- 15:30 - 15:50 **Pressureless Sintering of B₄C with TiO₂ Additions**
L. Levin, N. Frage, M.P. Dariel, Israel
- 15:50 - 16:10 **Densification Processes of Sintered Molybdenum**
Y. Ben-yehuda, M. Boas, D. Gorni, O. Aharon, Israel
- 16:10 - 16:30 **TEM Examination of the Interparticle Contact Area in Silver Compacts after Acid-Assisted Consolidation**
M. Pinkas, O. Yehezkel, M.P. Dariel, Israel
- 16:30 - 16:50 **Forming Technologies for P/M Aluminum Alloys**
D. Ringhand, Germany

16:50 - 17:10 **Energy Dispersive X-Ray Fluorescence Using Micro-Beam Excitation**
M. Haschke, Germany, A. Seaman, B. Scruggs, USA

Compound Semiconductors

Hall B

Chair: Y. Golan

15:30 - 16:00 **Grain Boundaries in GaN and their Impact on Electrical Conductivity – Invited**

J. Salzman, B. Meyler, C. Uzan-Saguy, R. Kalish, Israel

16:00 - 16:20 **MOCVD Growth of Gallium Nitride on Si Substrates**

S. Zamir, B. Meyler, E. Zolotoyabko, J. Salzman, Israel

16:20 - 16:40 **Czochralski Growth and Characterization of Stoichiometric and Slightly Off-Stoichiometric PbTe Single Crystals**

A. Jarashnelij, Z. Dashevsky, S. Shusterman, A. Horowitz, M.P. Dariel, Israel

16:40 - 17:00 **A New Approach to Thermodynamically Stable Contacts for Binary Wide Bandgap Semiconductors**

I. Shalish, Y. Shapira, M. Eizenberg, Israel

17:00 - 17:20 **GaN Layer Growth Optimization for High Power Devices**

A. Hass Bar-Ilan, S. Zamir, O. Katz, B. Meyler, J. Salzman, Israel

Simulation and Modeling

Hall C

Chair: S. Brandon

15:30 - 15:50 **Combined Modeling of Aggregation of Silica in Process of Silica Sedimentation, Using Monte-Carlo Approach with Thermodynamic Limitations**

F.A. Romm, V.M. Karchevsky, O.L. Figovsky, Israel

15:50 - 16:10 **Molecular Dynamics Study of Structure and Diffusion in a Molten Aluminium Surface**

A. Hashibon, J. Adler, W.D. Kaplan, Israel

16:10 - 16:30 **Microstructure of the Relaxed (001) Si Surface**

A.E. Kiv, T.I. Maximova, V.N. Soloviev, Israel

Polymer Blends II

Hall D

Chair: M. Gottlieb

15:30 - 15:50 **Conductive Polyaniline/Polymer Blends Through Aqueous Dispersions: Synthesis and Characterization**

E. Segal, Israel

15:50 - 16:10 **Toughened Poly (Butylene Terephthalate)/MBS Blends Compatibilized by Epoxy Resin**

H. Tai, China

16:10 - 16:30 **Interfaces Modification and Characterization in Three-Component Polymer Blends**

I. Fisher, A. Siegmann, M. Narkis, Israel

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- 16:30 - 16:50 **Thermo-Electric Behavior of PVDF/UHMWPE/Carbon-Black Immiscible Polymer Blends**
I. Mironi-Harpaz, Israel
- 16:50 - 17:10 **Impact Modification of HDPE for Injection Molding**
A. Shuster, J. Marchukov, Israel

Intelligent Processing of Plastics **Hall E**
Chair: G. Pinto

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- 15:30 - 16:00 **Intelligent Processing of Thermoplastics by Injection Molding – Invited**
S. Kenig, Israel
- 16:00 - 16:30 **Process-Properties Relationship in Styrenic Polymers - Invited**
N. Niessner, Germany
- 16:30 - 16:50 **Modeling and Computerized Simulation of Stretch Processing for Plastics Materials**
M. Omer, Israel
- 16:50 - 17:10 **Microwave Processing of Bulk Polyurethane Adhesives and Bonded Joints**
A. Buchman, H. Birnholtz, H. Dodiuk-Kenig, Israel
- 17:10 - 17:30 **Co-Injection on a Single Barrel Machine**
S. Kilim, UK

Smart & Functionally Graded Materials II **Hall F**
Chair: E. Abramov

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- 15:30 - 16:00 **Development of Smart Materials in NASA – Invited**
R.G. Bryant, USA
- 16:00 - 16:20 **Development of Functionally Graded Material Based on Heavily Doped PbTe<In>Crystals**
S. Shusterman, Z. Dashevsky, M.P. Dariel, Israel
- 16:20 - 16:40 **Evaluation of an Optical Fiber for Use as an Embedded Sensor in a Syntactic Foam**
D. Benderly, O. Breuer, Z. Glikman, M. Leibowitz, Israel
- 16:40 - 17:00 **Graded TiB₂/TiC Composites Infiltrated with Liquid Aluminum**
Y. Seidman, N. Frage, M.P. Dariel, Israel
- 17:00 - 17:20 **Dendri-Polyamide Cured Epoxies Compositions and Properties**
L. Moshinsky, R. Yroslavsky, S. Kenig, Israel

Novel Polymeric Systems		Hall G
<i>Chair: S. Hirsh</i>		
15:30 - 15:50	Transient Electroluminescence from Polymer-Based LED Devices <i>D. Davidov, A. Yakimov, V. Savvate'ev, Israel, H.-H. Hoerhold, Germany</i>	
15:50 - 16:10	Polymer-Metal Precursors for High Temperature Superconductors <i>Y. Najary, S. Dubinsky, O. Zrian, M.S. Silverstein, Israel, I. von Lampe, G. Hinrichsen, Germany</i>	
16:10 - 16:30	Innovative Modification of PVC Particles for Absorption of Toxic Chloro-Organic Compounds From Water <i>M. Shach-Caplan, Israel</i>	
16:30 - 16:50	Novel Polymeric Foams: Synthesis, Properties and Applications <i>A. Sergienko, M.S. Silverstein, Israel</i>	
16:50 - 17:10	Partially Crosslinked Polypropylene <i>A. Dagan, F. Weiner, S. Kenig, Israel</i>	
17:10 - 17:30	Stereocomplexation of D- and L- Poly (lactide) <i>A. J. Domb, J. Slajer, Israel</i>	
Composites II		Hall H
<i>Chair: H. D. Wagner</i>		
15:30 - 16:00	Studies of Interfacial Crystallinity in Model Composites - Invited <i>G. Marom, Israel</i>	
16:00 - 16:20	Abrasive Wear of Infiltrated Metal-Ceramic Composites Based on Nonstoichiometric Titanium Carbide with Fe-C Alloy Matrix <i>S. Moisa, N. Frage, S. Sabatello, I. Eskinazi, M.P. Dariel, Israel, C. Samoila, Romania</i>	
16:20 - 16:40	The Kinetics of Transcrystallization in Isotactic Polypropylene Based Composites <i>E. Assouline, H.D. Wagner, G. Marom, Israel</i>	
16:40 - 17:00	Origin of SiC Nano-Inclusions in Silicon Nitride/Carbide Nano/Micro Composites <i>P. Sajgalik, M. Hnatko, Z. Lences, Slovakia</i>	
17:30 - 17:50	Concluding Session	Hall E

List of Posters - Poster Session 2

- P2-1 **Investigation of Thermal Shock Resistance Properties of Plasma Sprayed Thermal Barrier Coatings**
G. Xiaohui, W. Xujun, G. Yu, China
- P2-2 **Fluidized Bed Carburising**
D. Dragomir, L. Druga, Romania
- P2-3 **The Organic Phase for Aluminum Batteries**
G. Levitin, R. Tel-Vered, C. Yarnitzky, S. Licht, Israel
- P2-4 **Reactive Wetting and Electron Microscopy of i-3A Materials**
S. Avraham, Israel, N. Claussen, Germany, W.D. Kaplan, Israel
- P2-5 **Surface Modification of PEP-Films by PEP-PEO Diblock-Copolymer Additives**
M. Heuberger, T. Kerle, Israel, J. Allgaier, Germany, J. Klein, Israel
- P2-6 **Effect of Processing Parameters and of Magnesium Addition on the Distribution and Wettability of Ceramic Reinforcements in Al/SiC(P) Composites**
P. Moldovan, D. Bunea, Romania
- P2-7 **Long-Term Changes in a Heat Exchanger Steel**
P. Schaaf, Germany, S. Ariely, R. Schnek, Israel
- P2-8 **Fractal Structures Formation by Diffusion Controlled Iron Reduction**
A. Petelin, Yu. Yusfin, Russia
- P2-9 **The Details of the Elastic Fields Associated with a Martensitic Plate**
R. Shneck, Israel
- P2-10 **Formation of Subgrains in α -Uranium Due to Phase Transition**
G.A. Frank, R. Shneck, A. Landau, Israel
- P2-11 **New Adhesives for Electronic Devices**
O.L. Figovsky, L.S. Sklyarsky, O.N. Sklyarsky, Israel
- P2-12 **Quantitative Method of Evaluating Corrosion Resistance of Post Metal Etch Treatments**
I. Abramovich, B. Stern, E. Aharoni, Israel
- P2-13 **Preparation of Porous Alumina Ceramic Bodies by Direct Solid-State Sintering of Spray-Dried Granules**
M. Kravchik, M. Berlin, A. Bar, R. Fischer, Israel

- P2-14 **Cutting Test for Evaluation of Graded Cermets**
B. Zolko, N. Frage, S. Moisa, L. Levin, S. Sabatello, M.P. Dariel, Israel
- P2-15 **Ultrasonic Determination of the Elastic Properties of Graded Cermets**
R. Marks, N. Frage, E. Zaretski, O. Yehezkel, O. Tevet, M. Shochat, M.P. Dariel, Israel
- P2-16 **Interfacial Bonding Effects on Ballistic Performance of Single-Polymer and Hybrid PE Composites**
H. Harel, G. Marom, Israel
- P2-17 **Composition Materials on Base of Polyvinylchloride and Butadienacrylonitril Elastomers Plasticized by EDOS**
E.M. Gottlib, M.L. Verizhnikov, A.V. Akhmerov, P.P. Chakirov, Russia
- P2-18 **Ultraswitching in Thin Polymer Films**
L. Resnick, A.V. Butenko, I. Shlimak, Israel
- P2-19 **Interacting Oligomers, Containing Terminated Epoxy and Cyclocarbonate Groups with Primary Aromatic Amines**
L. Shapovalov, N. Blank, O. Birukov, Israel
- P2-20 **Hybrid Epoxy Urethane Chemically Resistant Materials**
L. Shapovalov, F. Buslov, O. Figovsky, Israel
- P2-21 **HTSC Thin Film Preparation by Polymer Metal Precursor Technique**
I. von Lampe, F. Zygalsky, G. Hinrichsen, Germany, M. S. Silverstein, Israel
- P2-22 **Development in Organic Leds**
G. Smith, USA
- P2-23 **Blooming of Flame Retardant Additives in Plastics**
G. Titelman, I. Gorelik, S. Bron, Israel
- P2-24 **Polymeric Materials Upgrades their Protective Properties During Exposure in Strong Aggressive Media**
O.L. Figovsky, Z.E. Fiskin, Israel
- P2-25 **Recycling of Polymers**
D. Plevinski, Y. Bar-Yaakov, J. Scheinert, Israel
- P2-26 **Structure and Properties of Biodegradable Hydrophobic Polymer Materials**
M. Ioelovich, Israel
- P2-27 **Filled Porous Epoxy Beads for Lead Sorption**
L. Gottlieb, D. Winberger, E. Rabinovitz, Israel

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- P2-28 **R & D on the Thermoplastic Rubbers (TPR) Mechanical Properties Improving**
A. Shuster, G. Oren, Israel
- P2-29 **Porous Silicon Surface Treatments by Photo-Thermal Gas Phase Reactions**
S. Stolyarova, A. El-Bahar, Y. Nemirovsky, Israel
- P2-30 **Coextrusion of High Barrier Films Containing Liquid Crystalline Polymers**
A. Ophir, S. Kenig, F. Weiner, Israel
- P2-31 **Competitive Acceleration Mechanisms in Fast Diffusional Phase**
A. Katsman, L. Levin, Israel
- P2-32 **Thermal – Fatigue Kinetics in SMC Solder Joints by SEM/EDS Fractography**
E.E. Glickman, J. Davidson, O. Ichilov, Israel
- P2-33 **Structural Model of the Hexagonal Approximant in Al-Fe-V-Si Alloy**
V. Ezersky, A. Rochman, M. Talianker, Israel
- P2-34 **Joining of ZrO₂ - 4.5 wt% Y₂O₃ Ceramics Using Nanocrystalline Tape Cast Interlayers**
B.G. Ravi, R. Chaim, Israel
- P2-35 **How Israeli Scientists Could Participate in the MaTech Programme – New Materials for Key Technologies of the 21st Century**
V. Maly, Germany

Abstracts

Monday

Plenary Sessions

THE FUTURE OF EQUIPMENT DEVELOPMENT AND SEMICONDUCTOR PRODUCTION

Dr. Dan Maydan

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The semiconductor industry has experienced exceptional double-digit growth over the past 25 years, fueled by strong demand in end-use markets such as computing, communications, consumer appliances, and industrial applications. Its future, however, depends on the ability of semiconductor manufacturers and equipment suppliers alike to lower cost while pushing the technological limits of lithography, materials science, and further the development of new manufacturing techniques like dual damascene. This presentation will describe in detail those technology challenges that semiconductor manufacturers and equipment suppliers must overcome to extend Moore's Law into the next century, as well as highlight areas where equipment productivity has added, and can continue to add, tremendous value to the growth of the industry. Finally, a new paradigm called Process Module Systems will be put forth to address the increasingly complex manufacturing requirements as well as economic challenges that the industry as a whole faces.

**MECHANICAL PROPERTIES OF THIN FILM POLYSILICON
AND THEIR RELEVANCE TO MEMS DEVICES**

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MEMS (microelectromechanical systems) is one of the fastest growing fields in microelectronics. A brief survey of the field will be given, followed by a discussion of the mechanical behavior of polysilicon, the workhorse structural material in current surface-micromachined MEMS devices. In particular, data will be presented on the fracture toughness of polysilicon, generated using single edge notched beam fracture mechanics specimens fabricated by conventional MEMS surface micromachining technology.

Residual stresses during film formation is also a significant issue in fabrication of MEMS polysilicon devices. We have now developed a novel technology in which polysilicon films can be fabricated with zero residual stresses.

APPLICATIONS OF QUASI-PERIODIC MATERIALS

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As the structures of quasi-periodic (QC) materials are fundamentally different from those of periodic materials, so are their properties. Recent studies of these properties yielded a wealth of information relevant to their usefulness.

Several examples of existing commercial applications of QC materials include strengthening of commercial metallic alloys and wear protection coatings. Other properties call for future applications as thermal barriers, solar selective absorbers, hydrogen storage, thermoelectric refrigeration and power generation.

I will review of these properties and uses as well as QC activities at the Technion.

MAGNESIUM - AN OLD METAL WITH A PROMISING FUTURE

Barry L. Mordike

Institut fuer Werkstoffkunde TU Clausthal, Germany

Magnesium was discovered by Michael Faraday but was scarcely used until the advent of the first world war. The aircraft industry was developing rapidly and the magnesium alloy 'Elektron' (AZ 91) was developed. Cast, forged, extruded and rolled parts were exhibited in 1909 at the International Airtransport Exhibition. These fulfilled the requirements of price, low density, sufficient strength and yield stress, high ductility and good corrosion resistance in air and water. The same demands are made today!

VW, when it used magnesium for the gear box in the VW beetle, used alone 20% of the world magnesium production. The advantages of magnesium have been recognised for over 100 years and yet the major breakthrough has not been achieved.

In all continents major research programmes are in progress to develop applications for magnesium particularly in the automobile sector. The progress made in alloy development and process technology will be discussed and the components now possible in magnesium presented.

Monday

Parallel Sessions: 1

INTERPHASE CONTROL FOR CORROSION PROTECTION IN COATINGS

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Interfaces are known to accelerate corrosion attack and grain boundary corrosion is an established phenomenon. However, in many systems that are designed for corrosion protection, thermally grown interphases prevent corrosion or at least reduce corrosion attack. Chromium oxide formation, Cr_2O_3 , on stainless steels at room temperature is one example of a thermally grown oxide that prevents corrosion in certain environments. Liquid metal corrosive attack must be controlled positively in the production of galvanized zinc coatings. Hot dip zinc coatings containing small amounts of aluminum, rely on the formation of a nanometer thick thermally grown interphase, Fe_2Al_5 , to prevent liquid zinc attack of the underlying steel substrate. Unfortunately, the Fe_2Al_5 inhibition layer is unstable and concentrated attack through interfaces results in attack at the grain boundaries of the steel. A model of the simulation of the inhibition layer breakdown was developed using iron oxide as the interphase with identical results. Similarly, thin films of thermally grown oxides have been used to control high temperature gaseous corrosion in both oxidation and sulfidation environments. In corrosion attack of Fe-Al alloy coatings, the amount of aluminum in the coating may affect the formation and structure of the thermally grown alumina interphase allowing for growth of the corrosion product. A thin interphase layer of Al_2O_3 has been used to prevent the oxidation of Ni/ Ni_3Al composite coatings and it has been shown that corrosion attack may be dependent upon the ability of aluminum reservoirs in the coating to form alumina at the interface. The similarities between all of these different systems lead to a general concept for the control of corrosion in both liquid and gaseous attack by thermally grown interphases.

SURFACE TREATMENT OF TANTALUM TO IMPROVE
CORROSION RESISSTANCE

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Tantalum was treated by radio-frequency (r.f.) plasma technique in order to improve surface properties and corrosion resistance. In this study thin layers of tantalum carbide were produced by inductive r.f. plasma-assisted CVD (IPACVD) in a gas mixture of argon, methane and hydrogen. IPACVD approach combines plasma-diffusion treatments with chemical vapor deposition. The thickness of the layer and the phase contents (TaC and Ta₂C) were affected mainly by two distinct ranges: (a) power input of <1200 W and pressure of 20-30 mbar; (b) power input of 1600-2000 W and pressure of 40-60 mbar. Thin layer, less than 1 μm thick, and gradient diffusion profile were observed at processing condition of range (a). However, conditions at range (b) form thick layers (several μm) consisting of mainly TaC phase with uniform chemical composition and the highest hardness (≈25GPa). Intermediate effects were obtained between ranges (a) and (b) and between their combination.

The properties of the layer, such as corrosion resistance, hydrogen content, chemical composition and TaC/Ta₂C phase ratio, are presented and discussed.

HYDROXYAPATITE COATING OF DENTAL IMPLANTS BY HVOF SPRAYING

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Hydroxyapatite- $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ (HA) is an important implant material due to its excellent biocompatibility with osseous tissues. Special consideration is given to retaining the original crystalline structure of the HA particles during thermal spray coating of implants. Conventional plasma spraying takes HA particles through high temperature which leads to significant changes of the crystal structure, increases amorphous phase content resulted in increased solubility of HA coatings.

Application of HVOF (High Velocity Oxy Fuel) process opened new opportunity to develop relatively inexpensive HA coating for dental implants with retained high crystallinity ,phase stability and good mechanical properties. The powder particles velocity in the jet depends upon the type of the powder material and may reach 500-700 m/sec (hypersonic level).

The correlation between HVOF process parameters (particle size, gas pressure, spraying distance, surface preparation) and the microstructure, mechanical properties, adhesion and solubility rate will be discussed .

The work was carried out using a Ceramed Dental HA powder. The substrate was Ti-6Al-4V alloy.

Electrodeposition of Metallic Multilayers by a Pulse Method

Tzipi Cohen

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Metallic magnetic and non-magnetic thin films have been produced by a novel electrochemical deposition process. This process has several advantages over vacuum deposition methods, including reduced cost, ability to deposit over a wide area, and ability to uniformly deposit on complex surfaces. This latter feature allows electrochemical deposition to be used in conjunction with state-of-the-art nanofabrication processing to produce materials with submicron dimensions. In addition, operation of electrodeposition at room temperature minimizes the extent of metal mixing due to interdiffusion. The main advantage of using electrodeposition by pulsing is the improvement in the deposit properties. In this method we can minimize absorption of non-metallic contamination during deposition, and cause a large increase in density, ductility and conductance of the deposit.

A new method of formation of composition modulated alloys by electrodeposition from a single electrolyte was developed by Yahalom and Zadok. According to this method, which will be used in the present project, we can consider two metals A and B, where metal B is less noble, that is of a more negative reduction potential, and A is the nobler metal.

At a potential sufficiently negative to reduce the nobler constituent A, but above the redox potential of B- a layer of A is deposited. If the potential is then shifted to a value below the redox potential of B- an alloy of A and B is deposited. By lowering the concentration of A in the electrolyte, the deposition of the noble becomes mass transport limited without hampering the deposition of the less noble metal B. Thus it is possible to produce, from a single solution, compositionally modulated samples, consisting of layers of pure metal A, interspersed with layers of an alloy of B and A, by alternately pulsing between the reduction potentials of the two metals. The relative concentrations of the metals B and A, in the alloy layers, are dependent on their respective concentrations in the bath from which they are reduced. One can, therefore, in theory, cause the composition of the alloy layer to approach that of pure metal B as closely as one desire. The difference between the redox potentials of the two metals must be big enough. During the pulse duration of the deposition of the pure A layer-time t_1 - the current density is low since the metal A ions concentration is low in the bulk, and as a result the limited current is low.

During Metal B pulse -time t_2 , we can use high current densities, so the current deposition of this metal is much higher than that of metal A. As a consequence- the percentage of the metal A in the deposit of the metal B is very low (~1 %).

This electrochemical method was further developed to produce ternary multilayered systems such as Cu/Fe-Ni (Permalloy) and quaternary systems, such as Ni-Co-Fe/Cu.

Advanced Copper Wiring for Microelectronic Devices

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Copper interconnects are expected to play an increasingly important role in the post-PC internet world, for enabling applications in both network computers and information appliances. The implementation of Cu requires the introduction of new technologies such as: in-situ cleaning, deposition of a diffusion barrier layer, deposition of a seed layer followed by filling of Cu vias and trenches, and planarization by CMP. The suitable materials, technologies and hardware solutions will be discussed. Finally, the advantages of introducing a Copper wiring systems module, recently developed by Applied Materials, will be emphasized. The module requires only a single set of inputs and outputs, with a guaranteed output, including reduced defect density and lower cost.

ELECTROLESS DEPOSITION OF COBALT ALLOYS
FOR DIFFUSION BARRIERS IN ULSI CU METALLIZATION

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Cobalt alloys (CoW_xP_y and $\text{CoW}_x(\text{B})$) electrolessly deposited were studied as potential diffusion barriers for ULSI Cu metallization. The electroless processes and the influence of the solution parameters on the deposition rate and the film composition are described. The CoW_xP_y films were deposited on various seed layers (Cu, Co and Pd wet activation) on single crystal silicon or SiO_2 (thermally oxidized). Some of the samples were patterned to form MOS capacitors or metal-silicon contacts. The devices were annealed at temperatures in the 300 – 600 °C range.

It was found that the composition of the films was not influenced by the seed layers and was constant with depth. The films were crystalline, in the hcp structure of ϵ -Co, the orientation of which was dependent on the seed layer. For the Cu seed layer, a strong basal plane (0002) orientation was found, while for Pd wet activation, the ϵ -Co phase was randomly oriented. We found that increasing the phosphorus content in the film resulted in a trend to form an amorphous phase.

The MOS devices yielded good IV and CV characteristics. In contrast, the results of the Schottky diodes were difficult to interpret due to high noise level during the measurement. The minority carrier lifetime was found to be a sensitive monitor for the presence of Cu in the substrate due to the failure of the barrier.

**THE ROLE OF SURFACE AND GAS PHASE
KINETICS IN THE NUCLEATION AND GROWTH
OF COPPER USING CHEMICAL VAPOR
DEPOSITION**

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The usage of copper replacing aluminum as interconnect material in microelectronics is now well established since Cu shows a significantly lower electrical resistivity and a higher electromigration resistance compared to Al. Electroplating, the currently applied Cu filling technique, is expected to reach its gap filling limit as device dimensions shrink. A promising way for conformal gap filling in very small dimensions is to deposit a Cu seed layer by CVD. The microstructural and morphological characterization of these films was performed using TEM and SEM techniques as well as AFM. The elemental composition at the Cu/barrier interface is determined using EDS in the TEM and SIMS. For TiN a shorter incubation time and a higher nucleation density can be observed compared to Ta and TaN. CVD Cu films deposited on top of Ta or TaN show a high density of voids compared to TiN where void formation does not occur; this can be attributed to the barrier specific nucleation densities. On a PVD Cu underlayer virtually no incubation time and no voids in the Cu films are detectable. Moreover, the CVD Cu grains formed prior to the film merging on top of the barriers show a poor wetting. A thin carbon, fluorine and oxygen containing interlayer can be found between the developed films and the barrier, which explains the poor adhesion of these Cu films. This layer is due to the formation of byproducts that compete with the Cu deposition. The byproducts formation is fast and their desorption is slow on the heterogeneous Ta, TaN and TiN surfaces, whereas on Cu either the byproducts are not formed or their desorption rate is very fast compared to the rate of Cu deposition.

EFFECT OF LDD REACTIVE ION ETCHING ON THE ELECTRICAL PERFORMANCE OF SUB-MICRON CMOS DEVICES

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In MOSFET (Metal Oxide Semiconductor Field Effect Transistor) with LDD (Lightly Doped Drain) side-wall spacers, leakage currents of source/drain, drain/substrate and transistor brake-down voltage are caused by the crystalline defects at the side-wall and the field oxide edge. These defects occur after N^+ and P^+ implantation following the LDD spacers definition. During the post implant anneal, the defects grow into dislocations. Over-etch time is the excess etch time after complete removal of the LDD oxide from the flat topography regions. This work demonstrates that minimization of over-etch time in the LDD etch-back process, reduces the stresses at the spacers and field-oxide edge, resulting in reduced dislocations depth and improved transistor performance of submicron CMOS (Complementary MOS) devices.

This work investigates the effect of over-etch time in the LDD etch-back process on the transistor performance of submicron CMOS devices. Clear correlation is seen between the transistor or diode performance and the over etch time. Increased over-etch time enhances leakage in the transistors and diodes as well as increased field-oxide penetration in the birds-beak region and deeper penetration into the Si at the spacers edge. The penetration induces stress leading to dislocation formation after N^+ and P^+ anneal. Deeper over-etch causes deeper dislocations which enhance leakage currents and weaken the diodes causing lower breakdown voltage. Steep spacers shape results in a sharp profile of the N^+ and P^+ dopants distribution. This sharp profile induces strong electrical fields resulting in hot electron degradation.

Comparison is also done between different LDD spacers materials, TEOS, Nitride and PECVD oxide. The oxide spacers show lower reverse breakdown voltage compared to the Nitride samples. Two reasons may cause that: the higher dielectric constant of the nitride spacers, and the lower overlap of nitride spacers which reduces the Gate Induced Drain Lowering (GIDL) effect.

A model is proposed based on the effect of surface roughness, shape of the LDD spacers, shape of isolation layer and with respect to the lateral diffusion in the source and drain regions. All these parameters are controlled by the LDD etch back and affect the electrical performance of Sub-Micron CMOS Devices.

SUPERPLASTICITY AND JOINING OF CERAMICS

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The high-temperature, steady-state creep of submicron- and nanoscale-zirconia and alumina-zirconia composites, having different alumina distributions and volume fractions, has been studied as function of the strain rate, stress and temperature. The data have been analyzed using the standard creep equation with a threshold stress. Microstructural observations of deformed samples indicated that deformation was achieved mainly by grain boundary sliding (GBS). This microscopic characteristic was used to join pieces of these materials. When two pieces of ceramics are compressed in the superplastic regime, GBS will produce a junction between the pieces. Several types of strong junctions have been produced: i.e. between pieces of the same material having different grain sizes and between pieces of different materials. Scanning electron microscopy of the interface showed that the interface contained no cavities or cracks. Vickers indentations and high-temperature mechanical tests indicated that the interface is as strong as the matrix. This joining technique allows the construction of a complex shape or a functional gradient material.

**SOME STRUCTURAL EFFECTS OF PLASTIC DEFORMATION
ON TUNGSTEN HEAVY METAL ALLOYS**

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Tungsten-based heavy metal alloys containing 90-97% W are two-phase composites combining high density, high strength and relatively high ductility. W content and manufacturing parameters have a strong influence on the deformation and fracture behavior of the alloys. Specimens with different content of W were prepared. The microstructure was studied after successive stages of plastic deformation, allowing the delineation of the weak regions of importance as fracture starting points. A correlation between microstructure and mechanical properties was found and an explanation for the improved plasticity with lower tungsten content is given. A fractographical study of impact loading as a function of velocity was carried out.

EVALUATING THE FRACTURE ENERGY OF THIN CERAMIC LAYERS BY CLEAVAGE

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A new method is suggested aimed at evaluating G_{IC} , the mode I fracture energy of thin brittle layers. The fracture energy is obtained by cleaving a ceramic layer sandwiched between two metallic layers, joined by brazing. The driving force for the cleavage is the mismatch between the thermal expansion coefficients of the ceramic and the metallic layers. The fracture energy is calculated from the strain energy released in the cleaved sandwiched structure. Based on this method, the fracture energy of the ceramic layer can be evaluated if the cleavage temperature is known, either by visual inspection or instrumentation. The method is effective for thin ceramic layers, for the determination of the fracture energy of cleavage planes of single crystal brittle solids (provided the cleavage plane is within the plane of the specimen), and for the interfacial fracture energy of ceramic/ceramic or ceramic/metal joints. In order to verify and calibrate the test method, polycrystalline alumina thin plates were joined by brazing with Ti-6Al-4V alloy using Wesgo Cusil ABA alloy. The appropriate selection of materials and geometry, and some difficulties arising from this method are discussed.

EFFECT OF PROOF TESTING ON OPTICAL FIBER STRENGTH

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Two methods used in the characterization of optical fiber (OF) mechanical strength are gage testing (GT) and proof testing (PT). The results of these tests can be used for reliability estimates in the performance of OF.

GT is a test to failure, in which the ultimate strength is determined. PT is a screening test, in which fibers are loaded to a point much lower than the expected strength, to remove any inherent flaws. Performance of a PT is intended to guarantee a minimum fiber strength, equal to the PT level.

One concern in the performance of a PT is that the OF can be damaged, thus reducing the ultimate strength. The objective of this work was to evaluate the effect of running a PT, a commercial OF was subjected to three different load levels, with several repeated runs at each load level. A GT was then performed on the fiber, to determine the extent of damage. This procedure was carried out on three different PT machines – an internal “old” design, an internal “improved” design, and a “build to spec” design, constructed by a commercial supplier.

Comparison of the strength and strength distribution results showed that for most load and repeat levels, the fiber was not damaged as a result of PT. For the highest load and repeat level, on the “old” design only, significant damage was caused. The damaging effect of the “old” design was caused mainly by poor design of the components (reels, belts, etc.) and by relatively long unloading times.

INORGANIC NANOTUBES FROM LAYERED COMPOUNDS

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Using the paradigm of carbon fullerenes, it was shown by Tenne and co-workers¹ that nanoclusters of layered compounds, like MoS₂, are unstable in the planar form and fold into hollow cage structures of various shapes: from spherical and polyhedral to nanotubes of a μm length and cross-section of 10-20 nm diameter (IF). Following this discovery a few methodologies for the systematic synthesis of large amounts of MoS₂ and WS₂ nanotubes were pursued. Recently, nanotubes from various other layered compounds were synthesized. Also, recently, the first MoS₂ octahedra having 572 Mo atoms, were reported².

The structural, optical, electrochemical, and tribological properties of MoS₂ (WS₂) nanotubes were investigated in some detail. It was found by Frey et al.³, that the MoS₂ (WS₂) nanotubes are semiconductors with a tunable bandgap which scales with the diameter of the nanoparticles. The 2H polytype structure is locally preserved in these nanoparticles. Few extra Raman lines observed in the IF suggest a reduced symmetry compared with the bulk (2H) predecessor. The tribological properties of the fullerene-like particles was shown by Rapoport et al.⁴ to outperform the ubiquitous solid lubricants 2H-MoS₂ (2H-WS₂). This suggests an eminent range of applications for these nanomaterials. Other applications will be briefly discussed.

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FULLERENE THIN FILMS FOR PHOTOVOLTAICS AND OPTOELECTRONICS

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This paper summarizes results of the research of C₆₀ thin films in the Ben-Gurion University of the Negev. Correlation between conditions of vacuum deposition of C₆₀ thin films (material and temperature of a substrate, evaporation and deposition rates) and their crystalline structure is presented. The latter was studied by X-ray diffraction, AFM and STM. As a result of this study, we report on the extremely rapid (10-20 Å sec⁻¹) growth of polycrystalline C₆₀ films with a strong (111)-texture.

We also discuss a relationship between the crystalline structure of C₆₀ thin films and such properties of the material as dark and photo-conductivity, optical absorption, Electron Spin Resonance and Surface Photovoltage Spectra.

Finally, device structure and parameters of C₆₀- based solar cells are presented.

SINTERING STUDY OF NANOCRYSTALLINE TUNGSTEN CARBIDE

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WC powder with an average grain size of 6 nm was obtained by high energy ball milling of micron size powders, under protective atmosphere. The influence of heating time and temperature on the powder's densification was studied during sintering in a dilatometer, operated under vacuum at temperatures between 1000-1600 °C, for times between 1-24 hours. The effect of pressure on the powder's densification was studied by uniaxial hot-press. The microstructure and morphology of the powder were characterized prior and after different sintering conditions, by analytical TEM, XRD and high resolution SEM. Three stages of sintering were found during sintering in the dilatometer: (a) rearrangement of particles at low temperature (850 °C) without grain or particle growth, (b) neckformation between powder particles at 1000-1250 °C and initial grain growth at 1200 °C, and (c) pore elimination accompanied by massive grain growth at 1300-1450 °C. Sintering at higher temperatures did not increase the density. The maximum density obtained was 95%. The apparent activation energy of the second stage of sintering was calculated and found to be 76 KJ/mol. After sintering at 1300 °C a mixture of micron size and nanometer size grains were found. The grain growth occurred in preferred orientations.

