

# International Workshop on Interfaces

Rowland M. Cannon Memorial Workshop

NEW MATERIALS VIA INTERFACIAL CONTROL

June 22-26, 2008

Santiago de Compostela, Spain



**Instituto de Cerámica — Universidad de Santiago de Compostela**  
**Lawrence Berkeley National Laboratory, Berkeley, California, USA**  
**Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain**

**Chemistry – Engineering Conference Center**

Santiago de Compostela, Spain

**Gran Hotel Santiago**

Maestro Mateo s/n

15706 Santiago de Compostela, Spain

**Cover image courtesy of Dr. Christian Kisielowski (NCEM-LBNL, Berkeley, CA, USA)**

**Imaging columns of the light elements C, N, and O with sub-Ångstrom resolution**

**Phase of the Electron Exit Wave Interface: GaN [1100]/Sapphire [1120]**

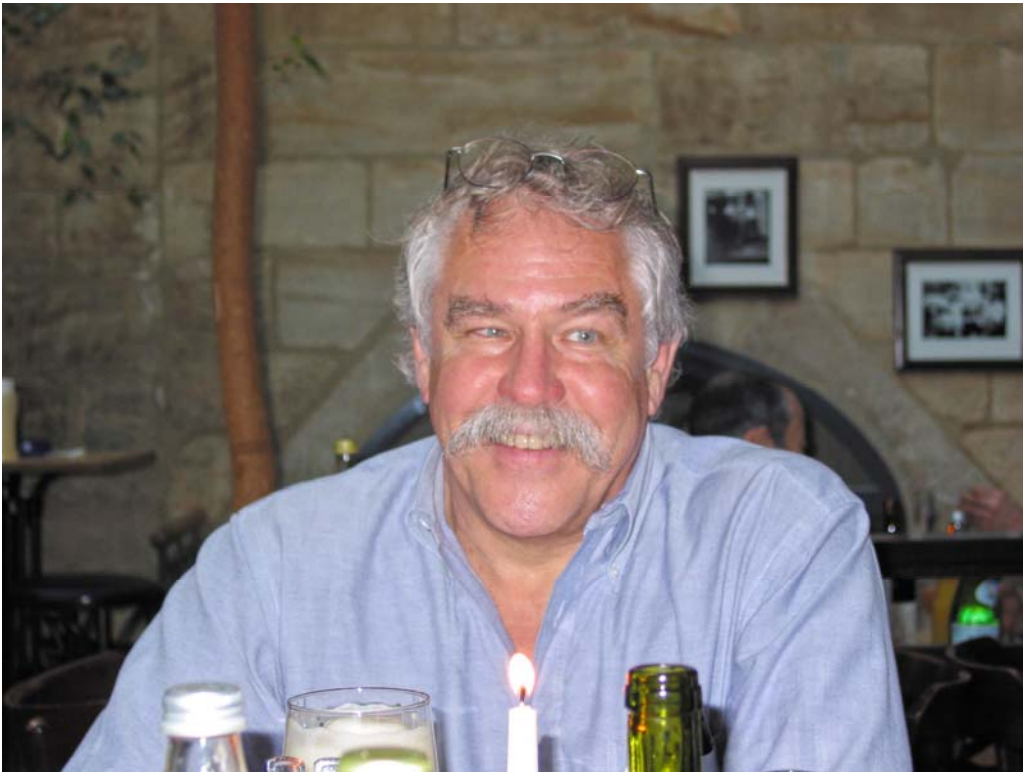
National Center for Electron Microscopy, Lawrence Berkeley National Laboratory

C. Kisielowski, C.J.D. Hetherington, Y.C. Wang, R. Kilaas, M.A. O'Keefe, and A. Thust, *Ultramicroscopy*, **89** (2001) 243-263.

*IN MEMORIAM*

Rowland M. Cannon Jr.

Lecturer in Materials Science and Engineering  
Staff Scientist  
Lawrence Berkeley National Laboratory  
UC Berkeley  
1943 – 2006



One of the best known ceramists in the world, Rowland Morrell Cannon Jr., died suddenly and unexpectedly in Berkeley, California on April 21, 2006. He was 63 years old. Rowland Cannon was born in Idaho Falls, Idaho, on February 17, 1943. He completed his undergraduate (B.S., 1966) and graduate studies (Sc.D., 1975) in the Department of Materials Science and Engineering at the Massachusetts Institute of Technology (MIT), where he also served as chapter president for the Phi Delta Theta fraternity. He enjoyed a long and successful career in ceramic science, beginning at AVCO Corporation in 1966-1971, continuing as an assistant and associate professor at MIT (1975-1982), and since 1983 serving as a research engineer, lecturer, and staff scientist at the University of California, Berkeley and the Lawrence Berkeley National Laboratory.

Throughout his career, he engendered respect, trust and affection from a wide network of friends, colleagues and collaborators across the globe, particularly as a frequent visiting scientist at the Max Planck Institute for Metals Research in Stuttgart, Germany. His work was rewarded with numerous honors, including the American Ceramic Society's Ross Coffin Purdy Award for best paper of the year (1991), being named an ACS Fellow (1994), earning an Alexander von Humboldt Senior Research Award (2000), and most recently completing a month long W. D. Kingery Distinguished Lectureship at MIT in February-March 2006. He also received the Sosman Memorial Lectureship from the American Ceramic Society in 1997.

The impact of his research in the thermodynamics, processing and structural properties of ceramics was extraordinary. He had recently been working with numerous groups around the world on the character, stability and properties of interfaces for ceramics and their role in microstructure development and mechanical behavior; this included the formation and adherence of ceramic-metal interfaces, wetting, interface mechanics, and kinetic behavior. Indeed, throughout his career, he was known for his profound understanding and insight into the mechanical behavior and microstructure evolution in ceramics. Based on this work, he published over 140 scientific papers and gave a remarkable 11 invited Gordon Conference lectures on topics that include sintering and microstructural evolution, mechanical properties, and interfaces. He collaborated in research with several national laboratories and with universities and institutes in Germany, Italy, Japan, China, Turkey, Great Britain, and Israel.

## **PURPOSE AND SCOPE OF CONFERENCE**

It is becoming increasingly recognized that new technologies require structures whose functions and properties are not achievable using monolithic materials. The combination of dissimilar materials creates interfaces whose properties and formation need to be understood before they can be applied commercially. Interfaces play a critical role in areas as diverse as metallic interconnects in semiconductors, adhesion of oxide scales on metal substrates, thermal barrier coatings, composites, bonding between bulk metals and ceramics, and biomaterials. In all of them, it is critical to correlate the interfacial structure to its properties and to the device behavior.

Talks will be complemented by poster presentations emphasizing aspects of materials interfaces. We seek a balance between theory and experiment, and fundamentals and applications. In-depth discussion and detailed interaction among participants is encouraged and expected. The workshop will assess what is known and highlight future research opportunities.

## **CONFERENCE HISTORY**

Previous International Workshops on Interfaces were held in Spain, mostly in Santiago de Compostela, every 3 years starting in 1990.

The 1990 workshop emphasized the importance of characterization. No topical issue was published.

The 1993 workshop stressed the correlation of processing, properties, and characterization, with an emphasis on ceramic/metal interfaces. The proceedings of this conference were published in Scripta Metallurgica et Materialia, Volume 31, Issue 8, 1994. (<http://www.elsevier.com>)

The 1996 conference dealt with the role of interfaces. The proceedings of this conference were published in Acta Materialia, Volume 46, Issue 7, 1998. (<http://www.elsevier.com>)

The 1999 conference took place in Sevilla, Spain, and dealt with the progress on ceramic and biomaterial interfaces. The proceedings were also published in Acta Materialia, Volume 48, Issue 18/19, 2000. (<http://www.elsevier.com>)

The 2002 conference was held in Oviedo. It summarized the progress in the field of interfaces during the previous three years. The proceedings were published in Journal of the European Ceramic Society, Volume 23, Issue 15, 2003. (<http://www.elsevier.com>)

The 2005 conference took place in Santiago de Compostela with a subtitle: Interfaces by Design. The proceedings were published in the Journal of Materials Science, Volume 41, Issue 16, 2006. ([www.springerlink.com](http://www.springerlink.com))

Each meeting summarized the state of the art, defined key outstanding problems, and promoted collaboration and exchanges that helped advance the field during the ensuing years. We anticipate that this 2008 workshop will serve again as a forum of discussion to define the main questions in the area, promote interaction, and stimulate new research efforts among established researchers and a new generation of materials scientists.

## ACKNOWLEDGMENTS

The International Workshop on Interfaces: New Materials via Interfacial Control is sponsored by the Instituto de Cerámica — Universidad de Santiago de Compostela, Lawrence Berkeley National Laboratory, and the Instituto de Ciencia de Materiales de Madrid, CSIC.

We wish to thank the following organizations for their contribution to this conference:

The European Office of Aerospace Research & Development (EOARD)  
Spanish National Research Council (CSIC)  
Ministry of Education, Spain  
University of Santiago de Compostela  
Regional Government of Galicia  
Joining and Welding Research Institute, Osaka University



## PROCEEDINGS

The proceedings of this international workshop will be published as a regular issue of the *International Journal of Materials Research*.

## **International Organizing Committee**

Jose S. Moya — Chairman (ICMM-CSIC)

Manfred Ruehle – (MPI-MF, Stuttgart – Germany) - Program Co-Chair

Tony Tomsia (Lawrence Berkeley National Lab) - Program Co-Chair

C. Barry Carter (Univ. of Connecticut)

W. Craig Carter (Materials Science, MIT)

Roger H. French (DuPont Co. Central Research)

Ron Kerans (WPAFB)

Kiyoshi Nogi (Osaka University)

Eugen Rabkin (Technion — Israel)

David Seidman (Northwestern University)

## **Local Organizing Committee**

Francisco Guitian (University of Santiago)

Jose S. Moya (ICMM-CSIC)

Victor Valcarcel (University of Santiago)

Carlos F. Gutierréz-González (ICMM-CSIC)

Carmen Cerecedo (University of Santiago)

Jaime Franco (Lawrence Berkeley National Lab)

# WORKSHOP PROGRAM

**SUNDAY, JUNE 22, 2008**

- 9:00 - 17:00      **INCEMS Meeting, Gran Hotel Santiago**
- 17:00 - 22:00      Registration at Gran Hotel Santiago
- 20:00 - 22:00      Welcome Reception at Gran Hotel Santiago

**MONDAY MORNING, JUNE 23, 2008**

## **Interface Micromechanics**

Session Chair: Tony Tomsia

- 8:45 – 9:00      Jose S. Moya - Opening remarks
- 9:00 – 9:30      Anthony G. Evans – University of California, Santa Barbara, USA  
**The influence of interfaces on the fuel efficiency of aeroturbines (OP1)**
- 9:30 – 9:40      Discussion
- 9:40 – 10:10      Robert O. Ritchie, D.H. Alsem, M. Launey, E. Munch, E. Saiz, and  
A.P. Tomsia – University of California, Berkeley, USA  
**Nature-inspired hybrid structural materials (OP2)**
- 10:10 – 10:20      Discussion
- 10:20 – 11:20      **Poster Session P1** & Coffee
- 11:20 – 11:50      Kevin J. Hemker – John Hopkins University, Baltimore, USA  
**Interfacial delamination in layered thermal barrier coatings (OP3)**
- 11:50 – 12:00      Discussion
- 12:00 – 12:30      Eric A. Stach, Z. Shan, M. Jin, A. Minor, S.X. Mao, J.M.K. Wiezorek,  
D.M. Follstaedt, J. Knapp, and J.W. Morris, Jr. – Purdue University, West  
Lafayette, USA  
**In-situ transmission electron microscopy studies of the deformation  
response of nanocrystalline metals (OP4)**
- 12:30 – 12:40      Discussion
- 12:40 – 13:00      **General Discussion**
- 13:00 – 15:00      **Lunch (and Posters)**



## MONDAY AFTERNOON, JUNE 23, 2008

### Modeling of Interfaces at Different Length Scales

Session Chair: Ron Kerans

- 15:00 – 15:30      Mark Asta – University of California at Davis, Davis, USA  
**The crystal-melt interface: Insights from atomic-scale computer simulations (OP5)**
- 15:30 – 15:40      Discussion
- 15:40 – 16:10      Andrew M. Rappe – University of Pennsylvania, Philadelphia, USA  
**Nucleation and growth mechanism of ferroelectric domain-wall motion (OP6)**
- 16:10 – 16:20      Discussion
- 16:20 – 17:00      **Poster Session P2** & Coffee
- 17:00 – 17:30      Catherine Bishop – Oxford University, Oxford, UK  
**A phase field model for grain boundaries in ceramics: strontium titanate (OP7)**
- 17:30 – 17:40      Discussion
- 17:40 – 18:10      L. Yue, D. Weygand, and Peter Gumbsch – University of Karlsruhe, Karlsruhe, Germany  
**Modelling grain growth in STO ceramics: linking physical properties to measurable statistical quantities (OP8)**
- 18:10 – 18:20      Discussion

