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## Schedule At A Glance

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### Sunday – August 4, 2013

Registration	3 – 5 p.m.	Plaza Foyer
Monday – August 5, 2013		
Registration	7 a.m. – 7 p.m.	Plaza Foyer
Opening Remarks	8 – 8:15 a.m.	Pavilion Ballroom
Plenary Session	8:15 – 9 a.m.	Pavilion Ballroom
Concurrent Technical Sessions	9 a.m. – Noon	Pavilion Ballroom, Broadway I-II & Broadway III-IV
Break	10 – 10:15 a.m.	Plaza Foyer
Lunch On Own	Noon – 12:55 p.m.	
Plenary Session	1 – 1:40 p.m.	Pavilion Ballroom
Concurrent Technical Sessions	1:45 – 5:15 p.m.	Pavilion Ballroom, Broadway I-II & Broadway III-IV
Break-sponsored by Netzsch	3:30 – 3:45 p.m.	Plaza Foyer
Welcome Reception & Poster Session 1	5 – 7:30 p.m.	Plaza Foyer

### Tuesday – August 6, 2013

Registration	7:30 a.m. – Noon	Plaza Foyer
Plenary Session	8:10 – 8:55 a.m.	Pavilion Ballroom
Concurrent Technical Sessions	9 a.m. – Noon	Pavilion Ballroom, Broadway I-II & Broadway III-IV
Break	10 – 10:15 a.m.	Plaza Foyer
Free time	Noon – 5 p.m.	
Poster Session 2	5 – 7 p.m.	Plaza Foyer
Banquet	7 – 9:30 p.m.	Pavilion Ballroom

### Wednesday – August 7, 2013

Registration	7:30 a.m. – 4 p.m.	Plaza Foyer
Plenary Session	8:10 – 8:55 a.m.	Pavilion Ballroom
Concurrent Technical Sessions	9:20 a.m. – Noon	Pavilion Ballroom, Broadway I-II & Broadway III-IV
Break	10 – 10:15 a.m.	Plaza Foyer
Lunch On Own	Noon – 12:55 p.m.	
Plenary Session	12:55 – 1:40 p.m.	Pavilion Ballroom
Concurrent Technical Sessions	1:45 – 4:30 p.m.	Pavilion Ballroom, Broadway I-II & Broadway III-IV

## 2013 ICCPS Plenary Speakers

### Pavilion Ballroom

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Monday, August 5th

8:00 a.m. – 8:15 a.m. Opening Remarks



**Monday, August 5th**

**8:15 a.m. – 9:00 a.m.**

**Ludwig Gauckler, ETH, Professor, Switzerland**

**Title: “Innovations Through Processing of Ceramics and Ceramic Composites”**

Gauckler received his degree in physics at the University of Stuttgart and his PhD in materials science in 1977. As senior scientist at the Max Planck Institute for Metals and Materials Research in Stuttgart he carried out research in the area of high performance structural and functional ceramics. He was research associate at the University of Michigan, Ann Arbor in 1977. From 1979 to 1988 he was responsible for the inorganic non-metallic materials development in the central laboratories of Alusuisse-Lonza AG. Since 1988 he is Professor for “Nonmetallic Inorganic Materials” in the Department of Material Science at the ETH-Zurich. He served as head of the Department from 1991-1993. He was guest professor at the Tsinghua University in Beijing in 1993 and at the MIT, Boston in 2001. Gauckler and his co-workers received several awards for their work on colloid chemistry for ceramic processing and high temperature SOFCs, among them the Award for Real Advances in Materials from NASTS and the Federation of Materials Societies. He is Fellow of The American Ceramic Society and served as President of the scientific advisory board of the Swiss Academy of Technical Sciences. He is member of the Academy of Ceramics, and serves on the boards of several high-tech companies and on the editorial boards of the Journal of Electroceramics, Ceramic International and Materials Science and Engineering A. He has published over 180 scientific papers and holds 15 patents.



**Monday, August 5th**

**12:55 p.m. – 1:40 p.m.**

**David Pine, Professor of Physics, Mathematics and Director of the Center for Soft Matter Research, New York University**

**Title: “Colloids with Directional Interaction”**

Pine earned his BS in physics and mathematics from Wheaton College in 1975, and his MS and PhD in physics from Cornell University in 1979 and 1982, respectively. Prior to NYU, he was a professor of chemical engineering and materials at the University of California, Santa Barbara for ten years. He chaired the chemical engineering department from 2001-2004. Pine was also a staff physicist at Exxon Research & Engineering. He has held several appointments in the American Physical Society’s division of condensed matter physics. Pine is also an adjunct professor of chemical engineering at KAIST. He was Co-Editor, The European Physical Journal E (Soft Matter). He is a fellow of the American Association for the Advancement of Science and the American Physical Society.