**TiO₂ COLLOIDS FOR MESOPOROUS ELECTRODES:
THE EFFECT OF THE PREPARATION CONDITIONS ON THEIR
PHYSICAL PROPERTIES**

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The high light to energy conversion efficiencies achieved with dye sensitized solar cells may be attributed to the nanoporous TiO₂ electrodes¹. These electrodes consist of small sintered TiO₂ colloids, and it is well accepted that the properties of the electrodes depend on the properties of the colloids used to fabricate them¹. This dependence includes properties such as colloid size, shape and crystal structure, thus in most cases, colloids of the same size, shape and crystal structure are considered to be similar regardless of the method used for their synthesis.

We report here a significant influence of the conditions under which the colloids are made on the physical properties of these colloids. Colloids of the same size, shape and crystal structure, which were made under different conditions, differ from each other by: (i) the surface crystal-face, and (ii) the temperature at which their structure transforms from anatase to rutile.

The TiO₂ colloids were prepared using the standard hydrothermal method reported in the literature². This method includes titration of titanium isopropoxide into acid followed by aging and hydrothermal process. The major difference between the two preparations relates to the type of acid used; nitric acid in one case and acetic acid in the other. When characterized by TEM, XRD, BET and elemental analysis the crystals seem to be similar.

In spite of the similarities found using the standard characterization methods the crystals prepared in different solutions differ by their phase transformation temperature. The colloids prepared in nitric acid experience phase transformation at lower temperature. At this point we cannot rule out the possibility that there are differences in the bulk of the colloids. However, we do see that there is a difference in the surface crystal-face when comparing the two samples. Using HRTEM it seems that the colloids prepared in acetic acid expose more of the 101 face in comparison with the colloids prepared in nitric acid. The significance of these results with respect to the performance of the mesoporous TiO₂ electrodes and dye sensitized solar cells will be discussed.

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PREPARATION AND SOLIDIFICATION FEATURES OF AS21 MAGNESIUM ALLOY

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AS21 magnesium alloy is used for high temperature applications that require adequate creep resistance. The aim of the present investigation was to obtain information regarding preparation procedure and phase constituents that are precipitated during solidification of AS21 alloy and can affect the performance of this alloy in service conditions. Magnesium alloys are usually alloyed with manganese to remove iron. Hence, the understanding the factors which influence the mutual solubility of Mn and Fe in molten magnesium is very important for establishing the efficient alloying procedure with Mn and other elements, particularly with Si in AS21 alloy. Silicon is added to Mg-Al alloys in order to enhance the creep resistance. It was believed that only Mg₂Si particles are formed through the solidification process of AS21 alloy. However, it is evident from the results of present investigation that additional phases are present in the AS21 ingots. The sequence of phase transformations occurring through the solidification process are discussed and the factors affecting the microstructure features were disclosed.

**THE INFLUENCE OF THE MICROSTRUCTURE ON THE CREEP
PROPERTIES OF AZ91 MAGNESIUM ALLOY**

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A qualitative model of the creep process of AZ91D is proposed on the basis of creep tests, optical metallography, SEM metallography and TEM study. This model answers a few questions concerned with creep results. The major deformation process is dislocation motion that is not restricted to basal planes (as in pure magnesium). In addition, secondary mechanisms such as cavitation at the grain boundaries in the case of ingot casting and grain boundary sliding in the case of pressure die casting were found to contribute to the deformation process. The microstructure of both, ingot casting and pressure die casting is unstable due to non-equilibrium solidification process. This instability causes secondary precipitation of β ($Mg_{17}Al_{12}$) phase during creep. These secondary β particles coarsen with time, lose their strengthening effect and the material loses, therefore, its creep strength.

In view of this model, creep resistance in case of interrupted creep tests (which are a better simulation of a vehicle component) should be inferior comparing to conventional creep test under the same parameters. Preliminary results of such creep tests support this claim and can be explained on the basis of the creep model.

Galvanic corrosion of magnesium alloys.

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Israel Institute of Metals

Magnesium alloys are being used primarily for engineering applications, where there is an obvious need for lightweight materials. The limited use of magnesium results mainly from its corrosion properties. Magnesium and its alloys are located in the first row of the galvanic series of important metals in seawater environment. They are the most anodic and therefore they are particularly vulnerable when they are coupled with other metals.

We investigated the galvanic corrosion behavior of magnesium alloys AZ91 and AM50 coupled to the following alloys: mild steel, cast iron, stainless steel, aluminum, zinc, copper and brass. Galvanic coupling was studied by monitoring the current generated between the galvanic couples in 0.1% solution of NaCl using a zero-impedance ampere meter.

The results showed that magnesium alloys behaved as the anode when coupled with the tested metals. The galvanic current decreased with time when the tested magnesium alloys were coupled with zinc and aluminum and it increased with time when they were coupled with mild steel and stainless steel. Galvanic currents for the couples magnesium alloys - zinc and magnesium alloys - aluminum were small in comparison to all other tested couples.

Weight loss measurements were recorded for several uncoupled and galvanically coupled samples in 0.1% NaCl solution and in the salt spray cabinet.

Several metallic and insulating coatings on mild steel and aluminum were studied with the purpose to decrease galvanic corrosion of magnesium alloys coupled with mild steel and aluminum.

Microstructural Analysis of Gas Tungsten Arc Welded Magnesium AZ91 Alloy

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The integrity and performance of a weld is controlled by the solidification behavior of the weld metal or fusion zone (FZ) and by the metallurgical behavior of the base metal immediately adjacent to the weld, the partially melted zone (PMZ) and the heat affected zone (HAZ). The microstructures and microhardness of the fusion zone, the partially melted zone and the heat affected zone of gas tungsten arc (GTA) welds of magnesium AZ91 alloy were studied. The details of the solidification behavior and the origin of the different morphologies were investigated by optical and scanning electron microscopy and the distribution of the alloying elements was measured by energy-dispersive (EDS) and wavelength-dispersive spectrometry (WDS).

The magnesium AZ91 alloy has a typical as-cast structure. It comprises of an incomplete network of the β -phase, $Mg_{17}(Al,Zn)_{12}$ in form of degenerate eutectic together with Mg dendrites exhibiting strong Al-rich coring. In addition, small particles of the Al_8Mn_5 phase, rich in iron (1-2 at%), are scattered in the matrix. The morphology of the Mg dendrites and the β phase vary with the location of the inspected area in the AZ91 cast ingots.

The fusion zone contains an increased fraction of interdendritic β phase, finer and richer in Zn than those observed in the base metal prior to welding. Some variation in weld grain morphology was observed, but equiaxed Mg dendrites showing Al-rich coring were dominant. The minor interdendritic constituent formed in the FZ consists of "pearlitic" precipitates; this arises from solid state discontinuous decomposition of the Mg-Al supersaturated solid solution. The discontinuous precipitation has commenced at a grain boundary that has then advanced into an adjacent grain.

FUNCTIONALLY GRADED MATERIALS

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The idea that an inhomogeneous material, under certain circumstances, may display improved performance as compared to a homogeneous one is very old. Only over slightly more than a decade, however, has the concept of inhomogeneous, or more specifically graded materials, been put in a more rigorous framework. The acronym FGM (Functionally Graded Materials) is being used to denote material parts in which a pre-designed gradient of properties has been introduced along one or more of their dimensions in order to improve the overall performance.

The talk will briefly review the constraints involved in implementation of the concept and the state of art with regard to processing FGMs. Some illustrative details based on the related activity that is being pursued at the Ben-Gurion University will be presented.

DIFFUSION INDUCED COMPOSITIONAL GRADIENT IN TiAlN THIN FILMS

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Functionally graded TiAlN coatings are promising coatings for improving wear and oxidation resistance. A new approach for fabricating graded TiAlN coating was examined. A bi-layer coating consisting of AlN on a Ti layer is heat treated in order to activate diffusion processes that may induce a composition gradient in the film. The heat treatment however, may also cause the formation of undesired compounds, which might degrade the coatings performances. In this work the formation of a composition gradient and the kinetics of interfacial reactions between the AlN and Ti layers were investigated.

Bi-layer AlN/Ti coatings were deposited on Mo substrates at different N₂/Ar ratios. The coatings were heat treated in an N₂ atmosphere in the temperature range of 600-800°C for 1-3 hours. The coatings were characterized prior and after the heat treatment using X-ray diffraction for phase identification and Auger electron spectroscopy in order to reveal the composition gradient in the films. Cross sectional TEM observations were performed for microstructural characterization. The results indicate that by choosing the proper deposition and heat treatment conditions it is possible to fabricate compositionally graded (Ti, Al)N coatings using the diffusion activated approach.

THERMAL TESTING OF FUNCTIONALLY GRADED MATERIALS USING LASER SHOCK METHOD

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A functionally graded material (FGM) is one of the advanced composite materials which is composed dominantly of technical ceramics on one side of the element, dominantly of metal on the other and an intermediate layer with composition changing continuously from ceramics to metal at a distance of the order of 100 microns. These materials are developed for the purpose of use as a protective coating for the metallic elements which are subjected to thermal loads due to high temperature environment (up to a temperature of 2000 K), cyclical changes of temperature, etc. We developed an algorithm of the thermal test experiment of a FGM coating using a local heating of a specimen by a power laser beam which results in formation of thermal stress field which, under certain conditions, causes fracture of a coating. We developed an analytical method for determining the temperature and thermal stresses distributions in a tested specimen whereby a coating is described as a laminated plate composed of n layers with the constant material properties within a layer. Temperature and stress distributions in a nonhomogeneous laminated plate are obtained in an analytical form using the method of integral transforms. Analysis of the limiting case of the derived distributions when the thickness of layer i $\Delta_i \rightarrow 0$ and the number of layers $n \rightarrow \infty$ allowed us to determine the stresses distributions in an FGM assembly and the operating parameters of the thermal shock method, e.g., the duration of heating, laser beam radius, etc. Analysis of numerical results obtained for the assembly composed of a substrate HS-steel and a FGM coating (tungsten carbide ceramics and HS-steel) shows that spatial distribution of ceramics in a coating influences essentially the level of tensile thermal stresses induced by a laser heating. This allows to design coating so as to minimize the stresses induced by the high temperature environment. The experimental results obtained during thermal tests of this assembly are discussed.

FUNCTIONALLY GRADED MATERIALS BASED ON
NONSTOICHIOMETRIC TiC INFILTRATED WITH A Fe-C ALLOY.

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Ceramic-metal composites based on sub-stoichiometric titanium carbide provide several degrees of freedom for the generation of graded structures. Thermodynamic calculations show that it is possible to control the carbon transfer from the melt to the carbide. Thus, depending on the ratio between the carbon concentration in the melt and in the ceramic phase, the microstructure of the metal phase can be altered. For experimental verification, solid ceramic skeletons, consisting of 3 and 4 superposed layers of TiC_x powders, each with a different carbon content, $0.8 < x < 0.98$, were infiltrated with Fe-0.8%C alloy. The microstructure of the metallic phase in the graded samples changed continuously from pearlite to ferrite in the as infiltrated composite and from martensite to ferrite in the as quenched composite. The compositional and microstructural changes in the FGM resulted in variation of the hardness. Thus, the hardness of the as infiltrated sample varied from 900HV on the $TiC_{0.98}$ side to 650HV on the low carbon content side. In oil quenched samples, the hardness varied from 1400HV to 650HV.

SYNTHESIS OF DENSE OXIDE-BASED *IN SITU*
COMPOSITES VIA THERMAL EXPLOSION / SHS UNDER
UNIAXIAL APPLIED PRESSURE

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Self-Propagating High-Temperature Synthesis (SHS) is one of the rapidly emerging cost-effective technologies used to synthesize *in situ* composites - materials in which desired reinforcements and matrices are formed during processing. The major problem to be overcome in the SHS processing of advanced materials is the high retained porosity of the synthesized products.

In the present research, *in situ* processing of $\text{Al}_2\text{O}_3/\text{TiAl}$, $\text{MgAl}_2\text{O}_4(\text{spinel})/\text{TiAl}$ and $\text{Al}_2\text{O}_3/\text{TiB}_2/\text{TiAl}$ particulate reinforced composites from fine Al-TiO_2 , Al-Mg-TiO_2 and $\text{Al-AlB}_2\text{-TiO}_2$ powder blends via thermal explosion mode of SHS under pressure is considered. In the 'reactive forging' method used, compacted powder blends were placed between the press rams preheated to 700-1000°C and a low uniaxial pressure (5-10 MPa) was applied. Under these conditions, full conversion of reagents into products and near full final density could be achieved. Rapid cooling from the combustion temperature due to the 'heat sink' action of the rams resulted in the fine submicron-scale microstructure of the *in situ* composites synthesized. Accurate temperature monitoring allowed to establish SHS ignition and combustion temperatures (T_{ig} and T_{comb}), and to determine kinetic parameters of the combustion consolidation process. Based on these data, the role of heat transfer in temperature evolution and product formation during reactive forging has been assessed.

NANO PARTICLE REINFORCED POLYMER COMPOSITES

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Polymer composites based on nano-particle reinforcement, possess unique properties due to interactions in the molecular level between the nano-particles and the polymer.

In the last decade the manufacturing technology of nano-particles made of aluminosilicates has been developed. The particles are 1-2 nano-meter thick and the dimensions of length and width are in the range of 1000 nano-meters.

On the basis of these nano-platelets, in combination of polyethylenes and polypropylenes, a few compounds were formulated and characterized with respect to their nano-structure, thermal properties and permeability to oxygen.

Preliminary results indicated that the surface treatment of the particles surface and their dispersion state, have the main effect on the mechanical and physical properties of the nano-composites.

CARBON NANOTUBES-BASED POLYMER COMPOSITES

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Recent experimental and theoretical results suggest that carbon nanotubes hold great promises as a possible reinforcing phase in composite materials of a new kind. Such developments still present, however, enormous practical challenges, in particular when attempting to probe the properties of individual nanotubes, for which most studies consist of computer simulations. Experimental observations of various deformation and fracture modes under compression of single multi-walled carbon nanotubes, obtained as a result of embedment within a polymeric film, are reported. Based on a combination of experimental measurements and the theory of elastic stability, the compressive strengths of thin- and thick-walled nanotubes are found to be about two orders of magnitude higher than the compressive strength of any known fiber. We report the first observation of the formation of damage doublets in adjacent carbon nanotubes embedded in a polymer matrix. Such damage clusters are comparable to those arising in fiber-reinforced composite materials, as a result of the redistribution of stress from a failed fiber to its unfailed adjacent neighbors. Bundles of single-wall carbon nanotubes under tensile stresses were observed to fracture in real-time by transmission electron microscopy. The expansion of elliptical holes in the polymer matrix results in a tensile force in bridging nanotubes. The polymer matrix at both ends of the bundles deforms extensively under the tension force, and fracture of the nanotubes occurs in tension within the polymer hole region rather than in shear within the gripping polymer region at the ends of the bundles. This provides evidence of significant polymer-nanotube wetting and interfacial adhesion. Recent advances in the potential use of nanotubes as sensors in composite matrices are also briefly outlined.

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APPLICATION OF FLUOROPOLYMERS BASED COMPOSITES
IN ELECTROLUMINESCENT FIBERS

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Fluoropolymers are known to be stable and nonflammable materials. A few of them based on PVDF copolymers possess high value of dielectric constant ($\epsilon > 8$) and can be successfully applied as binders for dielectric and electroluminescent powders in production of Electroluminescent Fibers (ELF). In some cases said materials are used as colored insulating layers of ELF.

Very high level of filling (volume fraction of filler can achieve 50%) requires from binder significant flexibility and low surface tension which avoids internal stresses on interfacial boundary between filler and binder. Fluoropolymers as having very low surface tension comply mentioned demand. PVDF of various composition allows to develop very flexible highly filled dielectric and electroluminescent layers without application of cross-linking agents and plastisizers.

Low surface tension of Fluoropolymers improves compatibility of filler and binder interior particular layer but results in poor wettability between two layers during dip coating process. This problem were solved with application of specially selected fluoro-based surfactants which modify surface tension of polymer solution and permit to obtain multi-layer system of high quality.

ELF for outdoor application comprising Fluoropolymer made insulating layer are stable for action of sun light , microorganisms , water vapors and can not be source of fire. This work contains results on behavior of photo-luminescent colorants in Fluoropolymers in terms of photo-stability and compatibility with fluoropolymeric matrix. The latter is very important in development of colored ELF for outdoor usage.

Kinetic scheme is presented which describes photo-fading of photo-luminescent dyes depending on nature of polymer matrix.

ELF are very sensitive to presence of water molecules. Application of PCTFE extrusion coating substantially increases life time of ELF in case of elevated humidity (RH more than 80%). ELF durability is presented depending on various polymeric protecting layers and environmental conditions.

GRAIN BOUNDARY AND TRIPLE JUNCTION MIGRATION
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The current status and latest achievements of grain boundary and triple junction migration in metals are reviewed.

The migration of 90° <112> planar tilt symmetrical and asymmetrical grain boundary in specially grown Bi bicrystals driven by magnetic driven force and the dependence of grain boundary mobility on temperature, driving force and the direction of motion are addressed.

The motion of low and high angle planar tilt <112> and <111> grain boundaries moved by shear stresses and the peculiarities of such a motion are considered. In particular, the sharp transition from low angle to high angle boundaries was observed.

In practically the motion of a straight grain boundary is the exception rather than the rule. The shape of a moving grain boundary is a source of new and useful findings concerning grain boundary migration. The experimentally derived shape of a grain boundary in Al-bicrystals was compared with theoretical calculations in the Lücke-Detert approximation.

A thermodynamic treatment of vacancy generation accompanying grain growth is discussed.

The experimental and theoretical results of a motion of grain boundary systems with triple junctions are presented. Their impact on the kinetics of microstructure evolution and, in particular, on Von Neumann - Mullins relation is outlined.

GRAIN BOUNDARY GROOVING PHENOMENA IN THE Ni-RICH NiAl

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Thermal grooves formed during the high temperature annealing of the Ni-41 at.% Al alloy at the intersections of the grain boundaries and the free surface were studied. The atomic force microscopy (AFM) imaging of the grooves topography demonstrated that the grooving process is accompanied by the relative shift of two neighboring grains. The correlation between the shift amplitude and the misorientational degrees of freedom of the grain boundaries was established. The theory of the grain boundary grooving with the simultaneous sliding process was developed and a good agreement between the calculated and measured groove profiles was demonstrated. Possible mechanisms for the relative shift of the grains during annealing are discussed. It is also demonstrated that the dynamics of grain boundary motion during the annealing can be deduced from the grooving studies.

INVESTIGATION OF ANNEALED STRUCTURES IN TERNARY U-Nb-W ALLOYS.

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Dilute uranium alloys with small additions of the second transition metal solute have been the subjects of extensive investigations. The elements that are most commonly alloyed with uranium are molybdenum, niobium, titanium and zirconium. In general, these alloying additions, extensively soluble in the high temperature uranium γ phase, are almost completely insoluble in the low temperature α phase. Therefore, on slow cooling the γ phase decomposes via diffusional phase transformation forming a two-phase structure of α phase plus either an intermetallic phase or an alloy rich γ_0 phase.

This work presents the results of study of the effect of addition of tungsten as a second solute element, on the structures formed in annealed ternary U-5.8wt%Nb-W alloys. It is known that the solubility of tungsten in uranium is extremely limited, less than 1at%, and there is no intermetallic compound in the uranium-tungsten system, however, there is full solubility of tungsten in niobium.

The niobium content in the alloys was chosen near the eutectoid composition of U-Nb system. The range of the tungsten content was between 0.5wt%W to 4.0wt%W.

In common with other U-Nb base alloys it was found that the microstructure of annealed U-Nb-W alloy consists of two main phases: orthorhombic α -uranium and γ_0 tetragonal phases that form as a result of eutectoid reaction. However, in addition to these phases the existence of small particles of new ternary U-Nb-W phase was revealed. The composition of this phase as determined by EDS analysis in TEM was Nb=49.7555at%, W=45.7055at%, U=4.5366at%, and the unit cell was identified as cubic BCC with the lattice parameter $a=3.185\text{\AA}$.

**THE OBSERVATION OF NON-EQUILIBRIUM
TRANSFORMATIONS AND METASTABLE STRUCTURES
IN TERNARY TITANIUM ALUMINIDE**

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Titanium aluminides are promising materials for aerospace applications, though their usage is limited due to low ductility at ambient temperatures. To improve materials' performance, microstructural optimization by thermo-mechanical treatment and alloying is employed. Iron is a potential candidate for property improvement due to presence of several ternary and quasi-binary cubic phases in the Al-Ti-Fe system. Yet the available data on phase and microstructural formation in this system is restricted to several cross-sections of ternary phase diagram. The knowledge on phase and microstructural formation under non-equilibrium conditions, which is essential for alloys' development, is lacking. In the presented work, non-equilibrium transformations and formation of metastable structures in the iron-alloyed titanium aluminide have been investigated. The observed microstructures have been characterized by x-ray diffraction, scanning and transmission electron microscopy, electron probe microanalysis. The particular emphasis have been made on formation of ternary Al_2FeTi phase (τ_2 , $Mn_{23}Th_6$ type) and its metastable modifications as well as on formation and decomposition of quasi-binary $FeTi(Al)$ phase (CsCl type). The influence of high temperature phase content and cooling conditions on structural state of the examined alloy at ambient temperatures has been analyzed and discussed.

Monday

Parallel Sessions: 2

Studies of copper and silver deposition, employing the EQCM

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Renewed interest in the electrochemistry of copper deposition stems from the transition from Al to Cu as the conducting medium in vias and interconnects in VLSI circuits, following the transition to 0.25 μm technology and below. The first stages of deposition are of paramount importance, since they may determine the bonding of the metal to the substrate. The EQCM is an excellent tool to study the formation and properties of very thin films, because of its high sensitivity.

The formation and stability of cuprous ions as intermediates in the deposition of copper is discussed. A Pourbaix-type diagram is proposed to show the regions of thermodynamic stability of cuprous ions.

Copper deposited on gold is stressed, because of the mismatch between the crystal parameters of the two metals. This stress can readily be detected by the EQCM. Some of the stress is relaxed with time on open circuit. In comparison, a layer of silver deposited under similar conditions shows no effect of stress, since the crystal parameters of Ag and Au are almost identical.

It is shown that the EQCM can serve as an additional important tool in the development of electroplating procedures, the study of the effect of additives and for quality control during manufacturing.

**THE EFFECT OF COMPOSITION AND MICROSTRUCTURE ON
THE CORROSION BEHAVIOR OF MAGNESIUM-ALUMINIUM
ALLOYS**

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The magnesium alloys have an excellent "strength-to-weight" ratio thus they have drawn much attention particularly in automotive applications. The use of magnesium alloys can reduce the vehicle weight, which is important for saving energy. The most common Mg alloy in used is the AZ91D which have adequate corrosion resistance. However, one of the major causes for corrosion deterioration is the presence of impurities, non-metallic inclusions and intermetallic phases. Some of these phases may be more cathodic comparing to the Mg matrix and hence it is important to understand their effect on the corrosion mechanism of the magnesium alloys.

In this study the corrosion behavior of magnesium AZ91 is investigated. The corrosion resistance was measured under salt spray, and immersion test conditions as well as under potentiodynamic polarization analysis in NaCl and Mg(OH)₂ electrolyte. A detailed discussion of the role of microstructure, especially β phase (Mg₁₇Al₁₂) and cathodic particles, on the mechanism of the corrosion process will be presented.

INHIBITION ABILITY OF SOME ORGANIC ACIDS IN ENGINE COOLANTS

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Israel Institute of Metals

Automobile engine cooling systems contain a variety of metals including steel, cast iron, copper, brass, lead solder and cast aluminum operating at the high temperatures, pressures, and flow rates. The possibility of corrosive attack on metals is high. The resulting metal damage, such as pitting and metal loss, causes perforations or other defects leading to fluid loss and system failure.

One well-recognized method for preventing the above mentioned phenomena is an addition of corrosion inhibitors to engine coolants based on ethanediol as an antifreeze material. However, a number of commonly accepted corrosion inhibitors have been found to cause environmental problems: amines and nitrites present together can form dangerous nitrosamines, phosphates are detrimental to the environment. In addition corrosion inhibitors such as phosphates, silicates, borates, nitrites, nitrates and molybdates are consumed after sometime, which further contributes to the corrosion effect on the engine system. Besides, many conventional inhibitors used in automobile cooling systems do not provide adequate protection against the pitting of aluminum parts especially in presence of Cl^- ions and traces of Fe^{+3} , Cu^{+2} , Zn^{+2} ions. Therefore, supplemental coolant additives are needed which are environmentally friendly and have a long term use.

A new class of inhibitors based on organic carboxylic acids has been adopted for engine coolants. In this work we studied several carboxylic acids and their combinations were tested as corrosion inhibitors in antifreeze. Linear polarization and weight loss methods were used to measure absolute corrosion rates, cyclic polarization was used to study localized corrosion of aluminum, and the impedance technique was used to determine the mechanism of inhibition of carboxylic acids. The most effective inhibitors combinations were tested according to ASTM D3306 specifications and similar specifications of other countries and were found to comply with their requirements.

CORROSION OF SINTERIZED STEEL IN SALTY WATER

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The corrosion processes in NaCl 5% solution were studied on steel samples, obtained by sintering from powders of different compositions, with a carbon content of 0.02 – 0.12%

The results are comparatively discussed with those obtained for steels with no special alloying elements and a carbon content of 0.2 – 0.45%.

Tests were done on a galvanostatic installation and current – potential data were used to obtain the Tafel plots. Measurements started by evaluating the static potential, then the intensity was stepwise modified and the potential was measured, both on the sample and on the platinum electrode, using a saturated calomel electrode as reference.

The kinetic parameters of corrosion were calculated: the density of the corrosion current, the mass index and the penetration index and the influence of the samples' composition is discussed. The values of the corrosion potential are evaluated and compared for the investigated samples, and the influence of the interfaces is outlined.

Using a SEM, the surfaces of the sintered steels were scanned and it was found that intercrystalline corrosion is likely to appear due to the reaction of the Cl⁻ ions with the iron.

ANALYSIS OF CORROSION FAILURES IN REFINERY EQUIPMENT

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Analysis of corrosion failures in the CCR Unit, FCC Unit, Crude Distillation Unit and BTX Plant has been carried out for two last years. The analysis showed the following: 60 % of the failures relate to general, pitting, galvanic and MIC corrosion. 30% of failures relate to dew point corrosion, erosion (cavitation), stress corrosion cracking and deterioration of polymers. 10% of the failures relate to corrosion under thermal insulation, grooving corrosion and deterioration of ceramics. Typical corrosion failures and their causes were analyzed. Remedial anti-corrosion measures were recommended for improving the corrosion situation. Analysis of the causes of corrosion failures showed that in 50% of the cases, human error is responsible due to wrong choice of materials and incorrect operating conditions.

MULTI-LAYER UNDER BUMP METALLURGY FLIP CHIP BONDING TECHNOLOGY

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In recent years Flip Chip technology has attracted much attention at the semiconductor device packaging processing world, as an alternative to the traditional wire bonding. The major promoters for this technology transition are speed, size and the increasing number of I/O in more complex ASICs and Microprocessor applications. The technology was first introduced by IBM over twenty years ago and was at the core of its Multi Chip Module (MCM) packaging. The most critical step of flip chip processing sequence is the bump, a solder ball placed on the Al I/O pad which replaces the traditional wire bond.

The process developed by IBM to make the bump is called "C4", which comprises the following three fundamental steps; (1) Deposition of the Under Bump Metallurgy (UBM), (2) Solder deposition, and (3) Reflow and ball formation. The first two steps are executed by means of evaporation of the materials through a stencil molybdenum masks. Traditionally, the UBM comprises a base layer of Cr, which provided for a good adhesion and diffusion barrier, followed by deposition of a "phased in" mixed layer layer of Cu and Cr, and capped with a Cu layer. The bimetal "phase in" layer is aimed to provide for good adhesion between the base Cr layer and the Cu-capping layer. A Au layer is then deposited on top of this metal multilayer structure to provide for the bonding geometries.

Lately, a number of companies have searched for more cost-effective alternatives to C4. As an example for such an approach, the UBM may be sputtered instead of being evaporated, the molybdenum mask is replaced all together by a standard photolithography processes, the solder is either deposited by electroplating or by printing techniques similar to ink-jet. Alternative the UBMs metal multilayer such as Ti/Cu/Au, or Ti/Ni/Au. Successful execution of such a sequential deposition processes, requires an equipment that is capable of sputtering of multiple metal layers (up to 4) , as well as a "phase in" deposition technique.

In 1998, Applied Science and Technology introduced a new in-line sputtering tool with these specific characteristics. The tool's unique architecture enables the deposition of most of the practical UBMs structures, with a high throughput of 60 wafers/hour. In this talk we will discussed some of the "phase in" techniques and results that have been achieved through a split target process (patent pending), aimed in effective replacement of the currently used flip chip evaporated process.

THE INTERFACE OF FLUORINATED AMORPHOUS CARBON WITH COPPER METALLIZATION

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In order to integrate low-k materials into new devices with rapidly decreasing dimensions, a few very important issues need to be considered. These integration issues include: adhesion, chemical and electrical stability, adhesion etc. The most probable areas to detect instabilities in the form of elements diffusion, and chemical reactions are the interfaces of the low-k layers with the adjacent materials e.g. the metals.

We are currently studying fluorinate amorphous carbon (a-F:C) films, prepared by HDP-CVD methods from CH_4 and C_4F_8 , as candidates for low-k inter metal dielectric applications. The dielectric constant of the film is ~ 2.7 , measured by C-V at 1MHz. In order to enhance the film's adhesion to the adjacent layers, an adhesion promoter layer is introduced. The film is stabilized by 30 minutes of annealing at 400°C in N_2 environment. The samples used for the current research consist of metal layer (Cu or its diffusion barriers Ta or TaN,) and the a-F:C film with the adhesion promoter sandwiched inbetween.

In order to learn about the film and interfaces stability the samples were annealed at 400°C and 500°C for 30 minutes. RBS, SIMS and AES analyses were used to study elements interdiffusion. HRTEM was used to observe the microstructure of the interfaces and possible formed inter-layers. These results as well as the stability of the dielectric constant after the 400°C and 500°C anneals will be presented.

**EFFECTS OF SILICON CONCENTRATION ON MECHANICAL
CHARACTERISTICS OF SILICON CONTAINING DIAMOND-LIKE
CARBON FILMS DEPOSITED BY PACVD**

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Hydrogenated, silicon containing diamond-like carbon (DLC) films have fascinating physical properties including a wide bandgap that can be tailored and photoluminescent characteristics, as well as hardness approaching that of diamond, and excellent wear resistance. The properties and structure of Si containing DLC are highly dependent on the deposition conditions and techniques used. In this study, Si containing DLC was deposited on a variety of substrates using plasma assisted CVD. Different combinations of tetramethylsilane and acetylene gas were used as precursors. The effects of Si/C ratio on various mechanical properties of the films were studied and the composition of the films was ascertained using XPS. Sessile drop measurements showed a decrease in surface energy with increasing Si/C ratio. The intrinsic compressive film stress was calculated from the substrate curvature. The film stress also decreased as Si/C increased. The hardness of the deposited films was investigated using a nano-indenter. Atomic force microscopy analysis showed the films to have roughness on the order of a few Angstrom. A surface morphology with a "granular" appearance was evident. The films proved to be resistant to chemical attack and provided excellent corrosion resistance when applied to copper and stainless steel substrates.

**SILICON NITRIDE DEPOSITION BY RTCVD
- AN OPTIONAL GATE DIELECTRIC REPLACEMENT
IN DEVICE FABRICATION**

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The ability to scale down devices has led to increasingly complex and sophisticated replacements for the gate dielectric SiO_2 film. An efficient way to reduce leakage current is to use a film with a relatively high dielectric constant. Silicon nitride is an attractive candidate for this purpose. However, conventional thin CVD nitride is leaky due to a high trap density in the film and the nitride forms a poor interface with silicon.

Ultra thin (<3 nm) $\text{Si}_3\text{N}_4/\text{SiO}_2$ stacked layers have demonstrated better properties compared to oxide films with identical thickness. The films have been found to provide a superior boron diffusion barrier and lower leakage current.

TWO-DIMENSIONAL DIFFUSION CHARACTERIZATION OF BORON IN SILICON USING REVERSE MODELING

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Accurate computer simulation of semiconductor structures and devices requires modeling the underlying physics of point defect and impurity diffusion interactions. For this reason, much effort has been invested in determining both models and parameter values with good predictive capability. There are sustained efforts to develop physical techniques for two dimensional (2-D) profile measurements, like lateral Secondary Ion Mass Spectroscopy (SIMS). However, to date, all these techniques are very expensive and time consuming. The 1-D analytical techniques give information on the dopant inside the bulk silicon, but only indirectly describe the concentration of silicon point defects: interstitials and vacancies.

This work deals with the physical basis for the simulation of impurity diffusion in 2-D silicon. Much effort has been directed at modeling the enhancement diffusion that occurs, after boron implantation, during oxidation of the silicon surface. We use, for the first time, the reverse modeling concept. It is based on comparison between 2-D process/device simulations and various electrical and physical measurements of sub-micron devices. We started by calibrating the process and device simulators by 1-D process modeling adjustment using results of SIMS, thickness calibration, and sheet resistance measurements. 2-D electrical modeling for the mobility and threshold voltage was compared to sub-micron device measurements and data from literature reviews.

Various experiments that measure electrical characteristics as a function of several parameters that affect the two-dimensional diffusion (for example, temperature, time, and implant dose) were studied in order to extract information regarding the two-dimensional diffusion of boron in silicon.

**BIOLOGICAL COMPOSITES OR ALL YOU WANTED TO KNOW
ABOUT SHELLS**

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Natural hard tissue-shells, teeth, and bone -- are ceramic/polymer laminated composites that utilize CaCO_3 (calcite or aragonite) or $\text{Ca}_5(\text{PO}_4)_3\text{OH}$ (hydroxyapatite) as the ceramic (mineral) phase and a variety of proteins as the polymer phase. These *biological* ceramic composites are produced at ambient conditions with a wonderful diversity of microstructures (microarchitecture) and with properties that are quite impressive, given the humble nature of the ceramic phase and the modest quantity of proteins present (the organic constituents in some mollusk shells can be as small as 0.5%).