**Free Evening**

## TUESDAY MORNING, JUNE 24, 2008

### Wetting and Segregation

Session Chair: Wayne Kaplan

- 9:00 – 9:30 S.M. Hong, T B. Reynolds, C.C. Bartlow, J.T. McKeown, and Andreas M. Glaeser – University of California, Berkeley, USA  
**Ultrarapid transient-liquid-phase joining of alumina (OP9)**
- 9:30 – 9:40 Discussion
- 9:40 – 10:10 Osamu Takai – Nagoya University, Nagoya, Japan  
**Interfaces at solution plasma (OP10)**
- 10:10 – 10:20 Discussion
- 10:20 – 11:20 **Poster Session P3** & Coffee
- 11:20 – 11:50 Eugene Rabkin, R. Levi and L. Klinger – Technion, Haifa, Israel  
**The role of dislocations in liquid metal embrittlement (OP11)**
- 11:50 – 12:00 Discussion
- 12:00 – 12:30 Kiyoshi Nogi – Osaka University, Osaka, Japan  
**Reactive wetting in liquid Cu alloy and graphite and SiC systems (OP12)**
- 12:30 – 12:40 Discussion
- 12:40 – 13:10 Diego Gómez García and A. Domínguez Rodríguez, University of Seville, Seville, Spain  
**On segregation and plasticity (OP13)**
- 13:10 – 13:20 Discussion
- 13:20 – 15:00 **Lunch (and Posters)**

## TUESDAY AFTERNOON, JUNE 24, 2008

**18:00 Excursion – Santiago Cathedral**

**20:30 Conference Dinner - The Parador Hostal de los Reyes Católicos**

## WEDNESDAY MORNING, JUNE 25, 2008

### Rowland Memorial Session

Session Chair: Manfred Ruehle

- 9:00 – 9:30      Eduardo Saiz and A.P. Tomsia – Lawrence Berkeley National Laboratory, Berkeley, USA  
**Solid-liquid interfaces at high-temperature (OP14)**
- 9:30 – 9:40      Discussion
- 9:40 – 10:10     R. F. Rajter and Roger H. French – DuPont Co. Central Research, Wilmington, USA  
**Van der Waals – London dispersion interactions for metallic and semiconducting carbon nanotubes from ab initio uniaxial optical properties (OP15)**
- 10:10 – 10:20     Discussion
- 10:20 – 11:20     **Poster Session P4** & Coffee
- 11:20 – 11:50     Carol Handwerker, A. Pedigo, J. Blendell, M. Williams, K. Moon, and B. Boettinger – Purdue University, West Lafayette, USA  
**Tin whisker formation: stress relaxation mechanisms in Sn and Sn-Cu electrodeposited films (OP16)**
- 11:50 – 12:00     Discussion
- 12:00 – 12:30     Martin P. Harmer, S.J. Dillon, and G.S. Rohrer – Lehigh University, Bethlehem, USA  
**Chemical control of grain boundary complexions (OP17)**
- 12:30 – 12:40     Discussion
- 12:40 – 13:00     **General Discussion**
- 13:00 – 15:00     **Lunch (and Posters)**

## WEDNESDAY AFTERNOON, JUNE 25, 2008

### Rowland Memorial Session

Session Chair: Dominique Chatain

- 15:00 – 15:30      David R. Clarke – University of California, Santa Barbara, USA  
**Ultra-low thermal conductivity oxides by design (OP18)**
- 15:30 – 15:40      Discussion
- 15:40 – 16:10      Arthur H. Heuer – Case Western Reserve University, Cleveland, USA  
**Interfacial issues in the materials science of silicon MEMS (OP19)**
- 16:10 – 16:20      Discussion
- 16:20 – 17:00      **Poster Session P5** & Coffee
- 17:00 – 17:30      Christoph T. Koch – Max Planck Institute for Metals Research, Stuttgart, Germany  
**Application of electron inline holography to interfaces in ceramics (OP20)**
- 17:30 – 17:40      Discussion
- 17:40 – 18:10      Michael J. Hoffman, T. Waschkies, and R. Oberacker – University of Karlsruhe, Karlsruhe, Germany  
**Role of interfaces for manufacturing and mechanical behaviour of MMCs produced by liquid metal infiltration of ceramic preforms (OP21)**
- 18:10 – 18:20      Discussion
- 18:20 – 18:45      **General Discussion**

**Free Evening**

## THURSDAY MORNING, JUNE 26, 2008

### Interfaces in Low Dimensional Systems

Session Chair: Jose S. Moya

- 9:00 – 9:30 Maria Varela, S. J. Pennycook, W. Luo, S. T. Pantelides, J. Garcia-Barriocanal, C. Leon, and J. Santamaria – Oak Ridge National Laboratory, Oak Ridge, USA  
**Oxide interfaces under the microscope (OP22)**
- 9:30 – 9:40 Discussion
- 9:40 – 10:10 Amanda K. Petford-Long – Argonne National Laboratory, Argonne, USA  
**The role of interfaces in the behavior of magnetic tunnel junction structures (OP23)**
- 10:10 – 10:20 Discussion
- 10:20 – 11:20 **Poster Session** & Coffee
- 11:20 – 11:50 Elizabeth Dickey – The Pennsylvania State University, Pennsylvania, USA  
**Size effects in the growth of semiconducting nanowires (OP24)**
- 11:50 – 12:00 Discussion
- 12:00 – 12:30 D. Li, A. Kolpack, M. He, J. Garra, A. Rappe, J. Vohs, and Dawn Bonnell – University of Pennsylvania, Philadelphia, USA  
**Controlling oxide-water interfaces to design patterned nanostructures (OP25)**
- 12:30 – 12:40 Discussion
- 12:40 – 13:00 **General Discussion**
- 13:00 – 15:00 **Lunch (and Posters)**

## THURSDAY AFTERNOON, JUNE 26, 2008

### Interfaces in Artificial Biomaterials

Session Chair: Shelley Wiederhorn

- 15:00 – 15:30      Ulrike G.K. Wegst – Drexel University, Philadelphia, USA  
**X-ray microtomography aids the design of new materials (OP26)**
- 15:30 – 15:40      Discussion
- 15:40 – 16:10      Ramon Torrecillas – Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain  
**Structural nanocomposites for top-end functional applications (OP27)**
- 16:10 – 16:20      Discussion
- 16:20 – 16:50      Coffee
- 16:50 – 17:20      Jérôme Chevalier and L. Gremillard – University of Lyon, Villeurbanne, France  
**Low temperature degradation of zirconia ceramics in the presence of water: implication on biomedical implants (OP28)**
- 17:20 – 17:30      Discussion
- 17:30 – 18:00      General Discussion and Closing Remarks

## THURSDAY EVENING, JUNE 26, 2008

**20:00** - Banquet at Gran Hotel Santiago

**ROWLAND M. CANNON – CELEBRATION OF A LIFE IN SCIENCE – TONY TOMSIA**

**POSTER SESSION**  
**MONDAY MORNING, JUNE 23, 2008**  
**P1: Theory and Processing**

**P1-1**

J-M. Albina, M. Mrovec, and Christian Elsässer

Fraunhofer Institute for Mechanics of Materials - IWM, Freiburg, Germany

**First-principles electronic-structure investigation of interfacial properties of electroceramic perovskite materials**

**P1-2**

Nicole A. Benedek<sup>1,2</sup>, A. L.-S. Chua<sup>2</sup>, C. Elsässer<sup>3</sup>, A. P. Sutton<sup>2</sup>, and M. W. Finnis<sup>1,2</sup>

<sup>1</sup>Department of Materials and <sup>2</sup>Department of Physics, Imperial College London, London, UK;

<sup>3</sup>Fraunhofer Institut für Werkstoffmechanik, Freiburg, Germany

**To what extent can interatomic potentials accurately describe grain boundaries in strontium titanate?**

**P1-3**

Marta Suárez, A. Fernández, J. L. Menéndez, and R. Torrecillas

Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain

**Atomic diffusion mechanisms in SPS and conventional sintering in pure and self-doping of Al<sub>2</sub>O<sub>3</sub>**

**P1-4**

Shigeru Fujino, K. Kunikawa, and T. Kajiwara

Department of Chemical Engineering, Kyushu University, Fukuoka, Japan

**Fabrication of transparent silica glass film by extrusion and its application for micro-imprinting**

**P1-5**

Isabel Álvarez and R. Torrecillas

Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain

**System Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>: conventional vs. spark plasma sintering**

**P1-6**

Olga García-Moreno, A. Fernández, and R. Torrecillas

Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain

**Oxide-based nanocomposites with null thermal expansion coefficient**

**POSTER SESSION**  
**MONDAY AFTERNOON, JUNE 23, 2008**  
**P2: Grain Boundaries**

**P2-1**

M. W. Finnis<sup>1,2</sup>, Nicole A. Benedek<sup>1,2</sup>, Shao-Ju Shih<sup>3</sup>, and D. J. H. Cockayne<sup>3</sup>

<sup>1</sup>Department of Materials and <sup>2</sup>Department of Physics, Imperial College London, London, UK;

<sup>3</sup>Department of Materials, University of Oxford, Oxford, UK

**An improved method for calculating grain boundary misorientation relations from electron microscopy data**

**P2-2**

Dominique Chatain<sup>1</sup>, E. S. Siem<sup>1</sup>, and W. C. Carter<sup>2</sup>

<sup>1</sup>Centre Interdisciplinaire de Nanoscience de Marseille CINAM-CNRS, Marseille, France;

<sup>2</sup>Department of Materials Science & Engineering, MIT, Cambridge, MA, USA

**Organization of pores and particles at interfaces**

**P2-3**

Karleen J. Dudeck and D. J. H. Cockayne

Department of Materials, Oxford University, Oxford, UK

**HREM study of the SrTiO<sub>3</sub> 3 (112) grain boundary**

**P2-4**

Shao-Ju Shih<sup>1</sup>, S. Lozano-Perez<sup>1</sup>, N. P. Young<sup>1</sup>, M. Bäurer<sup>2</sup>, M. J. Hoffmann<sup>2</sup>, and D. J. H. Cockayne<sup>1</sup>

<sup>1</sup>Department of Materials, University of Oxford, Oxford, UK;

<sup>2</sup>Institut für Keramik im Maschinenbau, Universität Karlsruhe, Karlsruhe, Germany

**Correlation between segregation and morphology of grain boundaries in Nb-doped polycrystalline SrTiO<sub>3</sub>**

**P2-5**

Michael Bäurer<sup>1</sup>, M. P. Harmer<sup>2</sup>, and M. J. Hoffmann<sup>1</sup>

<sup>1</sup>Institut für Keramik im Maschinenbau, Universität Karlsruhe, Karlsruhe, Germany;

<sup>2</sup>Center for Advanced Materials and Nanotechnology, Lehigh University, Bethlehem, PA, USA

**Grain growth in undoped strontium titanate**

**P2-6**

Ling Yue, D. Weygand, and P. Gumbsch

Universität Karlsruhe, Karlsruhe, Germany

**Study of grain growth in STO: influence of anisotropic grain boundary properties**



**POSTER SESSION**  
**TUESDAY MORNING, JUNE 24, 2008**  
**P3 Metal/Ceramic Interfaces and Nanomaterials**

**P3-1**

Wayne D. Kaplan<sup>1</sup>, H. Meltzman<sup>1</sup>, and T.M. Besmann<sup>2</sup>

<sup>1</sup>Department of Materials Engineering, Technion-Israel Institute of Technology, Haifa, Israel;

<sup>2</sup>Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN, USA

**Transient and equilibrium interface configurations between metals and oxides**

**P3-2**

Manfred Rühle

Max Planck Institute for Metals Research, Stuttgart, Germany

**Metal/oxide interfaces with different lattice mismatches**

**P3-3**

T. A. Guisard Restivo and Sonia R. H. Mello Castanho

Centro de Ciências e Tecnologia de Materiais – CCTM – IPEN, Cidade Universitária, São Paulo, Brazil

**Cu-Ni-YSZ anodes for SOFC by mechanical alloying processing**

**P3-4**

B. Piccione, John Blendell, and R. Edwin García

Materials Engineering, Purdue University, West Lafayette, IN, USA

**Response surface measurement for BiFeO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> multiferroic nanocomposite**

**P3-5**

Carmen Rial, F. Guitián, and A. Gil

Instituto de Cerámica, Universidade de Santiago de Compostela, Santiago de Compostela, Spain

**Grain growth of TiO<sub>2</sub> inks doped with metal oxides**

**P3-6**

Laura Esposito<sup>1</sup>, A. Piancastelli<sup>1</sup>, and H.-J. Kleebe<sup>2</sup>

<sup>1</sup>Institute for Science and Technology of Ceramics, National Research Council, Faenza, Italy;

<sup>2</sup>Institut für Angewandte Geowissenschaften, Technische Universität Darmstadt, Darmstadt, Germany

**Influence of the starting powders and processing features on the microstructure and transparency of YAG-based materials**

**POSTER SESSION**  
**WEDNESDAY MORNING, JUNE 25, 2008**  
**P4: Grain Boundary Films**

**P4-1**

F. J. Palomares<sup>1</sup>, Federico Soria<sup>1</sup>, J.S. Moya<sup>1</sup>, M. Burianek<sup>2</sup>, M. Muehlberg<sup>2</sup>, and H. Schneider<sup>2</sup>

<sup>1</sup>Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain;

<sup>2</sup>Institut fuer Kristallographie, Universitaet zu Koeln, Koeln, Germany