**Tuesday, August 6th**

**8:10 a.m. – 8:55 a.m.**

**Hiroaki Imai, Professor, Keio University, Japan**

**Title: “Bioinspired Techniques and Mesoscale and Microscale Hierarchical Assembly”**

Imai earned his BS and PhD in applied chemistry at Keio University in 1983 and 1990 respectively. After working at Nippon Sanso Cooperation, he joined the Department of Applied Chemistry, Faculty of Science and Technology, Keio University as a Research Associate in 1993 and was promoted to a Professor in 1999. He was a Visiting Scientist at Princeton University, USA from 1996-97. His research interests are bioinspired and biomimetic processing for hierarchically structured functional materials using self-organization and self-assembly.

## 2013 ICCPS Plenary Speakers Pavilion Ballroom

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**Wednesday, August 7th**

**8:10 a.m. – 8:55 a.m.**

**James J. Watkins, Director, Center for Hierarchical Manufacturing, University of Massachusetts**

**Title: “Roll-to-Roll Processing of Functional Materials and Devices”**

Watkins earned his BS and MS in chemical engineering from The Johns Hopkins University in 1987 and 1988 respectively, and he earned his PhD in polymer science and engineering from the University of Massachusetts in 1997. His research interests include macromolecular templates for functional device structures, materials synthesis and processing in supercritical

fluids, phase behavior and transport in multi-component polymer systems, scalable fabrication of nanostructure materials. Watkins has earned numerous honors and distinctions, including the Camille Dreyfus Teacher-Scholar Award (2000-05), the David and Lucile Packard Foundation Fellowship for Science and Engineering (1998-2003), and the CAREER Award, National Science Foundation (1998-2002).



**Wednesday, August 7th**

**12:55 p.m. – 1:40 p.m.**

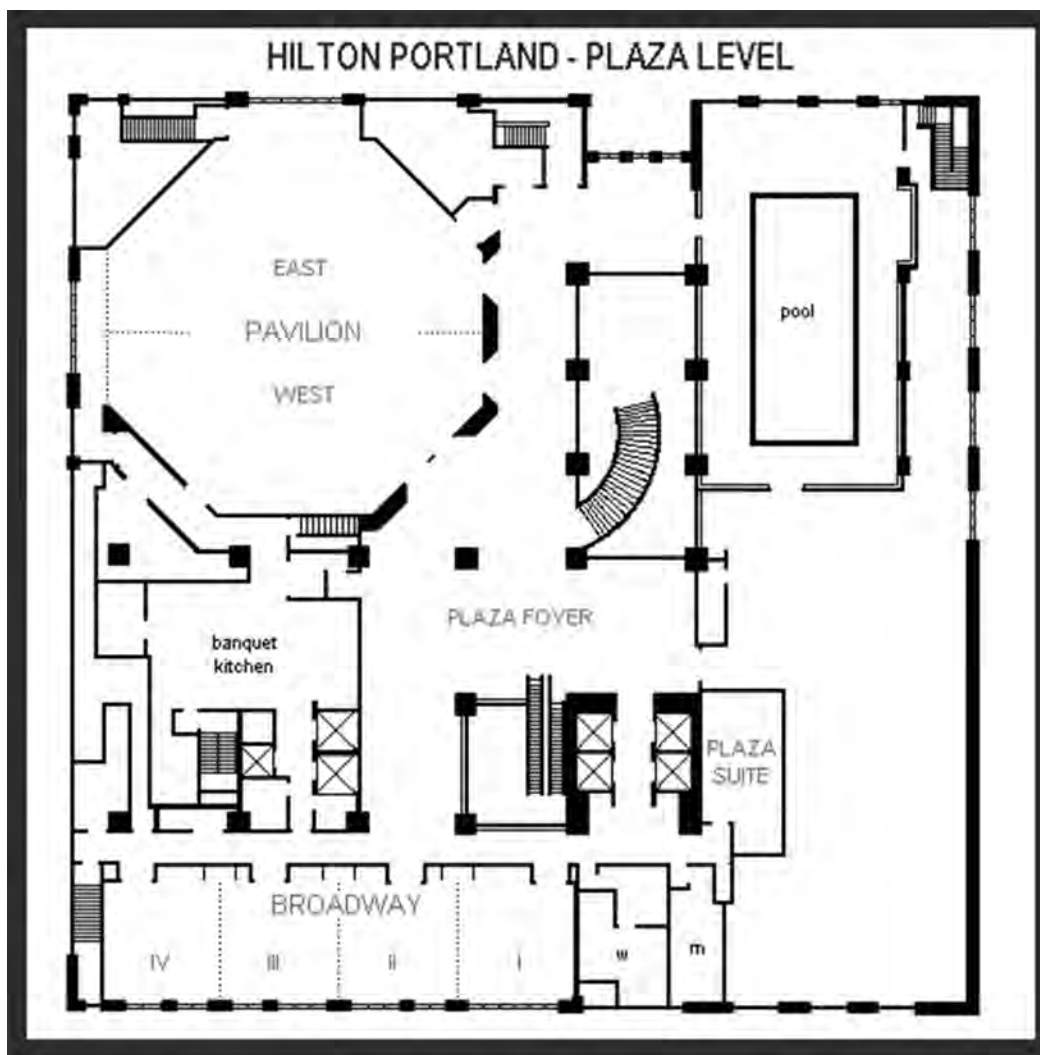
**Christophe Martin, Grenoble INP, France**