Structure/property studies of these laminated composites present exciting opportunities at the intersection of biology, materials science, and applied mechanics, as will be illustrated with results from the giant conch, *Strombus gigas*, an organism at the apex of molluskan evolution.

USING POLYMER SCIENCE TO INFLUENCE CELL BIOLOGY

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Spinal cord injury is a devastating disorder of the central nervous system for which there is no clinical therapy. We are investigating new strategies to enhance regeneration by specifically designing materials for *in vivo* application. There are two principle mechanisms to enhance nerve (axon) regeneration: contact-mediated signals and diffusible signals. For the former, we have modified fluoropolymers to evoke a specific cellular response. For example, we have modified poly(tetrafluoroethylene-co-hexafluoropropylene) (FEP) with cell adhesive peptides (i.e. YIGSR and IKVAV) and have patterned poly(chlorotrifluoroethylene) (PCTFE) with cell adhesive (peptide) and cell repulsive (polyethylene glycol) regions. We found that primary hippocampal neurons responded to our surfaces by extending a similar number and length of neurites on peptide regions as observed with positive controls (polylysine / laminin). For diffusible signals, we have studied the controlled release from biodegradable polymers, such as poly(lactic-co-glycolic acid) (PLGA), of bioactive neurotrophic factors, such as nerve growth factor (NGF), and investigated the effect of concentration gradients on cellular response. By co-encapsulation with ovalbumin, NGF release is bioactive for 80-90 days and degradation and percolation control its release. We found that there is a minimum concentration gradient to which neurons respond in terms of a directed cellular response.

SELF ASSEMBLY OF NANOMETER SCALE ELECTRONICS BY BIOTECHNOLOGY

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The realization that conventional microelectronics is approaching its miniaturization limits has motivated the search for an alternative approach based on self-assembled nanometer-scale electronics. Considerable efforts have been put in that direction but progress has been impeded by the fact that materials amenable to self-assembly (e.g. organic and biological molecules) display poor electrical characteristics while electronic materials (e.g. metals and semiconductors) are chemically too simple to show self assembly of elaborate structures. In an attempt to overcome that difficulty we have recently proposed a new concept based on the hybridization of biological materials with electronic ones. The inherent molecular recognition properties of the biological molecules are first utilized to construct a template for the subsequent assembly of electronic materials into a molecular scale useful circuit. In the talk, the concept will be reviewed together with the results of recent experiments where some of its key elements have been investigated. A fully self-assembled fabrication of a conductive metal wire, considerably narrower than the narrowest wire available in conventional microelectronics will serve as an example for the strength of the proposed approach. Open conceptual and practical questions related to self-assembly of complex molecular scale electronics will be discussed.

**Titanium Alloy grade 7 Under the Influence of Micro - Organisms
In Diluted NaBr Solution.**

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The Behavior of Titanium grade 7 in diluted bromide solution contaminated with Micro - Organisms, was studied. The exposure of the tested titanium alloy to aqueous environments combined with microorganisms was carried out in a unique cell under induced potential. During the exposure the potential between the anodic surface and the reference electrode was recorded. Heavily polluted environment with micro - organisms resulted in break down of the steady potential after an incubation period of about 2-4 hours.

Results clearly indicate that the aqueous media polluted with microorganisms inhibits the anodic passivation process and depolarizes the cathodic process resulting in higher corrosion current density. Potentiodynamic polarization measurements show that increasing the total bacterial counts (TBC) to a value above 10^4 /ml results in sensitivity to localized corrosion attack. As the TBC increases the anodic break down potential decreases from about 950 mV in 10^2 /ml to about 800mV in 10^7 /ml.

Additional phenomena that observed in the induced potential testing apparatus are:

- Preferential growth of pseudomonas bacteria in the vicinity of the titanium alloy grade 7 in the separated electrolytic cell were the platinum electrode acts as a cathode.
- Disinfecting effect of the solution in the separated electrolytic cell were the platinum electrode acts as an anode.

SCIENCE AND SERENDIPITY IN FUNCTIONAL CERAMICS- MATERIALS INTEGRATION AND MATERIALS INNOVATION

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Electroceramics research is driven by technology and device applications and covers every scale from the level of the atoms to that of final devices. This growing field includes a vast number of magnetic, dielectric, semiconducting, conducting and superconducting ceramics used in domains as diverse as transport, communication, and health. Important electroceramic materials have been discovered in the last decade, and are being developed for new applications.

Electroceramic research faces two important challenges :

- Material integration issues play an increasingly important role driven by the need to integrate electroceramic functions onto conventional semiconductor chips as well as by the evolution of multifunctional components and systems.
- Electroceramic materials are following the footsteps of conventional semiconductors with respect to further miniaturization, and are experiencing the same evolution from microtechnology towards nanotechnology. Nanometer-size effects and issues in nanotechnology become therefore increasingly important.

Recent research and basic trends in electroceramics will be discussed in the context of these two challenges.

KLTN - A New Crystal for Photorefractive and Electrooptic Applications at the Paraelectric Phase

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Potassium Lithium Tantalate Niobate (KLTN), is a new electrooptic crystal designed to operate at the paraelectric phase where the electrooptic effect is quadratic. KLTN possesses several unique properties that open several avenues in both the basic research and applied research arenas:

1. **Electroholography:** It was shown that at the paraelectric phase photorefractive (PR) space-charge gratings are transformed into birefringence gratings only when an external electric field is applied. This effect, known as the voltage controlled PR effect, opens the way for a generic beam stirring method coined **electroholography** (EH). KLTN doped with Cu and V was tailored to be the basic building block in EH devices for photonic routing and switching. Moreover, The paraelectric phase was found to be the optimal configuration for electric field multiplexing of volume holograms, which can be used for active filtering of WDM signals.
2. **The dielectric photorefractive effect:** A photorefractive mechanism originating from the formation of a spatial modulation of the (dc) dielectric constant was observed in KLTN slightly above the phase transition. This mechanism was attributed to the formation of ferroelectric clusters in the vicinity of the transition. This mechanism was used for developing a new fixing process for PR holograms, which requires a temperature change of only 4K.
3. **Paraelectric photorefractive spatial solitons:** Spatial solitons were demonstrated in photorefractive KLTN at the paraelectric phase. Such solitons can be used as the basis for the construction of optical elements with electrically controlled focal length. In particular, very close to the phase transition, spontaneous spatial solitons were observed due to the field-induced formation of minute ferroelectric clusters.

In summary, the understanding of the relation between the structure and composition of KLTN and its physical properties led to the synthesis of specific crystals tailored to meet specific technological requirements.

Gas Sensing in Thin Films of TiO₂ at Low Temperatures (100-325°C)

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Thin films of TiO₂ are prospective materials for gas sensors due to the dependence of their electrical conductivity on gas adsorption. In this work some low temperature gas-sensing properties of reduced TiO₂ thin films are presented. The TiO₂ films (~100-200 nm) were deposited on SiO₂/Si substrates by reactive ion sputtering. The films studied were reduced by post vacuum annealing at 400°C. These films have a microstructure consisting largely of a rutile phase with an average grain size of ~20-30 nm in an amorphous matrix.

The electrical conductivity and gas-sensing properties of the reduced TiO₂ films were studied at temperatures between 100 and 325°C during exposure to pulses of air (i.e. oxygen) pressure or during exposure to pulses of CO in air. The activation energy of conductivity strongly depended on the ambient atmosphere. At ~200°C the sensitivity became significant and thereafter increased with temperature. The changes in conductivity were reversible and reproducible.

The kinetics of the conductivity response, $\Delta\sigma(t)$, to a pulse of air was found to follow a logarithmic law, $\Delta\sigma(t) \propto -\ln(1+t/t_0)$, during the first stage (~5 minutes) of exposure, and a modified parabolic law, $\Delta\sigma(t) \propto -[t + \alpha(1 - \exp(-\beta t))]^{1/2}$, afterwards (α , β and t_0 are constants). This suggests that the conductivity is dominated by surface processes involving charge transfer from the TiO₂ film to chemisorbed oxygen during the first stage of exposure to air. Following this stage, the response is dominated by bulk processes wherein oxygen is incorporated into the reduced TiO₂ and the carrier concentration is diminished.

GROWTH AND CHARACTERIZATION OF BARIUM TITANATE BASED MATERIALS FOR HIGH STRAIN APPLICATIONS

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Ferroelectric single crystals combine high frequency response and high strain actuation. Current materials for high strain actuation are largely based on lead containing relaxor perovskites. Materials such as $\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ and $\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3$ in solid solution with PbTiO_3 show high strain levels but are not easily fabricated due to the high vapour pressure of lead at elevated temperatures.

Investigation of Barium Titanate based perovskite systems (BT) as candidates for alternative, lead-free materials, will be presented. These systems show promise for high strain electromechanical actuation and ease of single crystal growth. Results from electromechanical testing of both polycrystalline and single crystal BT will be shown. Piezoelectric coefficient and strain levels of polycrystalline BT were found to be comparable to those of PZT. Measurements of single crystal BT are presently in progress. The effects of single crystal growth parameters and composition on structure and properties will be discussed.

DEVELOPMENT OF PIEZOELECTRIC THIN FILM DEPOSITION TECHNIQUES FOR MEMS APPLICATIONS

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MEMS are originated from fabrication of sensors and actuators by IC fabrication technique, where the sensing and actuation is mostly conducted by electrostatic principle. Electrostatic actuation has some disadvantage such as large energy dissipation, difficulty of control, large surface area and necessity of high accuracy of micro machining. Piezoelectric principle of actuation and sensing is also candidate for MEMS to overcome these disadvantages of electrostatic MEMS.

Smart MEMS is now a subject of interest in the field of micro-actuators, micro pumps, micro optical devices etc. It can be fabricated by deposition of PZT thin films and micro machining. The PZT is mostly prepared by sol-gel and sputtering process. However PZT thin film of more than 1 micron is difficult to obtain due to the fact that the sputtering process has a low deposition rate and the sol-gel process accumulate thermal stress.

To overcome the difficulties of the common process we try to develop new techniques which has been introduced recently and can be used to deposit PZT layers between 1-10 micron thickness. Two techniques will be described: a) Cathodic Electro-synthesis, b) Jet printing.

The cathodic Electro synthesis technique was developed by Dr. I. Zhitomirsky and was adopted for thin film coating on Silicon wafer. The deposition rate and layer thickness could be controlled on selective area, which is of a great advantage over the common coating technique.

The jet printing is a new technique introduced recently for PZT layer deposition. Although first material parameters of PZT deposited layers have been investigated and reported, actual suitability of the deposition method for MEMS applications as micro gyro still has to be proofed. To do so, we develop the Jet Printing technique used to fabricate actuator elements suitable for actuation purposes in gyro. The technique principle was demonstrated using Alumina particles.

BASE METAL ELECTRODES FOR MULTILAYER CAPACITORS

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Recently, multilayer ceramic capacitors based on BaTiO_3 dielectrics have been processed at production scale with nickel electrodes. The properties in terms of dielectric constant, loss, dielectric breakdown strength and degradation are comparable to the more costly precious metal devices. In order to sinter the dielectric material without oxidizing the electrodes, the sintering is done in reducing atmosphere. The dielectric material is modified by various dopants, in order to maintain its electronic properties. However it is always plausible to be able to do the heat treatments (binder burn out, calcination, sintering, annealing) at as high oxygen partial pressure as possible.

In this presentation we will show how to modify the electrodes to substantially suppress the oxidation rate, with a relatively simple process. The underlying thermodynamics will be described. Experimental results of oxidation of unmodified and modified metals will be shown. We will also present a simple multi layer device with modified base metal electrodes.

**GRAIN REFINEMENT OF MAGNESIUM ALLOYS AZ31, ZK60
TOWARDS SUPERPLASTICITY STUDIES.**

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Super-plastic forming of wrought Magnesium alloys enables designers to produce complex automotive or aerospace parts, using low power press and extending dies life.

The super-plastic forming, takes place at elevated temperature ($>0.5T_m$) and low strain rate (in the order of 10^{-4} sec^{-1}). The requirement for fine grain microstructure (usually in the order of less than $10\mu\text{m}$) is essential for the occurrence of super-plasticity phenomena. Both high temperature DRX (Dynamic recrystallization) and strain annealing, namely, room temperature pre-deformation followed by recovery and recrystallization processes can be used for grain refinement of wrought magnesium alloys.

The restricted slip deformation mode (limited slip systems) of the HCP crystal structure of the magnesium at room temperature, imposes constraints in applying of cold work deformation. Low level of pre-deformation causes anisotropic strain distribution which results in large differences in the grain size at the final stage of the recrystallization process. Thus, preventing such problems by selecting appropriate conditions or special alloys will be important in characterizing the plastic response of the magnesium alloys in the super-plastic deformation state.

Preliminary studies mainly by metallographic observations and image analysis indicate that grain refinement in AZ31 is sensitive to the strain level, whereas the ZK60 is less sensitive due to the presents of zirconium.

The current study investigates the super-plastic behavior in extruded ZK60 and AZ31B which were grain refined by strain annealing process. The mechanical tests were conducted at temperature range of 200-300 °C in two strain rates : 10^{-4} and 10^{-2} sec^{-1} . Additional fractographic and TEM observations were performed in order to classify the fracture modes and deformation mechanisms in the super-plastic stage.

**MECHANOELECTROCHEMICAL BEHAVIOR
AND CREEP CORROSION OF MAGNESIUM ALLOYS**

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Attractive mechanical properties of such light materials as magnesium alloys cause their broad application in the transportation industry. Environmentally assisted delayed fracture significantly decreases resistance to stress corrosion and durability of high-strength alloys. Today stress corrosion is becoming a major problem in Mg alloys applications.

Various types of die-cast test specimens were used in this study of standard mechanical properties, mechano-electrochemical behavior (pH=10.5) and creep corrosion resistance (pH=9.3) of Mg alloys. Specimens of Mg-alloys (AM20, AM50 and AZ91 type) were cast using a cold-chamber machine. The method of a slow strain rate tensile test was developed for mechano-electrochemical studies, and an electrochemical cell was built in the tensile system.

A comparison shows a distinct difference in the mechanochemical effect magnitude at maximal and minimal Al contents, namely, the least effect is observed in AM20 alloy, and the greatest one - in AZ91 alloy. The evaluation of the mechanochemical behavior in alloys of various composition with the account for the deformation level makes it possible to optimize the choice and design of alloys with an elevated resistance to stress corrosion.

DEVELOPMENT OF HIGH STRENGTH MAGNESIUM ALLOYS

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Magnesium alloys have attractive increasing interest as a structural materials for automotive and aerospace applications because of the smallest specific weight. Two key factors limiting their wide application are relatively low mechanical properties and poor corrosion resistance. Hereafter we present the results of the study aimed at the development of commercial magnesium alloy having high mechanical properties at both room and elevated temperature. The purpose was realized on the basis of quasicrystalline precipitation within magnesium -based solid solution matrix. Quaternary Mg-Zn-Y-Zr system was studied. The alloys (5.1 – 7.1 wt.% Zn, 0.58-1.48 wt.% Y, 0.4 - 0.9 wt.% Zr, balance Mg) were prepared in conventional melting furnace with protective gas atmosphere (CO₂, air, SF₆), cast in massive metal mould and air cooled. Metallographic study revealed presence of quasicrystalline phase Mg₃₀Zn₆₀Y₁₀ within grain boundary eutectic in as-solidified structure. DTA showed the quasicrystalline phases primarily formed from by binary eutectic reaction below 550°C. The quasiperiodic phase was thermodynamically stable and its microhardness was several times higher than that of the alloy Mg-based solid solution matrix (700 and 50 HV, respectively).

It was found that the regime consisting of solution heat treatment at 500°C followed by water quenching and aging at 250°C produced the optimum structure of the tested alloys: Mg-Zn solid solution matrix with approx. 2 at.% of homogeneously distributed Zn strengthened by fine (about 100 nm) intermetallic precipitates. Room temperature UTS and YS of the heat treated alloy were 235 and 135 MPa, respectively, with elongation 8%.

These primary results are now followed by the study in two main directions: (1) application of plastic deformation in order to achieve the maximum mechanical strength of Mg-Zn-RE alloys; (2) fundamental study of strengthening mechanism of these alloys.

Mechanical Properties and Microstructure of Gas Tungsten Arc Welded 4 mm Thick Magnesium AZ91D Plates

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Gas Tungsten-Arc (GTA) welds were performed on 4 mm thick plates machined from as cast magnesium AZ91D ingots. The microstructure was determined using optical and scanning electron microscopy. Mechanical properties were determined by standard tension tests on small-scale samples. It was found that Mg AZ91D cast alloy could be GTA welded with negligible amount of defects. However very large heat affected zone (HAZ), in the order of a cm is created. The rapid melting and subsequently resolidification during the welding process induces microstructural refinement, which increase both the strength and ductility of the alloy. The HAZ zone consists of two regions: (i) partially melted zone (PMZ) near the fusion line, and (ii) a wide region that was heat affected without melting. In both regions the interdendritic β phase is coarser than the one found in the base metal. However, in the PMZ an almost continuous net work of β phase in grain boundaries is formed. This in turn decreases markedly the mechanical properties of the HAZ to levels well below of the mechanical properties of the as cast alloy and therefore limits the mechanical properties of welds.

MOLTEN SALT ELECTROLYTES FOR HIGH POWER THERMAL BATTERIES

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There are two main types of batteries which use molten salt electrolytes: thermal batteries and high temperature rechargeable batteries. In both cases, the advantages of molten salt electrolytes are their high conductivity that leads to very high power density (thermal batteries) or to high energy density (rechargeable batteries).

In thermal batteries, the LiCl-KCl eutectic has been used as the standard electrolyte. Typically, the end of life of these batteries occurs because of the freezing of the electrolyte at 352°C. For pulse-battery applications requiring very high current densities ($>1000 \text{ mA/cm}^2$), the factor limiting battery performance is the formation of a large Li^+ gradient at the anode as a result of severe concentration polarization. The use of an all-Li cation electrolyte should prevent this problem.

A study of all-Li electrolyte was undertaken at Rafael to evaluate its performance under the appropriate discharge conditions, for comparison to the standard LiCl-KCl eutectic. The effect of MgO-binder content of electrolyte-binder mixes, temperature discharge, and current density upon the electrochemical performance was examined using Li(M)/FeS₂ single cells. Validation tests were also achieved with appropriate full-sized batteries.

Electrochemical Deposition of Organic Semiconductors for Solar Cells

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The use of organic semiconductors (OSC) in photovoltaic cells has shown low efficiency of solar energy^{1,2}. Light absorption in such cells results in exciton creation that reacts with a hole selective material that dissociates (quenches) the exciton and enables usable charge separation¹. To complete the circuit, the charges have to be collected across the OSC and quencher layers. This process is dominated by the high electrical resistivity of the OSC, which limits the thickness of the OSC layer¹. As a result of the thickness limitation, the optical density and thus the conversion efficiency of the cells reported so far are low^{3,4}.

One of the ways to increase the optical density without thickening of the OSC layer is to increase the surface area of the cell using porous substrates³. This approach necessitates the ability to deposit the OSC into porous material. We developed a method that enables fabrication of OSC layers in porous substrates. The method is based on electrochemical deposition of OSC ions that are electrochemically produced during the deposition process.

The new electrochemical deposition method was developed for the PPEI (Perylene Bis(phenethylimide)) which is one of the promising OSC for solar cells. The deposition and its use for high surface area solar cells will be presented.

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DIELECTRIC PROPERTIES OF TUNGSTEN-SILICA NANOCOMPOSITES

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Composite materials are of great interest since changing composition, microstructure and size of the constituents can control their properties. Some of their properties are changed dramatically near the percolation threshold and can be utilized at that point for various applications. The behavior in the vicinity of the percolation threshold and the percolation threshold itself become difficult to predict when the constituent phases have different sizes or shapes. The goal of our study is to understand the influence of composition and microstructure on the electrical properties of W-SiO₂ nano-composites.

The composite specimens were produced by first ball milling of pure tungsten (1 μ m size) and silica (20nm size) powders at different compositions (10÷97%(wt) W), then pressing the resultant mixtures to form green compacts, and finally sintering at various temperatures. XRD as well as TEM combined with EDS were used to characterize the microstructure and composition of the specimens. Electrical capacitance and conductance measurements under ac and dc conditions provided data on the dielectric constant and the conductivity as a function of frequency and composition.

The dielectric constant of the composite green compacts is higher compared to green compacts made of pure nano-sized silica. The dielectric constant decreases with increasing frequency in the range of 1KHz÷1MHz. The effect of composition is dominant at about 20%(vol.) W, where the dielectric constant jumps to a higher value.

The effect of microstructure and composition on the electrical properties is discussed with an emphasis on the contribution of density and type of interfaces between particles to the specimen behavior near the percolation threshold.

THERMODYNAMIC CHARACTERIZATION AND
STATISTICAL THERMODYNAMICS OF THE
TiCrMn-H₂(D₂) SYSTEM

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Pressure composition (p-c) isotherms of the TiCrMn-H₂(D₂) system were measured over a wide temperature and pressure range (up to 1,000 atm H₂/D₂) which included the super-critical range (above T_c). The macroscopic thermodynamic parameters of the system and their isotope effects were evaluated. Also, hysteresis phenomenon between absorption and desorption isotherms were obtained as a function of temperature, for the two hydrogen isotopes. The experimental p-c isotherms were compared to model calculated ones, derived by simplified statistical thermodynamic treatments. From this comparison microscopic energy-related parameters were evaluated. These parameters included the average hydrogen-lattice interaction, the pairwise nearest neighbor hydrogen-hydrogen interaction and the average vibrational frequency of H(D) at the interstitial sites.

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- Practical hints
- Further information and assistance

NEW FAMILY OF LAMINAR - BAND
CERAMIC METAL COMPOSITES

A. Gorodnev

Ceramight Composites Ltd., P.O.B. 380, Yavneh 81103, Israel

Ceramight Composites Ltd. has developed and is producing a new family of laminar-band ceramic metal composite materials, called COMPOLENT. An original technology was created for manufacturing the COMPOLENT composites on the basis of thick films, made of refractory oxides, nitrides or carbides and metals. The final structure of these composites comprises continuous homogeneously distributed and randomly oriented band ceramic and metal inclusions with a thickness from 10 to 100 microns.

By now, COMPOLENT composites of various compositions, have won the close attention of academia and industry, due to their unique complex of physical and mechanical properties at broad temperature interval. For example, the layers of ceramics such as fully stabilized zirconia (FSZ) provide the composite with low thermal conductivity and high mechanical strength at high temperatures, whereas the layers of refractory metals, such as W or Mo impart high thermal shock resistance and high fracture toughness. The overall results for FSZ/Mo at 1500°C, is a synergistic bending strength of more than 500 Mpa and fracture toughness of $17 \text{ Mpa}\cdot\text{m}^{1/2}$. COMPOLENT composites show a good performance at temperatures up to 3000°C and more. The most prospective applications for this kind of COMPOLENT are parts that work under harsh conditions of severe thermal shocks, together with mechanical stresses at high temperature level.

The microstructure, physical and mechanical properties of COMPOLENT composites at temperatures up to 2000°C will be reported and discussed.

Non Destructive Strength Evaluation of Steel Wires and Cables

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<http://www.case-technologies.com>

Tensile strength is the most important property of steel wires (or wire ropes) used in applications such as elevators, cranes, ski lifts, rescue hoists, radio towers and many other engineering applications. The company's products measure the tensile strength of steel wires and cables non-destructively and on-line in real-time.

CaSE Technologies Ltd. recently developed an on-line, NDT steel cables monitoring system, based on a unique concept patented by the company. The system consists of a series of electromagnetic sensors mounted near the cable, and a central data acquisition and analysis system that can be located in a control room or any other suitable place. One central system will be able to monitor a few sensing stations each one located on a separate cable. The sensors monitor continuously and without contact the mechanical properties of the cable. The results are displayed on the control screen. The system alerts of any deviations from setup values, enabling the operator to take corrective steps.

Specially designed sensors and a proprietary computer algorithm were developed by CaSE. These are based on recent breakthroughs in the understanding of the correlation between the mechanical and magnetic properties of steel. This knowledge enabled CaSE to develop a new measuring method, which will furnish information regarding the strength of a cable as well as information about broken wires and loss of metallic cross section and other flaws.

RECENT DEVELOPMENTS WITH SAPPHIRE -
A MISSILE'S BEST FRIEND

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Sapphire is the first man-made crystal and in this century, sapphire crystals have been extensively used as fabricated hard articles, gems, optical windows for harsh environments, ruby and Ti-doped laser rods and as substrate material for the deposition of silicon (SOS devices), superconductors and GaN for blue LEDs and lasers. The superb strength, resistance to thermal shock and rain erosion, hardness and chemical inertness of sapphire have made it the material of choice for the external windows of modern infra-red air to air missiles. The unique combination of excellent thermo-mechanical properties of sapphire, far surpasses the slight drawbacks of optical non isotropy and relatively short cut-off wavelength of the optical transparency. In this presentation, the near-net-shape growth technology, developed for the first generation missiles, will be described. The solution to some of the problems that arose with the development of the larger domes for the second generation missiles will also be discussed. Sapphire is now starring in several missile projects worldwide. This has led to a renewed interest, as well as to systematic studies of the basic material properties. Extensive efforts are also invested in the strengthening of sapphire for even faster missiles. Some of these studies and efforts will be presented, as well as some recent results from experiments in diffusion bonding of sapphire components.

MILLENNIUM MATERIALS TECHNOLOGIES FUND L.P. - (MMT)

Nir Belzer- Chief Executive Officer

Millennium Materials Fund Management Co. Ltd
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MMT is a specialized fund, created to invest in the development and commercialization of novel advanced materials and industrial processes.

This area of investment has been selected for 4 reasons:

1. Israel is a leading country in the field of developing new materials and industrial process due to several reasons :
 - A. High quality of technological education.
 - B. Intensive research & development in the military field focus a lot of attention to the field.
 - C. Technological Incubators supported by the state encourages a lot of research in the field.
 - D. Immigration from the former USSR brought population with a lot of experience in the materials field.
2. The field of specialty materials is so specialized that there are very few other funds that invest in the field. We believe that together with our technical expertise we have an opportunity to look at many deals almost on an exclusive basis and invest on attractive valuations. It is also an area where potential returns are exceptionally high.
3. The field of specialty materials and the very early stage investments required is especially suitable for our high value added, management intensive approach, that has worked so well until now.
4. Development of a new material which has a variety of potential applications.

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3. Or-Maor group, a group who specializes in high technology new materials & new industrial processes. Or-Maor has already established successfully several "Start-Up" companies and is represented by Oren Gafri who is an expert in the field of materials & industrial processes.

Three major companies invested in the Millennium Fund:

Clal Industries – one of the largest and leading industrial holdings company in Israel,

Siemens AG & Henkel KgaA, two leading German multi national industrial companies.

**IMPROVING TRIBOLOGICAL PERFORMANCE OF MECHANICAL
COMPONENTS BY LASER SURFACE TEXTURING**

I. Etsion

Technion, Haifa, Israel

G. Halperin and G. Ryk

Surface Technologies Ltd., Nesher, Israel

Significant improvement in load capacity, wear resistance, friction coefficient etc. of mechanical components can be obtained by forming regular micro-surface structure in the form of micro-pores on their surfaces. A feasibility study was performed experimentally using Laser Surface Texturing (LST) technique to produce the micro-pores that can serve either as micro-bearings in cases of full lubrication or as micro-reservoirs for lubricant in cases of starved lubrication conditions. In all cases friction is reduced, wear resistance is increased and life is prolonged.

**GRADED COATINGS ON HIGH SPEED STEEL
PRODUCED BY LASER ALLOYING WITH TUNGSTEN CARBIDE**

M. Riabkina-Fishman and E. Rabkin, Technion, Israel; N. Frage and M.P. Dariel, Ben-Gurion University of the Negev, Israel; A. Weisheit, R. Galun, and B.L. Mordike, Institute für Werkstoffkunde und Werkstofftechnik, Clausthal, Germany

The aim of the research is to produce graded layers on M2 high-speed steel by using laser alloying technique with direct injection of WC powder. The powder injected in the melt pool is diluted with the substrate material to a degree determined by the depth of substrate melting and the rate of powder injection. The produced alloyed layer serves as substrate when laser alloying is repeated, therefore, multiple laser treatment with the powder injection results in a multi-layer coatings with tungsten content increasing from layer to layer. Variable experimental conditions were beam power (2 to 7 kW), beam travel velocity (0.25 to 1 m/min) and powder injection rate (3 to 7 g/min). Single, double and triple alloyed coatings have been produced. By changing laser treatment conditions and powder injection rate we have been able to obtain single alloyed layers having tungsten content of 12 to 60 wt.% and to achieve 70 wt.% W in the upper layer of a triple-layer coating. Laser treated M2 steel with no WC injected powder has 770 HV surface hardness. The hardness increases to above 1000 HV at about 50 wt.% W and reaches 1600 HV in the upper layer of triple-layer coating containing 70 wt.% W.

Laser Nitriding of Iron and Stainless Steel

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The irradiation of metals with short pulses (55 ns) of an excimer laser in nitrogen containing atmosphere leads to important changes in their surface if the laser irradiation melts the surface and forms a plasma. The depth of these modifications is ranging up to several micrometers. For iron a decisive take-up of nitrogen into the surface (nitridation) is found. Besides that, additional superimposed effects like fall-out deposition and crater formation can be observed [1,2]. All these superimposed effects can only be resolved by a combination of analyzing methods. The results of resonant nuclear reaction analysis (RNRA), Mössbauer spectroscopy (CEMS and CXMS), Rutherford backscattering spectrometry (RBS), transmission electron microscopy (TEM), and surface profilometry allow an analytical description of the nitrogen depth profiles with the number of laser pulses [2-5]. These nitrogen depth and lateral profiles and the additional effects (fall-out, melt expulsion, roughness) can be well described with simple models for heat transfer, plasma and fluid dynamics, dissolution and diffusion of nitrogen. The influence of the energy density will be presented as well as that of the lateral laser intensity distribution [5,6]. Finally, also the successful treatment of stainless steel will be shown, along with the improvement of the technical properties of their surfaces, i.e. corrosion resistance, wear resistance, and an increase in the surface hardness (microhardness). Here we concentrate on the microscopic changes observed in the TEM and HRSEM [7].

* Supported by the Deutsche Forschungsgemeinschaft (DFG Scha 632/3).

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Optimum Laser Surface Treatment of Fatigue Damaged Ti-6Al-4V Alloy

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A Laser Surface Treatment (LST) which enhances fatigue resistance of Titanium 6Al-4V alloy has been studied via 1.8 kW CW CO₂ Laser. The LST was applied to 3PB specimens loaded cyclically ($R=0.1$, $0.7\sigma_y$ on the surface), at different stages of fatigue life. Temperature surface fields were calculated by infrared camera. Two basic mechanisms were identified: one is related to Healing Mechanism (HM), which “erases” prior fatigue damage, and the other is connected to Microstructure Mechanism (MM). Healing was found to be effective for surface temperatures above 400°C. Microstructure changes had a general negative effect except for temperatures lower than 600°C and specific laser conditions. The combination of both effects lead to optimal LST of 2sec and 0.85kW/cm², for which a 50% increase in fatigue life was due to MM, in addition to a full healing. A positive correlation between hardness and fatigue life was also found.

NOVEL LASER -BASED PROCESSING OF SENSOR
AND ELECTRONIC DEVICES AND SUBSYSTEMS

D.B. Chrisey, A. Pique, J. Fitz-Gerald, R.C.Y. Auyeung,
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1-Israel Institute for Biological Research, Ness-Ziona, Israel
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We have combined some of the major positive advantages of laser induced forward transfer (LIFT) and matrix assisted pulsed laser evaporation (MAPLE), to produce a novel excimer laser driven direct writing technique which has demonstrated the deposition in air and at room temperature and with sub-10 μm resolution of active and passive prototype sensor and electronic circuit elements on planer and nonplaner substrates. We have termed this technique MAPLE DW (matrix assisted pulsed laser evaporation direct write). This presentation will outline the simplistic approach used to carry out MAPLE DW, give experimental conditions, and physical characterization results for the deposition of NiCr thin film resistors, Au conducting lines, yttrium-iron-garnet ferrite patches for inductor's, and multilayer depositions of Au conductors and BaTiO₃ dielectrics to produce prototype capacitors and RC circuits. In general, the electrical properties of the materials deposited (conductivity, dielectric constant, and loss tangent) are comparable or superior to those produced by other commonly used industrial processes such as screen printing. The mechanism of the MAPLE DW process, especially the novel aspects making it a powerful approach for direct writing all classes of materials (metals, oxide ceramics, polymers and composites), will also be outlined. An overview of other important aspects contained in the MAPLE DW approach, e.g., the ability to do *in situ* laser micromachining, surface pre-treatment, and annealing, will be given. Lastly, the future directions of this project and an appraisal of other chemical and physical direct write approaches outside of the scope of our efforts will be presented.

Monday

Posters

A STUDY OF PROPERTIES OF OXIDES CERAMIC COATINGS BY
MICROARC OXIDATION

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A compact oxide ceramics coating as thermal barrier coatings was built on aluminum metal by microarc oxidation in electrolysis solution. The microstructure of surface and the section of oxides ceramic coatings were observed through metalloscope and SEM. The coating was tested by thermal shock and microhardness tester. The compositions of ceramic coating were qualitatively and quantitatively analyzed by EDXRF.

The results showed that oxide ceramics coating consisted of two layers resulted from incomplete microarc oxidation. The inner layer was thin and homogeneous. The clear cleavage plane was observed in the ceramic grains of inner layer after magnified up to 500 times. The mark of remains of possible gas escaping was observed by SEM. The joints between inner layer and outer layer, inner layer and metal were very close. No obvious discontinuity was observed. The main composition of oxides ceramic coating was $\text{SiO}_2\text{-xAl}_2\text{O}_3$. In the inner layer, $\alpha\text{-Al}_2\text{O}_3$, $\gamma\text{-Al}_2\text{O}_3$ and unchanged aluminum grains were observed besides $\text{SiO}_2\text{-xAl}_2\text{O}_3$. Dramatic change was observed on the structure of ceramic coatings after thermal shock from 550°C .