**Surface chemical characterization of thermally treated mullite-type  $\text{Bi}_2\text{Ga}_4\text{O}_9$  single crystals**

**P4-2**

Y. Gaillard, E. Jiménez-Piqué, J. A. Muñoz, J. Valle, and Marc Anglada

Department of Materials Science and Metallurgical Engineering, Universitat Politècnica de Catalunya, (ETSEIB), Barcelona, Spain

**Study of near surface changes in yttria-doped tetragonal zirconia after low temperature degradation**

**P4-3**

Masato Yoshiya<sup>1,3</sup>, I. Tanaka<sup>2</sup>, H. Adachi<sup>2</sup>, and R. M. Cannon<sup>3</sup>

<sup>1</sup>Department of Adaptive Machine Systems, Osaka University, Japan; <sup>2</sup>Department of Materials Science and Engineering, Kyoto University, Japan; <sup>3</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, USA

**Microscopic roles intergranular glassy film in high-purity  $\text{Si}_3\text{N}_4$**

**P4-4**

Mehmet A. Gulgun<sup>1</sup>, S. Sturm<sup>2</sup>, R. M. Cannon<sup>4</sup>, C. Ozaydin<sup>1</sup>, B. Ozdemir<sup>1</sup>, V. Srot<sup>3</sup>, and P. A. van Aken<sup>3</sup>

<sup>1</sup>Sabanci University, Istanbul Turkey; <sup>2</sup>Josef Stefan Institute, Ljubljana, Slovenia; <sup>3</sup>StEM MPI-for Metals Research, Stuttgart, Germany; <sup>4</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, USA

**Yttrium segregation in Y-doped alumina: “disputes and clarifications”. A tribute to a great person being missed badly: Rowland M. Cannon, Jr.**

**P4-5**

Sašo Šturm<sup>1</sup>, M. A. Gülgün<sup>2</sup>, R. M. Cannon<sup>3</sup>, and M. Ruehle<sup>4</sup>

<sup>1</sup>Jožef Stefan Institute, SI-1000 Ljubljana, Slovenia; <sup>2</sup>Sabanci University, FENS, Tuzla, Istanbul, Turkey; <sup>3</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, USA; <sup>4</sup>Max Planck Institute for Metals Research, Stuttgart, Germany

**The effect of Si impurities on the microstructure evolution in Y-doped  $\text{-Al}_2\text{O}_3$**

**POSTER SESSION**  
**WEDNESDAY AFTERNOON, JUNE 24, 2008**  
**P5: Biomaterials**

**P5-1**

Leticia Esteban-Tejeda<sup>1</sup>, A. Esteban-Cubillo<sup>2</sup>, R. Pina-Zapardiel<sup>1</sup>, C. Pecharroman<sup>1</sup>, and J. S. Moya<sup>1</sup>

<sup>1</sup>Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid. Spain; <sup>2</sup>Tolsa S. A. R&D Department, Madrid, Spain.

**Antibacterial activity of silver monodispersed nanoparticles into sepiolite and sepiolite nAg doped glass**

**P5-2**

Sylvain Deville<sup>1</sup>, R. K. Nalla<sup>2</sup>, E. Saiz<sup>3</sup>, and A. P. Tomsia<sup>3</sup>

<sup>1</sup>Laboratory of Synthesis and Functionalisation of Ceramics, Cavaillon, France; <sup>2</sup>Intel Corporation, USA; <sup>3</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, USA

**Reverse engineering the coke formula through freeze-casting of porous ceramics**

**P5-3**

Miriam Miranda<sup>1</sup>, A. Fernández<sup>1</sup>, E. Saiz<sup>2</sup>, A. P. Tomsia<sup>2</sup>, and R. Torrecillas<sup>1</sup>

<sup>1</sup>Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain; <sup>2</sup>Lawrence Berkeley National Laboratory, Berkeley, CA. USA

**Application of new forming and sintering techniques to obtain hydroxyapatite and -TCP nanostructured composites**

**P5-4**

Jaime Franco, P. Hunger, E. Munch, E. Saiz, and A. P. Tomsia

Lawrence Berkeley National Laboratory, Berkeley, CA, USA

**Synthesis of porous ceramic materials**

**P5-5**

Etienne Munch<sup>1</sup>, M. E. Launey<sup>1</sup>, D. H. Alsem<sup>1</sup>, U. G. K. Wegst<sup>2</sup>, E. Saiz<sup>1</sup>, A. P. Tomsia<sup>1</sup>, and R. O. Ritchie<sup>1</sup>

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**Nature-inspired model hybrid composites**

**P5-6**

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Nanomaterials and Nanotechnology Research Center (CINN-CSIC), Oviedo, Spain

**Low-temperature sintered graphite nanocomposites from carbon nanofibers**

**OP1**

## **The influence of interfaces on the fuel efficiency of aeroturbines**

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One of the mechanisms that limits the durability of thermal barrier systems used on hot section airfoils in aero-turbines is the loss of adhesion at the interface between the thermally-grown oxide ( $\gamma$ - $\text{Al}_2\text{O}_3$ ) and the Ni (Al) bond coat alloy. The fundamental phenomena governing this phenomenon are described. The results of first principles calculations are used to characterize trends in the work of separation and the force/separation behavior as functions of the oxide termination, dopant species and impurity segregation. The ensuing parameters are used as input to an embedded zone simulation of crack extension that incorporates the plastic deformation occurring in the alloy, including a plasticity length scale. The results are connected to experimental measurements of the toughness of the interface obtained from buckle propagation measurements.

## OP2

### **Nature-inspired hybrid structural materials**

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The structure of materials invariably defines the mechanical behavior. However, in most materials, specific mechanical properties are controlled by structure at widely differing length scales. Nowhere is this more apparent than with biological materials, which are invariably sophisticated composites whose unique combination of mechanical properties derives from an architectural design that spans nanoscale to macroscopic dimensions. Moreover, they are generally able to defeat the “law of mixtures” by devising such hierarchical structures with weak constituents into strong/tough hybrid materials that display superior properties to their individual constituents. The fracture resistance of such materials originates from toughening mechanisms at each dimension; few engineering composites have such a hierarchy of structure. However, the biomimetic approach has not been that successful because of the difficulty of synthesizing such materials. In this presentation we describe attempts to develop a range of bone- and nacre-like structural materials using a new freeze-casting technique, which utilizes the intricate structure of ice to create hybrid materials with complex lamellar and/or mortar and brick structures modeled across several length-scales. Our initial results show ceramic-polymer and ceramic-metal hybrid materials with toughness well in excess of those expected from a rule of mixtures construction. The architecture and properties of the synthetic materials are compared to their natural counterparts in order to identify the mechanisms that control mechanical behavior over multiple dimensions and propose new design concepts to guide the synthesis of hybrid/hierarchical structural materials with unique mechanical responses.

## OP3

# Interfacial delamination in layered thermal barrier coatings

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This presentation will summarize a collaborative experimental and multi-scale modeling effort to develop a design code for one of the prevailing failure modes in layered thermal barrier systems used for aero-turbines. The failure mechanism to be addressed occurs in systems with two-phase bond coats and is manifest as abrupt delamination along the interface between the thermally grown oxide (TGO) and the intermetallic bond coat. The Wei-Hutchinson interfacial delamination model has been adapted for this problem and used to integrate several important time/cycle dependent phenomena. Atomic level descriptions of the work of separation and interfacial strength are linked to the steady state interfacial toughness by accounting for plasticity in the adjacent bond coat. Extensions based on strain gradient plasticity have been incorporated into the model and complimented by micro-tensile experiments and the use of nanoindentation to determine the strain gradient length scale parameter. Model predictions of interfacial toughness are compared with micro-bending experiments, which when combined with finite element analysis provide an experimental measure of the interfacial toughness. These comparisons point to the role of interfacial chemistry in governing the work of separation and highlight the effect that the work of separation, interfacial strength, bond coat strength, and length scale parameter have on the delamination process.

## OP4

# In-situ transmission electron microscopy studies of the deformation response of nanocrystalline metals

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It has long been known that the grain size of a material has a substantial effect on its mechanical strength, through the well-established Hall-Petch relationship. In the past decade or so, there has been a resurgence of interest in this topic resulting from the ability to create metals with grain sizes in the deep sub-micron to nanocrystalline scale via a variety of processing techniques. In these materials, it has been conjectured that it may no longer be possible to deform individual grains via simple unit dislocation processes, and other mechanisms may be required to achieve plastic flow.

Here we utilize the technique of in-situ transmission electron microscopy to directly image how deformation proceeds in materials with grains sizes in the sub-micron and deep nanocrystalline regime. In the first portion of the presentation, we will review our work in the area of in-situ nanoindentation of sub-micron and nanocrystalline evaporated aluminum, while in the second portion, we will compare these results with in-situ uniaxial straining of pulsed-laser deposited (PLD) nickel.

We have constructed a unique sample holder for transmission electron microscopy that allows us to perform localized nanoindentation into the edge of an electron transparent material. This permits us to dynamically observe the processes by which mechanical deformation proceeds. During the nanoindentation of sub-micron grains, we find that deformation induces grain growth, resulting from grain boundary migration, grain rotation and grain coalescence. In-situ studies of nanograined films suggest that the same mechanisms are operative, though the difficulty of these nanosized grains makes the evidence less clear (Jin, et al. *Acta Mater*, 2004). Additional observations of Mg incorporation on grain boundary migration will be presented. (Soer, et al., 2004).

Uniaxial straining experiments of PLD nickel provide additional strong evidence of grain rotation and grain agglomeration. Through the use of dark-field imaging, we have provided compelling evidence that when the grain size is in the order of 10 nm grain rotation can become a prominent deformation response (Shan, et al. *Science*, 2004). However, even at these small grain sizes, we find that dislocations are trapped within the grains, indicating that dislocation processes are still active (Shan, et al., *PRL* 2007). Additionally, we will discuss how grain agglomeration leads to the dimple fracture surfaces seen in nanocrystalline metals (Shan, et al., *PRL* 2008) and how we can determine internal lattice strains through in-situ experimentation combined with microdiffraction (Shan, et al., *APL*, 2008).

## OP5

# The crystal-melt interface: insights from atomic-scale computer simulations

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The properties of crystal-melt interfaces have long been a topic of substantial interest in materials science, primarily because of their role in governing crystal growth kinetics and solidification morphologies. While the importance of crystal-melt interfaces has long been appreciated, detailed information related to their properties has become available only relatively recently due to advances in both experimental and computational methods. This talk will highlight recent results obtained in the application of atomic-scale molecular-dynamics and Monte-Carlo computer simulations as a framework for calculating structural, thermodynamic and kinetic properties of crystal-melt interfaces. Examples will be discussed, highlighting the influence of crystal structure and alloy composition on crystal-melt interface properties, both faceted and molecularly rough. It will be discussed how the detailed information provided by atomic-scale simulations can be combined with continuum analyses to derive insights into complex morphological phenomena in alloy solidification. We also describe results derived for size-dependent interface kinetics in the faceted growth of nanowires from liquid catalysts.



## OP6

# Nucleation and growth mechanism of ferroelectric domain-wall motion

Andrew M. Rappe

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The motion of domain walls is critical to many applications involving ferroelectric materials, such as fast high-density non-volatile random access memory. In memories of this sort, storing a data bit means increasing the size of one polar region at the expense of another, and hence the movement of a domain wall separating these regions. Experimental measurements of domain growth rates in the well-established ferroelectrics  $\text{PbTiO}_3$  and  $\text{BaTiO}_3$  have been performed, but the development of new materials has been hampered by a lack of microscopic understanding of how domain walls move. Despite some success in interpreting domain-wall motion in terms of classical nucleation and growth models, these models were formulated without insight from first-principles-based calculations, and they portray a picture of a large, triangular nucleus that leads to unrealistically large depolarization and nucleation energies. Here we use atomistic molecular dynamics and coarse-grained Monte Carlo simulations to analyze these processes, and demonstrate that the prevailing models are incorrect. Our multi-scale simulations reproduce experimental domain growth rates in  $\text{PbTiO}_3$  and reveal small, square, critical nuclei with a diffuse interface. A simple analytic model is also proposed, relating bulk polarization and gradient energies to wall nucleation and growth, and thus rationalizing all experimental rate measurements in  $\text{PbTiO}_3$  and  $\text{BaTiO}_3$ .

**OP7**

## **A phase-field model for grain boundaries in ceramics: strontium titanate**

Catherine Bishop

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A phase field model for grain boundaries in ceramic materials will be introduced. The model includes energetic effects from the bulk chemistry and defect populations, local crystallographic orientations and order, and electrostatics. From bicrystal simulations in different environments, grain boundary charge, potential difference with bulk, disorder and defect populations can be determined. The model will be applied to strontium titanate using calculated defect formation energies and tuned using data from experiment such as cation ratio at grain boundaries.

## OP8

# Modelling grain growth in STO ceramics: linking physical properties to measurable statistical quantities

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The tailoring of grain microstructures can today be supported by the simulation of grain growth processes which is based on the physical properties of the interfaces. For STO ceramics, used in microelectronic devices, it is crucial to understanding the formation of intergranular films and their consequences on the grain microstructure.