**Title: “Simulations of Particle Packing Effects on Sintering Defects and Deformation”**

Martin earned his MS in Mechanical Engineering from MIT in 1992, his PhD in Materials Science from Grenoble INP in 1995, and his HDR in Materials Science from Grenoble INP in 2005. He was a Visiting Scientist at Kyoto University, Japan from 2000-01 and a Visiting Scientist at the University of Washington, Seattle, USA from 2006-07. Martin’s research

activities are focused on particulate materials for materials science applications. It spans from shaping (compaction, sintering) to the behavior of materials elaborated from powders. Materials for energy applications (porous electrodes, nuclear pellets, MLCCs, thermoelectricity) represent typical applications. Discrete element simulations offer a natural and powerful tool for this research. A numerical tool, dp3D, specifically oriented toward materials science applications has been developed since 2001. Simulations are confronted to experimental observations, such as X-ray tomography.

# Hilton Portland & Executive Tower Floor Plan



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## Oral Presenters

Name	Date	Time	Room	Page Number	Name	Date	Time	Room	Page Number
<b>A</b>									
Adair, J.H.	5-Aug	4:30PM	Broadway I - II	11	Kozuka, H.	7-Aug	10:15AM	Broadway I - II	15
Alem, A.	6-Aug	11:00AM	Pavilion Ballroom	13	Kupp, L.	7-Aug	10:30AM	Broadway III - IV	15
Andersson, L.M.	5-Aug	1:45PM	Broadway I - II	10	<b>L</b>				
Andrew, J.S.	6-Aug	11:30AM	Pavilion Ballroom	13	Lanfant, B.	7-Aug	3:30PM	Broadway III - IV	16
Avery, T.	7-Aug	10:30AM	Pavilion Ballroom	14	Leckie, R.M.	7-Aug	3:15PM	Broadway I - II	16
<b>B</b>					Lecomte-Nana, G.	7-Aug	4:30PM	Pavilion Ballroom	16
Baeurer, M.	7-Aug	11:00AM	Broadway III - IV	15	Lee, K.	7-Aug	9:00AM	Broadway I - II	15
Beck, M.	6-Aug	10:15AM	Broadway III - IV	14	Leite, E.R.	5-Aug	3:15PM	Pavilion Ballroom	10
Bell, N.	5-Aug	3:45PM	Pavilion Ballroom	10	Lemke, F.	7-Aug	11:45AM	Broadway III - IV	15
Bell, N.	7-Aug	2:45PM	Broadway I - II	16	Leriche, A.L.	6-Aug	9:30AM	Pavilion Ballroom	13
Bergstrom, L.	6-Aug	10:15AM	Pavilion Ballroom	13	Lewis, J.A.	5-Aug	2:30PM	Pavilion Ballroom	10
Besra, L.	5-Aug	10:15AM	Broadway I - II	9	Libanori, R.	5-Aug	10:30AM	Pavilion Ballroom	9
Bouville, F.	5-Aug	3:30PM	Broadway III - IV	11	Lichtner, A.	7-Aug	11:30AM	Pavilion Ballroom	14
<b>C</b>					Lombardo, S.J.	7-Aug	10:15AM	Broadway III - IV	15
Cabanas-Polo, S.	7-Aug	10:30AM	Broadway I - II	15	Lu, K.	7-Aug	11:00AM	Pavilion Ballroom	14
Cambier, F.J.	5-Aug	3:45PM	Broadway I - II	11	<b>M</b>				
Carty, W.M.	5-Aug	4:00PM	Pavilion Ballroom	10	Maas, M.	5-Aug	9:00AM	Pavilion Ballroom	9
Carty, W.M.	7-Aug	10:45AM	Broadway III - IV	15	Martin, C.L.	7-Aug	12:55PM	Pavilion Ballroom	15
Castro, R.H.	7-Aug	9:00AM	Broadway III - IV	15	Martinez, C.	5-Aug	9:30AM	Pavilion Ballroom	9
Chen, C.	5-Aug	10:30AM	Broadway I - II	9	Mathur, S.	5-Aug	11:30AM	Pavilion Ballroom	9
Chinn, R.E.	7-Aug	10:45AM	Pavilion Ballroom	14	Miller, S.	5-Aug	10:15AM	Broadway III - IV	10
Chlubny, L.	7-Aug	3:45PM	Broadway III - IV	16	Miyayama, M.	5-Aug	11:30AM	Broadway I - II	9