EXPERIMENTAL RESULTS CONCERNING HARDENING BY
NITROGEN DIFFUSION IN TITANIUM ALLOYS

Cristian Badina, and Leontin Druga
INTEC Bucharest Romania

The works consist of plasma and gas nitriding experiments on Titanium samples and structural study of the diffusion layers. The main reasons for developing the nitrogen diffusion layers on titanium is the increasing of wear resistance, hardening of the outer layer and the obtaining of high corrosion resistance layer. In this purpose were performed plasma nitriding experiments at 620⁰C / 7 hours with different ammonia contents and different partial pressure, and gaseous nitriding experiments at 700; 750; 800; 850; 900⁰C for different times in ammonia base atmospheres. The obtained layers contain Ti₂N and TiN₂ in the white layer, Ti₂N, TiN₂ and alpha enriched with nitrogen in the transition layer, and alpha enriched with nitrogen in the diffusion layer. If we use titanium alloys we can obtain alloying elements nitride that can increase the hardness of the outer layer.

CHEMISTRY OF A NOVEL DAY/NIGHT SOLAR CELL GaAs/Si with *In-Situ* METAL HYDRIDE STORAGE

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The variations of Solar Energy with weather and daylight conditions are an obstacle to its widespread utilization as a replacement energy source. We report an unusually high efficiency solar cell with a stable output independent of variations in illumination. The cell is a single physical/chemical device generating load current without any external switching, and continues our studies on efficient photoelectrochemical energy conversion, *in-situ* and *ex-situ* energy storage, and Multiple Bandgap Photoelectrochemistry.

We explore theoretical and experimental constraints of both one photon, and multiple photon, per electron Multiple Bandgap Photoelectrochemistry (MBPEC) processes. These processes improve spectral overlap and absorption, enhancing the energetics of solar to electrical energy conversion.

The AlGaAs/Si *in-situ* Metal Hydride Cell, an *In-situ* Storage Multiple Bandgap Bipolar Solar Cell, is insensitive to variations in illumination and converts solar energy at an overall 18% efficiency (including both conversion & storage of solar energy). This single cell, unlike conventional photovoltaics, provides a nearly constant energetic output in illuminated or dark conditions. The cell combines bipolar AlGaAs ($E_g=1.6\text{eV}$) and Si ($E_g=1.1\text{eV}$) and AB₅ metal hydride/NiOOH storage, and generates a light variation insensitive potential of 1.2-1.3V.

EFFECT OF OXYGEN ON WETTING OF ALUMINA BY LIQUID ALUMINIUM

George Levi and Wayne D. Kaplan
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The wetting behaviour of liquid alloys on solid ceramic substrates is a major limiting factor in liquid state processing of metal-ceramic composites and joints. Since wetting is extremely sensitive to the working gas and the presence of alloying elements and impurities, it is important to study the wetting behaviour as a function of these parameters.

Wetting of Al_2O_3 by liquid Al is a non-equilibrium phenomenon, characterised by the continuous oxidation of Al. Based on thermodynamic calculations, it was shown that the decrease of the liquid Al- Al_2O_3 interface energy causes the improvement in wetting and adhesion in this system. Oxidation of Al at the solid-liquid interface is assumed to be responsible for this change in energy, although until now there was no microstructural evidence to reinforce this statement.

Wetting experiments of Al on sapphire were conducted in an ultra high vacuum wetting furnace in the 660-1300°C temperature range, under varying $P(\text{O}_2)$: 10^{-7} , 10^{-8} , 10^{-9} and less than 10^{-10} torr. The interface microstructure of specimens from the wetting experiments was investigated in a Zeiss optical light microscope, a LEO 982 high resolution scanning electron microscope (HRSEM) and a JEOL 2000FX transmission electron microscope (TEM). The specimens were also characterised by electron back scattered diffraction in HRSEM, and energy dispersion spectroscopy in TEM.

The microstructural investigations have provided evidence reinforcing the statement concerning the role of O_2 in the improvement of the wetting and adhesion of Al_2O_3 by Al. The results and the role of oxygen in the wetting of Al_2O_3 by Al will be discussed.

CAPILLARITY PHENOMENA IN THE TiC / (Cu, Al) SYSTEM

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Negev.

The wetting behavior in the TiC/(Cu, Al) system has been studied by the sessile drop method over the entire Cu-Al alloy concentration range. The experimental results show that the presence of oxygen contamination on the TiC surface plays a major role in the capillarity phenomena. According to the experimental results and the thermodynamic predictions, the wetting kinetics in the presence of pure Cu are controlled by partial dissolution of the titanium carbide phase. The presence of oxygen contamination on the ceramic surface strongly inhibits this reaction. The wetting behavior in the TiC-(Cu, Al) system is controlled by reduction of the oxidized layer on the ceramic substrate surface. In-situ deoxidization of the Cu-Al liquid drop and of the surface of the TiC substrate by evaporation of aluminum oxide may also contribute to the wetting process.

Processing of Nano-Ni Reinforced Al₂O₃

Michael Lieberthal and Wayne D. Kaplan

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Recently, nanocomposites have drawn a great deal of attention. These materials, which are characterized by sub-micron particles dispersed in a micron size matrix, have promising mechanical properties, and indeed have shown improved fracture strengths compared with monolithic materials.

The majority of the work so far has focused on a ceramic matrix reinforced with ceramic inclusions, such as Al₂O₃ reinforced with nano-SiC. Difficulties in processing have limited research on ceramic matrices reinforced with *metallic* nano-particles.

A novel method has been developed whereby nano Ni particles are introduced into an alumina preform. This method is simple, fast and inexpensive and can be used to manufacture parts having intricate geometric shapes. The metal is formed *in-situ* during sintering.

The influence of processing parameters, such as sintering time and temperature, on the alumina microstructure has been evaluated using high resolution scanning electron microscopy and x-ray diffraction. The presence of the metal particles, as well as their structure and size, were investigated by transmission electron microscopy. Fracture strength measurements using 3-point bending have shown significant improvements compared with monolithic alumina.

**ALLOYED SILUMINS, CASTED AND HEAT TREATED IN
MAGNETIC FIELD**

Dr.ing. IOAN GIACOMELLI, Dr.ing. RODICA FLOREA
University "TRANSILVANIA" of Braşov, Romania

This paper presents several aspects regarding the influence of Al - Si alloys casting and solidification in magnetic field on different structural changes. The alloys obtained in this way were then hardened and aged, in order to be compared with witness specimens. Certain structural and mechanical properties changes have been observed.

DIRECT EXPERIMENTAL SUPERVISION OF OUTSTRIPPING DIFFUSION ON TRIPLE JUNCTIONS IN Al

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There is common opinion among the researchers that the triple junctions of grain boundaries are ways of accelerated diffusion in metals. And it is considered, that the diffusion penetration of triple junctions is higher, than grain boundaries. However, the authentic experimental data confirming this speculative representation, till now were absent. There were only separate indirect experimental results, one of which possible explanations is accelerated diffusion on triple junctions of grains.

In the given work the technique of preparation of aluminum samples with large grains with so called bamboo structure and with triple junctions spread through the thickness of sample is mastered. The layer of zinc was applied on sample surface using chemistry method. Then samples were subjected diffusion annealing in vacuum at temperatures 250-300°C within 30-100 hours. The developed technique of mechanical removal of layers by thickness 5-10 microns has allowed us to observe individual triple junctions at different depth from the initial layer of zinc. With the help of the microanalysis the distribution of concentration of zinc in grain boundaries near to the triple junctions was determined. For an estimation of diffusion coefficient in triple junctions the models given in the literature were used.

In result the meanings of the relation of triple junction (D_{tj}) to the grain boundary (D_{gb}) diffusion coefficients were received, which make 1000 at 280°C.

FERROGRAPHY AS A TOOL IN TRIBOLOGICAL STUDY

Z. Horowitz and I. Ronn

**Predictive Maintenance of Dynamic Systems (P.M.D.S.)
Malat Bldg. Technion City 32000 Haifa Israel**

In the study of practical tribology, the failure of dynamic components attracts both theoretical and economical interest. Ferrography is a useful method for characterization of wear/ failure mechanisms. Some of the advantages of this method are discussed. Essentially, Ferrography involves microscopic examination of wear particles isolated from oil sample under defined conditions. The isolated-on -slide and fixed assembly of particles is called a "ferrogram". The wear particles are classed by their morphologies, their size and their chemical composition. This technique can distinguish between various modes of wear action. It can qualify and quantify the damage to an assembly as to the number of particles formed, and as to their size. In addition, it focuses the attention to the actual part being damaged. In the poster we present a case study of wear in an industrial assembly making use of the ability of this technique to predict imminent failure by characterization of the wear mechanism. Ferrography followed by application of SEM/EDAX on the "Ferrogram" can pinpoint the actual alloy involved, and can supply very useful information to the maintenance personnel.

MORPHOLOGICAL STRUCTURES OF AN EXTRUDED
AZ-81 MAGNESIUM ALLOY

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Some preliminary experiments were made on the hot forming of AZ-81 magnesium alloy known as not suitable for hot working. These experiments included extrusions through different dies and after several solution heat treatments. Metallographic technique and x-ray diffraction were used to study the morphology and microstructures of the extruded samples.

The as cast AZ-81 magnesium alloy, at room temperature, is closed to equilibrium and contains a mixture of two phases α -Mg (hexagonal) and β -Mg₁₇Al₁₂ (cubic). At high temperature all additives are dissolved in Mg and the AZ-81 becomes a single phase, namely solid solution in α structure.

During the extrusion process, the residue β phase forms elongated grains while the α phase exhibits almost equiaxed recrystallized microstructure.

The contrast in morphology between α and β phases after extrusion can be explained by the fact that the two phases undergo different processes during hot work. While the main process in α phase is a continuous recrystallization, the β phase is consumed in a decomposition process; thus its fragments are drifted by the plastic flow forming elongated particles.

PROPERTIES OF REVERSIBLE $A + B \leftrightarrow C$ REACTION
DIFFUSION PROCESS

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The investigation of the multi-component diffusion accomplished by quasi-chemical reactions between diffusion components is one of the main approaches for the analysis of complex diffusion profiles in solids. The quantitatively new property, which is incorporated in diffusion profiles by a reaction, is a reaction front. The reaction front is a thin zone in which the reaction between reactants is concentrated. In this work the properties of the reaction front formed in a reversible reaction-diffusion process $A + B \leftrightarrow C$, with initially separated reactants is studied. The case of the mobile C component is considered. The dynamics of the front is described as a crossover between an “irreversible” regime at short times and a “reversible” regime at long times. It is shown that the reversible regime is characterized by scaling of the local rate of C production $R_{local} \sim t^{-1}$, and by scaling of the global rate of C production $R_{global} \sim t^{-1/2}$. It is established that through this crossover the macroscopic properties of the reaction front such as the global rate of the C production, the motion law of the reaction zone center, and the macroscopic distribution of the components outside the front reaction are conserved.

**EFFECT OF ELECTRIC FIELD ON PROTECTION OF Si WAFERS
FROM METALLIC CONTAMINATION DURING WAFER
PROCESSING TECHNOLOGY**

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Metallic contamination of Si wafers during wafer processing technology leads to decreasing the quality of Metal-Oxide-Semiconductor devices. Dry oxidation of Si wafers as well as annealing of oxidized Si wafers (Si/SiO₂ system) in an inert atmosphere at high temperatures are accompanied by significant contamination of alkali metals.

Therefore, protection of the Si/SiO₂ system from metallic contamination has become a serious consideration in advanced wafer processing and smaller design rules.

The metal impurities which were studied are Li, Na, and K. The electric field application gives a significant reduction of the Li and Na content compared to conventional wafer processing both for anneal and for oxidation processes. The electric field application tends to reduce K content during annealing and oxidation processes. Secondary Ion Mass Spectrometry and Capacitance-Voltage methods have been used to measure metallic contamination in oxide layers.

RF HEATING OF A CONDUCTOR FILM ON SILICON SUBSTRATE FOR THIN FILM FORMATION

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A new method of rapid thermal process (RTP), based on induction heating (IH) of a thin conducting film placed on a silicon substrate is proposed. AC magnetic field frequency in the radio-frequency range of 1-20 MHz is used in the method. The characteristic feature of the proposed method is that no additional susceptor is required in contrast to the traditional IH methods, despite the fact that the thickness of the conductor film/Si substrate structure is less than the skin-layer depth. Silicides, nitrides, borides, or conventional metallic films, etc., possessing low electrical resistivity can be the object to be treated by the novel method. A theoretical model based on numerical coupled solution of quasi-stationary Maxwell's equations and the non-stationary thermal equation, taking into account the dependence of the resistivity and the thermal capacity on temperature was developed. Using this model the calculations of the heating kinetics were carried out for the TiSi_2 film/silicon substrate structure on specimens in the form of thin strip. Experimental results confirming the calculations for TiSi_2 film formation and the feasibility of the proposed method are presented.

**THE EFFECT OF 1% PRIOR CREEP DEFORMATION ON THE
RESULTANT RESISTANCE TO FATIGUE CRACK GROWTH
(FCG) OF PRECIPITATION-STRENGTHENED
SUPERALLOY TYPE UDIMET-700**

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Modern mechanical design criteria assume that initial, undetected small cracks exist in every component of jet engines and of other advanced mechanical and aerospace systems. These cracks can grow during normal operation of these systems and the designers of such systems are required to show how planned inspections will lead to the detection of these growing cracks before they reach critical dimensions which will cause catastrophic failure of a component and of a whole system. These requirements led to extensive FCG studies of potential candidate materials for the aforementioned advanced systems. Exposure to high-temperature loading, such as encountered in service conditions, may result in microstructural changes that can drastically change the resistance of materials to FCG. In this study, we exposed a polycrystalline, precipitation-strengthened nickel-base superalloy type Udimet-700, to 1% high-temperature creep deformation which was obtained under different loading conditions: 1. Short-duration loading under a high tensile stress, 2. Long-duration loading under low level stress, and 3. Intermediate loading duration cyclic loading. The major microstructural changes during high-temperature creep of the alloy were rafting of the initial cuboidal precipitates and the formation of discontinuities along grain boundaries. The results of this study show that longer the high-temperature period of creep the greater was the damage of the alloy and the lower was its resultant resistance to FCG. These results also show that the amount of prior creep deformation in itself is not a measure of the damage caused to potential materials for elevated-temperatures service.

PLASTIC DEFORMATION IN IMPURE NANOCRYSTALLINE CERAMICS

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Application of advanced ceramics for high technologies steadily increasing due to the wide range of their physical, mechanical and thermal properties. Recent advances in the synthesis of the nanocrystalline ceramics enable to refine the ceramic microstructure for improved or unique properties in the nanostructured ceramics. Superplastic forming of such nanocrystalline ceramics thus become an ideal fabrication technique important due to their improved

Plastic deformation behavior of impure nanocrystalline ceramics (NCC) was modeled using the percolative composite model in conjunction with models for plastic deformation by grain boundary sliding. The 'glass transition temperature' concept was used to determine the threshold strain rate criterion below which the impure nanocrystalline ceramic would deform plastically. Threshold strain rate is stress independent. It increases with the temperature increase, and with the grain size decrease.

Using the dissolution-precipitation model, dependence of the strain rate on temperature, stress, and grain size in the nanometer regime for impure NCC's were calculated. As an example, the critical conditions for plasticity in impure yttria-tetragonal zirconia polycrystals (Y-TZP) were evaluated. At 600°C, strain rates as high as 10^{-4} s^{-1} were expected in 10 nm impure Y-TZP. Comparison of the published data extrapolated into the nanometer range, to the calculated threshold level showed that increase in the applied stress is associated with increase in the grain size and strain rate onsets for plastic deformation.

ALUMINA CERAMICS FOR BALLISTIC PROTECTION

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Different structural ceramics used for ballistic protection have been reviewed from mechanical, manufacturing, and cost standpoints. Dense alumina ceramics is an excellent ceramic armour material. It has high mechanical properties, excellent manufacturing ability, and comparatively low cost. High alumina ceramics (Al_2O_3 content is approximately 98%) producing by Ceramic Protection Corporation (Canada) is successfully used as an armour material both for personal and vehicle protection. The alumina armour products have various designs, e.g. flat tiles and curved plates (single, double, and triple curve) with different sizes and thickness. The alumina ceramics is manufactured by slip casting and pressing techniques in dependence on the required design and quantity. The main properties of this ceramics such as hardness, fracture toughness, flexural strength, elastic properties (Young's Modulus and others) effecting on the ballistic protection were examined and analyzed as a function of microstructure. Manufacturing process optimization is needed to ensure that the produced ceramics has the best combination of properties.

The Microstructure of Laser Treated α -Al₂O₃

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The microstructure of laser treated α -Al₂O₃ was investigated. α -Al₂O₃ (96% purity) was laser treated using a CW-CO₂ laser beam at laser powers ranging from 200W to 800W, and scan speeds from 0.33 – 4.0 cm/s. The microstructure formed under various atmospheres was studied by SEM and TEM. The distribution of elements and macro-segregation in the laser treated zones were investigated by EDS-SEM and EDS-TEM microanalysis.

The melted zone of Al₂O₃ has a microstructure typical of a material solidified under uni-axial heat flow; that is a layer of randomly oriented small grains, which gradually increase in size towards the surface, followed by elongated dendrites, and finally a zone of small alumina grains.

Orthoenstatite, spinel and mullite were bonded at alumina grain boundaries in samples which were laser treated in air.

Crystalline γ -Al₂O₃ was found after laser treatment under forming gas, in the form of spherical inclusions. EDS-TEM microanalysis of grain boundaries confirmed that in addition to amorphous CaO, SiO and MgO at grain boundaries and triple junctions, amorphous Si forms at grain boundaries.

NEW TYPE OF GLASS-CERAMIC ZIRCON BEADS

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Glass and ceramic beads are currently used for the fine milling and mixing of powders and liquids in a wide field of technologies. Ceramic beads have a higher milling efficiency, but they cause high wear of the equipment and higher costs. A new type of glass-ceramic zircon beads was developed providing improved wear properties of the ceramic beads due to a significant content of glassy phase.

Ceramic beads are currently produced at Rami by dropping the powder/polymer slurry, followed by drop coagulation by reaction with a salt solution. It was shown that additives to the zircon, which aid the creation of the liquid phase during sintering, sharply improve sintering of the beads, decrease their sintering temperature and increase the density and fineness of their structure. As a result, the bead wear resistance is sharply increased at optimal content of the glassy phase, based on this liquid.

The wear of the zircon glass-ceramic beads was studied in conditions of dry and wet erosion. The bead wear kinetics are characterized by the achievement of a constant wear rate, the level of which depends on the bead structure. A decrease in the zircon grain size, as well as in the bead porosity are the decisive factors increasing the bead wear resistance - from 2.4% mass loss in the attritor for ceramic zircon beads, to 0.5-0.6% for glass-ceramic ones (for 16 h testing). Together with this, losses of the attritor disk are also decreased - from 15 to 5%. A further decrease of the zircon grain size by additional milling of the raw materials led to very low bead losses in the attritor test - 0.15-0.20%.

The new type of zircon beads is being produced currently at Rami under the trade name **Rimax**, in bead sizes of 0.6 to 2.8 mm diameter.

SOME INTERESTING STRUCTURAL ASPECTS OF NANOCRYSTALLINE ZIRCONIA

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The nanocrystalline state of the materials, specifically of ceramics, has been realized as a novel state exhibiting many interesting features to be still better understood. In the family of advanced ceramics, zirconia is a technologically important candidate material due to its applications from structural components to oxygen sensors and fuel cells. Efforts were made to synthesize and study the zirconia nanoparticles in pure and doped forms. The effect of preparative and processing conditions such as the refluxing time of hydrolysis, and annealing temperature etc. on the phase transformation in zirconia is investigated. Hydrolyzing the aqueous solution mixture of the raw materials for more than 48 hours stabilize the monoclinic phase of zirconia at room temperature, rather a lesser time hydrolysis leads to a metastable tetragonal phase in pure zirconia at room temperature. But on heating the powder above 800 °C, the metastable tetragonal phase transforms to the stable monoclinic phase. Two routes of phase transformation in pure zirconia were identified in association with the small particle size (≈ 10 nm). The competing effects of the particle size and the dopants (Y_2O_3 and CaO) on phase stabilization in zirconia were analyzed. The results of XRD, TEM and SEM on pure and doped zirconia nanoparticles will be discussed in the conference.

**EXPERIMENTAL STUDY OF THE PACKING BEHAVIOUR OF
BINARY MIXTURES OF POWDERS INTENDED FOR POWDER
INJECTION MOLDING**

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The selection of optimal powder parameters when preparing feedstocks for powder injection molding (PIM) is critical for enabling cost-effective production of high quality components. Attainment of high densities in injection molding offers potential for reducing the shrinkage and distortion that occurs during subsequent sintering. The requirement for small particle dimensions has led to some concerns regarding the potential cost of the process, making PIM a relatively expensive route for production of larger components. Employment of bimodal blends of powders has been investigated in order to evaluate the potential for replacing monosized particles with bimodal blends. A brief description of the difference between the mono-component powders and the blends of powders with respect to various aspects of PIM is given. An experimental study has been performed on mixtures of stainless steel powders for various particle sizes and morphologies. The evolution of packing density for different compositions of such binary powders has been studied and considerations regarding potential packing density gain are made in each case. Carefully chosen binary blends of powders can provide higher packing densities than their components, and the employment of blends that incorporate high concentrations of water atomized powders could also provide significant economic benefits.

Determination of pore fractal dimensions and porosity of porous materials from the dielectric response at percolation.

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The dielectric properties of the different porous materials as a function of temperature and frequency are considered. The dielectric properties are investigated using Broad Band Dielectric Spectroscopy (in the frequency range of 10 to 10^6 Hz) in the broad temperature interval enabling to cover most of the main dynamic processes occurring in these complex systems.

The analysis of the dielectric relaxation spectrum at percolation was used for determination of the pore fractal dimensions and porosity of porous samples. The percolation phenomenon in the porous glasses is related to the transfer of the electric excitation within the developed network of open pores due to the self-diffusion of charge carriers in the pores. The dielectric spectroscopy technique allows us to identify the relaxation process related to the percolation and to extract the fractal dimensions of the paths of excitation transfer within the porous medium from analysis of the non-Debye behavior of corresponding macroscopic dipole correlation functions. The random fractal model describes the geometrical disorder of the porous matrix. In the framework of this model, the relationship between the porous space fractal dimension and the porosity of the medium has been obtained. An analysis of the dielectric parameters enables us to characterize the porosity of the materials.

SOLIDIFICATION OF TERNARY QUASIPERIODIC PHASES IN Mg-Zn-Y SYSTEM

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Alloys of nominal compositions $Mg_{30}Zn_{60}Y_{10}$ and $Mg_{60}Zn_{30}Y_{10}$ were chosen in order to study a solidification process and a structure of phases co-existing in ternary system Mg-Zn-Y. Both compositions were prepared by melting of chemically pure elements in induction furnace with Ar protective atmosphere. As-solidified structure of each alloy was studied firstly by SEM and EDS techniques. Three co-existing phases were found for as-solidified $Mg_{30}Zn_{60}Y_{10}$ composition: $Mg_8Zn_{75}Y_{17}$, $Mg_{25}Zn_{67}Y_8$ and $Mg_{66.8}Zn_{33.2}$, which surface fractions were 8, 78 and 14 %, respectively. With using analytical TEM and X-ray diffraction it was found that $Mg_{25}Zn_{67}Y_8$ phase was quasiperiodic and two other phases were both crystalline. Nominal composition $Mg_{60}Zn_{30}Y_{10}$ alloy contained several co-existing phases, among which at least one - $Mg_{35.67}Zn_{55.39}Y_{8.85}$ had quasiperiodic structure and occupied about 63% of specimen surface. Oversaturated solid solution of Zn in Mg (94.44 at.% Mg and 5.48 at.% Zn) occupied about 33% of specimen surface and contained traces of Y. Additionally, small amounts of several crystalline phases were found: $Mg_{60.44}Zn_{37.89}Y_{1.67}$, $Mg_{72.3}Zn_{27.6}$ and $Mg_{49.9}Zn_{47.47}Y_{2.63}$. Application of DTA showed that for both found quasicrystalline phases were thermodynamically stable, but $Mg_{25}Zn_{67}Y_8$ quasicrystalline phase formed by binary peritectic reaction while $Mg_{35.67}Zn_{55.39}Y_{8.85}$ phase formed by primary crystallisation from melt. Additionally, it was found that quasiperiodic phases found had several types of quasiperiodic structure, which can co-exist within the single grain.

CORRELATION OF VISCOELASTIC PROPERTIES OF DIE-CAST MAGNESIUM ALLOY WITH PROCESSING CONDITIONS

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Stability and durability of Mg components operating under elevated temperatures are mainly limited by low creep and stress relaxation characteristics of the most widespread die-cast alloy AZ91D (Mg - 9%Al - 1%Zn).

Regression equations are obtained connecting these viscoelastic properties with the porosity of the alloy and with such parameters of die casting as casting temperature, die temperature, and injection rate.

Creep and stress relaxation characteristics of AZ91D alloy were almost independent of the content of discontinuous β -phase. Strain hardening of the alloy is connected both with hardening of solid solution by fine continuous precipitates formed in the grains and with the formation of other obstacles impeding dislocation motion. According to obtained values of the apparent activation energy of creep, diffusion processes (dislocation climbing, etc.) may control the process of creep strain at the temperatures above 175°C. Both porosity and elevated temperature facilitate dislocation motion and lead to the work softening of the alloy.

According to obtained relations, to improve viscoelastic properties, one should choose a set of conditions serving to obtain high-quality articles.

MATHEMATICAL MODELING OF LASER ALLOYING
FOR GRADED SURFACE LAYERS FABRICATION

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On the basis of the partial solution of the heat transfer differential equation, the time-dependence for the temperature distribution and the depth of a molten metal bath in the course of a laser treatment were obtained. Dimensionless process parameters that take into account property variation in the layers of a graded coating and characterize the laser-beam energy and the injected powder consumption were suggested. The dimensionless layer depth and the volume ratio between incorporated WC powder and molten metal (dilution) were considered. Comparison of the experimental data for the laser-alloying of tool steel by direct injection of tungsten carbide powder with the calculated predictions confirmed the validity of the developed model and allowed to optimize process parameters for producing a graded coating free of defects (cracks, pores, etc.). The results of the structural analysis and the hardness profile within the graded coating will be presented.

MODELING OF REACTIVE SYNTHESIS IN B_4C -Ti
POWDER BLENDS APPLIED TO THERMAL EXPLOSION
IN THE CONDITIONS OF ENHANCED HEAT TRANSFER

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A model describing non-isothermal reactive diffusion in B_4C -Ti powder blends leading to thermal explosion (TE/SHS) in the condition of enhanced heat transfer into the surrounding ambience was developed. The model explicitly incorporates the reaction kinetics and is based on previously reported experimental kinetics of isothermal growth of diffusion layers at a flat B_4C /Ti interface. In spite of a number of simplification (spherical geometry, sequential reaction of TiB and TiB_2 formation, etc.), a good agreement between the model calculation and experimental result on TE/SHS in B_4C -3Ti dense samples placed between the preheated rams of a press has been obtained. B_4C powder particle size was found to significantly affect the ignition of thermal explosion, and the combustion process could be controlled by addition of small amounts of very fine powder fraction.

THE THEORETICAL BASE OF SOFTWARE
FOR CONTINUOUS CASTERS TECHNOLOGICAL LINE DESIGN
AND CASTING CHARACTERISTICS VERIFICATION

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Productivity and reliability of continuous casters with the vertical and radial molds, quality of ingots is determined to a great extent by axes coordinates and technical condition of the ingot supporting rollers. For designing and verification of these parameters it is necessary to consider a complex of technological requirements, the default of which results in occurrence of surface and internal cracks, instability of casting process.

The accepted initial data are: the ingot thickness, width, casting speed, crystallization factor, allowable inner deformation of shell bulging and unbending. The total relative deformation should not exceed 0.3 ... 0.6 %, depending on the type of cast metal. Analytical dependence between the base radius, curved coordinates of the initial and finishing unbending zone's sections were found. Possibility to improve productivity and casting quality by optimizing a rolling spacing was showed.

The developed software gives opportunity to determine temperature, stress and deformation distribution in the ingot shell and may be used for checking causes of cracks formation in existing casters and for their modernization.

MODELING IN GENERAL DIMENSIONAL ANALYSIS

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The general tendency in modeling approaches who are referring at very complexes phenomena (like solidification, diffusion, etc.) there are the discretisation, either using finite element or boundary element.

This situation is generated by mathematic's impossibilities to put togheter, in analytical shape, more than six variables.

In science of materials field, complexity of phenomena, ghtered, as influence parameters, 9...12 factors. Experimentally, there is possible to study two-dimensional or three -dimensional dependence between this factors, but it means, in industrial applications, that the rest of factors there are constant (invariable) conducting some times at irreal situations.

For modeling of this complex phenomenon, is necessary to know a mathematical relation between entire list of variables.

General Diomensional Analysis (GDA) is the method which allow to build these relations based on the strong mathematics and on the experiment. G.D.A. combine experimental physics methods with new mathematical theorems. The result of this approach is an analytical relation who connected entire list of variables.

The paper explain the G.D.A. approaces appealing at a few examples from science of materials.

CORROSION OF Mg-Zn-Y-Zr ALLOYS IN CHLORIDE CONTAINING WATER

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Corrosion behavior of as-cast and heat treated Mg-Zn-Zr-Y alloys, in chloride containing water, was studied. Electrochemical measurements and microscopic techniques were used to characterize initiation and development of corrosion damage. It was shown that the as-cast alloy is the least resistant to pitting corrosion. Mg-Zn solid solution matrix occupying about 90% of the alloys' surface was found to be the structural constituent responsible for corrosion susceptibility due to the presence of Zn-lean regions. Homogenization of Zn distribution within the matrix by aging significantly increased the corrosion resistance of the tested alloys. Increasing the total Zn content in the matrix had non-monotonous effect on the corrosion resistance of Mg-Zn-based alloy. When Zn content in alloy exceeded 6 wt.%, structural inhomogeneity of its solid matrix became very strong and unavoidable by solution heat treatment, because of low temperature melting of the grain boundaries. Grain boundaries, which contain intermetallic phases, were found to resist corrosion attack, thus impeding corrosion zone propagation. Intrinsic protective properties of the corrosion product layer were found enhancing by pre-exposure at negative potential or at OCP conditions, which, apparently, causes formation of more uniform and dense corrosion product layer enriched by Zn-oxide – hydroxide.

RESEARCH REGARDING THE ACHIEVEMENT OF
THE TOTAL HIP PROSTHESIS

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In this article are shown the researches that had been made with a view to accomplish the total hip prosthesis formed of Ti6Al4V ELI femoral stem and a CoCrMo femoral head, according to the product international standards ISO 5832/3 and ISO 5832/12. The materials were selected on the basis of the biocompatibility and biomechanical investigations at which the permanent implants must correspond: high characteristics of mechanical resistance, elastic modulus similar to that of the bone, a low specific weight and corrosion resistance with a good biocompatibility. The obtaining of this product in original conception has required different variants of plastic deformation and heat treatment in order to obtain a complex of mechanical and structural characteristics imposed by product international standards.

SMALL-SIZED FILTER DEVICE FOR COMPREHENSIVE DRINKING WATER TREATMENT

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Dr. A. Gor

Nowadays it has become quite a regular practice to use different filter devices for drinking water purification prior to its consumption in domestic and others water installations

The above mentioned systems for domestic water treatment will bring to the improvement of water quality in such terms as taste, odor, softening, suspensions withdrawal. All the treatment methods do not concern the level of microbiology contamination for "purified" water, and it becomes well above the permitted numbers after water dechlorination and storage at room temperature

We propose the novel type of portable or in-line water filter devices. It will include all the regular water treatment options of improved design. In addition to the regular treatment, some preserving agent will be incorporated into filter design. It will perform as preserver against the rapid growth of bacterial contamination on its surface. It will also eliminate or retard the bacteria growth in the water filtrate to a great extent for the 24 - 48 hours period at least, whilst the retardant rate is anticipated as 50 - 100% in regard to initial one. *This component will impair neither the water taste quality nor MCL standards levels.*

The new water purification device has some advantageous features that differ from standard, market available items.

The filter design will enable to maintain the following features:

1. Small-sized and compact design that will be convenient in any installation;
2. Extremely large specific area of contact interface between the filter media and water filtrate;
3. Long lasting contact between filter media active components and water filtrate;
4. Filtration through one layer at a time.

Preserving Component incorporation onto the filter surface and its controllable release into the purified water will bring to the elimination or essential retarding of bacteria growth both on the filter surface and in water filtrate, thus enabling to keep on with high water quality and legal standards.

Production and Characterisation of Hydroxyapatite Derived from Australian Corals for Artificial Eyes

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Investigation of coral for maxillofacial, orthopaedic and ocular implants was first proposed in 1986 [1]. Coral is primarily of calcium carbonate and structurally very porous. Through the hydrothermal exchange, the coral structure can be converted to calcium phosphate (hydroxyapatite) [2]. The product hydroxyapatite is biocompatible and has the same porous and the chemical structure of bone. The hard or soft tissue of the body can grow into the pores due to its porous nature.

A modified hydrothermal process was developed to produce hydroxyapatite. A coral named new species, available from the Great Barrier Reef-Australia was used as raw materials. This coral is mechanically strong and exhibits an interconnected porous structure that resembles that of trabecular bone.

The coral used showed the presence of the aragonite phase of calcium carbonate. The special treatment such as the boiling and a specific chemical treatment can be used to remove the organic matter from the coral. The pre-treated coral was converted into hydroxyapatite by a chemical exchange reaction with ammonium hydrogen phosphate under hydrothermal conditions.

The conversion was carried out under saturated steam pressure, at 250° C and at a pressure of 35 bar. X-ray diffraction analysis (XRD), X-ray fluorescence (XRF), Fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA) and Scanning electron microscopy (SEM) were employed to characterize the coral and to optimize the processing parameters as well as to confirm the hydroxyapatite formation.

The hydroxyapatite obtained in block form retained the microstructure of porous carbonate skeletal structure of the coral and is a useful biomaterial for orthopaedic, maxillofacial and ocular (orbital) implant applications.

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COMPUTER AIDED MONITORING OF THE HEAT AND THERMOMECHANICAL TREATMENTS

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INTEC Bucuresti, ROMANIA

The use of automation elements and process interfaces due to their compatibility with the compatible IBM hardware AT structures make possible the development of a data acquisition, processing and registering system for the characteristic parameters of a technological process.