I will first present an improved three dimensional vertex dynamics simulation [1] which allows to include anisotropic grain boundary (GB) properties. The vertex dynamics method is a mesoscopic method which is optimized to simulate the collective grain growth behavior of large grain populations. Based on experimentally motivated grain boundary energy and mobility information in STO [2], a systematic study of the time evolution of statistical observables of the grain structure is compared to experiments. A relatively small GB energy dependence on the absolute direction of GB normal is found to alter the dynamics quite drastically. Any mean field expectation would be far off. On the contrary, a similar variation in the GB mobility is found to have a negligible effect on the overall dynamics.

Furthermore the occurrence of abnormally growing grains due to an increased GB mobility, e.g. caused by a wetting transitions, is observed only if all GBs of the abnormally growing grain are affected. This suggests that the formation of an intergranular film will be observable at the grain structural scale only after complete wetting of the grain. Such modelling allows to explore the consequences of different assumptions for GB properties on the resulting micro structure to improve thermal treatments.

[1] D. Weygand, Y. Brechet, J. Lepinoux, and W. Gust, *Phil. Mag.* B 79 (1999) 703.

[2] T. Sano, D.M. Saylor and G.S. Rohrer, *J. Amer. Ceram. Soc.* 86 (2003) 1933.

## OP9

# Ultrarapid transient-liquid-phase joining of alumina

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The vast majority of man-made devices require joints between similar or dissimilar materials. Ideally, joining is fast, reliable, and inexpensive. Conventional joining technologies often fail to provide these characteristics for applications involving materials with fragile microstructures or materials intended for high-temperature applications. Difficulties arise because joining temperatures are sufficiently high to degrade the microstructure, the joining time is excessive, or the use temperatures of the joined assemblies are constrained. Transient-liquid-phase (TLP) joining is an elegant means to reduce the joining temperature, and thus protect temperature-sensitive materials. However, TLP bonding is often time-intensive. We show that microengineered Ni/Nb/Ni multilayer interlayers that develop transient-liquid films allow rapid, reliable, transient-liquid-phase bonding of refractory, high-purity, high-strength  $\text{Al}_2\text{O}_3$  ceramics. Ultrahigh strengths, averaging 500 MPa, are attainable with isothermal holds as short as 5 min at 1400°C, and yield interlayers with remelt temperature above 2200°C. The wetting behavior of the TLP on  $\text{Al}_2\text{O}_3$ , the rapid redistribution of Ni within the interlayer, and the joint fracture characteristics will be described. Using multilayer interlayers with similar thermokinetic properties, it should be possible to extend this novel approach to other materials combinations.

## OP10

# Interfaces at solution plasma

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Plasma in gas phase is widely used in many industrial fields such as electronic device manufacturing processes, surface treatment processes and so on. Plasma in solid phase has been utilized finally for surface plasmon resonance (SPR) spectroscopy, nanoparticles, etc., and plasmonics is developing as a new research field.

On the other hand, plasma in liquid phase is not generally well-known, although it has been partially utilized in water treatments and electrical discharge machining. The fundamentals of plasma in liquid phase have not been established, including its generation techniques, its state, and activated chemical species in it. However, it would be reasonable to expect a higher reaction rate under lower-temperature conditions, and the greater chemical reaction variability since the molecular density of liquid is much higher than that of the gas phase.

Fig. 1 shows the three categories of plasma corresponding to the pressure-temperature relation of three phases. This figure summarizes the present status of plasma. We have named the plasma in liquid phase “solution plasma” because we can make variety of plasma by choosing the combinations of solvents and solutes in solutions, and are developing solution plasma processing (SPP). We can use aqueous solutions, nonaqueous ones, liquid nitrogen, supercritical fluids, etc. for SPP.

The detailed structure of solution plasma is unclear at present. A model of the solution plasma is shown in Fig. 2. The plasma is located in the center and surrounded by a gas phase which surrounded by liquid phase. There are two interfaces: plasma/gas interface and gas/liquid interface. The plasma is confined by a condensed phase, which produces unique features of the solution plasma and realizes fast reaction. We need basic studies on the properties of solution plasma and the novel reaction kinetics. Therefore the solution plasma is interesting from the viewpoints of both physics and chemistry. The solution plasma has many potential applications such as nanoparticle syntheses, dispersion of nanoparticles, surface modifications, water treatments, sterilization, recycle of rare metals, decomposition of toxic compounds, etc. This lecture reports on the structure of interfaces at solution plasma and the applications of SPP.

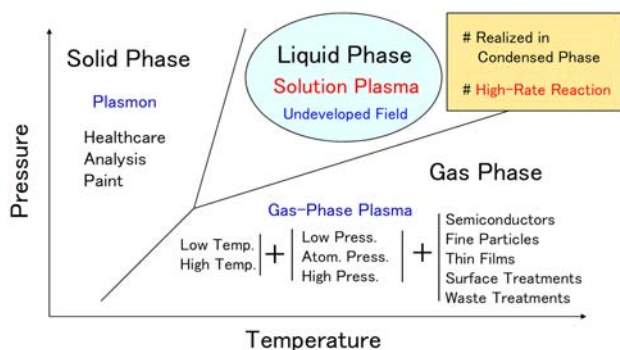


Fig. 1. Three categories of plasma corresponding to the pressure-temperature relation of three phases.

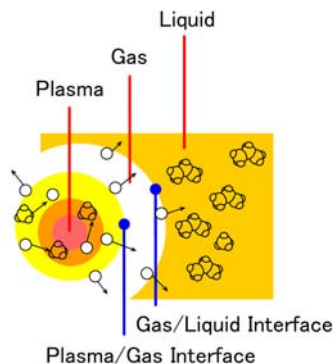


Fig. 2. A model of solution plasma.

## OP11

# The role of dislocations in liquid metal embrittlement

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We considered chemical interdiffusion along a grain boundary in a semi-infinite bicrystal subjected to external stress normal to the boundary plane. Plating out of diffusing atoms in the grain boundary partially relaxes applied stress and modifies the driving force for diffusion. The resulting diffusion wedge formed at the grain boundary can be described in terms of ensemble of continuously spread edge dislocations. We showed that this diffusion wedge exhibits a time-independent shape, with all its linear dimensions growing with annealing time,  $t$ , according to the  $t^{1/3}$  law. The initial stages of development of the grain boundary diffusion wedge were discussed in terms of the recent atomistic model of Nam and Srolovitz. The proposed phenomenological model was applied to the analysis of literature data on liquid metal embrittlement in the Fe-In and Cu-Bi systems.

In the experimental part of this work we studied the liquid metal embrittlement of a martensitic steel (AISI 4340) by Ga-In eutectics. The notch tensile strength of the hardened and tempered samples tested in air was about 2 GPa and did not depend on the deformation rate. Tensile tests of the notched specimens wetted with liquid Ga-In eutectics revealed that the notch tensile strength significantly decreases with a decreasing deformation rate. The activation volume of deformation determined from the experimental data was about twice the atomic volume of steel. Such low value of the activation volume was interpreted in terms of heterogeneous dislocations nucleation at the root of grain boundary groove formed at the solid/liquid interface, in accordance with the Nam-Srolovitz mechanism of liquid metal embrittlement.

**OP12**

## **Reactive wetting in liquid Cu alloy and graphite and SiC systems**

Kiyoshi Nogi

Joining and Welding Research Institute, Osaka University, Japan

Graphite and SiC, which are used as high temperature material, are not usually stable to liquid metals. Reactive wetting phenomena between liquid Cu alloy containing reactive metals such as Ti, Si and Cr and graphite and SiC were investigated from view points of thermodynamics and kinetics.

**OP13**

## **On segregation and plasticity**

Diego Gómez García and Arturo Domínguez Rodríguez

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This talk outlines the role played by cation segregation at the grain boundaries on the plasticity of ceramic polycrystals. A survey on the correlation between limited interface mobility and the steady-state creep response is discussed carefully. A special emphasis is devoted to segregation effects in nanostructured systems, particularly in tetragonal zirconia polycrystals with mean grain sizes within the nanoscale. Several open questions are still under debate on this topic, which will be analysed in detail.



## OP14

# Solid-liquid interfaces at high-temperature

Eduardo Saiz and Antoni P. Tomsia

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Interfacial adhesion and atomic kinetics at high temperature plays a determining role in many modern technologies, from brazing and soldering to composites and thin films. Experiments involving high-temperature liquids are frequently the only available probe to measure the fundamental physical parameters (e.g., work of adhesion or interfacial diffusivity) that control the mechanical behavior of these interfaces, and their formation and evolution. In this presentation, a comprehensive approach that combines analysis of macroscopic and microscopic phenomena such as liquid spreading or capillary driven mass transport with interfacial characterization down to the atomic level is used to study interfacial bonding and dynamics. Particular attention will be given to the role of segregation of “active” elements and reactivity in metal/metal and metal/oxide systems. By outlining the effect of these phenomena a framework for the theoretical analysis of reactive spreading emerges.

## OP15

# Van der Waals - London dispersion interactions for metallic and semiconducting carbon nanotubes from ab initio uniaxial optical properties

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As the drive towards nanoscale assembly continues, the need to understand all the fundamental interactions becomes increasingly important for the purposes of proper and successful experimental design. The van der Waals - London dispersion (vdW-Ld) interaction is a universal component of the long-range van der Waals interaction and arises from the transient induced dipoles, quantified by the optical properties of a material as derived from its interatomic bonding and electronic structure. Dispersion interactions play a role in interface and surface energies, wetting, the formation and stability of surficial and interfacial films, and have increased importance in the nano-scale sciences due to their long range nature and our desire to determine, design and control long range interactions in complex systems.

The calculation of the van der Waals - London dispersion (vdW-Ld) interactions often appear impractical because of the lack of full spectral optical properties and the proper geometrical formulations for meaningful results. These obstacles are removed through 1) the use of ab initio full spectral, uniaxial, optical properties calculated from electronic structure calculations, and 2) with extensions of the Lifshitz formulation to more complex geometrically and optically anisotropic configurations. We previously compared and contrasted the new formulations to simpler geometries, noting their overall similar structure but also highlighting the new and interesting effects (such as vdW-Ld torques) arising from newly introduced terms. Here focus is placed on the usage of spectral mixing formulations for the creation of effective optical properties of more complex systems. Specifically we analyze the effects of the SWCNT core and surfactant materials at near and far distances. Much like the new formulations, proper understanding and usage of these mixing formulations has wide range of utility for many others studying other nanomaterials and biological systems because it allows us to quantify vdW-Ld interactions for more complex, real-world systems.

R. H. French, *J. Am. Ceram. Soc.*, 83, 2117-46 (2000).

R. Rajter, R. Podgornik, V. A Parsegian, R. H. French, W. Y. Ching, *Phys. Rev. B.*, 76, 045417 (2007).

R. F. Rajter, R. H. French, *Journal of Physics: Conference Series*, (2007).

## OP16

# **Tin whisker formation: stress relaxation mechanisms in Sn and Sn-Cu electrodeposited films**

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With the transition to Pb-free solders, the microelectronics industry continues to face the risks of spontaneous Sn whisker formation from electrodeposited Sn coatings on electronic components. Addition of Cu to Sn electrodeposited films has been recognized by numerous researchers, including Tu et al. and Williams et al. as exacerbating Sn whisker formation. The transition from tin whisker formation in Sn-Cu coatings to hillock formation as the Cu concentration is decreased provides insight into the processes available for relaxation of the compressive stresses in the films. By monitoring the film stresses over time using the curvature of plated cantilever beams along with measuring the concurrent microstructural changes including abnormal grain growth in the films, a clearer picture can be obtained of the diffusion and interface processes operating as a function of Cu concentration. The processes operating in Pb-free Sn-based coatings and the possible role of Pb in changing those processes to eliminate whisker formation will be discussed.

**OP17**

## **Chemical control of grain boundary complexions**

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Recent theoretical and experimental work, which builds extensively upon the longstanding and pioneering contributions of the brilliant Rowland M. Cannon, has revealed the existence of grain boundary complexions. Complexions are equilibrium interface ‘phases’ that have associated thermodynamic properties such as excess volume, entropy and adsorption. Alumina has been shown to exhibit as many as six complexions under different conditions of temperature, dopant type and concentration and crystallographic grain misorientation. Each complexion has an associated distinct grain boundary mobility, which can vary by as much as four orders of magnitude between complexions. Through control of the complexions, the grain boundary transport rate and hence the material performance can be engineered towards a given application requirement. The current presentation explores the relationship between chemistry, interfacial energy and complexion stability (number). This has led to the postulation of a criteria for additive selection based upon the relative interphase energy (for second phase precipitation) to grain boundary energy ratio.

## OP18

# Ultra-low thermal conductivity oxides by design

David R. Clarke

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Thermal protection systems for high temperatures are normally based on zirconia as it has, apart from silica-glass, the lowest thermal conductivity at temperatures above 1000°C. Even though its conductivity is unusually low ( $<2.5$  W/mK at 1000°C), there is now a need for materials with even lower conductivity that can be used in air and are also mechanically robust. In the search for oxides with ultralow thermal conductivity, a number of microstructural design strategies have been adopted. These include the use of interfaces, the introduction of mass disorder, complex, incommensurate crystal structures and the nanophase dispersions. These approaches will be compared and used to illustrate the recent identification of several oxides with conductivity of 1 W/mK or less.