Colombo, P.	7-Aug	2:15PM	Pavilion Ballroom	15	Miyazaki, H.	6-Aug	11:00AM	Broadway III - IV	14
Conrad, J.	7-Aug	9:30AM	Pavilion Ballroom	14	Miyazawa, K.	5-Aug	11:30AM	Broadway III - IV	10
Cordeiro, M.L.	5-Aug	2:45PM	Broadway I - II	10	Mukherjee, A.K.	7-Aug	3:15PM	Broadway III - IV	16
<b>D</b>					Mullens, S.	6-Aug	9:30AM	Broadway I - II	13
Dang, F.	5-Aug	10:45AM	Pavilion Ballroom	9	<b>N</b>				
DeCarlo, K.	5-Aug	4:15PM	Pavilion Ballroom	10	Nakanishi, K.	5-Aug	11:00AM	Broadway III - IV	10
Deville, S.	5-Aug	9:30AM	Broadway III - IV	9	Ni, C.	7-Aug	3:00PM	Broadway I - II	16
Dillon, S.	7-Aug	2:15PM	Broadway I - II	16	<b>O</b>				
<b>F</b>					Ohtaki, M.	5-Aug	4:30PM	Broadway III - IV	11
Fahrenholtz, W.G.	6-Aug	11:00AM	Broadway I - II	14	<b>P</b>				
Francis, L.	7-Aug	9:30AM	Broadway I - II	15	Pascall, A.J.	7-Aug	4:00PM	Pavilion Ballroom	16
Franks, G.V.	5-Aug	9:00AM	Broadway III - IV	9	Pine, D.J.	5-Aug	12:55PM	Pavilion Ballroom	10
<b>G</b>					Porter, M.M.	5-Aug	10:45AM	Broadway III - IV	10
Garnier, V.	6-Aug	9:30AM	Broadway III - IV	14	Poterala, S.F.	5-Aug	1:45PM	Broadway III - IV	11
Gauckler, L.	5-Aug	8:10AM	Pavilion Ballroom	9	Pouchly, V.	7-Aug	11:30AM	Broadway III - IV	15
Gonzalez, J.	7-Aug	2:15PM	Broadway III - IV	16	<b>R</b>				
Grigoryev, E.	7-Aug	3:00PM	Broadway III - IV	16	Rhee, Y.	7-Aug	4:00PM	Broadway III - IV	16
Günster, J.	5-Aug	4:00PM	Broadway I - II	11	Richards, B.T.	5-Aug	2:30PM	Broadway I - II	10
Gupta, S.	5-Aug	10:30AM	Broadway III - IV	10	Roosen, A.	7-Aug	10:45AM	Broadway I - II	15
<b>H</b>					Rosignol, F.	5-Aug	3:00PM	Pavilion Ballroom	10
Hendriksen, P.V.	6-Aug	10:30AM	Pavilion Ballroom	13	<b>S</b>				
Hoffmann, M.J.	7-Aug	9:00AM	Pavilion Ballroom	14	Sakamoto, W.	5-Aug	9:30AM	Broadway I - II	9
Hotza, D.	7-Aug	3:30PM	Broadway I - II	16	Sakka, Y.	5-Aug	2:00PM	Broadway III - IV	11
<b>I</b>					Sander, J.	5-Aug	10:15AM	Pavilion Ballroom	9
Iijima, M.	5-Aug	5:00PM	Pavilion Ballroom	10	Schaffoener, S.	7-Aug	4:15PM	Pavilion Ballroom	16
Imai, H.	6-Aug	8:10AM	Pavilion Ballroom	13	Schmidt, I.	7-Aug	9:30AM	Broadway III - IV	15
Itatani, K.	6-Aug	9:00AM	Broadway I - II	13	Sciti, D.	6-Aug	11:30AM	Broadway I - II	14
<b>J</b>					Seabaugh, M.	5-Aug	10:45AM	Broadway I - II	9
Jiang, D.	6-Aug	10:30AM	Broadway I - II	14	Shinagawa, K.	5-Aug	3:15PM	Broadway III - IV	11
<b>K</b>					Sigmund, W.	7-Aug	1:45PM	Pavilion Ballroom	15
Kakegawa, K.	6-Aug	11:15AM	Broadway III - IV	14	Standing, T.	7-Aug	3:15PM	Pavilion Ballroom	15
Kakimoto, K.	5-Aug	4:00PM	Broadway III - IV	11	Stevenson, A.J.	6-Aug	11:15AM	Pavilion Ballroom	13
Kato, K.	5-Aug	11:00AM	Pavilion Ballroom	9	Stiglich, J.	6-Aug	11:15AM	Broadway I - II	14
Kikkawa, S.	6-Aug	11:45AM	Broadway III - IV	14	Studart, A.R.	6-Aug	9:00AM	Pavilion Ballroom	13
Kirihara, S.	7-Aug	2:30PM	Pavilion Ballroom	15	Suzuki, T.S.	5-Aug	3:00PM	Broadway III - IV	11
Kleebe, H.	6-Aug	10:15AM	Broadway I - II	13	<b>T</b>				
Kobayashi, K.	5-Aug	1:45PM	Pavilion Ballroom	10	Tanaka, S.	7-Aug	10:15AM	Pavilion Ballroom	14
Kobayashi, M.	5-Aug	9:00AM	Broadway I - II	9	Tatami, J.	5-Aug	4:30PM	Pavilion Ballroom	10
					Taylor, B.	5-Aug	3:00PM	Broadway I - II	10
					Tikare, V.	5-Aug	2:30PM	Broadway III - IV	11