In the field of heat and chemical treatments, this achievement, due to the complexity both of the phenomena and working equipment require the use of monitoring aided by computer like solution for the optimal operating of the processes and for the quality products assurance.

This paper study the monitoring aided by computer themes, like answer and solution for the necessity to modernise the heat treatment departments endowed with equipment with different functions.

Taking account the particularities of the thermochemical and heat treatment processes this paper studies next issues :

- the monitoring aided by computer ;
- the premises of the monitoring aided by computer ;
- the enclosing of the area from the heat treatment department for the monitoring aided by computer ;
- the performances in monitoring aided by computer.

DEVELOPMENT OF NANOCRYSTALLINE FILMS OF PBS WITH HIGH IR SENSITIVITY

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The objective of our study is to develop and improve the sensitivity of IR detectors at room temperature. Nano-crystalline thin films of lead sulfide were deposited on glass substrates by the hot wall epitaxy method. The grain size varied in wide limits (from 50 to 100 Å) by varying the growth rate and the substrate temperature. The grain boundaries are assumed to influence the optoelectronic properties of nanocrystalline films by generating potential energy relieves, estimated to reach 0.1 eV. We investigated the influence of the barrier height by introducing impurity atmosphere near the block boundaries. Oxygen was introduced into the films by annealing in air and in oxygen at various temperatures. Also indium impurity was introduced from a gas source (In_4Te_3 compound).

Hall effect was used to measure the free carrier density. Conductivity was measured in the 90-300K range. It rises with increasing temperature, indicating that conductivity is a thermal activation process. In the same time, in PbS films annealed in In atmosphere the conductivity decreases with temperature like in bulk PbS crystals. Photoconductivity was observed in films annealed in oxygen which was significant up to room temperature. Carrier lifetime and diffusion length were found to decrease with rising temperature and be larger in the films annealed in oxygen.

A model is proposed to explain the optoelectronic behavior of the nanocrystalline films. It is proposed that grain boundaries set up potential barriers for free carriers so that the carriers generated by an illumination become spatially separated: electrons become localized at the maxima of the potential while holes are localized at the minima and this separation retards their recombination and increases their lifetime.

INSTRUCTION IN MATERIALS SCIENCE AND ENGINEERING:
FROM BLACKBOARD AND CHALK TO MODERN TECHNOLOGY

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Materials Science and Engineering (MSE) has been created by combining disciplines which are several thousands years old (Metallurgical Engineering) with quite recent ones (Solid State Physics). It includes atomic and molecular interactions and structures, synthetic chemistry, phase equilibria and phase diagrams, a variety of characterization methods, mechanical testing and fracture mechanics, viscoelasticity, rheology, materials processing from forging through sintering to injection molding, as well as prepreg making and composite manufacturing.

Different sub-disciplines of MSE require often different instructional approaches. Teaching a single basic MSE course requires a combination of approaches. Fortunately, we have more and more instructional tools: computer modeling (watching a known process); computer simulations (learning about an insufficiently understood process); interactive computer programs; TV broadcasting of lectures followed by discussions by phone - both videorecorded; the *Journal of Materials Education* ; and traditional textbooks and class lecture notes. Moreover, MSE also exhibits certain unifying features (not limited to just, say, metals or semiconductors); these features will also be discussed.

Tuesday

Plenary Sessions

THE MEANING OF STRATEGIC RESEARCH AS
EXEMPLIFIED BY OUR WORK ON THE
AROMATIC COPOLYESTERS

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University of Illinois
Materials Science and Engineering
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Early work on the aromatic copolyesters is briefly reviewed. This includes comments on the emergence of the field of liquid crystalline (LC) polyesters as well as the more recent elucidation of the sequence ordering in the (LC) copolyesters.

Recent progress on the development of a new family of thermosetting copolyesters which grew out of the original LC polyester work is described. This new system solves many of the shortcomings of the commercially available thermosets by providing a system which is stable in air at 350°C. One unique feature is that it represents the only thermosetting resin which can be recycled back to a reactive oligomer by using a high temperature interchain transesterification reaction (ITR) with controlled amounts of acetic acid. Another unusual characteristic is that coatings can be first cured and then the coatings consolidated at 300°C to form a very strong adhesive bond through solid state ITR at the interface.

In the current environment, it is no longer sufficient to design a new polymer with a number of interesting properties. It is essential to demonstrate possible areas of advantage (in the context of this talk such work is considered "Strategic Research"). Hence, in this presentation a number of specific examples are put forward as illustrative of Strategic Research, where we have tailored the copolyester to solve specific problems.

TAILORING OF INTERFACES IN POLYMER COMPOSITE MATERIALS

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While polymer composite materials have been used for thousands of years, it has only been during the last half century that applications have become so demanding that the tailoring of well-bonded, durable interfaces (or "interphases") between the matrix and reinforcement has become a critical concern. The development of glass, carbon and high modulus organic fiber reinforced composites ushered in an era of new light weight, durable structural composite materials. The use of coupling agents, chemically reactive with both matrix and reinforcement, and/or chemical modification of the surfaces of one or both constituents have been the most successful means of chemically bonding the matrix to the encapsulated reinforcement. Many years of research have been invested in characterizing the molecular structure of the interphase region and its relation to mechanical and chemical stability.

An overview of research on the characterization of interphases in glass and carbon fiber reinforced organic polymers will be presented. While protection of the reinforcement from environmental damage and improvement of adhesion between phases have been well documented, neither the characterization of the properties of the interphase nor its consequences on the durability of a composite material product have been definitively resolved. It is generally agreed, however, that synthetically created interphases are often mechanical weak links and a potential source for the initiation of defects in a structure.

Recent research has focused on the attainment of monomolecular, self-assembled, reactive surface layers that form strong chemical bonds with both constituents. In this case, the polymeric "interphases" are reduced in dimension to as close to a true "interface" as molecular dimensions allow. Since sharp boundaries between constituents with large differences in physical properties can also cause mechanical difficulty, the tailoring of such an interface is a challenge. Recent work in this area and speculation on the properties required will be presented.

MATERIALS FOR THE ELECTRONIC INDUSTRY

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The semiconductor industry has over the past thirty years provided exponential increases in product performance as well as concomitant cost reductions at a level never before seen in this century. The rapid development and implementation of new materials has been key to improvements in circuit density for semiconductors, storage density for magnetic disk drives, and resolution for flat panel displays. Extending this pace of improvement into the future will demand significant further materials innovations.

This seminar will begin with an overview of the key materials that have enabled these advances in the semiconductor industry. It will then focus upon a discussion of the efforts to extend these technologies and the development of new materials for the 21st century.

INSTRUCTION IN MATERIALS SCIENCE AND ENGINEERING:
FROM BLACKBOARD AND CHALK TO MODERN TECHNOLOGY

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STRUCTURED ELECTRICALLY CONDUCTIVE POLYANILINE/POLYMER BLENDS

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Polyaniline, PANI, processability can be enhanced by doping with functionalized protonic acids [1] such as dodecyl benzene sulfonic acid (DBSA), p-toluene sulfonic acid (pTSA) or camphor sulfonic acid (CSA). Doping with these acids enhances solution [2] and melt [3] processability. In another approach to enhancing processability, PANI colloidal dispersions can be used [4,5]. Melt blending of binary PANI/Polymer systems and ternary PANI/Polymer-A/Polymer-B systems [3,6] has shown that the structure of the PANI particles and the associated electrical conductivity are related to the levels of interaction between PANI and a specific polymer. Thus, in the ternary blends, PANI's location may be selective, i.e., within one of the phases, at the interface, or even combinations thereof. Stable PANI-DBSA aqueous dispersions can be obtained by aniline polymerization in the presence of DBSA, with DBSA acting as the acidifying agent, surfactant, dopant and also as the final dispersion's stabilizer [4]. PANI-DBSA/Polymer blends can be easily produced by mixing the aqueous PANI-DBSA dispersion with an aqueous emulsion of the matrix (PS, PMMA, PVAc, etc.). Dried samples of such blends exhibit significant electrical conductivities already at very low PANI-DBSA contents (~0.5%). The conductivities are found to be independent of the polymer matrix present, but dependent on the surface characteristics of the PANI particles and on the polymers constituting the matrix. Thus, dissimilar surface characteristics may lead to a significant segregation of the PANI particles and therefore the generation of fine conductive networks at very low PANI concentrations.

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Exploring Technical Properties on the Molecular Level –
Modules for the Design of Smart Materials in the Next Millenium

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Technical properties of materials are controlled by the molecular structure and dynamical processes of their components. Surface properties like wetting and adhesion are highly dependent on the molecular structure and dynamics of the surfaces and their interfaces. Even large-scale industrial materials manufactured by "classical" conventional industrial processes, exhibit distinct and well defined molecular structures on the nanometer scale. These structures are needed to provide well-defined technical surface properties.

Consequently, a key task for the control of technical properties are investigations of the underlying molecular structures and dynamical processes of materials. Of particular value are structure-property correlations on the molecular level. A-priori correlations do not exist for most complex technical systems, therefore empirical or semi-empirical relationships need to be developed. Model systems often assist in such investigations.

Of particular interest are quantitative correlations. Surface properties like adhesion may quantitatively modeled by e.g. chemical surface reaction rates, surface and interfacial diffusion or segregation phenomena in the system. Such investigations usually require a broad approach of fundamental studies. However, once the correlations have been obtained one can directly derive improvements to materials or process conditions of the system.

Moreover, the "molecular" knowledge of technical properties in a particular system can be utilized for the design of new materials in a modular fashion. Examples of technical surface phenomena such as wetting and adhesion, in particular the approach to smart materials like reversible adhesion and ultra-hydrophobic surfaces will be presented and discussed.

Tuesday

Parallel Sessions: 3

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HARD TiN-BASED COATINGS ON SURGICAL TITANIUM ALLOYS

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An original PIRAC nitriding method was used for surface modification of Ti-based surgical alloys. Ti alloys were annealed in sealed stainless steel containers that allowed selective diffusion of nitrogen atoms from the atmosphere. The relationship between the microstructure, nitrogen concentration and microhardness was studied for the samples PIRAC nitrided at 850-1100°C. Phase identification and microstructure characterization were performed by XRD, SEM/EPMA and High Resolution SEM. PIRAC nitrided surfaces were found to have a layered structure with a TiN/Ti₂N coating followed by nitrogen-stabilized α -Ti. In contrast to previous investigations of surface nitrided Ti-6Al-4V alloy, a Ti₃Al intermetallic phase was detected at the Ti₂N/ α -Ti interface acting as a barrier for nitrogen diffusion. The effect of PIRAC coating on fretting behavior of Ti-6Al-4V was studied in vitro under conditions simulating in vivo conditions at the interface of modular orthopedic implants, and a significant reduction in friction coefficient and fretted areas was measured for the coated samples. The adhesion of the surface layers to the substrate was estimated in a three-point bending test. No delamination of the coating was observed even at stresses exceeding the yield point of the corresponding substrate. Residual compressive stresses were measured in both TiN and Ti₂N layers by x-ray diffraction using the $\sin^2\psi$ method.

PROTECTIVE COATING BEHAVIOR ON C - C MATERIAL

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Carbon - carbon materials are generally being woven from strips or thin rods that possess high strength directional properties. By weaving these elements in a three dimensional structure, a part having high non-directional strength can be processed, strength that is not deminished also at exposure to high temperatures. These two combined properties of strength and heat resistance makes C-C structures suitable for applications working at extreme temperatures regimes. The main draw-back of C-C materials is their sensitivity to oxidation even at quite low temperatures. At temperatures over 500°C, these materials begin to lose weight due to oxidation at a rate increasing steeply with the temperature rise. By applying protective coating on the surface of the C-C parts, this oxidation can be deminished appreciably. The kind of coatings, that were evaluated in this work, consisted mainly of SiC together with other constituents in smaller quantities. The coatings are processed by reactions of the carbon surface with mixture of powders or gases at high temperatures. Protective coatings are produced by cementation of the C-C parts packed in a graphite container together with powder mixture of active nature at 1900°C in an inert atmosphere.

Another more elaborate process for the creation of this kind of conversion coatings is by CVD process. Working with CVD reactor requires more complicate techniques and routines in comparison with pack cementation. But the CVD process is more technically suited for coating applications of service parts.

In the current work, protective coatings on graphite samples were produced by pack cementation procedures. The research aims were to try to characterize the better powder mixture able to produce effective conversion coating. The main criterion for establishing the coating behavior was its ability to lower the oxidation rate during exposure to oxidizing environment combined with increasing temperatures. Another powder property which is limited to pack cementation process, is the difficulty of loosening the samples out of the packed cementated powder.

The protection of the coating is based on different reactions of the coating constituents with oxygen to seal the surface of the graphite. Samples were characterized to define their phases by x-ray diffraction after being exposed to raising temperatures.

**ELECTROPHORETIC DEPOSITION:
THE TECHNOLOGY & ITS APPLICATIONS**

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Cerel - Advanced Ceramics & Technologies Ltd

**Technion - Israel Institute of Technology*

The electromigration of charged particles in an electric field, has been known for over a hundred years, while electrophoretic deposition (EPD) from a liquid suspension, defined as the formation of a dense deposit of electromigrating particles at an electrode, has been studied for at least sixty years.

Renewed interest in EPD as an industrial process has been fueled by current demand for reliable powder technologies with improved control of linear dimensions, composition or microstructure. EPD is capable of generating defect-free coatings and near-net shape micro-components having graded or laminated microstructures. Geometrical patterning of coated layers has also been demonstrated. Provided the particle size is in the appropriate range, any combination of powders, ceramic, metal, polymer or semi-conductor, can be successfully deposited, both sequentially and from powder mixtures.

Surface preparation of the conducting substrate is critical for either adhesion (EPD coatings) or easy release (free-standing EPD components). So is the consolidation of the green EPD composite, either by conventional sintering or by alternative gas and liquid phase processing options.

Comparatively few basic studies of the EDP process have been published, but the general features are well understood and will be described, together with some potential applications of the process.

ELECTRODEPOSITED NANO-COMPOSITE COATINGS

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Electro-deposited composite coatings have been widely adopted in industry. These include nickel matrix containing carbides, oxides, nitrides, sulfides and miscellaneous polymers. The second phase in these composite coatings is a particulate usually in average diameters ranging from 3-8 micrometers. The volume fraction of the second phase range from 5-25%.

Although these composites have proven to be very effective in wear resistant applications, it has become possible to obtain orders of magnitude improvement in performance of these coatings through the use of particulates that are in the nanometer size range, at lower volume fractions. This paper describes the methods by which composite coatings of nano particles of silicon carbide dispersed in nickel can be produced on aluminum substrates, and their effect on wear and elastic modulus. Work is continuing at Western Michigan University to further improve the performance of these coatings.

GeSi SELF-ASSEMBLED DOTS

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Recently, attention has been turned to the possibility of incorporating self-assembled GeSi dots (islands) into active Si devices. Such islands have a small band gap enabling optical activity in the near IR range. Also, they can be more optically active than bulk material as a consequence of the reduced dimensionality. However, it is not yet possible to grow islands of desired size, shape and density. Results on island distribution of Ge on Si obtained using low pressure chemical vapour deposition will be presented. The size distribution on (001) Si can be mono- bi- or three modal, depending on the growth conditions. Nucleation on high index planes is expected to narrow the island distribution. High index planes were generated by using selective epitaxial growth of Si for the formation of faceted mesas. An important aspect of self-assembling is the vertical ordering of islands. In the present talk these items will be discussed for GeSi as well as device applications such as light emitting diodes.

MICROSTRUCTURE AND ELECTRICAL PROPERTIES OF CoSi_2 CONTACTS ON $\text{Si}_{1-y}\text{C}_y$

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The addition of substitutional carbon to Si, to create $\text{Si}_{1-y}\text{C}_y$ is advantageous by, at least, two reasons: a) a decreases the diffusion rate of B and therefore enables a decrease in the vertical dimension of devices (e.g. Bipolar transistors). b) a modulation in conductance band, which enables creating hetero-devices based on Si technology. The use of CoSi_2 as a contact for $\text{Si}_{1-y}\text{C}_y$ layers epitaxially grown on Si was investigated.

Cobalt films (30nm thick) were evaporated on MBE grown p-type (boron doped) $\text{Si}_{1-y}\text{C}_y$ ($0 \leq y \leq 0.81\%$) layers (200nm thick) on top of p+ (100) oriented Si substrates. The samples were subsequently annealed for 30 min at 450, 600 and 700 °C.

Microstructure and chemical composition characterization by AES, SIMS, XRD, and TEM will be presented as well as results of electrical characterization including current-voltage (I-V) characteristics, and admittance spectroscopy.

No strain relaxation in the $\text{Si}_{1-y}\text{C}_y$, and no pile-up of carbon or boron were found, adversatively to the known behavior for Co (or Ti) metallization on $\text{Si}_{1-x-y}\text{Ge}_x\text{C}_y$ epilayers. The temperature of CoSi to CoSi_2 phase transformation increased with increasing carbon content in the $\text{Si}_{1-y}\text{C}_y$ layer. In addition, the CoSi_2 grown on carbon containing layers had a [100] preferred orientation, while on pure Si it was randomly oriented. This can be explained by a reduction in the diffusion rate of cobalt through the silicide layer, in the presence of carbon.

No majority deep states were found, up to ~0.5 eV above the valence band.

In conclusion, the use of CoSi_2 metallization to $\text{Si}_{1-y}\text{C}_y$ is possible, in spite of some drawbacks such as: inhomogeneous thickness of the silicide layer, an increase in transformation temperature to CoSi_2 , and an increase in sheet resistance.

Acknowledgments

The support of the Israel Ministry of Science to this research is acknowledged.

Y. Roichman would like to acknowledge Israel Ministry of Science for granting him Eshkol scholarship.

**Surface photovoltage spectroscopy of two dimensional structures
and devices**

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GaAs/AlGaAs single quantum well (QW) *p-i-n* structures for solar cells applications and InGaAs/GaAs/AlGaAs p-HEMT structures have been characterized using surface photovoltage spectroscopy, which is a room temperature, contactless, non-destructive method, for characterization of novel semiconductor devices.

In the first example, experimental and numerical studies of the photovoltaic response of single QW *p-i-n* structures are presented. It is shown that the photovoltaic (PV) response is governed by the QW parameters, in particular the dependence on the QW width is discussed. Numerical calculations show that the combined quantum effect on the oscillator strength and carrier lifetime in the well results in an increased PV response beyond the blue shift effect. The domination of the carrier lifetime at the well over recombination processes at the entire structure is discussed. The effective carrier lifetimes in the well layer/interface for the different studied samples are estimated.

In the second example, the contribution of the different layers of the p-HEMT device to the surface PV spectrum are demonstrated. The fingerprints from each layer is utilized for its characterization. In particular, the dominant contribution of the substrate/buffer interface and the external cap layer to the spectra and its implications to device performance is discussed.

NANOSCALE CHARACTERIZATION OF SEMICONDUCTOR ELECTRONIC
PROPERTIES USING KELVIN PROBE FORCE MICROSCOPY

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The shrinkage of semiconductor devices to the submicrometer level has led to the need for direct measurement of electrical properties with nanometer resolution. A combination of scanning Kelvin probe and atomic force microscope is implemented for the study of electronic properties of semiconductors on nanometer scale.

We present measurements of two-dimensional potential distribution of operating light emitting diodes. These measurements enable us to find the dependence of the surface built-in voltage on applied external bias. As the device is forward biased, the junction surface built-in voltage decreases up to flat band conditions, and then inverted. It is shown that this behavior is governed by self-absorption of the diode-emitted light.

A new method for measuring minority carriers' diffusion length is presented in the second work. The method is based on measuring the surface photovoltage between the microscope tip and the surface of an illuminated semiconductor *pn* junction. The photogenerated carriers diffuse to the junction and produce surface potential changes at the edge of the depletion region of the junction. The method is applied to GaP epilayers where a fitted electron diffusion length of $2.0 \pm 0.05 \mu\text{m}$ is obtained. This value is in good agreement with other measuring techniques.

**ON THE INELASTIC RESPONSE OF Al_2O_3 UNDER
SHOCK LOADING**

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The dynamic response of Alumina under shock compression was studied using planar impact experiments with different widths. Results show steady spreading of the inelastic shock profile with increasing tile width. Further analysis indicates an expected change in failure mechanisms in pressure in pressure of about twice the HEL. HEL decay phenomenon in alumina is presumably a measured artifact, resulting from the relatively slow response times of manganin gauges.

ENERGY ABSORBER MATERIALS FOR IMPACT AND CRASH PROTECTION

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The research and development of more efficient energy absorbing materials and technology are one of the issues of major importance to saving lives in roads and the highways during the last two decades.

Crash cushions and impact attenuators are protective devices that prevent errant vehicles from impacting fixed objects hazards. The contribution of these devices are their ability to absorb energy at a controlled rate. The first concept of crash absorber design involves the transfer of the momentum of a moving vehicle to the expendable mass of materials located in the vehicle's path (for example: the well known containers filled with sand). The second and more sophisticated concept of impact attenuators design involves absorption of the kinetic energy of a moving vehicle by plastically/brittle deformable materials.

Because of the special form of the compressive stress-strain curve, light metals based on cellular or foam solids are capable of absorbing large amounts of energy at a relatively low stress. A typical loading stages include initially almost linear deformation followed by plastic collapse and final densification.

Special mechanical properties can be achieved by careful selection of the matrix material and processing.

The paper presents a review of the topic and the up-date advanced technology for the preparation and characterization of energy absorber materials.

DEVELOPMENT OF ADIABATIC SHEAR BANDS IN URANIUM ALLOYS

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Very localized deformation is observed in metallic materials when high strain rates are applied. The results are discussed in term of an adiabatic shear band (ASB) mechanism, believed to occur under these conditions.

Under high strain rate loading the heat, generated as a result of local plastic deformation, is confined mostly within the deformation zone. When the deformation zone is narrow enough, the generated heat results in a localized high temperature rise and change of mechanical properties, which lead to the formation of localized shear bands.

Different stages of ASB has been characterized in uranium alloys by optical and electron microscopy. Observations and simulation will be presented and the mechanism will be discussed.

**NONLINEAR DYNAMICAL (CHAOS) ANALYSIS OF THE
CRACK SURFACE IN SAPPHIRE**

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The dynamical behavior of a spatial fracture mechanism in a rapid crack in a single crystal brittle solid was investigated. Sapphire strips were fractured in three point bending. The specimens' low energy cleavage planes were inclined to the K_I path, which caused the generation of large sets of surface perturbations.

The perturbations were photographed and digitized and the results were then studied using non-linear dynamical analysis. It was shown that the governing equation of motion in the analyzed region is differential equation with a minimum of seven independent dynamical variables, in contrast to a single independent variable used in analytical calculations. This work indicates that even the highly complex perturbations of the crack surface, which may seem randomly generated, are governed by physical variables.

DIRECT MEASUREMENTS OF FORCES BETWEEN SURFACES BEARING POLYMER BRUSHES

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End functionalized poly(ethylene propylene) (PEP) was verified to form a solvated polymer brush in the good solvent cyclohexane. On this model system, normal and shear forces were investigated. Two opposing polymer brushes exhibit a very low friction coefficient when rubbed against each other. The friction coefficient remains low for high loads, in contrast to earlier studies with PS brushes, due to the low glass transition temperature of the PEP. It is proposed that when the compressed PEP brushes slide at low shear velocities there is some interpenetration that results in weak entanglements between the opposing brush layers. As the shear amplitude is increased, the brush layers are forced to disentangle, and the friction drops, leaving weak interdigitation. This causes some drag force which is the main friction factor in this case. Our results provide a detailed picture of the relaxation of the brushes when the shear motion is stopped.

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Shear and Normal Forces between Smooth Solid Surfaces, in Solution of Adsorbing PEO in Toluene

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Direct measurements of the normal forces between two atomically smooth mica surfaces immersed in toluene have been carried out as a function of surface separation. Then poly(ethylene oxide) (PEO) was introduced into the toluene, which is a good solvent for PEO. Following full overnight adsorbance of PEO ($M_w=37k$ and $M_w=112k$) a force-distance law was indicated on rapid compression / decompression of the surfaces, with repulsion beginning at a distance of $\sim 8R_g$ (radius of gyration) and increasing monotonically at shorter distances. Measurements of the shear response were then carried out as a function of surface separation at the same conditions. Uniform back and forth shear motion was applied in a series of separations, from some tens of nanometers down to closest approach distance. A significant shear response was observed at a distance of $\sim 2R_g$ and increased monotonically at shorter distances. In low molecular weight, normal force measurements performed immediately after shear showed no forces up to separation of 5nm, below which stronger attraction was indicated. This shows that during shear PEO was detached from the surfaces. We estimated that $\sim 10\%$ of the polymers were still on the surface and the attraction between the surfaces is due to bridging effect. In the high molecular weight, the polymer was not removed from the surface and under high compression, the shear forces showed weak increase with the normal load.

BROMINATED FLAME-RETARDANT BLOOMING PREVENTION BY MOLECULAR MODELING

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The goal of this research was to understand the brominated fire-retardant (BFR) blooming mechanism in High Impact Polystyrene (HIPS) and to recommend ways to prevent it. Two different main software tools were used. The first was Quantitative Structure Analysis Relationship (QSAR), in which a database of blooming results was the input. The output was a family of empirical equations created by an innovative statistical algorithm, enabling the user to analyze ways to reduce blooming by varying several variables. Although limited understanding of the blooming process can be achieved in this way, fast analysis of the blooming database and conclusions concerning its reduction are gained.

The second tool used was a full atomistic simulation, enabling the user to gain both understanding of the blooming mechanism as well as finding ways to prevent it.

One QSAR analysis showed that elevated melt temperature in injection molding process, leads to a substantial reduction in the BFR blooming, specifically, an increase of melt temperature from 260°C to 270°C was tested.

Atomistic simulation was used to calculate (indirectly) diffusion rates of BFR's in PS matrix, the cohesive energy and miscibility parameter of all the participants, as well as phase diagrams of the BFR's in PS. The results showed that the BFR's differ in compatibility with PS and with butadiene phases.

Based on both computational results and experimental evidence, a blooming mechanism was suggested, in which the mass transfer of the BFR is taking place through the butadiene inclusions which are less compatible with the BFR, leading to its high rate of diffusion. The BFR depletion in the Butadiene/PS interface leads to concentration gradients and to its diffusion from the PS matrix to the interface.

DISCOLORATION OF PP-BASED COMPOUNDS CONTAINING MAGNESIUM HYDROXIDE

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Magnesium hydroxide is extensively used in the plastic industry as a flame retardant and smoke suppressant in various polymeric matrices (polyolefines, nylons etc). It was found that compounding $Mg(OH)_2$ with polymers leads to the formation of various colors ranging from light gray to rather dark beige.

The effect of the temperature and processing technology, and of various properties of $Mg(OH)_2$ (impurities, particle size and morphology) on color formation was investigated in this work. For instance, it was found that various colors occur when compounding is performed with different polymers at different temperatures. One of the causes for the discoloration appears to be the interaction between the $Mg(OH)_2$ and antioxidants containing phenolic groups. This reaction takes place rapidly when the former is added to the polymer melt containing the antioxidant.

In flame retardancy applications, magnesium hydroxide is commonly added in rather large concentrations (55-65% by weight). The powder is usually coated with various materials to ensure a good compatibility with the polymeric matrix. The amount and type of coating, and its continuity over the particles surface (dependent on the powder morphology and coating technology) may represent a way to prevent the chemical interaction between the filler and plastics components and avoid discoloration.

A method for the quantitative evaluation of coating quality and continuity was developed. The results of this evaluation were compared with color measurements of plastics, as compounded in our lab. Based on this research, ways to improve the quality of magnesium hydroxide as fire retardant were suggested.

COMPOSITES BASED ON SEMICONDUCTING AND LIGHT EMITTING POLYMERS

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We describe polymer-based composites within two complementary contexts. Firstly, extending the properties found in neat polymers so as to allow the making of more complex or sophisticated devices. In this context we describe a method of controlling the refractive index of solution processable polymer composites in the range of $n=1.6$ to $n=2.7$. This was used to realise waveguides and DBR mirrors using solution casting techniques. Hence this method provides building blocks for all-polymer optoelectronic and lasers. Secondly, we use composites as a tool to explore the basic photo-physics of conjugated polymers. Here we describe a method of probing and manipulating the non-emissive triplet excitons, which are known to play an important role in affecting the efficiency as well as degradation of polymer LEDs. We also show how this study provides an insight towards the possible realisation of electrically pumped polymer lasers.

**MICROSTRUCTURAL STUDIES OF
POLYSTYRENE/POLYBUTADIENE/ORGANO-BROMINE-FR
TERTIARY SYSTEMS**

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High impact polystyrene (HIPS) is produced by polymerizing styrene in the presence of a rubber, usually poly(1,3 - butadiene) resulting in improved impact properties of the material. The material is commonly used in consumer products such as small appliances and electronic devices. The addition of Organo-bromine based flame- retardant (FR) is known to alter the mechanical properties of HIPS and affect its processability. Long term stability, microstructural and thermodynamic stability of the system is also of interest.

In this work we report a study on the effects of two chemically different Bromine-based FR's on the properties and microstructure of HIPS. Microstructural information was obtained by direct visualization using transmission electron microscopy (TEM). Samples were cryo-microtomed from model bars, and treated with OsO₄ which selectively stains the rubbery phase. The composition of the different phases was determined by means of energy dissipating spectroscopy (EDS). Measurements were complemented by DSC/DTA analysis. Large differences in microstructure and FR distribution have been observed between the samples containing the two different FR's. These differences are correlated with property and stability observations.

SEGREGATED STRUCTURES IN CARBON BLACK CONTAINING IMMISCIBLE POLYMER BLENDS

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Immiscible polymer blends are interesting host multiphase systems for the incorporation of low concentrations of fine electrically conductive fillers, such as carbon black (CB). These conductive blends achieve high levels of conductivity at surprisingly low CB loadings, in comparison to their individual constituents. The low filler content is of practical advantage due to the tendency of CB to diminish mechanical properties and complicate polymer processing.

The critical amount of CB necessary to build up a conductive network and accordingly make the polymer conductive is referred to as the percolation threshold. In immiscible blends, the CB particles do not distribute uniformly, but rather accumulate preferentially within one of the phases and/or at the interface. When these segregated regions form continuous structures, conductive pathways are obtained, and double-percolation occurs.

The main purpose of this study was to investigate the behavior of CB-containing multiphase polymer systems based on high impact polystyrene (HIPS) blended with either linear low density polyethylene (LLDPE), ethylene-vinyl acetate copolymer (EVA) and ultra high molecular weight polyethylene (UHMWPE). The nature of the blend components, the blend composition, CB content and shear level were found to determine the structure and the resultant electrical properties. The morphology, resistivity and flow properties of these blends are closely interrelated, and the conductive blend properties may be tailored according to these considerations. Double-percolation phenomena were emphasized and discussed in relation to the general percolation theory.

O. Breuer currently at RAFAEL, Israel Ministry of Defense.

STRUCTURE AND PROPERTIES OF TERNARY IMMISCIBLE POLYMER BLENDS

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Immiscible polymer blends are interesting host multiphase systems for the incorporation of fillers. Such systems may exhibit several morphologies: filler particles and minor polymer particles separately dispersed in the matrix, filler particles located at the interface between the continuous matrix and the dispersed polymer particles, and filler particles encapsulated by the minor polymer phase.

The actual morphology developed during the blending process is determined by thermodynamic parameters, e.g., polymer/filler interfacial tensions or spreading coefficients, and by kinetic factors, e.g., polymer viscosity.

The main objective of this study was to investigate the dominant parameters that have led to a preferred morphology and to relate the rheological, mechanical, and thermal behavior of some multicomponent blends to their morphology.

A series of ternary blends with varying compositions was chosen, comprising polar EVOH as the minor phase, a semi-crystalline matrix of varying polarity (PP, LLDPE, or EVA) and mica as the filler.

The morphology of the blends studied has revealed the existence of two dispersed phases: EVOH encapsulated mica particles and some separately dispersed EVOH particles in the matrix. The structure of the multicomponent blends studied has a significant effect on their behavior as depicted by their thermal, rheological, static and dynamic mechanical properties. The structure itself can be controlled by careful selection of the blend's components, its composition and processing details.

FUNCTIONAL POLYMERIC NANOPARTICLES OF NARROW SIZE DISTRIBUTION: DESIGN, SYNTHESIS & BIOMEDICAL APPLICATIONS

BY

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During the past decade Prof. Margel research group have pioneered in the development of Polymeric Nanoparticles ranging in size from a few nanometers to a few microns. These particles having very narrow size distribution, spherical shape and high surface area. They may also contain different dyes and variety of surface functional groups (i.e. aldehydes, hydroxyls, carboxylate, amines and thiols), and may provide a unique solution to a variety of basic and practical problems in materials science and biotechnology.

During the past ten years six major types of uniform functional monodispersed nanoparticles have been prepared in the laboratory:

- 1) Solid nanoparticles suspended in aqueous and/or organic solvents microencapsulated with different dye compounds and/or magnetic properties. These nanoparticles were prepared from a variety of functional polymeric materials, e.g. polyacrolein, polyglutaraldehyde, polymethyl-(hydroxymethyl)acrylate, polychloromethylstyrene, polyvinyl -amino acids, cellulose and silica;
- 2) Core-shell particles composed of cores from micron-sized monodispersed polystyrene particles and shells from different metals or inorganic materials (e.g. gold, silica, titania and magnetite).
- 3) Non-magnetic and magnetic silica hollow micron-sized particles composed of sintered silica nanoparticles.
- 5) Supported nanoparticles prepared by covalent binding in a monolayer structure of functional nanoparticles, i.e. polyacrolein, onto solid substrates such as glass and polystyrene.
- 5) Template nanoparticles based on polystyrene have been used for preparation of other uniform functional nanoparticles, e.g. PMMA, polyvinyl alcohol, etc, and for in situ surface modification with functional vinylic monomers such as polyacrylic acid, polyacryl amide, etc. This technology is also used for encapsulating into the template particles different materials such as a variety of fluorescent dyes.
- 6) Biodegradable, non-toxic, magnetite (Fe_3O_4) nanoparticles of very narrow size distribution in sizes ranging from approximately 20 nm up to 0.1 μm . The process for preparation of these monodispersed magnetic nanoparticles is simple and the yield of the particles is almost 100%. These particles are superparamagnetic, i.e. they magnetized in the presence of a magnetic field, but no remanence is observed in the absence of a magnetic field.