## OP19

# Interfacial issues in the materials science of silicon MEMS

Arthur H. Heuer

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MEMS—Micro Electro Mechanical Systems—add sensing and actuation, to the electronic functionality that is the hallmark of the semiconductor revolution. A majority of current commercial devices employ Si as the structural material to take advantage of the extensive Si technology developed for consumer and other devices. However, the complex device geometry and novel applications of many Si MEMS devices has spawned some unusual interfacial issues which had not been observed or been troublesome in purely electronic devices. Three will be addressed in this lecture:

- i) Galvanic corrosion during “release”;
- ii) Control of residual stresses during polysilicon (polycrystalline silicon); and
- iii) Mechanical damage during high cycle fatigue.

## OP20

# Application of electron inline holography to interfaces in ceramics

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A thorough characterization of materials, such as ceramics, whose properties are controlled by internal interfaces, includes the characterization of individual interfaces within the material. Transmission electron microscopy is an excellent tool for investigating structure and composition of such internal interfaces at very high resolution. For thin specimen the phase shift of the electron wave traveling through the sample is proportional to the samples mean inner potential. Profiles of the projected inner potential across interfaces give insight into segregation and accumulation and extent of space charge.

The phase of an electron wave passing through a specimen can only be measured by interfering it with an external reference wave (off-axis holography) or itself (inline holography) [1]. Some of the advantages of inline holography over off-axis holography are: (a) very simple experimental setup (works in any TEM), (b) possibility to record holograms far away from the specimen edge, (c) large fields of view because there is no need to oversample, and (d) specimen drift may easily be compensated, even during exposure.

I will give an introduction to nonlinear electron inline holography, including examples of its application, covering mapping of the magnetisation of magnetic nanostructures, correction of lens aberrations in electron microscopes, reconstructing atomic structure information from HRTEM micrographs, and mapping potential profiles across interfaces.

Emphasis will then be put on tomographic mapping of potential profiles across grain boundaries in ceramics, and how information about space charge layers at these boundaries can be extracted.

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1. C.T. Koch, *Ultramicroscopy* **108** (2008) 141

## OP21

# **Role of interfaces for manufacturing and mechanical behaviour of MMCs produced by liquid metal infiltration of ceramic preforms**

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Applications of light metal castings can be substantially enhanced by local strengthening of highly loaded zones with ceramic preforms. In the present paper we discuss the role of metal-ceramic interfaces for manufacturing and mechanical properties of appropriate composites based on an aluminium-silicon alloy reinforced with alumina. The MMC was prepared by liquid metal infiltration of alumina preforms with unidirectionally aligned pore channels which were produced by freeze-casting of water based alumina suspensions. Pores of the preform are formed by the replica of the former ice crystals. It will be shown that the size and structure of the pore channels of the green preform depend on the water/ice-alumina interface energy and the particle mobility.

The sintered preforms with dense lamellae, separated by pore channels, were subsequently infiltrated with liquid metal by squeeze- and die-casting. The infiltration behaviour depends on the wetting of the ceramic preform by the aluminium melt, the preform permeability and casting pressure. A simple model is derived, which quantifies the contribution of interfacial (wetting) and the hydrodynamic (permeability) effects for a given casting pressure. The model is then used to compare the potential of preforms with different pore structures for liquid metal infiltration. Freeze casted preforms with a lamellar pore structure turn out to be superior at high infiltration velocities, where other preforms with an isotropic pore size distribution fracture from pressure loss.

Finally it will be shown that the properties of the final composite materials depend strongly on the interfacial strength between the solidified metal and the ceramic. We modified the interfacial strength by coating the preform with Ni, Cu, and Ag prior to infiltration. Characterization of the resulting mechanical properties reveal a strong dependency on the nature of the interface.



## OP22

# Oxide interfaces under the microscope

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Oxide interfaces show a very rich range of physical behaviors, some of them absent in the constituent materials. One example is the enhanced ferromagnetism reported at the ferromagnetic/insulating  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$  (STO) interface when a few unit cells of the nominally antiferromagnetic  $\text{LaMnO}_3$  (LMO) compound are inserted [1]. Another can be found in manganite/superconducting interfaces, where new effects have been reported such as giant magnetoresistance [2] or long range proximity effects [3]. Aberration corrected electron probes constitute an irreplaceable tool towards simultaneously probing the structure, chemistry and physical properties of these systems. This talk will review the state of the art of the field and discuss some applications to complex oxide thin films and interfaces of interest in spintronics. The chemistry and electronic properties of several oxide systems such as ferromagnetic/insulating  $\text{LaMnO}_{3+\delta}/\text{SrTiO}_3$  interfaces will be discussed. Our results show how these properties are not consistent with simple ionic models. Other examples will include studies of superconducting/ferromagnetic  $\text{YBa}_2\text{Cu}_3\text{O}_7/\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$  superlattices. A significant amount of charge is transferred from the ferromagnet into the superconductor due to interface band bending. These and other manganite based heterostructures will be discussed, together with pertinent results of density-functional calculations.

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## OP23

# The role of interfaces in the behavior of magnetic tunnel junction structures

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The novel properties of nanoscale layered magnetic structures depend critically on their microstructure and composition, with variations on the atomic scale leading to variations in properties. Magnetic tunnel junctions (MTJs) represent one example of such a structure. In their simplest form MTJs consist of two ferromagnet layers on either side of a nanoscale oxide tunnel barrier. The magnitude of the spin-polarized current that tunnels across the barrier depends on the relative orientation of the magnetization in the two oxide layers. This effect – tunnel magnetoresistance (TMR) is the phenomenon on which a number of technological applications are based, such as read-heads in hard disk drives, and magnetoresistive random access memory. The TMR phenomenon depends critically on the nature of the interfaces between the oxide tunnel barrier and the ferromagnetic layers on either side, as these control the magnetotransport by affecting parameters such as spin polarization, tunnel barrier height, and indeed the shape of the tunnel barrier. We have used high resolution electron microscopy (HREM), transmission electron microscopy (TEM) chemical mapping and atom probe tomography (APT) analysis to obtain high spatial resolution information about the interfaces in MTJs. We have used TEM to study not only the microstructure of the materials, but also to carry out in-situ *local* measurements of their transport properties, which can be directly correlated with the structure. The results of these studies will be presented.

## OP24

# Size effects in the growth of semiconducting nanowires

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The reliable and controllable fabrication of semiconductor heterostructures has for decades made possible the development of many new devices as well as the improvement in speed in existing devices. Similar enabling research is now necessary for nanowire devices. It has been shown that axial-heterostructured nanowires can be grown in which the composition of the nanowire is changed along the length during growth. Such devices have great potential in optical and electronic applications in which quantum confinement leads to behavior differing from the bulk. In addition, radial heterostructures hold great promise for many unique devices, such as nanowire field-effect transistors and nanoscale optical cavities. One critical feature of these nanowire-based structures is the ability to growth nanowires of controlled composition and with high-quality interfaces.

This presentation will address several size-dependent growth phenomena in the synthesis of nanowires by the vapor-liquid-solid mechanism. We quantitatively measure chemistry of individual nanowires at the nanometer length scale and elucidate crystalline defects that can result from heterostructure growth. First, the dependence of alloy  $\text{Si}_{1-x}\text{Ge}_x$  nanowire composition of nanowire radius will be discussed. Then the ability to produce chemically abrupt interfaces in  $\text{Si-Si}_{1-x}\text{Ge}_x$  axial heterostructures will be examined. And finally, the criteria that govern the growth of epitaxial, defect-free axial or radial heterostructures in the nanowire geometry will be addressed along with the prospects for developing strained devices to enhance carrier mobility.

## OP25

# Controlling oxide-water interfaces to design patterned nanostructures

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It is well known that photochemical reactions on oxides depend upon crystal orientation at the solid-liquid interface. Recently, ferroelectric domain orientation has been used to control conditions at solid-liquid interfaces such that oxidation/reduction reactions become domain specific. Controlling this interface enables the design of complex multi component nanostructures on single crystal thin film substrates such as PZT and BaTiO<sub>3</sub>. Here two approaches are taken to probe interactions at these interfaces. For the case of ferroelectric substrates in aqueous metal salt solutions, the factors affecting kinetics (illumination power and reactant concentration) and energetics (substrate band structure and redox potentials) are correlated to the deposition of Ag and Au nanoparticles. To explore the atomistic mechanisms that operate at ferroelectric surfaces, atomic structures and molecular adsorption of a variety of compounds (water, CO<sub>2</sub>, pyridine, alcohol, etc) are quantified. These results are combined to develop a model of interface behavior based on the effects of adsorption on surface band structure.

## OP26

# **X-ray microtomography aids the design of new materials**

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Synchrotron-generated X-rays provide scientists with a multitude of investigative techniques well suited for the analysis of the structure, composition and interfaces of all classes of materials. Discussed will be the properties of synchrotron- versus desktop-generated X-rays and the opportunities and challenges that they provide for the characterization of complex biological and engineered materials. Case studies on the structure and function of insect mouthparts, nutshells, woods and bamboo, freeze-cast biomaterials and a meteorite will be presented to illustrate the qualitative and quantitative techniques of absorption and phase contrast tomography available and the challenges and opportunities that each of these provide for materials research in general and the design of new materials in particular.

## OP27

# Structural nanocomposites for top-end functional applications

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Currently ceramics and composites are not suitable for the industrial development of many technological innovations due to the impossibility of combining in the same material high mechanical performance with critical functional material properties.

Ceramic-ceramic and metal-ceramic nanocomposites entirely made of ceramic and metallic nanoscale particles or nanoscale phases, is a term that denotes a broad class of engineered materials where unique and otherwise unattainable properties can be revealed. The industrial applications of nanocomposites rely on the successful consolidation of these materials preserving their nanostructures. Traditional processing techniques have strong limitations of not being able to retain the nanoscale grain size if conventional nanoparticles are used as starting raw materials.

When it is sought to obtain a dense composite formed by a matrix and a homogeneous distribution of a nanosized second phase, many problems arise for two reasons. First, in conventional powder processing, it is essential to synthesize nanoparticles that have to be non-agglomerated and preferably monodispersed. Second, the obtention of a homogeneous distribution of second phases on the nanometric scale is quite complex.

Additionally hazard studies demonstrate that ultrafine or nanoparticles produce more potent adverse effects in the form of inflammation and subsequent tumors compared with larger-sized particles of identical chemical composition at equivalent mass concentrations or intratracheally-instilled doses. Surface properties, such as surface chemistry and area, may play a significant role in nanoparticle particle toxicity. The very high size-specific deposition of nanoparticles when inhaled as singlet ultrafine particles rather than aggregated ones also contributes to their effect.

During the last years a colloidal processing route was proposed by the authors of this work in order to resolve the above mentioned problems avoiding nanoparticle handling. This route is based on the control of heterogeneous precipitation of different precursors on the surface of ceramic and/or metallic particles and the subsequent crystallization of nanoparticles by an adequate thermal treatment.

Depending on the application field of the obtained nanocomposites, different systems have been studied. In this paper the results corresponding to biomedical, optical and extreme conditions fields will be presented and discussed.

## OP28

# Low temperature degradation of zirconia ceramics in the presence of water: implication on biomedical implants

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The mechanisms responsible for low-temperature degradation (LTD) of zirconia ceramics are first reviewed. The detrimental consequences for biomedical devices are then discussed with a special emphasis on the critical issue of zirconia degradation actually observed for hip prostheses.

The consequences of LTD on the long term performance of zirconia implants are shown, on the basis of hip joint heads retrieval analysis and clinical study: LTD is associated with roughening and microcracking. This impacts the wear performance of hip joint heads, as roughening increases the wear rate of the antagonist part, whereas the coupled effect of surface microcracking and wear generates pull out of zirconia grains. The biological interaction of wear debris with the immune system cells then results in inflammation, osteolysis and implant loosening. For the worst cases, as it was observed in 2001-2002 for Saint Gobain Prozyr® heads, LTD leads to slow crack growth and delayed failure. The potential negative impact of LTD on currently developed alumina-zirconia composites is examined.

# **POSTER ABSTRACTS**



## **P1-1**

# **First-principles electronic-structure investigation of interfacial properties of electroceramic perovskite materials**

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Electroceramic perovskite materials are very attractive for micro- or nanostructured functional devices, e.g., for information or energy storage, for mechanical sensors or actuators, etc., whose functions are controlled to a large extent by interfacial properties. For a targeted optimisation of such devices, fundamental insights into interfacial structure-property relationships at the atomic level are highly valuable but still lacking or difficult to obtain by means of experimental high-resolution techniques. Alternatively, first-principles electronic-structure calculations based on density functional theory are very powerful to provide such insights with strong predictive power. In this contribution, results for interfacial electronic states and energy barriers at a selection of grain boundaries in cubic SrTiO<sub>3</sub> and phase boundaries between SrTiO<sub>3</sub> and metallic or oxidic electrodes will be presented.

## P1-2

# To what extent can interatomic potentials accurately describe grain boundaries in strontium titanate?

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The technological importance of polycrystalline Strontium Titanate (STO) is directly linked to its interfacial and grain boundary properties, which are at present poorly understood. A complete understanding (including links with experiment) requires information from many length scales, including electronic and atomistic, to microstructural and mesoscopic. We have tested the ability of a number of interatomic potentials from the literature to accurately describe at least the structures of some simple grain boundaries in STO. The potentials we have tested are of two types: rigid-ion model with either fixed formal or partial charges and shell model. We have also performed a detailed first-principles Density Functional Theory (DFT) study of the same boundaries and used the resulting interface structures and energies to validate the interatomic potentials. Our conclusion is that none of the chosen potentials can reproduce the energy ordering of the boundaries predicted by the DFT calculations. The boundary structures produced by some of the potentials do however agree reasonably well with the DFT structures.

### **P1-3**

## **Atomic diffusion mechanisms in SPS and conventional sintering in pure and self-doping of $\text{Al}_2\text{O}_3$**

Marta Suárez, A. Fernández, J. L. Menéndez, and R. Torrecillas

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This work explores the use of aluminum alkoxides as additives in order to minimize grain growth during sintering of alumina submicron sized particles. The alkoxides are transformed into alumina in the process of sintering, therefore avoiding light scattering by second phases and favouring the obtention of harder and more transparent alumina materials. The presence of the aluminum alkoxides dispersed on the alumina grains changes the surface energy of the alumina grains and, therefore, the sintering behaviour is modified. The atomic diffusion mechanisms are studied for pure and self doped alumina materials sintered by conventional sintering and spark plasma sintering. It is shown that:

1. Doping with aluminium alkoxides allows obtaining a finer final grain size than in the pure materials for similar final density values.
2. The diffusion mechanisms are modified from volume diffusion driven in the non doped materials to grain boundary in the doped ones.
3. The sintering mechanisms operating in spark plasma sintering and in conventional sintering are the same, but the activation energy is reduced by a factor of 2 in spark plasma sintering.

## P1-4

# **Fabrication of transparent silica glass film by extrusion and its application for micro-imprinting**

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Transparent silica glass is an important material with many desirable properties—low thermal expansion coefficient, low electrical conductivity, high chemical resistance, high UV transparency, etc. It is frequently used for high-efficiency lamps, crucibles for melting high-purity silicon, IC photo-mask substrate, and lens material for excimer stepper equipment. In our previous study, we fabricated transparent silica glass by slip-casting and powder sintering method [1]. The purpose of this paper is to describe the fabrication of transparent silica glass film by sintering a green sheet, which is fabricated using high-purity silica glass powder, organic binder and distilled water. The silica green films were extruded at room temperature by a high-vacuum extrusion machine. It could be formed into a sheet of less than 100  $\mu\text{m}$  in thickness. We found optimum fabrication conditions such as mixture ratio of raw materials, flow characteristic, heating schedule for transparent silica glass films.

Transparent silica glass films were fabricated by sintering a green compact above 1400 C and high-vacuum ( $10^{-4}$  Pa) atmosphere conditions. Microscale surface modification of silica glass films were examined by applying conventional stamp mold. The glass green film imprint was proceeded through the simple steps of pressing (6 MPa) at room temperature. The imprinted transparent films were also fabricated by high-vacuum sintering method. The microimprinted glass surface transferred reversely from the mold.

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## P1-5

# System $\text{Al}_2\text{O}_3\text{-TiO}_2$ : conventional vs. spark plasma sintering

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-alumina has been doped with different percentages of  $\text{TiO}_2$  by a colloidal processing route. The oxidation state of titanium in the resulting powders was analysed by XPS and resulted to be  $\text{Ti}^{3+}$ . The resulting powders were sintered conventionally and by Spark Plasma Sintering. Different sintering behaviour has been found to occur in both techniques. During air sintering, the  $\text{Ti}^{3+}$  remains in solid solution with the  $\text{Al}_2\text{O}_3$  so grain growth is promoted up to 2000 ppm  $\text{TiO}_2$ , when aluminium titanate begins to precipitate and grain growth is inhibited. When  $\text{Al}_2\text{O}_3$ -doped- $\text{TiO}_2$  is sintered by SPS, due to the high heating rates,  $\text{TiO}_2$  remains on grain boundaries and depending on the added quantity of  $\text{TiO}_2$ , the temperature when alumina sintering begins and ends is modified. Grain growth is also promoted when increasing the percentage of  $\text{TiO}_2$  and the formation limit of aluminium titanate in SPS was also found at 2000 ppm of  $\text{TiO}_2$  but the precipitation of this compound is less enhanced than in conventional sintering.

## P1-6

# Oxide-based nanocomposites with null thermal expansion coefficient

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Very low or null thermal expansion materials have many applications from cookware to aerospace industry. Among others, lithium aluminosilicates (LAS) are the most studied family with low and negative thermal expansion properties. However, LAS materials are quite poor in mechanical properties. Nanocomposites using LAS as the matrix component are promising in many applications where better properties are required thanks to the addition of second phases with better properties. In this study we will show the capability of the design of an alumina-LAS nanocomposite. The main scope of this work is to study the solid state compatibility of the alumina and LAS phases, to control the sinterability of the composites and to design a composition for a null thermal expansion nanocomposite. For this purpose, Taimei alumina (TM-DAR) powders and an ad-hoc synthesized  $\beta$ - eucryptite phase were used to fabricate the nanocomposite. XRD phase compositions and microstructures are discussed together with data from dilatometries in a wide temperature range. The obtained results, in terms of solid state compatibility, make it possible to design a nanocomposite with null thermal expansion coefficient.

## P2-1

# **An improved method for calculating grain boundary misorientation relations from electron microscopy data**

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Much of our understanding of the structure of grain boundaries comes from High Resolution Transmission Electron Microscopy (HRTEM) data. The determination of a grain boundary misorientation relation for a particular interface usually starts with the accurate measurement of the positions of a number of poles in each grain. This information is then used to calculate a misorientation axis and angle. The calculation can be performed ‘by hand’, however this approach is tedious and prone to errors. Rotation matrix methods can be implemented into computer programs and this helps to reduce calculation errors. The disadvantage in this case is that there are two different but equivalent definitions for the rotation angle and the answer depends significantly on which definition is chosen to perform the calculations. We present an alternative procedure for calculating misorientation axes and angles, which is based on the quaternion formulation of rotations. The method is formally rigorous and does not suffer from the non-uniqueness problems which plague rotation matrix methods. In addition, the quaternion method provides a route for calculating the error in the rotation. We demonstrate the new approach by calculating misorientation axes and angles for grain boundaries in Strontium Titanate.

## P2-2

# Organization of pores and particles at interfaces

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How do several finite size, small phases like pores or particles distribute at grain boundaries once they have achieved their equilibrium shape? A wealth of configurations can be generated because interfaces with solids are anisotropic and can have facets. These configurations depend on the relative sizes of the phases in contact and on the wetting anisotropy associated with the interface anisotropy. Impact for applications such as self-organization of small particles at interfaces is presented.



## P2-3

### HREM study of the SrTiO<sub>3</sub> 3 (112) grain boundary

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Grain boundaries play a dominant role in determining electronic and structural properties of polycrystalline ceramic materials such as SrTiO<sub>3</sub>. In order to develop an understanding of how grain boundary structure affects bulk properties, quantitative atomistic structural information is required. Due to their highly controlled nature, low angle coincident site lattice grain boundaries in the form of bicrystals provide ideal candidates for quantitative studies, as experimental results can be compared to modelled structures [1].

HRTEM and HAADF STEM are ideal tools for structural characterisation, however, due to the complex relation between specimen structure and recorded images, image interpretation is not always straightforward. In this work aberration corrected transmission electron microscopy and image simulation have been combined to determine quantitative information about the structure of the symmetric tilt SrTiO<sub>3</sub> 3 (112) boundary. Initially the [111] projection has been studied. Aberration correction allows for spherical aberration to be tuned to optimal values for imaging of light elements [2], while image simulation via multislice calculations allows for comparison between experimental images and modelled structures [3].

Atomic column positions are being determined with high precision and accuracy with the aim of comparing experimentally determined structures with model structures. Attention has been paid to careful data collection and image processing to improve signal-to-noise ratio of the images in order to obtain high precision quantitative data.

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## Correlation between segregation and morphology of grain boundaries in Nb-doped polycrystalline SrTiO<sub>3</sub>

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Grain growth behaviour of donor-doped polycrystalline SrTiO<sub>3</sub> has been widely studied because the dopants improve its semiconductor properties [1, 2]. When the sintering temperature is above the eutectic temperature, the grain boundaries (GBs) contain continuous liquid films which assist interface diffusion resulting in fast growth and causing abnormally large grains, known as abnormal grain growth (AGG) [3, 4], although it is not clear if this film is only stable in the presence of Si impurities [1]. However, in this study, AGG has been found below the eutectic temperature in Nb-doped SrTiO<sub>3</sub> even though no intergranular film is observed.

Using electron back scattered diffraction the abnormally large grains were observed to have frequent {100} and near-{110} GBs. The morphology and the chemical composition of these {100} and near-{110} GBs of abnormally large grains were characterized by high resolution transmission electron microscopy and by energy-dispersive X-ray spectroscopy. HRTEM showed that the {100} boundaries were disordered and planar while near-{110} boundaries were stepped. Ti enrichment was observed at the stepped near-{110} but not at the planar {100} GBs. The experimental results will be discussed in terms of models which consider the substitution of Ti by Nb during growth, and how this might influence grain growth and interface morphology.

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## P2-5

# Grain growth in undoped strontium titanate

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Controlling the microstructure of ceramic materials is essential to achieve good performance in respect to mechanical and electrical properties. The grain growth kinetics after complete densification of the material is depending on the mobility of the grain boundaries. Changes of the mobility with temperature which do not follow an Arrhenius law indicate a change in the composition and structure of the grain boundaries. By this it is possible to monitor changes at the interfaces occurring at high temperature by a microstructural analysis.

In this study grain growth in Strontium Titanate has been studied in the temperature range from 1200°C to 1600°C with a cation stoichiometry ranging from Sr/Ti=1.005 to Sr/Ti=0.996. Several changes in the mobility have been observed, indicating interfacial changes. In a temperature range from 1425°C to 1500°C abnormal grain growth occurs for samples with Sr/Ti<1.002. For selected points of annealing time and temperature, the stoichiometry of grain boundaries has been analysed by energy dispersive x-ray spectroscopy in STEM showing the existence of neutral and Titanium-rich boundaries in all samples, with different occurrence frequency for different annealing parameters and stoichiometries. These differences are in good agreement with the observed grain growth and it can be concluded that the Titanium-rich boundaries reduce the coarsening rate of the microstructure.

## P2-6

# Study of grain growth in STO: influence of anisotropic grain boundary properties

Ling Yue, D. Weygand, and Peter Gumbsch

The grain growth for STO is investigated, using a three dimensional vertex dynamics model, based on the model described [1]. In this approach grain boundaries are discretized and their time evolution is derived from the minimization of the grain boundary (GB) energy. The GB energy is dissipated by the motion of the GBs and triple lines. The original model [1,2] has been extended to handle disorientation and inclination dependent GB energies and mobilities and triple line drag.

It is known that grain boundary properties (energy and mobility) depend on the misorientation and interfacial orientation. In this investigation the grain boundary properties are parametrized by a function, motivated by experimental observation in STO [3]. A systematic study of the influence of structure dependent grain boundary properties on statistical observables, e.g. grain size distribution function, grain growth dynamics and correlation function between grain size and number of neighboring grains is presented and compared to experiments.

Furthermore the improved vertex model is used to explore the three-dimensional analogue of the Neumann-Mullins law for isotropic and anisotropic conditions.

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### P3-1

## Transient and equilibrium interface configurations between metals and oxides

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Preferred orientations of crystallographic planes with solid substrates are known to develop during thin film deposition and solidification. In some cases the preferred orientation can be in the form of a fully defined orientation relationship, and depending upon the specific system, heteroepitaxial growth can occur. However, the relative orientation resulting from thin film deposition is usually a metastable state, and significant changes can occur to the preferred orientation of thin films during thermal annealing. This work explores these issues for FCC metals on the basal plane of  $\alpha$ - $\text{Al}_2\text{O}_3$  (sapphire), via solid-state and liquid-state dewetting of thin metal films. While the preferred orientation of as-deposited FCC films is usually (111), a (200) preferred orientation can develop during annealing as the film approaches equilibrium. The partial pressure of oxygen, both during the deposition process and during the annealing cycle, can significantly influence the preferred orientation. This effect is due to segregation and reconstruction at the metal-alumina interface, which has been explored by aberration corrected transmission electron microscopy.

## P3-2

# **Metal/oxide interfaces with different lattice mismatches**

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Metal/Oxide interfaces can be generated by evaporation of the metal onto the surface of an oxide (usually a single crystalline specimen). Coherent, semi-coherent and (in the mathematical limit) also incoherent interfaces can be generated. Structure and relaxations can be observed and quantitatively analysed by transmission electron microscopy. A coherent interface can be observed at Al/MgAl<sub>2</sub>O<sub>4</sub> and also at Pd/SrTiO<sub>3</sub>. Detailed HRTEM and EELS and ELNES studies were performed at those interfaces. Cu/Al<sub>2</sub>O<sub>3</sub>, Cr/SrTiO<sub>3</sub>, Nb/Al<sub>2</sub>O<sub>3</sub>, are semi-coherent interfaces. In addition to the above studies, also relaxations of atom columns can be analysed. Information on the shear stresses at the interfaces can be obtained.