The functional groups of the particles have been used for covalent binding, via different activation procedures, of ligands such as drugs, proteins, enzymes, antigens and antibodies to the nanoparticle surfaces. The non-conjugated and the conjugated particles were then used for applications such as imaging, catalysis, biocatalysis, specific cell labeling and cell separation, diagnostics, affinity chromatography, specific blood filtration by hemoperfusion, heavy metal ions detoxification and carriers for oligonucleotide synthesis and various bio-chemical reactions.

The dielectric relaxation in complex materials

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The presented contribution is centered around the area of condensed matter physics for investigation of the structure, dynamics, and macroscopic behavior of materials which can be characterized as *complex systems (CS)*. "Complex" here refers to the fact that the systems possess at least one degree of freedom beyond the usual translation one in "simple" fluids and/or non-crystalline solids. CS are a very broad and general class of materials which include associated liquids, polymers, biomolecules, colloids, etc.)

CS involve the appearance of a new ("*mesoscopic*") length scale, intermediate between molecular and macroscopic. Fast as well as ultra-slow molecular rearrangements take place in the presence of the microscopic, mesoscopic and macroscopic organization of the systems. The complete characterization of these relaxation behaviors requires the use of variety techniques in order to span the relevant ranges in frequency. To obtain this information, noninvasive methods such as Dielectric Spectroscopy (DS) is very advantageous. The unique technique with wide frequency (10^{-3} - 10^{11} Hz) and temperature (-170 °C ÷ $+300$ °C) ranges of that method is more than any others appropriate for such different scales of molecular motions.

The dielectric relaxation behavior in porous glasses for example can provide the information on the geometry of pore size distributions, fractal dimensions of the porous matrixes and the mesostructural features of water which can easily be absorbed on silica layers. The obtained information about the cooperative relaxation processes of water molecules absorbed in the confined volumes allowed us to understand the molecule structure and properties of water absorbed on the pores' surface and determine the morphology and structural transitions in the porous systems.

The complex dielectric behavior of the studied porous glasses versus frequency and temperature can be described in terms of the four distributed relaxation processes. For a quantitative analysis of the dielectric spectra for the first, second and third processes a formula of superposition of Havriliak-Negami (HN) and Jonscher's empirical term $(i\omega)^{(n-1)}$ has been fitted to the isothermal dielectric loss data. In the case of the fourth process the superposition of the HN function and a conductivity contribution was used.

The study of the porous glasses leads to several new features in their dielectric behavior: (i) The dielectric relaxation of porous glasses is very sensitive to the cooperative dynamic of water molecules filling the pores and geometrical micro- and mesostructural features of the porous matrix; (ii) The dielectric relaxation behavior of different porous glasses is associated with the complex dynamics of water molecules and reflects their interactions with the pores surface; (iii) Analysis of the dielectric spectra of the porous glasses enables us to calculate the fractal dimension and porosity of the materials.

DOMAIN ENGINEERING FOR NONLINEAR OPTICAL DEVICES

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New generation of quasi-phase-matched nonlinear optical devices allows to develop laser sources in spectral regions where conventional lasers are unavailable. It is based on engineered micrometer-scale ferroelectric domain configurations and represents a new concept in material science. Applied technique exploits tensoric properties of ferroelectrics where specifically designed domain structure with periodically inverted spontaneous polarization provides needed alternative change of sign of nonlinear optical coefficient.

Ferroelectric LiNbO_3 , LiTaO_3 , KTP and isomorphic crystals are mainly used due to their high nonlinear optical coefficients and stable built-in domain structure. Detailed studies showed those fundamental processes of minimization of depolarization field, domain nucleation, anisotropy of domain walls velocity determine quality of tailored domain configurations and strongly affect parameters of optical converters.

Studies of dielectric spectroscopy, DC conductivity, polarization switching, peculiarities of ferroelectric domain propagation in KTP and isomorphic crystals allowed developing a method of fabrication of periodic domain structures. It is based on low temperature polarization switching and use a phase transition from superionic to insulating state in flux-grown KTP and family crystals.

MULTIFUNCTIONAL NONOXIDE CERAMIC LAMINATES

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Multifunctional $\text{Si}_3\text{N}_4/(\beta\text{-SiAlON}+\text{TiN})$ layered composites were prepared by tape casting and subsequent hot pressing. The bending strength and fracture toughness of layered materials measured in direction normal to the layer interface were substantially greater (by 82 and 107%) in comparison to the bulk $\beta\text{-SiAlON}+\text{TiN}$ composite. High anisotropy was achieved for the electrical resistance of the layered materials in parallel ($6 \cdot 10^{-2} \Omega\text{cm}$) and perpendicular ($5 \cdot 10^{11} \Omega\text{cm}$) direction to the layer alignment.

Layered material design and in situ reactions were used for the preparation of $\text{SiC}/(\text{Ti-Nb-C-Al})$ laminated composites. While the layered structure and the addition of 5 vol% $\beta\text{-SiC}$ seeds contributed to the improvement of fracture toughness of this rather brittle composite, the reaction sintering allowed to combine materials with high melting point. The fracture toughness of TiC-based layer was increased by metallic binder.

**MATERIALS REQUIREMENTS FOR LIFE STEAM TEMPERATURES
UP TO 700°C**

**Erich Tolksdorf
VGB Headquarters Essen, Germany**

The current tendency toward increasing efficiency of fossil fired power stations require higher life steam temperatures and hence materials to withstand this under long term application. Up to 650°C the developments aims at ferritic/martensitic steels with sufficiently high creep properties, however, stainless steels or Ni-base alloys are required at 700°C. The currently available materials, their properties, and damage development under long term high temperature service will be discussed. The main properties of interest are the creep and corrosion properties. The 9-12%Cr-steels under development or those which are commercially available present sufficient creep properties, however lack corrosion resistance when compared to X20CrMoV 12 1 steel. At even higher temperatures, i.e. 700°C only Ni-base alloys like IN 617 are sufficient resistance, however, may have some high temperature corrosion problems when exposed to sulfur containing flue gas.

**THE MECHANICAL BEHAVIOR OF CERAMIC/METAL
LAMINATES UNDER THERMAL SHOCK LOADING**

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A new material system for applications involving thermal shock is proposed. The system consists of thin layers of ceramics and thinner metallic interlayers. The dominant behavior is the absence of interaction between the biaxial cracking mechanisms in an individual layer, and localization of the damage to those layers experienced sufficient tensile stresses. The result is a dramatic increase of the residual strength after thermal shock. In addition, R-curve behavior upon mechanical loading due to plastic deformation of the metallic interlayer was observed.

In this study, the ceramic/metal laminate was constructed from Coor's ADS96R thin plates alternating with thinner Wesgo Cusil ABA interlayer foils and joined in active brazing. The laminates were quenched at their bottom surface only in a specially designed apparatus.

The residual strength was evaluated in three point bending. Thickness effect is described, as well as damage evolution and plastic deformation of the metallic interlayers.

CARBON/CARBON COMPOSITES WITH RANDOMLY ORIENTED REINFORCEMENT

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Mario Gerchovich, Israel Engelberg
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The main applications of Carbon/Carbon (C/C) composites are in aircraft break pads and in missiles hot parts. The expensive C/C types used in these systems are either bi-directionally or multi-directionally reinforced materials. The aim of the present research was to develop C/C composites randomly reinforced by chopped fibers, densified in cost-effective processes, to achieve properties satisfactory for aerospace systems. The full study will include few composition parameters. Among them: the fiber type, its length and its content (volume/weight fraction), the matrices content (glass-like carbon from resin precursor, pitch precursor polycrystalline graphite, or Chemical Vapor Infiltrated-CVI carbon or a combination of those). Another variable is the carbon additive (filler) type and content. Several types of densification processes are applied, as well as heat treatments and the Graphitization. All these processes were developed in RAFAEL for bi- and multi-directional C/C composites, and adapted to the processing of random composites. The evaluation of the composites will be based on the mechanical properties, such as the strength and the modulus in flexure and compression. Other quality indicative properties parameters include the voids content, the hardness and the wear rate. The strength and moduli values expected in random C/C composites with chopped fibers are significantly lower than the obtained with the continuous bi-directionally reinforced C/C composites. Hence, any result in the same scale of order will be satisfactory for most applications. Preliminary studies have already revealed strength and moduli results comparable to those of a bi-directionally reinforced C/C. The study is still running and further results are expected.

**THE EFFECT OF THE STRESS INTENSITY FACTOR (ΔK)
ON THE MORPHOLOGY OF THE FRACTURE SURFACE
DURING FATIGUE-CRACK-GROWTH (FCG) STUDIES
OF A POLYCRYSTALLINE SUPERALLOY**

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As part of studying the effect of prior high-temperature creep on the resultant microstructure and resistance of a polycrystalline, precipitation-strengthened nickel-base superalloy to FCG, we examined the morphology of the fracture surface during FCG. Depending on the level of ΔK , there was a continuous change in the morphology of the fracture surface. Starting with intragranular cleavage at low ΔK values, continuing with intragranular fatigue striations, then moving into mixed intragranular and increasing intergranular fracture, while becoming totally intergranular fracture at high ΔK values. Microstructural changes which took place during prior creep deformation, such as rafting of the initially cuboidal precipitates and separation along grain boundaries, did not seem to affect the existence of the aforementioned 4 zones of fracture morphologies.

SOLID STATE REACTIONS IN BN-Ti SYSTEM

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Boron-nitride (BN) is increasingly used in a variety of industrial applications, including high temperature applications in direct contact with metals. Among all the metals, the reaction between Ti and BN is of great interest due to the high affinity of Ti for both boron and nitrogen, and the stability of the resulting titanium borides and nitrides. The primary goal of the research was to establish the phase composition and morphological features of the reaction zone formed at BN/Ti interface after annealing at 1000 - 1200°C. The main experimental configuration employed was a BN sample immersed inside Ti powder and annealed under high vacuum. Phase identification and characterization was performed using XRD, SEM, EPMA, HRSEM, TEM and PEELS. Annealing hexagonal and cubic boron nitride samples in Ti powder at 1000 - 1200°C in vacuum was found to result in the formation of thick multi-layer coatings on the ceramic surface composed of titanium borides - TiB_2 , TiB , and nitrides - TiN , Ti_2N , α - $Ti(N)$. Phase formation and morphological features of reaction layers at BN/Ti-powder interface are discussed and compared with conventional BN/Ti diffusion couple. Kinetic and thermodynamic considerations are used to explain the obtained results.

THE SHS REACTION IN Al-Ni MULTILAYERS

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The SHS (Self-Propagating High Temperature Synthesis) reaction is a self-contained exothermic combustion reaction, that generates heat fast enough to be self-sustaining. The excessive heat, generated in the SHS reaction may be used for joining materials, without heat input from an external source.

As a possible configuration we have examined multilayered, sputter-deposited Al-Ni foils as the reactive medium inserted between the parts to be joined. The Al-Ni foils were of various average compositions, total thickness and layer periodicity. The study consists of developing methods for automated sample deposition, for structural characterization and for flame velocity and reaction heat release measurements. These methods will be described, several results will be shown, and the joining potential of the Al-Ni multilayers will be demonstrated.

GRAIN BOUNDARY POROSITY IN NiAl INDUCED BY Cu DIFFUSION

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We studied the chemical interdiffusion of Cu and Ni-49 at.% Al alloy at different temperatures. No Kirkendall porosity was observed in the bulk diffusion zone, however, the formation of elongated pores at the grain boundaries was revealed. Temporal evolution of the pores size and morphology was investigated. The pores are generally connected with the original Cu/NiAl interface, but in some cases they migrated a substantial distance inside the NiAl thus disconnecting from the original interface. We also developed a theory of pore growth induced by the vacancy flux along the grain boundary and controlled by the surface diffusion mechanism. The calculated pore shapes are in a good qualitative agreement with the experimentally observed ones. It is shown that the maximal value of the grain boundary vacancy flux exists above which the steady-state inward migration of the pore is not possible and a break-up of the pore occurs.

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Tuesday

Parallel Sessions: 4

PRESSURELESS SINTERING OF B₄C WITH TiO₂ ADDITIONS.

L. Levin, N. Frage, and M.P. Dariel

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Covalent bonding prevails in boron carbide and, consequently, pore eliminating mass transport sintering mechanisms such as grain boundary and volume diffusion become effective only at elevated temperature, close to the melting point. The addition of TiO₂ improves the sintering behavior of B₄C. Thus the addition of 40 wt.% TiO₂ to B₄C powder with 17 m²/g specific area allows reaching 95% relative density after sintering for 1 h at 2160 °C. The striking improvement of the sintering behavior of B₄C by TiO₂ additions is attributed to the formation of sub-stoichiometric boron carbide. Departures from stoichiometry presumably induce changes either by altering the crystal structure or by introducing structural defects that promote mass transport. Alternatively enhanced densification could be attributed to an evaporation-condensation mechanism caused by increased evaporation rates of boron from the carbon-lean carbide particles. Metallographic examination of the compact sintered at 2160°C revealed a two-phase microstructure with 10 μm B₄C grains. The *in situ* formed TiB₂ are of the order of 5 to 7 μm and act as very efficient boron carbide grain-growth inhibitors. Higher density affects positively the mechanical properties as evidenced by the 420 MPa bending strength reached in the compacts sintered at 2160°C.

DENSIFICATION PROCESSES OF SINTERED MOLYBDENUM

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A process for obtaining nearly full density powder metallurgy derived Molybdenum products is described. Since it is not possible to achieve full density in pure Molybdenum by pressureless sintering process, other densification processes must be implied following the sintering.

Raw powder was cold compacted and sintered in two stages to 93% of TD. HIP process was utilized to increase the density to 99.4-99.8% of TD. Plastic deformation of the as sintered material was also examined as a densification increase method.

Mechanical properties were studied before and after hipping. The elongation increased from practically zero in the as sintered condition to about 9% after hipping. The ultimate strength increased by about 10% due to hipping at 1500°C.

As sintered specimens were hot rolled. The dependence of the bending angle on the amount of reduction in the thickness was studied. After 90% rolling reduction, a bending angle of 180° without cracking was feasible.

Density changes of as sintered specimens as a function of forging to various reductions were studied. 34% and 57% reductions yield density of 97.4% TD and 99.4% TD, respectively. The elongation after 60% reduction increased to about 5%, while the ultimate tensile strength is 22% lower than the hipped specimens value.

TEM EXAMINATION OF THE INTERPARTICLE CONTACT AREA IN
SILVER COMPACTS AFTER ACID-ASSISTED CONSOLIDATION

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Surprisingly elevated stiffness and strength values were observed in unsintered silver powder compacts that had undergone acid-assisted consolidation. This material is considered as a viable mercury-free dental restorative material to conventional amalgams. These results were attributed to the surface-oxide removing potential of the acid that enabled some cold-welding to take place between adjacent particles. In order to gain additional insight into the phenomenon on a microscopic scale, acid-assisted silver compacts were prepared for TEM examination. The results revealed the presence of nanometric silver particles in the interparticle voids. These observations suggest that 'dissolution-precipitation' in the liquid acid medium which leads to some mass-transport may be responsible for the strengthening effects found in the acid-treated silver compacts.

FORMING TECHNOLOGIES FOR P/M ALUMINUM ALLOYS

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The search of weight reduction in automotive and aerospace engineering leads to increased demands that cannot be fulfilled with conventional ingot metallurgy (I/M) aluminum alloys.

Powder metallurgical (P/M) aluminum materials offer improved mechanical and thermal properties that result from the alloy composition and the rapid solidification of the melt. The alloys can be tailored to specific application demands like high strength, high temperature stability, low thermal expansion or high wear resistance. Forming technologies like the extrusion of rods produce materials with full density from previously spray deposited billets or atomized powder.

The forming of parts from extruded rods appears to be the most promising technology for industrial scale production. For the layout of appropriate forming processes the specific properties of the P/M aluminum alloys such as increased flow stress and reduced formability were considered.

The feasibility of forming P/M aluminum alloys was investigated by cold and warm forging of parts from extruded grades. Pistons and connecting rods were forged with temperature controlled dies in order to achieve reproducible forming conditions. The precision forging of connecting rods demonstrates the feasibility to produce components with complex geometries, improved surface and reduced flash-volume-fraction from dispersion strengthened P/M aluminum alloys. FEM simulations of the forming processes provide essential information on the optimization of the process layout and the manufacturing conditions.

ENERGY DISPERSIVE X-RAY FLUORESCENCE USING MICRO-BEAM EXCITATION (μ -XRF)

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The elemental composition of small areas or individual particles has conventionally been determined using electron beam EDS methods. The concept of using X-rays as an alternative energy source is of great interest because it offers several advantages over the use of electrons.

Recent developments in capillary optics provide an efficient method of transforming the wide-angle beam emergent from a conventional (low power) X-ray tube into a high intensity small diameter beam suitable as the primary excitation source for XRF applications. Beam diameters in the range of $<100\mu\text{m}$ are possible which in turn provide the possibility for elemental analysis having lateral resolutions in the region of $30\mu\text{m}$.

Thus the opportunity to perform applications similar to electron beam microanalysis techniques is achievable, albeit not with the same ultimate spatial resolution, but with the following advantages:

- Improved sensitivity. The absence of Bremsstrahlung gives a reduced spectrum background resulting in improved limits of detection.
- No special sample preparation methods or correction methods needed for electrically non-conducting materials.
- Zero damage to electron-beam sensitive materials.
- Possibility to measure samples in air, enabling wet or biological samples to be analysed, or even liquids.
- Analysis of deeper layers in the sample is possible due to the higher penetration of incident X-rays compared to incident electrons.

The high sensitivities and spatial resolutions now possible offer many new application opportunities for the already versatile XRF technique. For example, now it is possible to isolate small particles and inclusions from their surroundings and to produce elemental distributions.

This paper will describe the features of the instrument and discuss its analytical performance for a range of applications including:

- Forensic applications (glasses, ink pigments, pigment distribution methods to reveal erased script)
- Metal samples (alloy segregation, small particles)
- Printed Circuit Boards (element distributions for components and coatings)
- Distribution analysis and mapping of biological materials

With the μ -XRF technique a new dimension is added to the already mature energy-dispersive XRF analysis technique.

GRAIN BOUNDARIES IN GaN AND THEIR IMPACT ON ELECTRICAL CONDUCTIVITY

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Technological breakthroughs in GaN doping and contact technologies have resulted in numerous devices, including the blue GaN-based laser. Unfortunately, GaN films are typically grown on substrates to which they are both lattice and thermally mismatched. As a result, even device grade GaN films possess a high density of grain and sub-grain boundaries, dislocations, as well as numerous point defects. This results in a characteristically large density of electronic gap states, which affect device performance. In particular, electrical transport in thin GaN layers exhibit anomalous behavior which include: *i)* persistent photoconductivity; *ii)* a large UV photo-response gain; *iii)* an increase in electron mobility with increasing doping; and *iv)* a large optical gain regardless of the defect density.

One could consider that the columnar microstructure of GaN thin films can lead to a transport mechanism similar to that used to model Poly-silicon conductivity^{1,2}. According to this model, each boundary poses a potential barrier, the height of which is the main parameter affecting the transport. This model of grain boundary controlled transport (GBCT) may inclusively account for all the above mentioned observations.

In this work, we study the applicability of the GBCT model to thin GaN layers. The GaN layers for this work were produced by Organometallic Vapor Phase Epitaxy (OMVPE) on sapphire. Optimization of the growth process for obtaining high quality samples was performed by a statistical multi-parameter design of experiments³. After growth, the background doping of the samples was varied in a well controlled way by the compensation effect of radiation damage (ion implantation), and the sample resistivity and its temperature dependence vs implantation measured.

The GBCT model predicts a non-linear functional dependence of the electrical conductivity on implantation dose. We present experimental results of ion implantation (H^+ , He^+) with increasing doses, and conductivity measurements showing a dramatic increase of 10 orders of magnitude in the resistance in the implanted samples. The measurements closely follow the prediction of the model.

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MOCVD GROWTH OF GALLIUM NITRIDE ON Si SUBSTRATES

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GaN is a wide, direct band gap (3.4 eV) semiconductor, useful to manufacture LEDs and Laser Diodes emitting in the range of blue and UV, and solar blind UV detectors. GaN is grown mainly on sapphire substrates. The growth of GaN on Si substrates will allow the integration of Si mature electronics and GaN photonic devices. There are some serious problems in the growth of GaN on Si substrates: significant lattice mismatch (~ 17%), large thermal expansion mismatch (~ +56%), as well as problems related to the growth technique.

In this work we succeeded to grow 1 μm thick GaN layers on 2" Si(111) wafers, by MOCVD at ~1000°C. We found that using a high temperature (>1000°C) nucleation layer of AlN and avoiding any precleaning inside the reactor improves the GaN layer quality.

As grown layers exhibit intense spontaneous band to band emission (at 365 nm) and strong basal plane preferred orientation, the latter giving rise to rather narrow (0002) X-Ray diffraction peaks (FWHM = 600 arcsec in best samples).

A model relating the crystalline quality of the film to the AlN growth temperature is proposed.

CZOCHRALSKI GROWTH AND CHARACTERIZATION OF STOICHIOMETRIC AND SLIGHTLY OFF-STOICHIOMETRIC PbTe SINGLE CRYSTALS

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PbTe is the best known $A^{IV}B^{VI}$ compound semiconductor. The elevated electron mobility in the heavily doped state and the low lattice thermal conductivity of these compounds make them useful for thermoelectric energy-related applications.

In our study, single PbTe crystals of stoichiometric composition and containing excess of Te (*p*-type) or excess Pb (*n*-type) were grown by the Czochralski technique. Different process parameters were examined in order to optimize the growth process. These included the thermal gradient, the growth velocity, the rotation rate, the pressure and the composition of the melt. Crystals of up to 100 mm length and 30 mm in diameter were grown under a variety of growth parameters. The crystals were examined with regard to electrical homogeneity over their length using the 'hot probe' technique. The relevant thermoelectric transport properties (Seebeck coefficient, electrical and thermal conductivity) of the best crystals obtained were investigated over a wide temperature range up to 900 K.

**A NEW APPROACH
TO
THERMODYNAMICALLY STABLE CONTACTS
FOR
BINARY WIDE BANDGAP SEMICONDUCTORS**

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One of the main bottlenecks in the design of high-temperature electronic devices, based on SiC or GaN, is the thermodynamic stability of the substrates with the contact metallizations. "Silicide-like" contacting technology, as is widely used in silicon devices, would be highly desirable, if feasible. However, for various reasons such a technology is still lacking for binary semiconductors.

In this work, a new approach is suggested, where a bi-metal contact is constructed. In this approach, each metal preferentially reacts with another component of the semiconductor, leading to a thermodynamically stable sequence of metallic layers. This approach is studied on several systems: <SiC>\Ti\Co, <SiC>\Ti\Pt, and <GaN>\Ti\Pt. For concept demonstration, we show preliminary results obtained at the <SiC>\Ti(30nm)\Pt(30nm) system. Depth profiles and phase identification were made using x-ray photoelectron spectroscopy on as-deposited and annealed samples (500 to 900 °C for 1h duration). Crater edge Scanning Auger Microscopy was used on the same samples.

GaN LAYER GROWTH OPTIMIZATION FOR HIGH POWER DEVICES

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GaN and related alloys are considered a future material for high power electronic devices. Many laboratories around the world are investing enormous efforts to optimize GaN layers. Different research groups explored the influence of individual growth parameters on a single, or a pair of layer properties. These experiments are mostly based on the change of one growth parameter at a time¹⁻³, although it is clear from kinetic modeling that growth parameters are inter-related⁴. The impressive progress in the layer quality in many laboratories can not hide the fact that epitaxial GaN films are still far from optimal, and more efforts are needed to improve the material quality. Surprisingly, we found no report of a comprehensive, statistical multi-parameter design of experiments for growth and optimization of GaN and related materials.

Statistical multi-parameter design of experiment (DOE) is relatively common in the semiconductor research and development⁵. The DOE methodology is used to plan a group of experiments in which specified changes are made simultaneously to several process inputs in order to establish potential cause-and-effect relationships between **process inputs** and **process outputs**. Design of experiments can be used to efficiently characterize, improve and optimize a process, establish sensitivity of the response to the different factors, and find the factors that have the greatest impact on process performance. The effect of one factor at different levels of another factor (factor interaction) can be estimated systematically. The model can be represented as a polynomial regression model, which is a truncated Taylor series expansion.

This work presents a statistical multi-parameter design of experiment. The layers were grown by low-pressure metal organic chemical vapor deposition (MOCVD), in a vertical GaN reactor. The layer parameters (output variables) that were optimized are X-ray diffraction rocking curve full width at half-maximum (FWHM), background doping concentration, and Hall mobility at room temperature.

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³ S. Keller et al.; *Jpn. J. Appl. Phys.* **35** (1996), p. L285

⁴ D. D. Koleske et al.; *J. Appl. Phys.* **84** (1998), p. 1998

⁵ R. L. Mason et al.; *Statistical Design and Analysis of Experiments with Application to Engineering and Science* (1989), John Wiley and Sons, Inc.

**COMBINED MODELLING OF AGGREGATION OF SILICA IN
PROCESS OF SILICA SEDIMENTATION, USING MONTE-CARLO
APPROACH WITH THERMODYNAMIC LIMITATIONS**

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Modeling of silica aggregation in solid phase during process of silica sedimentation from aqueous solution is very complex problem, because of difficulties of simulation of formation of the branched and cross-linked silica aggregates. Traditional method of simulation based on the analogy with macromolecules does not work for such complex structures. Numerical methods (Monte-Carlo) do not work but for random systems.

The problem of modeling of formation of multi-unit branched cross-linked structures had been solved recently by new approach – the statistical polymer method. This approach has been tried for some systems, comprising silica gel. The results of calculations are in very good correlation with experimental data. However, for discrete systems with limited number of elements (corresponding to the start of sedimentation) such thermodynamic approach is not enough appropriate.

For the correct description of the starting stage of silica sedimentation we have proposed the combination of Monte-Carlo approach with the statistical aggregate method. The obtained results allow modeling of the process of cluster growth and estimation of several parameters of the obtained silica structure.

MOLECULAR DYNAMICS STUDY OF STRUCTURE AND DIFFUSION
IN A MOLTEN ALUMINIUM SURFACE

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We present the results of a molecular dynamics study of atomic surface diffusion and structure in molten free aluminium surfaces in the temperature range: 700K-1500K. The model system is composed of a slab with a free surface on one side, and a portion of a rigid solid plus a semi-infinite "virtual" bulk on the opposite side, and periodic boundary conditions in the other two directions. We simulated systems with up to 1944 atoms, using a manybody type of interatomic potential, fitted to aluminum properties. Four different orientations of the crystalline surfaces were considered: $[100]$, $[111]$, $[0\bar{1}1]$, and $[\bar{2}11]$.

The diffusion coefficients were calculated from the long-time dynamics of the atomic mean square displacements. The behavior of the diffusion coefficient parallel to the surface was studied as a function of distance from the free surface. The atomic disorder at the surface was evaluated via the static structure factor and the radial distribution function, which were obtained as functions of distance from the surface. The results for the various orientations and temperatures are compared and discussed.

Microstructure of the relaxed (001) Si surface

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We have applied molecular dynamics method and semi-empirical potential [1] to obtain the realistic picture of Si surface layers relaxation.

The starting configuration was taken as a parallelepiped containing 864 atoms. There were 12 layers with 72 atoms in each one. Periodic boundary conditions were used in two dimensions.

Investigation of full Si surface relaxation leads to structural defects and dangling bonds arising as it was shown in the case of amorphous Si modeling [2].

The result is that during relaxation a rebuilding and a re-hybridization of chemical bonds take place. Such procedure leads to arising of non-usual polygons with $n=3-9$ and nodes with 1 – 3 electrons that do not participate in chemical bonds creation.

Thus there are nodes with more than one dangling bond. Computer calculations allow obtain the radial distribution function (RDF) and the angle distribution function (ADF) (the angle between chemical bonds directions are implied).

The chief conclusion is that a quasi-disordered phase (QDP) arises as a result of free Si surface relaxation. The RDF and ADF are similar to those for a-Si. Each of upper four layers has his specific structural characteristics. The third layer is the most “perfect” one.

Only this layer in a surface QDP is without nodes that have two dangling bonds. But at the same time the third layer is the most distorted. Atoms of this layer are characterized by the largest displacement with reference to their normal position in the crystallographic plane. Thus the third layer in surface QDP plays a special transitional role between the crystal volume and the surface QDP.

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**CONDUCTIVE POLYANILINE/POLYMER BLENDS THROUGH
AQUEOUS DISPERSIONS: SYNTHESIS AND
CHARACTERIZATION**

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The applicability of ICP's (Intrinsically Conductive Polymers), such as polyaniline (PANI), is restricted by their poor processability and mechanical properties. PANI can be processed without altering its structure by melt blending with some conventional polymers. These blends are designed to combine the desired properties of both components, i.e., electrical conductivity of PANI along with processability and properties of the matrix polymer.

Recently, a unique process of polymerization in an aqueous dispersion of anilinium-Dodecyl Benzene Sulfonic Acid (DBSA) complex was developed in our laboratories. Conductive blends of PANI-DBSA dispersions with other aqueous emulsion polymers (e.g., PVAc, polyacrylates, PS and XPS) were prepared by simple mechanical mixing of the latices. The PANI-DBSA percolation threshold of the dried blends was extremely low (about 0.5 %wt).

PS/PANI blends exhibit a segregated very fine PANI dispersion within the PS matrix. The PANI-DBSA behavior is of a unique "polymeric filler" with very high surface area and strong interaction with the matrix, evidenced by a significantly higher glass transition temperature of the matrix. The conductivity of PANI-DBSA/polymer blends prepared by the present method was found independent of the type of the matrix polymer used. This unique behavior was analyzed and explained.

The research was conducted under the supervision of Professor Moshe Narkis.

**TOUGHENED POLY(BUTYLENE TEREPHTHALATE) / MBS
BLENDS COMPATIBILIZED BY EPOXY RESIN**

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In the development of new polymeric materials, multiphase polymer blends are of great importance. Toughness of poly(butylene terephthalate) (PBT) was greatly improved by in-situ reactive extrusion of PBT and MBS (methyl methacrylate-butadiene-styrene copolymer) with high-molecular-weight epoxy resin as reactive compatibilizer. Mechanical properties, rheology, morphology and thermal properties of these blends were investigated. We explored the correlation between properties and structure of this blend as well.

Results indicated that both of epoxy resin and blending steps play an important role in improving the properties of PBT/MBS blends. Best results in terms of toughness/tensile strength balance, with impact strength exceeding 600 J/m (20 J/m for neat PBT), was achieved when PBT was blended in twin-screw-extruder with both MBS and epoxy resin compatibilizer. The outstanding performance of such PBT blends, containing 15% and 20% MBS and 0.8% ~ 1.2% high-molecular-weight epoxy resin compatibilizer, respectively, was attributed to interfacial coupling of epoxy resin to the PBT/MBS blends.

INTERFACES MODIFICATION AND CHARACTERIZATION IN THREE-COMPONENT POLYMER BLENDS

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Much of today's high performance composite technology is based on interfaces investigations. The effect of interface characteristics on the properties of three-component polymer blends comprising PP/EVOH/glass beads (GB) was investigated (polypropylene - PP, ethylene-vinylalcohol - EVOH). The system selected is based on the binary PP/EVOH immiscible blend representing a semi-crystalline apolar polymer (PP) and a semi-crystalline highly polar copolymer (EVOH), where PP serves as the matrix. The structures observed by SEM analysis consisted of the filler particles engulfed by the EVOH phase, with some of the minor EVOH component dispersed within the PP matrix. The effects of silane treatment (GB/EVOH interface) and compatibilization, using a maleated-PP compatibilizer (PP/EVOH interface), were studied in relation to the generated structures and properties. The compatibilizer was added in a unique procedure by which the encapsulated GB/EVOH structures were preserved. The characterization methods used included morphology by Scanning Electron Microscopy, thermal properties and crystallization behavior by Differential Scanning Calorimetry, shear viscosity by capillary rheometer, mechanical properties by tensile testing and dynamic characteristics by Dynamic Mechanical Thermal Analysis. The work done has shown that structure-performance relationships in the three-component blends can be varied and controlled.

Acknowledgement

The partial support of the Lower Saxony Ministry of Science and Culture, and the Fraunhofer Institute of Chemical Technology, Germany is gratefully acknowledged.

THERMO-ELECTRIC BEHAVIOR OF PVDF/UHMWPE/CARBON-BLACK IMMISCIBLE POLYMER BLENDS

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The electrically conductive properties of various polymer systems containing conductive fillers such as carbon blacks, carbon fibers and metallic powders has been extensively investigated. An interesting phenomenon, exhibited by some conductive polymers, is a PTC (positive temperature coefficient) effect. The main feature of PTC materials is that upon heating, the conductive polymer shows a sharp resistivity increase near the melting region of the semi-crystalline polymer matrix. The PTC effect is sometimes followed by an NTC (negative temperature coefficient) effect, namely, a resistivity decrease. PTC materials can be used as self-regulating heaters, current limiters, over-current protectors, etc. From a practical point of view, the NTC effect is undesired and should be eliminated, and the material must have electrical reproducibility upon heating and cooling cycles.

Carbon black (CB) filled polyvinylidene fluoride (PVDF) / ultra high molecular weight polyethylene (UHMWPE) blends were prepared by melt mixing. This system represents an immiscible polymer blend with preferential location of CB at the interfaces. This unique structure induces a reduced critical amount of CB necessary to build-up a conductive network. The PVDF/UHMWPE/CB system depicts a double-PTC effect, followed by an NTC effect. Crosslinking of the dispersed phase (UHMWPE particles) by γ -irradiation, prior to melt mixing, modifies the system's morphology and increases both PTC and NTC effects. Irradiating the whole system diminishes the undesired NTC effect by significantly reducing the freedom of movement of the CB particles upon melting/crystallization cycles, which also greatly improves the electrical reproducibility.

The research was conducted under the supervision of Professor Moshe Narkis.

IMPACT MODIFICATION OF HDPE FOR INJECTION MOLDING

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It is well known that the impact resistance of HDPE is steady within the temperature range of -40 to $+40$ C, compared to Polypropylene compositions.