### P3-3

## **Cu-Ni-YSZ anodes for SOFC by mechanical alloying processing**

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Solid oxide fuel cell (SOFC) has been considered to participate in energetic generation systems wide world due to its high conversion efficiency and energy output besides lower pollutant release. The anode concentrates several functions like fuel oxidation, electronic conductivity, O<sub>2</sub>- ion transport and direct reforming of hydrocarbon or alcohol fuels. Biofuel direct feed is attractive regarding carbon poisoned anode problem can be overcome. Among the new processing methods employed, copper additives are said to improve the anode tolerance to biofuels.

The work shows some results concerning a new cermet material 40 vol% [(Cu)-Ni]-YSZ processed by mechanical alloying (MA) followed by sintering by activated surface method (SAS). The projected cermet microstructure for this application is expected to reach microstructure characteristics that lead to better electric and ionic percolating, higher electrocatalytic activity and fuel reforming. The powder samples prepared by MA optimized conditions show homogeneous mixture. TEM analysis has demonstrated the powder particles are nanosize structured after 2 hours of MA. The aggregate lamellar internal structure was not observed for longer milling time. Suitable sintered pellets could be obtained from these powders, within the required porosity and microstructure. Sintering kinetics studies for pellets Ni-YSZ and Ni-Cu-YSZ indicate 2-steps sintering processes. Cu-bearing anode features the smallest activation energy for sintering and higher densification. Sintered pellets samples were analyzed by MEV showing liquid phase sintering process. X-ray profiles after sintering revealed the Cu is partially alloyed with Ni, while the original peaks remain. Electrical and thermal conductivity measurements were performed to evaluate the materials.

### P3-4

## **Response surface measurement for BiFeO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> multiferroic nanocomposite**

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A thin film BiFeO<sub>3</sub> - CoFe<sub>2</sub>O<sub>4</sub> multiferroic nanocomposite has been investigated to determine the coupling between the two phases in a constrained film by combining piezoelectric force microscopy (PFM) and an externally applied magnetic field (up to 1 kGauss). (The film was provided by R. Ramesh and H. Zheng) Three perpendicular PFM scans were measured to determine the magnitude and direction of then polarization vector on the scale of the microstructure, and the minimum electromagnetic energy required to switch ferroelectric domains 180° was determined. The electric field for switching was found to be strongly dependent on the magnetic field and a minimum magnetic field to locally switch the polarization state was found. It has been proposed that the coupling mechanism between the ferroelectric BiFeO<sub>3</sub> and the ferromagnetic CoFe<sub>2</sub>O<sub>4</sub> is due to the epitaxial alignment of the two phases both in the plane of the film and normal to the film. In addition, a numerical model of the system was developed to evaluate the effect of the spatial interactions at the underlying microstructural features of the film on the magnitude of polarization order parameter. This model is based on the Finite Element Method and includes magnetic, electric, thermal thermodynamic fields, as well as all the relevant electromechanic, thermomechanic and magnetostrictive interactions. Alternative microstructures which would improve multiferroic properties are suggested for future nanocomposites.



### P3-5

## Grain growth of TiO<sub>2</sub> inks doped with metal oxides

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Direct-ink writing (DIW) is a high efficiency and rapidly technique that can be used to fabricate three dimensional micro-periodic structures of metal oxides which are inaccessible by traditional processing routes. This technique involves materials assembly through a layer-by-layer deposition of inks. These structures may find application for photonic materials, gas sensors, micro-fuel cells, biomaterials, etc. Sol-gel inks of functional oxides are able to build photonic crystals by DIW. The main problem in the design of these structures is the control of ink composition, drying parameters and mechanical stability of the structures after calcination. To calcinate the ink structures is essential to transform it to metal oxide structure. Calcining at high temperatures involves significant grain growth and surface roughness that degrade optical properties while a low temperature lead poor mechanic stability.

We have been studied the grain growth of TiO<sub>2</sub> by doped of the inks precursors with several metal oxides at different temperatures. We use TiO<sub>2</sub> because it has advantageous physicochemical properties of our interest as the transparency in the near infrared and a high refraction index. Our results show that it is possible to calcinate the inks at high temperature without a significant grain growth of TiO<sub>2</sub>. That will allow us calcining the ink structure at high temperatures in order to achieve mechanical stability without to degrade the optical properties of the system.

### P3-6

## **Influence of the starting powders and processing features on the microstructure and transparency of YAG-based materials**

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Polycrystalline YAG-based ceramics are currently studied to replace single crystals as solid state laser sources. The transparency higher than 80% requested for this application can be obtained only with fully dense, pore-free materials. In addition a homogeneous microstructure with grains not larger than 10 μm would be preferable.

The materials have been prepared by reactive sintering under high vacuum and in a clean atmosphere furnace using powders previously mixed by wet ball milling, dried and cold isostatically pressed. The results after sintering (density, microstructure by SEM-EDS and TEM and in-line transmittance) have been reported. The influence of the powder grain size, morphology and purity level on the microstructure evolution and phase formation during sintering have been analyzed. In particular the study focused on the mechanism that during the densification process leads to the presence of closed porosity which works as light scattering source for the laser transmission. The different reactivity of the micrometric powders versus the nanometric ones, the presence of aggregates and impurities in the powders, have been also considered as key factors affecting the optical characteristics of the sintered body.



## P4-1

# Surface chemical characterization of thermally treated mullite-type $\text{Bi}_2\text{Ga}_4\text{O}_9$ single crystals

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$\text{Bi}_2\text{Ga}_4\text{O}_9$  with mullite-type crystal structure has recently received great interest for applications in solid oxide fuel cells, oxygen sensors and gas separation membranes (1). In all these cases, both the chemical properties of the material and the stability of the surface are essential to fulfil the expected performances during operation at high temperature. The growth of  $\text{Bi}_2\text{Ga}_4\text{O}_9$  single crystals with mullite-type structure is influenced by incongruent melting associated with low solubility of the compounds in  $\text{Bi}_2\text{O}_3$ , and the steep liquidus curve in the growth range between  $950^\circ\text{C}$  and about  $1080^\circ\text{C}$ . Therefore, the Top Seeded Solution Growth (TSSG) technique has been chosen to produce the single crystals. To avoid melt supercooling and spontaneous nucleation growth rates were kept below  $1.5 \text{ K/d}$ . Clearly transparent, homogeneous and nearly inclusion-free single crystals of  $\text{Bi}_2\text{Ga}_4\text{O}_9$  were obtained up to about  $20 \times 20 \times 50 \text{ mm}$  in size. In the present investigation  $\text{sc-(110)}$  surfaces were submitted to thermal treatments ( $500^\circ\text{C}$ - $800^\circ\text{C}$ ) under ultra high vacuum conditions and irradiation by low energy ions ( $\text{Ar}^+$ ). The surface chemical modifications induced upon these different processes have been followed by *in situ* X-ray photoelectron spectroscopy (XPS) experiments. XPS results showed a preferential sputtering of O atoms bonded to Bi, which produced a reduction of  $\text{Bi}^{3+}$  to  $\text{Bi}^0$ , and the metallic Bi enrichment of the surface; on the contrary,  $\text{Ga}^{3+}$  is not affected by ion bombardment (Fig 1). Upon annealing, simultaneous O and Bi interdiffusion from the bulk and segregation onto the surface, and sublimation of metallic Bi present on the outermost surface layers were observed. These dynamical processes favored a change in the chemical composition of the surface, which above  $600^\circ\text{C}$  recovers the nominal (110) surface stoichiometry and the corresponding  $\text{Bi}^{3+}$  and  $\text{Ga}^{3+}$  oxidation states. This effect was also found to be reversible by annealing the single crystal in air at  $700^\circ\text{C}$ .

1. H. Schneider and S. Kormaneni, Mullite, Wiley-VCH, 2005

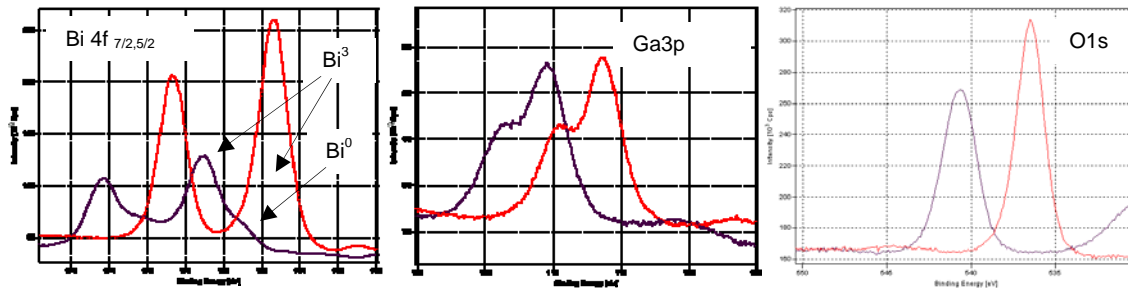


Figure 1.  $\text{Bi}4f$ ,  $\text{Ga}3p$  and  $\text{O}1s$  core level XPS spectra taken with Al-K radiation of  $\text{Bi}_2\text{Ga}_4\text{O}_9(110)$  single crystal for the as-received (red) and ion irradiated (black) surfaces. Spectra are displayed after a shift in energy by the sake of clarity. Similar spectra to as received sample are obtained in UHV annealed samples at  $T = 600^\circ\text{C}$ .

## P4-2

# Study of near surface changes in yttria-doped tetragonal zirconia after low temperature degradation

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Zirconia ceramics doped with different concentrations of Yttria are playing a key role in biomedicine and engineering since they are used in diverse important applications such as in dental ceramics (Zirconia doped with 5-6 mol%  $YO_{1.5}$ ), thermal barriers coatings on Nickel superalloys blades in aero engines (7-8 mol%  $YO_{1.5}$ ), and in solid oxide fuel cells (15 mol%  $YO_{1.5}$ ). In all these three major applications the main concern is related to reliability-durability because of degradation in service under different extreme conditions of temperature, humidity, stresses, etc. In particular, full tetragonal Zirconia alloys with compositions close to 5.8 mol%  $YO_{1.5}$  have high strength and fracture toughness, arising from their submicron average grain size and from the transformation of the tetragonal phase to monoclinic in front of the crack tip with an increment of volume of about 4%, but their reliability is affected by the presence of aqueous environments. In fact, in the presence of humidity the tetragonal phase at the surface can spontaneously revert into monoclinic, producing residual stresses, which originate surface microcracking and loose of structural integrity and reliability, a process well known as hydrothermal degradation or low temperature degradation.

The objective of this work is to evaluate the changes in microstructure, elastic modulus and hardness that occur near the surface of 5.4 mol%  $YO_{1.5}$  tetragonal Zirconia polycrystals during hydrothermal degradation. For this, specimens were subjected to hydrothermal ageing in water at 131°C during different times in order to achieve different levels of degradation. The elastic modulus and the hardness of the degraded surface layer of the aged specimens were extracted from measurements of nanoindentation up to different depths. The degraded layer was examined by scanning electron microscopy in a section perpendicular to the surface which was prepared by Focus Ion Beam machining and revealed the existence of microcracks and transformed grains. The results of nanoindentation are discussed in terms of microcracking and in terms of the depth of the surface degraded layer.

### P4-3

## Microscopic roles intergranular glassy film in high-purity Si<sub>3</sub>N<sub>4</sub>

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Characteristics of intergranular glassy film (IGF) in Si<sub>3</sub>N<sub>4</sub> often determine mechanical properties, such as fracture toughness or creep resistance via crack propagation or grain sliding, respectively. Although much attentions have been paid to the IGF for decades and it has been revealed that IGF contained O that came from surface oxidized layer of starting powder in addition to Si and N, fundamental knowledge on network structure of Si-O-N in IGF and the reason of the existence of IGF at two-grain boundary is still limited. Thus, in this study, three dimensional network structure of amorphous-like Si-O-N film of about 1 nm in thickness was calculated by the use of molecular dynamics simulation and simulated annealing technique. It is found that, while dry grain boundary without the IGF showed larger concentration of dangling bond at grain boundary, the presence of the IGF greatly reduced the dangling bond, thereby accommodating two Si<sub>3</sub>N<sub>4</sub> grains with smaller amount of defects and strain in the vicinity of grain boundary. Furthermore, although bulk Si-O-N glasses prone to have dangling bond due to mixture of atoms with different number density of chemical bond and thus it is unstable in bulk, certain amount of both N and O is needed in the IGF to minimize strain and defects at grain boundary: N is needed to reduce chemical mismatch with Si<sub>3</sub>N<sub>4</sub> grain and O is needed to give greater degree of freedom to accommodate grains with misorientation.

#### P4-4

### **Yttrium segregation in Y-doped alumina: “disputes and clarifications”. A tribute to a great person being missed badly: Rowland M. Cannon, Jr.**

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<sup>2</sup>Josef Stefan Institute, Ljubljana, Slovenia

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Although no one disputes the positive influence of yttrium in creep resistance of alumina, there exists a large variation in the amount of yttrium segregation levels at Al<sub>2</sub>O<sub>3</sub> GBs. Our recent work on precipitation behavior of yttrium doped alumina revealed the unexpected existence of YAP precipitates in the microstructure. Consequent studies showed that the excess yttrium at GBs is different for the microstructures that contain different precipitates as predicted by the activity of yttrium in various features of the microstructure:

$$(\mathbf{a})_x^{\text{bulk}} = (\mathbf{a})_x^{\text{GB}} = (\mathbf{a})_x^{\text{surf.}} = (\mathbf{a})_x^{\text{precipitate}}$$

Since then we have mapped out the temperature, concentration, and time dependencies of precipitation behavior in Y-doped alumina. Our current aim is to understand the temperature dependence of yttrium segregation levels. The most recent results of these investigations will be also presented and discussed.

## P4-5

# The effect of Si impurities on the microstructure evolution in Y-doped $\text{-Al}_2\text{O}_3$

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In spite of the many reports in the literature about the influence of a low concentration of  $\text{SiO}_2$  impurity in an  $\text{Y}_2\text{O}_3$ -doped alumina system there is still a lack of a detailed and systematic structural and compositional analysis of all the observable microstructural features present in the investigated ceramic bodies. In this study a detailed structural and compositional analysis of the precipitates, the triple-point pockets and the grain boundaries was performed. This allowed us to clarify the correlation between the changes in the micro-chemistry and the microstructures in *clean* and in *SiO<sub>2</sub>-contaminated*  $\text{Y}_2\text{O}_3$ -doped alumina. It was shown that *clean*,  $\text{Y}_2\text{O}_3$ -doped alumina can be successfully sintered to densities in excess of 99% of the theoretical density without experiencing any abnormal grain growth. The Y grain-boundary coverage was fairly homogeneous, with a mean value of  $\gamma=5.5$  at./nm<sup>2</sup>. No impurities other than Y were detected in this specimen within the detection limits of the analytical technique used. In contrast, AGG was observed in *Si-contaminated*  $\text{Y}_2\text{O}_3$ -doped alumina samples. The Y-Al-O precipitates observed in the *clean*,  $\text{Y}_2\text{O}_3$ -doped alumina specimen were YAP. In contrast, only the YAG phase was present in *SiO<sub>2</sub>-contaminated*  $\text{Y}_2\text{O}_3$ -doped alumina samples. A critical amount of Y and Si at the grain boundaries (4-5 at./nm<sup>2</sup>) in these samples is believed to have caused the structural transformation of the grain-boundary core. Such a transformation could accelerate the diffusivity along and across the grain boundary, which resulted both in lower kinetic constraints in the formation of the YAG second phases and at the same time allowing grain boundaries to move at high rates by a solution–re-precipitation process, thus inducing AGG.



## P5-1

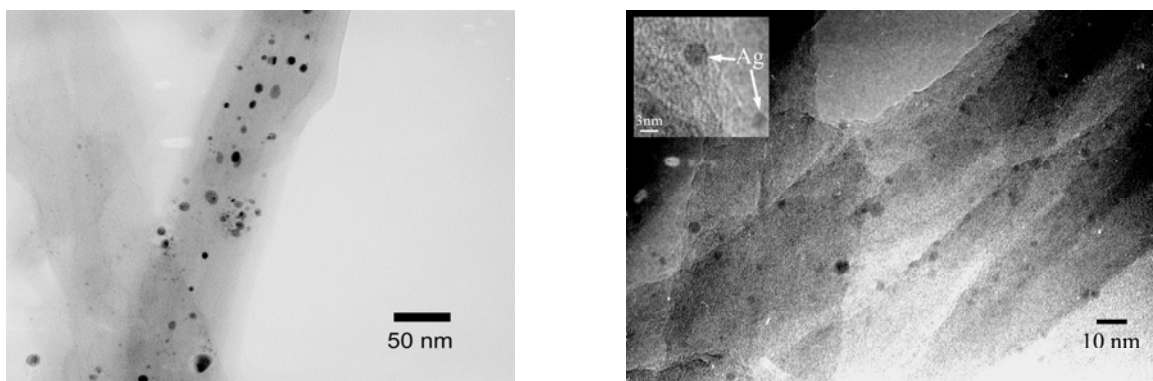
# Antibacterial activity of silver monodispersed nanoparticles into sepiolite and sepiolite nAg doped glass

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<sup>2</sup>Tolsa S. A. R. & D. Department, Ctra. Vallecas-Mejorada del Campo, 28031 Madrid, Spain.

Nowadays, nanotechnology is playing an important role in the field of health. It is well known that silver has antibacterial capabilities, then it has a great deal of interest in biological and biomedical applications (1). In the present investigation silver monodispersed nanoparticles (2-15 nm) embedded into submicron particles of sepiolite ( $\text{Mg}_8\text{Si}_{12}\text{O}_{30}(\text{OH})_4(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}$ ), suitable to be used for this kind of applications have been obtained by a specific treatment and a subsequent reduction process (2). The antibacterial activity of Ag/Sepiolite particles has been tested following the “Shake Flash Method” according to the ASTM E 2149-01. The microorganism used for the test were *Staphylococcus aureus* and *Escherichia coli*. This method is based on the reduction of the bacterial concentration in function of the shake time. The bacterial suspensions were kept with agitation during 72h. Antibacterial activity was identical to the Triclosan (chlorinated aromatic), a commercial broad-spectrum antibacterial/antimicrobial agent, so that these silver monodispersed nanoparticles were able to decrease the starting microorganism concentrations of *Staphylococcus aureus* or *Escherichia coli*. On the contrary the pure sepiolite showed any antibacterial activity. The high effectiveness of the Ag/Sepiolite system is the combination of the effect of silver spreaded along the sepiolite and the large specific surface of sepiolite. Antibacterial activity of silver depends on as much as free surface on liberate ions, therefore the role of sepiolite to avoid agglomeration is crucial. Considering the complex rheological behaviour of sepiolite fibres (3) and in order to increase their field of applications, i.e. as polymers filler or paints, a similar study were performed for a sepiolite-nAg (0.5-10 wt %) + low melting soda-lime glass mixture which was CIP into pellets, sintered at 750-900°C and subsequently attrition milled to obtain a nAg doped-glass powder (<35  $\mu\text{m}$ ). The antibacterial activity of this powder is also reported.



**Figure 1.** TEM micrographs of Ag/Sepiolite. Detail of n-Ag particles embedded in the fibers is shown.

1. A. Esteban-Cubillo, C. Pecharromán, E. Aguilar, J. Santarén and J. S. Moya, Antibacterial Activity of copper monodispersed nanoparticles into sepiolite, *J. Mater Sci*, 2006
2. A. Esteban-Cubillo, R. Pina-Zapardiel, J. S. Moya, M. F. Barba and C. Pecharromán, The role of magnesium on the stability of crystalline sepiolite structure, *J. of European Ceramic Society*, 2008
3. M. Alkan, O. Demirbas and M. Dogan, Elektrokinetic properties of sepiolite suspensions in different electrolyte media, *J. of Colloid and Interface Science*, 2004

## P5-2

# Reverse engineering the coke formula through freeze-casting of porous ceramics

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Materials that are strong, ultra lightweight and tough are in demand for a range of applications from automotive to medical. These requirements will best be met by new composite materials whose components and interfaces are engineered at the molecular level, and whose architectures are carefully designed from the meso- down to nano-scale dimensions while combining the favourable characteristics of several components. Although extensive efforts have been put into the development of porous scaffolds for bone regeneration, with encouraging results, all porous materials have a common limitation: the inherent lack of strength associated with porosity. Hence, the development of porous hydroxyapatite scaffolds has been hindered to non-load bearing applications. Nacre (seashells) and bone are frequently used as examples for how nature achieves this through biomineralized, hybrid organic-inorganic composites, highly optimized for specific functions. Unfortunately, it has proven extremely difficult to transcribe nacre-like clever designs into synthetic materials, in part because their intricate structures need to be replicated at several length-scales. We describe here how nacre-like materials can be obtained by controlling the freezing of ceramic slurries followed by subsequent ice sublimation and sintering, leading to multilayered porous ceramic structures with homogeneous and well-defined architecture, which can be subsequently filled with a selected second phase to obtain dense complex composites. Using the unique relationships between the slurries formulation and the structural characteristics and properties of the porous ceramics, and based on coke-based slurries formulation, we are working toward resolving the coke formula through a reverse engineering approach based on the use of various additives.

### P5-3

## Application of new forming and sintering techniques to obtain hydroxyapatite and $\beta$ -TCP nanostructured composites

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Calcium phosphate based bioceramics have an important position in the bone substitutions field, because of their excellent biocompatibility, bioactivity and osteoconduction characteristics. To enhance the bone ingrowth between implants and natural bone a hydroxyapatite/ $\beta$ -tricalcium phosphate (HA/ $\beta$ -TCP) composite has been chosen. The  $\beta$ -TCP is a bioresorbable material, but its biodegradation is too fast, so it is combined with HA, that is stable in body fluids.

The composites were forming by the robocasting technique, which can print ceramic samples with different and controlled shapes, sizes and macroporosities. A ceramic ink is deposited through a specific nozzle of highly concentrated colloidal suspensions (inks) capable of fully supporting their own weight during assembly. The next step is sintering these samples, and with this aim the system HA/ $\beta$ -TCP was studied in conventional furnace, testing different sintering conditions. The final composition in each sample was analyzed by X-Ray Diffraction and quantified with the Rietveld method.

A powder mixture with the same composition of HA/ $\beta$ -TCP was formed and sintered by another no conventional technique: Spark Plasma Sintering, which used pressure and pulsed current through a graphitic die that contain the powder to be sintered; this technique allows to obtain dense materials employing high heating rates and short stays, avoiding grain growth. Finally, the results and microstructures getting with both techniques were compared.

**P5-4**

## **Synthesis of porous ceramic materials**

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One of the major challenges in materials science is the development of porous ceramic structures designed to fulfill an increasing number of functions in technological fields as diverse as transportation, energy or health. The need to combine complex mechanical, biological or thermal responses requires the development of sophisticated materials that associate the intrinsic properties of a given ceramic with a hierarchical architecture whose design spans nanoscale to macroscopic dimensions. This work presents a brief overview of several techniques used to process innovative porous structures in practical dimensions with a wide variety of ceramics: robocasting, freeze casting and sol-gel templating.

Robocasting is a computer guided micro-extrusion process that permits the printing of ceramic scaffolds with very complex geometries. The printing inks can be processed both from ceramic powders or sol-gel precursors. In freeze casting, porous scaffolds are templated by controlling the directional growth of ice crystals on ceramic-based suspensions. The porous structure is modified by changing the composition of the ceramic colloidal suspension, by controlling the freezing conditions and by patterning the cooling surface. In sol-gel templating the porous structures are formed by the controlled crystal growth of volatile inorganic salts inside ceramic sol-gels. This technique creates highly porous interconnected structures. Mechanical response, biological compatibility and specific surface areas are compared in order to evaluate the technological potential of the various structures.

## Nature-inspired model hybrid composites

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Natural materials are sophisticated composites whose properties are invariably far superior to their individual constituent phases. This unique response derives from an architectural design that spans nanoscale to macroscopic dimensions, with precisely and carefully engineered interfaces. This work is focused on developing a new processing technology for the fabrication of a next generation structural materials, inspired by natural structures. The notion of biomimicry and hierarchical organization has received much interest in the materials communities; however, advances have been few if any, primarily due to the lack of processing techniques able to produce such materials in practical dimensions. We have developed a technique, *freeze casting*, which uses the intricate microstructure of ice to template the microstructure of complex lamellar alumina scaffolds. These scaffolds are the base for the fabrication of brick-and-mortar structures through the application of external pressure. Lamellar and brick-and-mortar ceramics are subsequently infiltrated with a second “soft” lubricating phase. The goal is to fabricate hybrid composites with complex hierarchical structures that display far superior properties than their individual constituents. The versatility of the technique allows a systematic exploration of the effect of interfacial adhesion and structural parameters on the materials properties. The work presented here focuses on a model  $\text{Al}_2\text{O}_3$ /Polymethylmethacrylate system. The materials architecture is related to the processing conditions. The mechanical response is discussed and compared to the mechanical behavior of natural composites such as nacre or bone that has attracted much attention in recent years and is still the subject of discussion in the scientific community.

## P5-6

# Low temperature sintered graphite nanocomposites from carbon nanofibers

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Graphite nanocomposites of high density and low temperature sintered are interesting for a wide range of industrial applications. The extraordinary electrical, thermal and mechanical properties of carbon nanofibres (CNFs) have prompted the development of attractive advanced materials with improved properties through its incorporation, for examples, in ceramic matrix composites. It is necessary that these nanocomposites were completely dense, which is particularly difficult in the case of materials with high carbon content. Spark plasma sintering (SPS) technique is presented as a good alternative to the densification of these materials.

The use of electrical discharge machining (EDM) in ceramics is challenging as many ceramic materials are not conductive. A promising solution could be the incorporation of secondary phases in order to increase their conductivity. CNFs were also successfully used to convert insulating nanoceramics to nearly metallic conductive composites.

In this work, they have been studied two systems; CNFs raw material and reinforced CNFs with 20 vol% of  $\text{Al}_2\text{O}_3$ . In both cases, completely dense materials were obtained by spark plasma sintering (SPS) technique. The combined effect of small diameter (20-80 nm) CNFs and high purity grade alumina nanopowder, lead to excellent composites with greatly improved mechanical and electrical properties. Consequently, CNFs emerge as potentially attractive materials low temperature sintered.