On the other hand, the disadvantage of HDPE high-flow injection grades is the comparatively low impact resistance at ambient temperature as compared to new PP grades (especially to new High Impact and Super High Impact Resistant Heterophasic Block Copolymers)

The impact modification of injection molding grades of HDPE is presented in this lecture.

INTELLIGENT PROCESSING OF THERMOPLASTICS BY INJECTION MOLDING

S. Kenig - Israel Plastics & Rubber Center

Injection molding (I.M) is among the most widely used techniques for processing of thermoplastics. From the transport phenomena point of view the process is very complicated, composed of unsteady state mass, momentum and energy transfer. Over the years, commercial software packages became available for calculating the various stages in I.M. As far as the process control is concerned, state of the art I.M. machines have close loop controls, where microchips are used for preprogramming of the process variables and accordingly control them. However, since the process variables are not absolutely constant during processing due to changes in machine performance and materials properties variations, the need for a more intelligent processing operation is needed as demands for in-line quality assurance is of primary importance for technical applications. The more advanced intelligent I.M. process should comprise of an adaptive process control where changes in materials and processing conditions could be monitored and consequently by the intelligent system, the process variables would be changed to yield accepted product with predefined specifications.

The intelligent system which is under development by the Israel Plastics & Rubber Center and Rafael and funded by the Ministry of Science, uses in line measurements of pressure profiles in the cavity, temperatures in various parts of the system, velocity of injection etc. An artificial neural network (ANN) is used to process the various parameters. The A.N.N possesses the basic requirements for an intelligent system like: self teaching, learning ability, on line process variable measurements and on line adaptive control of the I.M. process to meet predefined product specifications.

Process-Properties Relationship in Styrenic Polymers

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BASF was the first company to commercialize polystyrene in 1930 and is today the world's second largest producer of this polymer. Although the life cycle curve has flattened, a variety of new developments in styrenic polymers has contributed to product-"relaunch" in many cases. Basis for many of these innovations is the anionic polymerization.

In contrast to the free radical polymerization the anionic process allows to a much wider extent the control of molecular architecture. By introducing a tapered styrene/butadiene transition into the well known S/B/S block copolymers we were able to increase the phase volume ratio of "soft" and "hard" phase while maintaining a high overall styrene content, resulting in improved stability at high impact values (Styrolux®).

Adapting a similar concept to thermoplastic elastomers yields Styroflex®, a material displaying elastomeric properties at a butadiene-content of not more than 35 %. This unique feature results from the incorporation of styrene into the butadiene "soft" phase by statistical copolymerization.

In the last part of the lecture we present a new process, developed by BASF, the "retarded anionic polymerization".

MOLDING AND COMPUTERIZED SIMULATION OF STRETCH PROCESSING FOR PLASTICS MATERIALS

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Plastics materials processing by stretching is a wide-spread process for manufacturing of hollow containers (Blow molding) and large surface area products (Thermoforming). Stretch processes have technical and commercial advantages compared to other processes such as injection molding, due to the lower cost needed for tools and machines.

Molding of the Stretch process requires the knowledge of the material behavior under extensional stresses. Therefore it is necessary to use an adequate model which describes the dependence of stress strain during the stretching process. It is worth while to point out that effects such as strain induced crystallization and orientation have dominant influence on the stretch forming process.

In the presentation, the virtual design of products by CAD simulation of Blow molding and Thermoforming will be demonstrated. The software integrates pertinent rheological models (K-BKZ) and allows for optimizing the thickness distribution as function of process condition and product geometry using the materials properties and their dependence on strain rate and temperature fields. Moreover, the simulation enables the calculation of the cycle time and the draw ratio at each point on the product.

MICROWAVE PROCESSING OF BULK POLYURETHANE
ADHESIVES AND BONDED JOINTS

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Microwave energy is being used for polymer drying, cooking, sintering and as a heating method in processing.

Adhesive bonding using microwave radiation is a new application which is evaluated in this research as an alternative curing method for polyurethane adhesives. This method was first tested on curing bulk polyurethanes and further on adhesive joints with glass or polycarbonate adherends. The studies showed that the required curing time is substantially reduced by the use of microwave energy compared to conventional oven curing method. Optimal microwave conditions (energy and time) resulted in high quality bonds with high adhesive strength, a homogeneous product, with reduced residual stresses.

FTIR, DSC and dielectric measurement were used to characterize the curing mechanism compared to conventional thermal methods.

Microscopic, optical and spectroscopic analysis were used to study the failure surfaces. Microwave processing proved to be a valuable alternative method for rapid uniform and selective curing of polyurethane adhesive joints at low input power levels.

CO-INJECTION ON A SINGLE BARREL MACHINE

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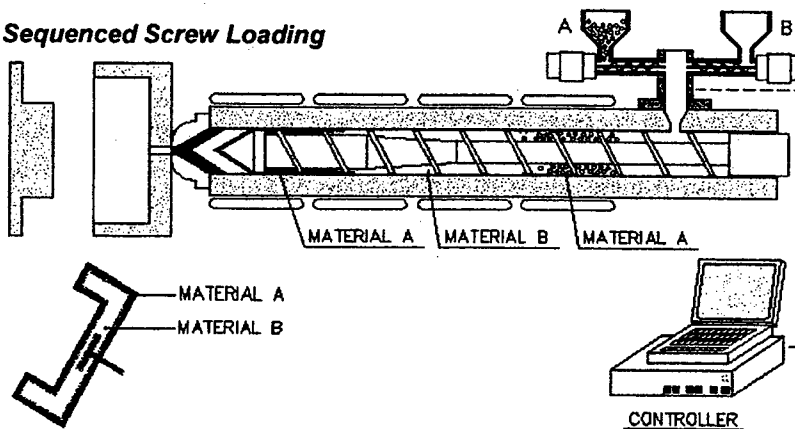
Tel: 0181 459 7477 Fax: 0181 459 7433

Website: <http://www.addmix.co.uk>

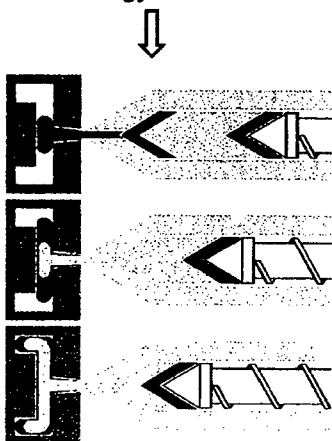
CONCEPT

By combining the ability to predict the formation of layers in mold cavities with knowledge of the transporting behaviors of a plastic injection molding machine screw, it is possible to produce co-injection molded components by feeding in a controlled manner two materials in a sequence to the throat of the barrel of an injection-molding machine.

Sequenced Screw Loading



Technology Based on



DESCRIPTION

The diagram above shows a computerized dosing system, attached directly to the feed throat of an injection-molding machine.

To achieve a co-injection molded component, different plastic granules, A and B are fed into the feed throat in a predetermined sequence. The material travels through the machine's barrel while still maintaining the feed sequence.

Molded component has a different skin and core, due to fountain effect taking place in the mold filling process.

Development of Smart Materials in NASA

Robert G. Bryant

During the past several decades, NASA has developed advanced materials and structural concepts for aerospace vehicles. These high performance materials and structures have met the objectives of creating advanced systems for lighter stronger load-bearing components. Recently, research at NASA has begun to focus on material systems that serve as structural components while providing an additional dynamic response to their surrounding environment. Material systems that behave in this fashion are termed "smart materials". NASA has developed several unique actuators and sensor systems that extend the performance of current piezoelectric materials. These devices have posed a challenge in terms of design integration and testing. This paper will outline current smart material developments and activities at NASA and also describe the forth coming activities into biomimetic research and technology goals for the aerospace industry.

DEVELOPMENT OF FUNCTIONALLY GRADED MATERIAL BASED ON HEAVILY DOPED PbTe<In> CRYSTALS

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Several methods for achieving high efficiency of thermoelectric conversion by increasing the figure of merit Z of thermoelectric materials over a wide temperature range have been put forward over the past years. One of these methods promotes the use of a thermoelectric "graded" material" with constant Z over a wide temperature range (100 – 600°C). Semiconductor compounds of type $A_{IV}B_{VI}$ (tellurides of VI group elements) are an advanced class of materials that display unique and valuable combinations of properties which are very useful in thermoelectric energy conversion. An increased, useful temperature range can be attained by eliminating the negative influence of the minority carriers in n -type PbTe, doped with indium up to 2at. %.

PbTe crystals were grown by the Czochralski technique. Large, 25-30 mm diameter crystals were grown at 2 cm/h growth rate and $\delta = 0.5$ 1/s rotation speed. Indium was introduced into the PbTe lattice from a gaseous phase. Indium atoms were transported into the PbTe crystal surface from a In_4Te_3 vapor source and the diffusion anneals were carried out in the 600 to 750°C temperature range for 25 to 200 hours long durations.

The dopant penetration into the semiconductor was determined by measurement of the Seebeck coefficient along the sample using a 5 μ m diameter "hot" probe. It was shown that an indium concentration ≥ 0.1 at % can be reached to a depth ≈ 3 mm. The figure of merit of samples with this thickness was measured over the 100 to 600°C temperature range. The results indicate a practically constant value of Z over the whole temperature range, supporting the initial premise that high efficiency energy conversion can, in principle, be realized with a well designed FGM structure.

EVALUATION OF AN OPTICAL FIBER FOR USE AS AN EMBEDDED
SENSOR IN A SYNTACTIC FOAM

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Optical fibers are being widely studied for use as embedded sensors in laminated composite materials. Appropriately chosen sensors can monitor parameters such as strain and temperature, either during curing or for continuous health monitoring.

The objective of this work is to develop and implement a methodology to determine the suitability of a commercial optical fiber for use as an embedded sensor. In this work, the host material is a syntactic foam which polymerizes at high temperatures. Characterization performed included the strength and optical attenuation of the fiber after exposure to temperatures encountered during syntactic foam production, effect of temperature exposure on the fiber coating, compatibility of the fiber coating with the components of the syntactic foam (liquid monomer, curing agent) and expected failure mode of an embedded fiber/syntactic foam sample.

The evaluation results showed that the candidate optical fiber seems suitable for use as an embedded sensor, despite the fact that the processing temperature exceeds the usage temperature recommended by the manufacturer. Use of liquid low molecular weight components as the matrix precursor also does not seem to present a problem.

GRADED TiB₂/TiC COMPOSITES INFILTRATED WITH LIQUID
ALUMINUM

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The mechanical properties of a ceramic-metal composite depend on its ceramic-to-metal ratio. Infiltrating a ceramic preform that has graded porosity with a liquid metal can produce graded composites. Graded porosity is achieved by compacting stacked powder layers consisting of various mixtures of ceramic compounds that sinter at a different rate. It is well established that the sinterability of TiC is higher than of TiB₂ and that both compounds are well wetted by liquid Al. The main objectives of this work were to establish a database for the densification of TiB₂-TiC mixtures in the 1400-1600°C range and to prove the feasibility of this approach for the manufacture of graded composites. The porosity observed ranged from 7 to 49% for mixtures sintered for 60 min at 1600°C. After infiltration with Al, the hardness ranged from 150 to 2000 HV, and the bending strength from 300 to 500 MPa. Graded preforms were made of layers of different mixtures and had a porosity that varied for more than 20% over their thickness. Subsequent infiltration with aluminum yielded graded samples in which the hardness changed from 900 to 2000 HV. The results illustrate the feasibility of the approach that was followed.

DENDRI-POLYAMIDE CURED EPOXIES COMPOSITIONS AND PROPERTIES

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Properties of highly branched (dendritic) polymers are different from conventional linear polymers. Highly branched polymers are characterized by a large number of reactive groups and unique rheological properties. These properties have found special applications in adhesive systems.

Using an innovative process, highly branched star-type polyamide-amines (PAMAM) were prepared aimed at curing of epoxy resins. The developed process is relatively simple and low cost in addition to being environmentally friendly, compared to existing processes. The dendri - polyamides have a molecular weight of 6000 to 15,000 gr/mole and functionality 30 - 45.

Formulations of dendri - polyamides with epoxy (DGEBA) were used to bond standard aluminum and steel specimens. Experimental results showed good shear strength of 12 MPa for aluminum and up to 17 MPa for steel. Peel strength measured is 1 N/mm.

In addition the dendri-polyamides impart self-grasping properties, which are of importance in assembly lines, where the positioning of the bonded parts has to stay unchanged in a short duration.

Transient electroluminescence from polymer-based LED devices

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Presentation:

Oral Presentation

Abstract

Transient electroluminescence (EL) is a powerful technique for studying new mechanisms of EL emission in polymers. We describe here studies on LED devices based on PPV and MEH-PPV conjugated polymers using a newly developed voltage pulse source based on a photoconducting switch and a Nd:YAG laser. We are able to achieve pulse heights up to several kV and pulse widths down to few nanoseconds. Such short and strong pulses allow the observation of the EL spectra without significant degradation of the sample and allow a study of the evolution of the EL spectra as a function of the pulse height. We discuss the prospectives of using strong and short voltage pulses to achieve electrically pumped lasing in conjugated polymers.

In order to decrease the RC-time constant of the LED device (which is indispensable in studies using such short voltage pulses) we worked with thick films ($\geq 2000 \text{ \AA}$) This allowed to observe a completely new result - transient EL spikes under the so-called capacitive coupling condition. That is, **no** carrier injection tooks place during the electric field pulse but nevertheless strong EL flashes are detected. The EL flashes are attributed to a mechanism involving carrier pumping from trap levels situated above the HOMO band. A "pump-probe" experiment allows to estimate a characteristic relaxation time of these carriers and to find the trap energy (0.15 eV above the HOMO band).

POLYMER-METAL PRECURSORS FOR
HIGH TEMPERATURE SUPERCONDUCTORS

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Today, high temperature superconductors (HTSC) are produced using either solid state reaction methods (relatively fast, but containing undesirable phases), or co-precipitation methods (purer product, but slower). It is possible that using polymer-metal precursors will provide a relatively simple and fast method to produce HTSC. The objectives of this research were to create polymer-metal precursors for HTSC production and to characterize their structure and properties. For this purpose, copolymers containing either acrylic acid (AA) or methacrylic acid (MAA), both of which have functional groups that can interact with inorganic salts, were prepared. Acrylates and/or methacrylates were used as comonomers to improve the mechanical properties. Films were cast from solutions containing a copolymer and a mixture of inorganic salts with a suitable stoichiometry in a common solvent, such as water. The polymer-salt interaction was characterized in solution and bulk through various spectroscopic methods. The dielectric constants were determined as a function of temperature or frequency and the T_gs were determined using thermal analysis. The T_g increases with an increase in the inorganic salt content while the dielectric constant decreases. The changes in T_g and dielectric constant demonstrate that there is a strong interaction between the copolymer and the inorganic salts.

Acknowledgement

The partial support of GIF is gratefully acknowledged.

**INNOVATIVE MODIFICATION OF PVC PARTICLES FOR
ABSORPTION OF TOXIC CHLORO-ORGANIC COMPOUNDS
FROM WATER**

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New two-component porous PVC (polyvinyl chloride) particles were developed for the purpose of removal, via adsorption/absorption, of halogenated organic compounds from water. The preparation of the two-component porous polymer particles is based on a polymerization/crosslinking method developed in our laboratory of a monomer (styrene or methyl methacrylate) absorbed within a suspension PVC-type porous particle. The influence of composition, process parameters and nature of the polymerizing/crosslinking constituents on the modified PVC particle structure, was studied by SEM (scanning electron microscopy), and porosity measurements. The level of grafting between the PVC and the crosslinked polymer was determined by gel content. Dynamic-mechanical properties of the modified PVC were investigated by DMTA (Dynamic-Mechanical-Thermal-Analysis). The modified PVC particle's structure shows that the polymerization/crosslinking process took place mainly within the PVC constituting the particles. The kinetic absorption experiments of a typical halogenated organic compound demonstrate that both the unmodified and modified PVC particles absorb the halogenated organic compound from water. The presence of crosslinked methyl methacrylate within the PVC particles was found to have a different effect than the crosslinked polystyrene presence.

The financial support of the Water Research Institute, Technion and Levi Eshkol scholarship, Israel Ministry of Science, is gratefully acknowledged. This presentation is based on the PhD work of the author, supervised by Prof. Moshe Narkis.

NOVEL POLYMERIC FOAMS: SYNTHESIS, PROPERTIES AND APPLICATIONS

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Porous polymeric foams are well-known materials and produced by a wide variety of techniques. Although these materials are widely used, their structure is often irregular and difficult to control. The objectives of this research were to produce foams with regular structures using a novel method based on high internal phase emulsions (HIPE), to characterize the molecular structure and properties of the foams and to alter their interaction with liquids through surface modification. The presence of two immiscible liquids, one of which is water and the other an organic (oil) is required to yield a HIPE. The HIPE then serves as a template for the synthesis of novel highly porous materials through the polymerization of the organic phase. The foams were characterized using scanning electron microscopy and various mechanical and thermal analyses. The morphology and the mechanical properties of the foams were suitable for their application as a porous absorption medium. Active species, able to participate in adsorption and/or absorption processes, were introduced to the foams through surface modification. Such activated foams could serve as purification media for water that is contaminated with organic compounds.

Acknowledgement

The partial support of the Water Research Institute is gratefully acknowledged.

PARTIALLY CROSSLINKED POLYPROPYLENE

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Crosslinking is a widely used method for the modification of polyolefins. This process involves the formation of three dimensional structures which cause substantial changes in material properties.

The procedures employed for the initiation of the crosslinking reaction involve macroradical formation via thermal decomposition of organic peroxides, high energy irradiation (gamma or electron beam) and grafting of silane groups which form crosslinks via hydrolysis of alkoxy silane groups and condensation of the resulting silanols.

While crosslinking is the dominant reaction in polyethylene, crosslinking as well as degradation occur simultaneously in the case of polypropylene.

Polypropylene crosslinking is complicated due to the beta scission and disproportionation. Therefore, an effective coagent for crosslinking is required to suppress the chain scission of polypropylene macroradicals and to increase the efficiency of crosslinking.

At the Israel Plastics & Rubber Center experiments were conducted with the aim of obtaining partially crosslinked polypropylene. The advantages of the partial polypropylene crosslinkage are based on an increase melt viscosity combined with a restriction of flowability. The material can be used for foamed films and sheets and for injection of high temperature resistant products.

STEREOCOMPLEXATION OF D- AND L-POLY(LACTIDE)

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Complexation of polymers is a well-known phenomenon. Often these complexes are based on electrostatic interactions or hydrogen-bridge formations. In the last few decades more and more findings are reported on complexation of two polymer chains based on mere difference in stereochemistry. Steric interactions, coming forth from the opposite chirality of two isotactic polymers, are thought to form the basis of stereocomplex formation.

PLA as a non-toxic biodegradable polymer is highly popular for its usage in the field of medicine. It is used for many applications varying from microencapsulation of bioactive compounds to biodegradable implants. In 1986 several patents have been issued by Du Pont de Nemours & Co. on the formation and usage of stereocomplexes of L- and D- polylactic acid (PLA). The patents disclose different ways to obtain stereocomplexes of PLA, their fabrication in molds and their use in the preparation of microspheres, encapsulating bioactive agents.

Complexed PLA behaves differently compared to isotactic PLA in several ways. Its melting temperature is found to be 50 °C higher than D- or L-PLA as was measured by Differential Scanning Calorimetry. $T_m=180^\circ\text{C}$ for D- or L-PLA, $M_w=100.000$; $T_m=230^\circ\text{C}$ for PLA-stereocomplex of similar molecular weight. The PLA-stereocomplex appears to be slightly soluble in dichloromethane and good soluble in hexafluoroisopropanol (HFIP). By casting a low concentrated solution on mica crystals were obtained of PLA-stereocomplexes. As opposed to enantiomeric PLA, crystallizing into lozenge shaped crystals, the stereocomplex forms lamella triangular crystals.

By linking poly(ethylene glycol) (PEG) to isotactic PLA we have been able to investigate the influence on the morphology of the formed stereocomplex crystals.

PLA-PEG copolymers were synthesized by ringopening polymerization of D- or L-lactide and methoxy-PEG-OH as initiator. Stereocomplexes were prepared in different solvents mixing equimolar solutions at slow rate for one week. AFM studies were performed by casting a 0.1% solution of polymer in different solvents on mica and evaporating the solvent. Powder Diffraction patterns were obtained on a STOE image plane system. X-ray tubes were filled with the remaining film after casting on a microscope glass and evaporation of solvent using the solutions prepared for AFM.

Powder Diffraction experiments show evidence that stereocomplexes are formed from the enantiomeric block-co-polymers. The PEG chain appears to crystallize undisturbed by the PLA part, which form the stereocomplex. From AFM studies the influence of different solvents was made clear. Using Atomic Force Microscopy (AFM) the block-co-polymers were found to form structures like rods and coiled coils. Mixing equimolar solutions of PEG-PLA in water did not result in any difference in shape compared to the morphology of the enantiomers. Casting from a dioxane solution, however, resulted in the formation of disc-like structures (about 1 μm in diameter, 100 nm height). In acetonitrile needle shaped crystals occurred as well as discs. It is hypothesized that differences in supra structures occur as a result of crystallization of the stereocomplex in layers. Packing of these layers as two dimensional structures leads to different shapes, which is influenced by macroscopic effects. Stress along crystalline rods, for instance, may cause coiling into formation of coils. Similarly few coiled structures emerge from dioxane and acetonitrile solutions.

STUDIES OF INTERFACIAL CRYSTALLINITY IN MODEL COMPOSITES.

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The insertion of fibres into thermoplastic polymer composites may result in changes in the morphology and crystallinity of the matrix, wherein a relatively high nucleus concentration at the fibre surface induces nucleation and crystal growth which is restricted laterally. As a result the matrix spherulites grow from the fibre surface in the radial direction only, to produce a cylindrical layer of columnar crystallinity around the fibre, termed transcrystallinity. The thickness of transcrystallinity could vary from a thin layer confined to the fibre surface to a thick bulk that invades most of the matrix volume.

Transcrystallization depends on the chemical and physical nature of the constituents as well as on the processing conditions of the composite material. Because of the large variability of composite materials, where innumerable fibre-matrix combinations are feasible, it is impossible to approach the issue of transcrystallinity in general terms. Rather, each case should be approached individually and in reference to a specific question such as the mechanism and rate of nucleation, the kinetics of transcrystallization, the crystalline structure of the transcrystalline layer and its effect on the mechanical properties of the composite material.

In view of the compositional variability of composite materials, and in turn, of transcrystallinity, an extensive research work is required before general rules and conclusions can be drawn. In this paper we scan and review our main observations on a number of specific systems which we investigated recently, contributing to the accumulation of sufficient relevant data on the basis of which some generalizations could be eventually attempted. Most of the experimental work was carried out with microcomposites and model materials containing transcrystallinity of significant thickness. Different combinations from an array of constituent materials - comprising polyolefin and polyamide matrices and polyethylene, polypropylene, aramid, carbon and glass fibres - were used to make composite materials. In a number of cases neat transcrystalline specimens were isolated by microtoming and tested for their mechanical, structural and physical properties. X-ray diffraction analysis was used to determine the specific orientation of the polymer backbone in the transcrystalline layer with respect to that of the fibre.

The main conclusion concerning the effect of transcrystallinity on composites properties notes that this orientation (of the polymer backbone in the transcrystalline layer with respect to the fibre direction) is a major factor.

ABRASIVE WEAR OF INFILTRATED METAL-CERAMIC
COMPOSITES BASED ON NONSTOICHIOMETRIC TITANIUM
CARBIDE WITH Fe-C ALLOY MATRIX.

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Cermets, based on the titanium carbide phase incorporated in carbon steels, display some unique characteristics because they can be annealed and worked by normal machining techniques and subsequently hardened by the martensitic transformation that takes place in the metallic phase. The phase transformation and the ability to undergo hardening strongly depend on the initial chemical composition of the ceramic and metal phases. In this study the wear behavior of the infiltrated cermets based on nonstoichiometric titanium carbide (TiC_x , x varied from 0.98 to 0.60) with a Fe-1.0%C matrix before and after thermal treatment was investigated. The wear was determined by weigh losses incurred during dry sliding in ambient air on abrasive SiC (180mesh) paper. The wear resistance of the cermets varied according to their microstructure and hardness. The dimensionless Archard coefficient was estimated for each composition and a mechanism of the wear behavior was put forward.

THE KINETICS OF TRANSCRYSTALLIZATION IN ISOTACTIC POLYPROPYLENE BASED COMPOSITES

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The kinetics of monoclinic α and hexagonal β transcrystallization of isotactic polypropylene on various fibers (aramid kevlar 149 fibers, glass fibers and high modulus carbon fibers) was investigated. This is an interesting aspect of transcrystallization since it greatly influences the thickness of the transcrystalline interface, which in turn will affect the mechanical properties.

No difference was found between growth rates of bulk spherulites and transcrystalline layer. Therefore Hoffman's theory led to same results: regarding α transcrystallization, a transition between regimes II and III occurred near 137°C and the ratio of the slopes of the two regimes was close to the theoretical value (2). Regarding β transcrystallization, only regime II was exhibited in the studied temperature range.

However, induction time was strongly influenced by the kind of fibre. Ishida's approach is linked to induction time and nucleation rate and it enables to calculate the free energy difference at the fibre-crystallite interface. It was concluded that α transcrystallization is more likely to occur on kevlar 149 fibres than on high modulus carbon fibres.

Gradient-thermal measurements were performed for α transcrystallinity and permitted to estimate the activation energy of transcrystallization for the different composites. Activation energies of transcrystallinity promoted on kevlar 149 and high modulus carbon fibres were found similar and lower than activation energy of bulk crystallization.

**ORIGIN OF SiC NANO-INCLUSIONS IN SILICON
NITRIDE/CARBIDE NANO/MICRO COMPOSITES**

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Abstract

SiC/Si₃N₄ nano/micro composites were prepared from starting mixture of crystalline β-Si₃N₄, amorphous SiNC, Y₂O₃ and/or Al₂O₃. The role of SiC inclusions with respect to the mechanical properties is important. Two types of inclusions were identified within the same sample; the inclusions with the "clean" interface between SiC and Si₃N₄ host grain and the inclusions containing oxygen rich surface layer at this interface. These both have a different origin. Free carbon introduced along with the amorphous SiNC influences the silicate melt chemistry and thus, the formation of the SiC inclusions and the creep behaviour of the nano/micro composite.

Tuesday

Posters

INVESTIGATION OF THERMAL SHOCK RESISTANCE PROPERTIES
OF PLASMA SPRAYED THERMAL BARRIER COATINGS

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Several kinds of plasma sprayed thermal barrier coatings composed of bonding layer (Ni, NiCrAl, NiCrAlCoY) and ceramic layer (Al₂O₃, ZrO₂-Y₂O₃, ZrO₂-MgO, Al₂O₃-TiO₂-MgO, Al₂O₃-TiO₂) on aluminum alloy were tested by thermal shock. It was found that the thermal shock fracture process was a continuous process of four-step, which occurred resulting from residual stress, the difference of thermal expansion coefficient and elastic modulus. The microstructure of coatings of interface between metal/ceramic and metal/metal layers was also studied through Metalloscope, Transmission Microscope, SEM and X-ray diffractometer before and after thermal shock. The result showed that the properties of thermal shock resistance were mainly affected by the following factors: i) the reaction at the interface, the type of physical/chemical bonds and the density of the bonds at interface, ii) the discontinuity of interfacial phase and the oxides film on metal surface, iii) residual stresses left from preparation of coatings, iv) porosity: existence of apt porosity and submicroscopic crack could establish strain mechanism to reduce elastic modulus and improve the properties of thermal shock resistance of coatings, iv) additive: some additive can change microstructure, suppress the growth of grains and form solid solution at grains interface to improve fracture toughness at interface.

FLUIDIZED BED CARBURISING

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The fluidized bed carburizing is essentially an extension of the conventional gaseous carburizing; the reactions in gaseous phase and the gas-solid reactions are almost identical. The main difference between these types of carburizings is the mass transfer coefficient value, at the atmosphere - metallic material interface that is twice at the fluidized bed carburizing than the similarly achieved at the conventional carburizing. The turbulent motion and rapid circulation rate of the solid particles under the influence of fluidizing gas and the widely solid-gas interfacial areas provide this phenomenon. Described in this paper are some aspects concerning the mass transfer coefficient β in fluidized bed carburising and the results of the fluidized bed carburising experiments performed in an atmosphere based on methane.

THE ORGANIC PHASE FOR ALUMINUM BATTERIES

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ABSTRACT

The electrochemical behavior of Al anode in nonaqueous electrolytes was investigated. Common contemporary electrolytes for Li batteries were selected, based on their activity toward Al anodic discharge. Negative shifts of $E_{Al/Al^{3+}}$, by up to 1 volt, occur in several electrolytes. Enhanced electrolytes include $(C_2H_5)_4NCl$ (TEA), and $AlCl_3$ in solutions of γ -butyrolactone (BLA), and acetonitrile (ACN), such as $AlCl_3/BLA$, TEA/ACN, and concentration optimized $AlCl_3/KCl/BLA$. Further investigation revealed that solution activators (dissolved metals salts) have a substantial effect on aluminum behavior in this media. Unlike Sn(II), Sn(IV), or Ga(III), both Hg(II) and In(III) significantly activate Al redox behavior. 1 M $AlCl_3/BLA$ and 0.3 M TEAC/ACN with of In or Hg salts, shifts negative open circuit voltage and decreases polarization losses. Extended galvanostatic measurements of these systems, show high Al $3e^-$ coulombic efficiencies (up to 90% and more) for Hg activated electrolytes. Addition of only 10 mM In salt induces (for example, at 10 mA cm^{-2} discharge) a 500 mV negative shift enhancement in the anodic potential (over the non-activated case), which is maintained throughout 40 mAh cm^{-2} deep discharge of the aluminum anode. Addition of mercury to the same electrolyte can result in even larger improvements of the potential (850 mV).

REACTIVE WETTING AND ELECTRON MICROSCOPY OF i-3A MATERIALS

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Ceramic matrix composites possess a wide range of applications due to an increased fracture toughness, strength and thermal shock resistance compared to monolithic ceramics. A relatively new approach to process ceramic matrix composites for elevated temperature applications is based on the reactive infiltration of aluminum into a ceramic preform. During infiltration, a reaction takes place between the Al and the ceramic phases ($\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) resulting in an infiltrated alumina aluminide alloy (i-3A).

The processing of i-3A materials is based on infiltration of a liquid metal into a porous ceramic. The contact angle of the infiltrating liquid determines in part the infiltration kinetics. The wetting of the metal (or alloy) to be infiltrated into the ceramic is characterized by sessile drop experiments on fully dense substrates. The pressureless infiltration kinetics are measured on porous substrates.

Preliminary results of the sessile drop experiments and the microstructure analysis will be presented.

SURFACE MODIFICATION OF PEP-FILMS BY PEP-PEO DIBLOCK-COPOLYMER ADDITIVES

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The modification of polymer-surfaces is of high interest in new technological applications. Surface-modifications give rise to new surface-properties that play an important role in many applications, such as compatibilisation at interfaces, control of wetting and dewetting, tuning of lubrication and friction properties.

Recent experiments showed a reduction of the friction between sliding surfaces by grafting polymer-brushes on the surfaces. Polymer-brushes are normally formed through densely-grafted polymer-chains tethered on a surface.

We seek to create a polymer-brush out of a polymer-matrix with a diblock-copolymer, where one block is of the same material as the matrix and the other part is a polymer, which is energetically incompatible with the matrix. Enthalpic reasons should be the driving-force for the segregation of the diblock-copolymer to the surface and out of the bulk material. Excluded volume interactions between the neighbouring chains and a sufficiently high density of the diblock-copolymers at the surface should stretch the incompatible part of the copolymer to the air and form polymer-brushes.

We investigated surface-enrichment and a change of the wettability of a poly(ethylene-propylene)(PEP) matrix blended with the diblock-copolymer poly(ethylene-propylene-ethylene-oxide)(PEP-PEO).

The surface-segregation was monitored by nuclear-reaction depth-profiling-experiments and the change of wettability was measured via contact-angle-measurements on the initial and modified polymer-surfaces.

EFFECT OF PROCESSING PARAMETERS AND
OF MAGNESIUM ADDITION ON THE DISTRIBUTION
AND WETTABILITY OF CERAMIC REINFORCEMENTS
IN AL/SiC_(p) COMPOSITES

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ABSTRACT

In this work was studied the influence of processing of Al/SiC_(p) composites on the distribution of the ceramic particles and the interface phenomena at the matrix/SiC particles.

It is established that addition of surface-active element, i.e. Mg, has a very important effect in the uncoated ceramic particles wetting process by the melt, and in the distribution of reinforced ceramic particles in the composite.

To avoiding the chemical reaction between SiC particles and liquid aluminium is necessary that the processing temperature must be maintained in the vicinity of the matrix-alloy liquidus temperature and also the preheating of the uncoated SiC particles before their dispersion into melt.

LONG-TERM CHANGES IN A HEAT EXCHANGER STEEL

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The steel ASTM A213 P22 is used as a material for heat exchanger in power plants. During the duty cycles the temperature is in the order of 545°C, which last over tens of years. The microscopic changes in these steels under working conditions are hard to analyze, although they are most probably responsible for the failure and cracking of such devices. If one would be able to predict a future failure with the investigation of the microscopic changes, that would increase the reliability and save a lot of money.

We report on the investigation of such changes in ASTM A213 P22 steel in use for 30 years as a Superheater header at Haifa power station. The microscopic changes were analyzed using Mössbauer Spectroscopy and optical microscopy. As a result it seems that the change of the carbide phases M_3C , M_7C_3 and $M_{23}C_6$ and their morphology are the most prominent changes and most probably responsible for material failures.

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Fractal Structures Formation by Diffusion Controlled Iron Reduction

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It is presented the solid phase interactions in Fe-O-Al system by temperatures lower than the aluminum melting temperature. In experiments were used the Fe_2O_3 and Fe_3O_4 samples, which were pressed from oxide powder and then sintered in the vacuum by 1200°C . Aluminum layer was applied to the oxide surface with the help of vacuum atomization. The thickness of the aluminum layer was about 1 – 5 micrometers.

The process was executed during 1 – 6 hours in inert atmosphere. After heat treatment the samples were investigated by optical and electron scanning microscopy. Local chemical composition was determined by micro spectroscopic methods.

As a result of aluminum – iron oxides interactions in the surface zone of the samples were formed fractal structures with the direction of the long axis from surface to the middle of the sample. These structures consist from solid aluminum-iron oxides and from finely divided metallic iron grains.

It is discussed the diffusion controlled mechanism of fractal structure formation.

The Details of the Elastic Fields Associated with a Martensitic Plate

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Martensitic transformations take place in metals and ceramics by a rapid cooperative movement of atoms which prevents the relaxation of the transformation stresses. Twinning and dislocation slip are invoked to relieve the stresses. Although the stresses are reduced by these internal deformation mechanisms, several observations e.g., large undercooling and microcracking in steels, autocatalytic growth of martensitic crystals and the phenomena of training in shape memory alloys, show that the stresses do not vanish. This conflicts with several theoretic predictions indicating that the stresses should decay to zero as the sub-structure of the martensite crystal become finer. We report the results of a detailed calculation of the elastic fields and the elastic energy generated by a martensitic plate-shaped crystal, undertaken to investigate this controversy. The crystal is constructed of an alternate array of domains with tetragonal transformation strains, lying parallel to a $(hk0)$ invariant plane and the solution was obtained by the method of Fourier transformation. We follow the evolution of the stress localization at the corners of the domains as the sub-structure becomes finer and describe the stress interaction between the domains. We found that the stresses do not vanish as the sub-structure becomes finer thus giving rise to a non-vanishing elastic energy even for the finest sub-structures. This result confirms with the experimental hints mentioned above and with a former analysis by Mura *et al.* (1976). Our analysis may contribute to the understanding and improving of training routines of shape memory alloys.

FORMATION OF SUBGRAINS IN α -URANIUM DUE TO PHASE
TRANSITION

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Uranium has three phases: γ (775-1132°C) with cubic structure - *cI2*.
 β (668-775°C) which is a complex tetragonal structure with thirty atoms per
unit cell- *tP30* and α (below 668°C) which has orthorhombic structure - *cO4*.
Metallographic examination of α -uranium after slow cooling from the β
phase reveals cellular microstructure. The cells in that structure are
subdivided into numerous subgrains.

These subgrains are thought to be creep cells arising from stress which
accompanies the $\beta \rightarrow \alpha$ phase transformation. This assumption is based on
the relatively high change in the volume per atom -1.12% during the phase
transition and the unisotropic behavior of both phases. As the β phase is
harder than the α phase, most of the plastic deformation takes place in the
growing α cells. It is reasonable to believe that traces of this deformation
could be found in room temperature examinations. This led scientists
(Cahn & Tomillinson) to look for correlation of the misorientations
between the subgrains and the active slip systems of high temperature α
uranium which conform with Frank Bibbly equation.

This work presents new ways to determine misorientations by the use of
electron backscattered diffraction (EBSD). The validity of the dislocation
model is checked by calculations of stresses generated by the phase
transformation.

NEW ADHESIVES FOR ELECTRONIC DEVICES

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It is known that various polymeric materials, including adhesives, are used in electronic industry. In particular, the gluing of electronic components in addition to usual soldering increase reliability of electronic appliances. Adhesives for this purpose should have certain properties: adherence to a big variety of substrates, non-corrosivity on metal and also they should be reworkable.

In this study, room temperature setting polydiene urethane adhesives based on new hydroxyl-containing oligodiene urethanes and isocyanate hardeners were investigated for their mechanical and dielectric properties and for their compatibility with electronic components. It was found that the adhesives were highly elastic, stable against temperature and humidity and had good dielectric characteristics. Oligodiene urethanes were used also for modification of an adhesive composition based on copolymer butylacrylate – acrylonitrile. These adhesives were tested after exposing to thermal shock. Lap shear strength of these adhesives remains unchanged after thermal shock effects involving -60° - $+125^{\circ}\text{C}$.

The tests on printed circuit boards with the bonded electronic components were carried out in various conditions. It was shown that these adhesives did not degrade the parameters of electronic components and could be used for assembling of electronic devices.

QUANTITATIVE METHOD OF EVALUATING CORROSION
RESISTANCE OF POST METAL ETCH TREATMENTS

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Abstract

The effect of post metal etch treatments on suppressing Cl induced corrosion of conductors was investigated using a quantitative method. Patterned wafers of Al-Cu alloys with Ti barrier and Ti/TiN anti-reflective coating were used to evaluate the effect of actual product galvanic cells on corrosion development. Wafers after treatments were exposed to a high humidity atmosphere ("Wet Box") to accelerate Cl induced corrosion. The defect density versus time was monitored using KLA2132 automated inspection system. The wafers were also inspected optically and EDX analysis has been performed on suspected defects to verify accuracy of corrosion classification. Reference wafers were tested under ambient conditions and N₂ atmosphere. It was found that the majority of defects, on samples which developed corrosion, tend to appear during the first 24 hours. In some cases the corrosion resistance was not effected by the efficiency of the etch byproducts removal. Wafers after in-situ O₂+H₂O plasma ash and di-ionized water rinse had good corrosion resistance, although strip was not completed. Additional treatments by some of the solvents strip tested or by oxygen plasma in barrel asher, tended to re-activate corrosion. Corrosion was inhibited by storing the wafer after etch in free humid N₂ environment.

PREPARATION OF POROUS ALUMINA CERAMIC BODIES BY DIRECT SOLID-STATE SINTERING OF SPRAY-DRIED GRANULES

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Porous ceramics have many industrial applications such as various filters, catalyst supports etc. The major requirements for filter materials are sufficiently high open porosity, high mechanical strength, and uniform pore size. However, conventional preparation procedures - polymeric sponge method and foaming method - are environment-unfriendly and cannot provide both high porosity and high strength, since an increase in strength usually results in decrease in porosity. Hot isostatic pressing (HIP) provides obtainment of porous bodies with high porosity and improved mechanical properties, but the required equipment is very expensive.

A new method of porous ceramic bodies preparation by direct solid-state sintering of spray-dried (SD) alumina granules is proposed. Several types of SD powders (commercially available and prepared at ICSI) with various granules size were heated in a wide range of temperatures. Porous compacts were prepared using these SD granules and various additions of fine alumina powder. The methods studied included:

A. Powders were dry mixed in the mixer, binder was added, compacts were pressed at different pressures and sintered;

B. Dispersed suspension of the fine powder particles was prepared, coarse SD granules were added, suspension was flocculated, the mixture was dried in an oven, and further proceeded as in A.

C. The initial steps were same as in B, but the mixture was dried on a vacuum filter and the resulted discs were sintered

D. Gel - casting of discs with the following sintering.

It was found that the best combination of properties - a very high open porosity of 40 - 53% and excellent mechanical strength of 20 -30 MPa - was obtained using methods B and C, based on sedimentation of fine particles from suspension in water

The proposed method is environment-friendly, simple and based on the existing equipment.

CUTTING TEST FOR EVALUATION OF GRADED CERMETS

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No standard method is universally accepted for determining the wear resistance of a graded material. A novel cutting test method was put forward and applied in $TiC_x(Fe-C)$ composites in which gradual change of the pearlite-ferrite ratio in the infiltrated metal with a corresponding hardness profile had been established. The hardness values were determined and incisions were made with a 0.3mm thick diamond blade in a plane parallel to the hardness profile. Two independent micrometers measured the depth of the incision and its distance from a reference surface. In parallel, conventional abrasive wear resistance tests were performed on homogeneous samples of various compositions (and hardness), and the weight losses were determined.

The depth of the incision is a linear function of the cutting time and it depends strongly on hardness. Excellent correlation was found between incision depth, hardness values and the results of the wear test. It was concluded that the depth of the incisions is a useful and reliable parameter that can be used as a measure of wear resistance in a graded material.

ULTRASONIC DETERMINATION OF THE ELASTIC PROPERTIES
OF GRADED CERMETS

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The elastic moduli of solids can be derived from the longitudinal and transverse sound velocity and the density. In a graded ceramic-metal composite, the elastic properties vary in a pre-determined manner along one dimension. The property gradient can be established by varying the ceramic-to-metal ratio. The main objective of the present study was to determine the correlation between the compositional gradient and the elastic moduli.

A data base was established for the density and the longitudinal and transverse wave velocities of the bi-phased homogeneous TiC-TiB₂ ceramic composites of various ratio of the components after sintering at 1550°C for one hour, and for the metal-ceramic composites after subsequent infiltration with molten Al. The data base was used as a reference for graded samples fabricated by the "stacking of different layers" method composition.

INTERFACIAL BONDING EFFECTS ON BALLISTIC PERFORMANCE OF SINGLE-POLYMER AND HYBRID PE COMPOSITES.

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Polyethylene (PE) fibre reinforced polyethylene matrix composites, classified as 'single polymer composites', belong to a new sub-family of composites characterized by *physical* heterogeneity. Whereas classical composites comprise chemically different constituents, those of single polymer composites are chemically identical but morphologically different. Accordingly, the PE/PE system comprises ultra high molecular weight (UHMW) PE fibres of extended chain morphology embedded in low (LDPE) or high (HDPE) density polyethylene semicrystalline spherulitic matrix of folded chain morphology. The interface which no longer marks a boundary between chemically different phases, now separates morphologically different phases and may even possess its own typical morphology, e.g., transcrystallinity. A comprehensive research has laid a foundation for developments of different PE/PE composite systems for various engineering and biomedical applications.

This paper describes a development project of PE fibre reinforced PE composite plates for ballistic impact protection. Based on the thermal analysis and mechanical characterization of the unidirectional composites it was confirmed that PE/LDPE and PE/HDPE composites can be manufactured without causing fibre relaxation and loss of mechanical properties. Ballistic impact testing showed that both exhibited impressive ballistic resistance. This was explained by fracture analysis, revealing extensive fibre breakage and fibrillation in the vicinity of the bullet holes and by extensive delamination and bulging of the plate. It was found that using Dyneema SK75 instead of SK65 fibres improved the ballistic performance due to the difference in the melting onset temperature of the fibre. It caused a poor interfacial bonding between the fibre and the matrix and enabled a better delamination during the impact. The ballistic performance of hybrid composite materials with Dyneema (PE), carbon or aramid fibres in different lay-up sequences, intimate, segregated or sandwich, was tested to find the influence of the type of hybridization and the ballistic performance according to the type of fibre in the outer skin of the target.

The ballistic impact tests revealed that the performance of the composites was highly depended on the fibre/matrix interfacial bonding which in turn was determined by the kind of fibre and the lay-up sequence, as these parameters controlled the size of the damage zone.

**COMPOSITION MATERIALS ON BASE OF
POLYVINYLCHLORIDE AND BUTADIENACRYLONITRYL
ELASTOMERS PLASTICIZED BY EDOS.**

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Composition materials on base of polyvinylchloride (PVC) and butadienacrylonitril elastomers (BNE) are effectively plasticized by EDOS, which is a mixture of 1,3 dioxane derivatives with a main component - symmetric formal of 4-methyl - 4-hydroxyl - 1,3-dioxane .

EDOS chemically interacts neither with PVC nor with BNE. For the first polymer this is a dissolvent plasticizer, as for the latter it only restrictedly swells in EDOS.

Similarly to dibutylphthalate (DBP) and dioctylphthalate (DOP) EDOS decreases glass transition temperature of BNE and PVC. In the first case its plasticizing effect is the same as DBP's, irrespective of acrylonitril content in the elastomer composition. At the same time for PVC materials EDOS is less effective as plasticizer in comparison with DOP.

This can be connected with EDOS higher polarity factors. This leads to intensification of intermolecular interactions (IMI) in the systems PVC - plasticizer. For BNE plasticized by EDOS IMI are on the contrary lower as compared to DBP. This stipulates lesser decrease of network structure density of vulcanizers.

EDOS improves adhesive properties of composition materials on base of PVC and BNE.

Another important preference of EDOS over DOP and DBP is that it is twice cheaper and less toxic .

Due to better compatibility with PVC, EDOS decreases its optimal processing temperature as compared to the case of DOP use. As for rubber mixtures on BNE base our plasticizer accelerates vulcanization reaction and increases oil and benzine resistance of vulcanizers.

ULTRASWITCHING IN THIN POLYMER FILMS

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Effect of spontaneous electrical switching between high-resistance ($>10^9$ Ohm, OFF) and low-resistance (0.1-1 Ohm, ON) states was observed in a metal-polymer-metal system with *poly(dimethylsiloxane)* layer of 3-10 mkm thickness and gold electrodes, at room and higher temperatures. This new and intriguing phenomenon manifests itself at relatively small voltages of 1-2 V, which is at least two order of magnitude less than electrical breakdown voltage for given polymer. The effect has been studied on a 25-specimen system by variation of the polymer viscosity, temperature, electrical field and polymer thickness. The ability of the switch-ON state to bear sufficiently high current is also investigated.

INTERACTING OLIGOMERS, CONTAINING
TERMINATED EPOXY AND CYCLOCARBONATE GROUPS
WITH PRIMARY AROMATIC AMINES.

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Hardening of epoxy oligomers by primary aromatic amines is widely used for synthesis network cross-linked polymers with extremely high chemical resistance. Modification of such polymers by forming secondary nonisocyanate polyurethane interpreted network give materials with advanced mechanical properties. But early we have studied joint hardening of mixture of epoxy and cyclocarbonate (CC) oligomers "in situ" only for aliphatic primary amine hardening [1].

A study of interacting epoxy and CC oligomers and aromatic amines is carrying out at the model system including the epoxy oligomer "DER-324" and CC-oligomer "Laprolat-803" by the data of IRS. The synthesis was provided at the temperature 20-150°C.

As a result of IRS-investigation (for carbonyl group of CC-oligomers the characteristic absorption band is near 1800 cm⁻¹ [2]) we have placed interaction epoxy and aromatic oligomer happens by usual mechanism and increasing of temperature promotes of the cross-linked process; after 2 hours at 120°C any absorption bands characterized for epoxy groups are not observed.

The CC-groups do not practicably interact with the any primary aromatic amines up to 150°C, that is shown by the IRS data, because the characteristic for CC-oligomers ' carbonyl group absorption band is really stayed invarible at the all the temperature range.

The result of this investigation gives the possibility for the synthesis a new kind of CC-oligomers for epoxy resins' modification.

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**HYBRID EPOXY URETHANE
CHEMICALLY RESISANCE MATERIALS.**

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As was shown early epoxy coating in which using as a matrix epoxy bisphenol resins and aromatic primary diamines have a good thermostability and high level of acid resistance, but mechanical properties and adhesive strength needs to increasing [1]. Conventional epoxyurethane materials are used mainly for epoxy elastomers and shown limited chemical resistance because urethane groups in these elastomers are very sensible to acid or alkali hydrolysis [2].

The chemical resistance of network polymers contained intermolecular hydrogen bonds, which are typical, for nonisocyanate polyurethane is 1.5-2 times greater tat for conventional polyurethanes [3]. Modification of epoxy resins was carried out by adding different types of polyfunctional cyclocarbonate oligomers leads to improvement of all the mechanical properties (impact and abrasion resistance, specific tensile strain, adhesion strength, etc.). Chemical resistance of elaborated epoxyurethane materials for protective coating is similar to chemical resistance of well known epoxy materials based on epoxy bisphenol resins hardened by aromatic primary diamines. The synthesized network matrix polymer had in all the cases the gel fraction containing not less than 96% by weight.

The structure of epoxyurethane polymers was studied by IRS method and such investigations have shown a long time structure stability during exposure in aggressive media.

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HTSC THIN FILM PREPARATION BY POLYMER METAL PRECURSOR TECHNIQUE

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There exists a large variety of techniques for the production of HTSC films: thermal and electron beam evaporation or co-evaporation, thermal or laser molecular beam epitaxy (MBE), on- and off-axis high pressure sputtering, on- and off-axis laser ablation, metal organic chemical vapor deposition (MOCVD), sol gel and plasma spray deposition (1). We used the polymer metal precursor technique for the HTSC preparation, because this method offers various advantages: The installations are as simple as inexpensive (spincoater, oven) and because of the homogeneous distribution the reaction times are short. In addition the photolithographic patterning of precursor films saves process steps. Large films can be produced in the same high quality (2-5). In our contribution results received by thermogravimetric, IR spectroscopic and thermodynamic analysis of polymethacrylic acid, polyacrylic acid and novolak precursors are presented. Measurements of specific conductivity show that we were able to improve the HTSC properties of Bi-Sr-Ca-Cu-O and Y-Ba-Cu-O thin films by the knowledge of precursor properties.

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DEVELOPMENT IN ORGANIC LEDS

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Organic LED displays are the highest growth segment in the display industry. This new technology allows organic thin films to be used as electroluminescent layers for the production of colorful, bright displays, initial product applications are taking advantage of the unique characteristics of OLED's, such as their ability to be applied to flexible (plastic) substrates. The small handheld display market in general (such as those used on cellular phones) provides potential for OLED sales of \$600 Million by 2004. Currently OLED sales are near zero, and are projected to be \$400 Million by 2002. Larger sales potentials for OLED displays hinge on the ability to coat larger area substrates with organic materials.

Currently, there are at least 65 companies involved in the development of OLED display applications. Yet, the only product offering announced is from Pioneer Electronics in the form of a 5" by 2" display for a car radio. The use of this attractive and colorful OLED display on the model DEH-46DH will help Pioneer gain market share by creating a line of electronics displays which are more aesthetically pleasing and informative than other available electronics displays.

The Kurt J. Lesker Company is actively involved in the sales and development of material sources for organic material depositions. The Lesker Low Temperature evaporative source and associated power supplies are a prime example of the technology available at the Kurt J. Lesker Co. for the production of clean organic films with uniformities <5%. Process development at KJLC can address film growth on various substrate sizes, and KJLC equipment manufacture can build complete 'turkey' OLED deposition systems with associated transparent ITO and metallic contact layers.

BLOOMING OF FLAME RETARDANT ADDITIVES IN PLASTICS

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Blooming appears as white stains on the surface of plastics and it is the result of migration of the additives from the bulk of the specimen. Usually, it may be recognized visually or with an optical microscope. The methods employed today for the investigation of blooming are either qualitative or interacts heavily with the blooming process itself.

Within the framework of the present investigation, a new, quantitative method for the evaluation of blooming of bromine-containing compounds was developed. The method relies on SEM (Scanning Electron Microscopy), EDS (Energy Dispersive Spectroscopy) and IM (Image Analysis). The specimens are tested before and after aging at 65°C for various periods of time.

The advantages of the method are:

- The evaluation of blooming is quantitative;
- It does not interfere with the blooming process;
- It is a complementary tool for the investigation of blooming mechanism;
- Allows for the microscopically observation of blooming phenomenon, much before the white stains are visible on the specimen surface.

It was found that the blooming was enhanced when the flame-retardant (FR) additive melts during processing and is unable to crystallize within the polymeric matrix. Thermal analysis and XRD showed that the FR became amorphous and was acting as a plasticizer. On the other hand, FRs able to preserve their crystallinity during processing showed little or no blooming at all.

Various parameters, such as the type of polymeric matrix, the presence of other additives, the interaction with solvents and oils, were investigated in this work in relation with the blooming phenomenon.

Based on these results, a mechanism for blooming of bromine-containing FR's in HIPS was suggested. Accordingly, various ways for the diminishing or suppression of the blooming were proposed and experimentally evaluated.

POLYMERIC MATERIALS UPGRADES THEIR PROTECTIVE PROPERTIES DURING EXPOSURE IN STRONG AGGRESSIVE MEDIA

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For upgrading chemical resistance characteristics of polymer composite materials, anti-corrosive additives (ACA) have been proposed. ACA are inorganic powders reacting with aggressive environment they are introduced to, with formation of a new phase of high-strength hydrate complexes. This enhances bonding occurs upon the penetration of aggressive media into the ACA-containing polymeric composite materials. The chemical resistant properties of such materials are activated by harsh environmental conditions where polymer systems without additives remain defenseless to chemical corrosion.

ACA provide a number of distinct enhancements for polymers offering manufacturers products with stronger, corrosion-resistant finished materials. Chemical resistance tests have been carried out for a lot of polymer systems over one year. The results revealed that ACA-containing polymeric composite materials significantly outperform those materials without the additive. Furthermore, extensive testing has shown that ACA can increase composite material life by some 20 times offering substantial saving for users who can extend the life of their polymer-based products whether it be pipes, floorings, reinforced plastics or other materials exposed to corrosive media.

Composite materials enhanced with ACA yield higher impact strength than those without the additive, moreover, the material permeability is reduced 15-25 times. The percentage of ACA mixed with polymer matrix

RECYCLING OF POLYMERS

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The recycling of plastic goods has become a major task to be addressed by countries with developed economies, and the recycling industry faces new challenges as a result of political and economic pressures.

The European Community, led by Germany and the Scandinavian countries, is trying to set more stringent rules concerning the recycling of plastic materials.

Flame-retarded plastics, whose source is the housing of electronic devices (televisions, computers, printers, photocopying machines) and connectors, will probably form a significant part of the materials to be processed by recycling applications in the future. The major plastics in this case are styrenic copolymers (ABS, HIPS and alloys) and engineering thermoplastics (PBT and nylons). Many of them are flame-retarded by brominated flame-retardants.

The Dead Sea Bromine Group offers a contribution to the development of recycling technologies by providing technical data on the behavior of its proprietary flame-retardant systems under extreme recycling conditions. Properties of typical flame-retarded compounds are evaluated by several recycling steps and by an accelerated aging test at high temperature.

Projects on the recycling behavior of PBT, flame-retarded by poly (pentabromobenzyl acrylate), and of ABS, flame-retarded by tris (tribromophenyl) cyanurate, will be discussed.

STRUCTURE AND PROPERTIES OF BIODEGRADABLE HYDROPHOBIC POLYMER MATERIALS

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Hydrophobic synthetic polymers are widely used for packaging materials production. These polymers protect products and goods from environmental factors, but at the same time used packaging materials are highly resistant to degradation in the environment and stored in excess in wastes thus polluting environment. To solve the burning ecological problem of plastic wastes it's necessity to develop new types of biodegradable hydrophobic polymer material (*BHM*).

To create of *BHM* an especially composite based on natural and synthetic biodegradable polymers has been developed. The material had sandwich-type structure. Inner thick layer of the materials is cellulose, and external protective thin layers are formed from hydrophobic biodegradable polymer composition.

Superior properties of *BHM* can be achieved by means of progressive technological methods. The developed *BHM* is water resistant, low vapor and gas permeated, strong, cheap and completely biodegradable under composting conditions. Owing to biodegradable nature this material can be used for production of disposable packages, everyday items, sanitary and other environmentally friendly products and goods.

After using one part of *BHM* – products may be recycled, and other part may be destroyed in the environment.

Filled Porous Epoxy Beads for Lead Sorption

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Abstract

Lead sorption performance of porous epoxy beads filled with spent, dead biomass (*Saccharomyces cerevisiae* from beer fermentation process) which was shown to have good lead ion sorption capacities has been evaluated. The porous epoxy beads were prepared by a combination of: (1)-ionotropic gelation of a sodium alginate paste containing all components (biomass, diepoxide and polyamine by Ca^{2+} ions). (2)-partial epoxide crosslinking (Bisphenol A Diglycidylether with a polyaminoamide). (3)-porosity introduction by dissolving the polyelectrolyte (by decalcification with in a phosphate buffer). (4)-final crosslinking. The resulting 1.5-2.0 mm beads were studied by a combination of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and photoacoustic FTIR spectroscopy (PAS-FTIR). Among the more important factors that influence the lead sorption efficiency are the filler percentage, the degree of partial crosslinking (step 2) and the epoxide to amine ratio. The most dramatic effect on sorption was by the filler percentage. More surprising was the fact that even an inert filler towards metal ion sorption like poly α -methylstyrene gave good lead ion sorption capacities. This is probably a result of the important contributions of the epoxidic matrix and calcium alginate gel in the bead to the sorption process. The beads were tested in a column for lead ion sorption and shown to be useful for several loading-elution cycles.

R&D ON THE THERMOPLASTIC RUBBERS (TPR)
MECHANICAL PROPERTIES IMPROVING.

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A large selection of elastomers and T.P.R's (S.B.S, S.E.B.S, T.P.O, E.V.A, Santopren etc') were tested for, special applications with a high friction coefficient.

A lot of T.P.R compounds, with different components variations (include fillers, plastisizers and carriers change and modifications) were checked in this investigation for general targets, like friction coefficient increasing.

The mechanical properties like friction coefficient, hardness, elongation and tensile strength were analyzed and compared for above mentioned different T.P.R. compounds.

The final products from the different materials were injected, and tested in field conditions.

The mechanical properties vs. different materials will be shown in this report.

POROUS SILICON SURFACE TREATMENTS BY PHOTO-THERMAL GAS PHASE REACTIONS

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Porous silicon has been treated by UV enhanced photothermal reactions in NF_3 , N_2 , H_2 as well as in vacuum environment. The UV photons are provided by excimer lamp. The UV/ NF_3 treatment strongly enhances the photoluminescence of porous silicon. The increase of PL intensity is about 1 – 2 orders of magnitude at treatment temperatures in the range of 300 - 400°C. Using AFM and Auger measurements, it is found that the PL enhancement can be correlated with the growth of about micron thick SiO_x layer with the x value close to 2, and incorporation of fluorine. The overall effect of photoluminescence enhancement is suggested to be due to the NF_3 /UV photothermal etching of the as-formed native oxide, as well as, to the cleaning and passivation of the porous silicon surface with fluorine, followed by a rapid growth of a more stoichiometric oxide SiO_x ($x \approx 2$) layer in air.

**COEXTRUSION OF HIGH BARRIER FILMS CONTAINING LIQUID
CRYSTALLINE POLYMERS**

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Liquid Crystalline Polymers (LCPs) possess the lowest oxygen and humidity permeability among commercial polymers. However, LCPs are highly incompatible with conventional polymers (polyolefins, polyesters), due to their unique composition and morphology.

To compatibilized LCPs with conventional polymers, a variety of polymers were characterized having various functional groups like maleic anhydride, epoxy, acrylic acid etc.

Three and five layer films containing one LCP layer were prepared by coextrusion. Experimental results have shown that a 10 μ m LCP layer with polypropylene or polyester and appropriate compatibilizers, increased the barrier to oxygen by a factor of 130 compared to neat polypropylene and by a factor of 40 compared to neat polyester, respectively.

These unique barrier properties of LCPs containing multi-layer films can compete commercially with EVOH and PVDC containing coextruded films.

Competitive Acceleration Mechanisms in Fast Diffusional Phase Growth

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Rapid phase growth in thin film couples and in some bulk diffusional couples during an initial stage of reactive diffusion is analyzed. Two competitive mechanisms of the diffusion acceleration are considered: high volume diffusion due to vacancy supersaturation and grain boundary diffusion. In analyzing the first case, the moving interphase boundary was assumed to be a source of excessive vacancies. Two distinct cases of substantial vacancy supersaturation were considered: the very initial stage, when the non-equilibrium vacancies are distributed along the whole new phase layer, and the next stage, when the non-equilibrium vacancies are located within a thin layer near the interphase boundary. The system parameters which determine the dominating mechanism of the process were obtained. It was shown that different mechanisms of diffusion acceleration can intensify each other, and its sequential operation may lead to non-parabolic phase growth.

THERMAL- FATIGUE KINETICS IN SMC SOLDER JOINTS BY SEM/EDS FRACTOGRAPHY

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We submit evaluation of thermal fatigue (TF) reliability of SMC solder joints (SJ) by high resolution SEM / EDS fractography technique . This technique is shown to be in particular efficient in the observation of the early stage of TF damages -which is missed by traditional surface inspection techniques- and in revealing the weak interface links in the joint microstructures .

SEM /EDS inspection has been performed after standard accelerated thermocycling (ATC) of Cu / 63Sn37 Pb /FR-4 joints followed by pull-out test to expose the fracture surface which contains (i) fatigue striations zone and (ii) the dimpled zone of the overload ductile failure . It is shown that in the joints under investigation:

- The normalized surface area FF of the fatigue cracks which extend in the solder parallel to the lead-solder joint and very close to the intermetallic /solder interface grows exponentially with the number N of thermocycles ([0-100]C, one cycle per hour) in the range $N \leq 2674$. The average crack velocity is estimated as 0.15 $\mu\text{m}/\text{cycle}$.
- At $N=4300$, 90% of the joints are expected to have FF between 70% and 20% with the average fraction of the damaged interface $FF = 40\%$.
- TF threshold falls in the range $0 < N_{TH} < (600- 800)$, and is likely closer to the lower boundary of the interval.
- Although fatigue crack formation were observed in all parts of the joints, from 50% to 100% of the cracks form near the toe of the joints. This points likely to adverse wetting conditions and "a notch effect" caused by damage to the toe region from the cutting of the lead.
- Up to 40% of all thermal fatigue cracks form close to voids , in particular to larger voids which diameter exceeds 30 μm ; these large voids are shown to originate from the entrapment of the flux in the joint and its outgassing in the sandwiched solder during reflow and concentrate mainly along the Cu lead -solder interface .

It is concluded that amassment of the FF(N) SEM/EDS fractography data for various SMC solder joints under ATC conditions will create a good basis for unbiased comparative assessment of their reliability and soldering technology beyond traditional inspection routines and standards.

STRUCTURAL MODEL OF THE HEXAGONAL APPROXIMANT IN AL-FE-V-SI ALLOY

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Abstract

The structure of the new hexagonal approximant phase $Al_{14.6}(Fe,V)_3Si_{1.3}$ (P6/mmm) with cell dimensions $a=2.52nm$ and $c=1.26nm$ was investigated by high-resolution transmission electron microscopy. This phase was found for the first time by Koh et al[1] in strip-cast Al-Fe-V-Si alloy. We observed this phase in commercial Al8009 alloy produced by rapid solidification technology combined with powder metallurgy processing. It appears in small amounts, non detectable by X-ray diffraction, as particles growing in the vicinity of the nanoparticles of cubic $\alpha-Al_{12}(Fe,V)_3Si$, a well known approximant phase described by Cooper [2].

Electron crystallographic technique based on processing of the high-resolution images by CRISP-program[3,4] and combined with the analysis and simulation of electron diffraction patterns was successfully used for developing the structural model of the new phase.

Its structure was described as sixfold symmetry packing of complex icosahedral units, forming the unit cell with 502 atoms. The arrangement of atoms in each unit can be represented as twelve-coordinated $FeAl_{12}$ polyhedra joined by edges and corners, similarly to that found for other aluminium-rich intermetallic phases with iron or manganese [5].

The simulated HRTEM images and diffraction patterns based on proposed structural model of the hexagonal approximant phase are in good agreement with the experimental results.

The model also explains the experimentally observed orientation relationship between the $\alpha-Al_{12}(Fe,V)_3Si$ phase and the new phase.

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**JOINING OF ZrO_2 - 4.5 WT% Y_2O_3 CERAMICS USING
NANOCRYSTALLINE TAPE CAST INTERLAYERS**

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Nanocrystalline Y-TZP tape casts were used as interlayers to join conventional Y-TZP ceramic pellets. The joining experiments were carried out by hot pressing at temperatures 1000°C to 1300°C under constant pressure of 55 MPa. Two types of joints were obtained with and without a nanocrystalline interlayer. Successful joining was enabled at 1100°C only with the interlayer; four point bending test results revealed an average joint strength of 206 ± 10 MPa. The joint strength increased with the joining temperature. The specimens joined at 1300°C with an interlayer exhibited a joint strength of 613 ± 40 MPa, which is ~ 90% of the strength of the ceramic pellets. The interlayer at the joint exhibited homogeneous and crack free microstructure and preserved its nanocrystalline nature at all temperatures. Specimens joined without the interlayer revealed crack-like defects that resembled the non-bonded areas at the joint interfaces.

**How Israeli scientists could participate in the MaTech Programme –
New Materials for Key Technologies of the 21st Century**

*Dr. Vladimir Maty
Project Management Organization, NMT, of the
German Federal Ministry of Education and Research
Research Center Jülich, Germany*

The *MaTech* Programme has been rapidly implemented since its start in mid-1994 and has been fully accepted by the industrial as well as the institutional materials research groups in Germany. A total of DM 563 million was appropriated (until Dec. 1998) to 134 collaborative projects with 608 R&D partners. By adding the industry's share, R&D funding of roughly DM 1.2 billion has been set in motion in the field of materials technology.

The strategic goals of the MaTech Programme are:

- 1 strengthening of an internationally leading, sustainable technology position in the field of materials technology,
- 2 focusing on existing resources and competences,
- 3 linking the individual value-added stages in the innovation process,
- 4 application orientation to internationally important technology fields,
- 5 acceleration of the transfer of results into practical applications,
- 6 Resources and protection conservation of the environment by the application of new materials.

With a total annual budget of approx. DM 120 million, MaTech focuses pre-competitive research and development for the production and processing of new materials for innovative applications in materials-related key technology areas important for Germany on the following funding priorities:

Material innovations for:

- *information technology (IT)*
- *energy technology (ET)*
- *traffic and transport (TT)*
- *medical engineering (ME)*
- *manufacturing technology (MT)*

and for applications across material classes and in different technology fields:

- *new fields and interdisciplines (NFI).*

The BMBF objectives presently aim at an intensified formulation of main topics, stimulated competition and sustainability in research funding. Taking into account the results of the Interim Report on the MaTech Programme and the recommendations by the BMBF Strategy Group for Materials Research, the support measures under the MaTech Programme are to be implemented to an increasing extent by way of announcing thematic priorities. This will include an expansion of the collaborative research support concept. The announcement of new support measures in 1999 involves the following topics for collaborative projects:

- 7 Nanostructure materials (collaborative projects, closed),
- 8 New materials for innovative manufacturing (collaborative projects, closed),
- 9 Multifunctional materials (1st call ends 31.01.2000, 2nd call 15.08.2000)
- 10 New materials for machines (in preparation)
- 11 Lightweight construction materials (in preparation)

The announcements published in the Federal Law Gazette are disseminated by the NMT Project Management Organization through the information network and on the Internet at <http://www.fz-juelich.de/nmt>

The design of collaborative projects on topics which are not a subject of announcements will also be possible in the future according to the bottom-up procedure, i.e. deliberation of and on project outlines, formulation of applications, funding decision after evaluation.

The MaTech Programme offers opportunities for Israeli scientists to participate in collaborative research projects. This topic will be further elaborated upon the lecture/poster session.

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