# Presenting Author List

## Oral Presenters

Name	Date	Time	Room	Page Number	Name	Date	Time	Room	Page Number
Tolbert, S.	7-Aug	1:45PM	Broadway I - II	16	Wang, Y.	6-Aug	10:45AM	Broadway III - IV	14
Trunec, M.	6-Aug	10:30AM	Broadway III - IV	14	Watkins, J.	7-Aug	8:10AM	Pavilion Ballroom	14
					Wegst, U.	6-Aug	12:00PM	Pavilion Ballroom	13
					Wei, T.	5-Aug	5:00PM	Broadway I - II	11
Vaidhyanathan, B.	7-Aug	1:45PM	Broadway III - IV	16	Wu, Y.	6-Aug	9:00AM	Broadway III - IV	14
Vaucher, S.	5-Aug	3:15PM	Broadway I - II	11	Wu, Y.	7-Aug	2:30PM	Broadway III - IV	16
Videcoq, A.	5-Aug	2:00PM	Pavilion Ballroom	10	<b>Y</b>				
Voorhees, P.	5-Aug	2:00PM	Broadway I - II	10	Yamaguchi, T.	7-Aug	11:15AM	Broadway I - II	15
					Yi, E.	7-Aug	3:00PM	Pavilion Ballroom	15
					Yoshimura, M.	5-Aug	11:00AM	Broadway I - II	9
					<b>W</b>				
Wang, S.	7-Aug	3:30PM	Pavilion Ballroom	16					

## Poster Presenters

Name	Date	Time	Room	Page Number	Name	Date	Time	Room	Page Number
Alexandrova, E.	5-Aug	5:00PM	Plaza Foyer	13	Ku, N.	5-Aug	5:00PM	Plaza Foyer	12
Andersson, L.M.	5-Aug	5:00PM	Plaza Foyer	12	Lecomte-Nana, G.	5-Aug	5:00PM	Plaza Foyer	11
Arakawa, S.	5-Aug	5:00PM	Plaza Foyer	12	Leo, S.	5-Aug	5:00PM	Plaza Foyer	12
Bell, N.	5-Aug	5:00PM	Plaza Foyer	12	Li, X.	5-Aug	5:00PM	Plaza Foyer	12
Camerucci, M.A.	5-Aug	5:00PM	Plaza Foyer	12	Lombardo, S.J.	5-Aug	5:00PM	Plaza Foyer	11, 12
Choi, Y.	5-Aug	5:00PM	Plaza Foyer	12	Masubuchi, Y.	5-Aug	5:00PM	Plaza Foyer	12
Deiner, L.	5-Aug	5:00PM	Plaza Foyer	12	Oh, J.	5-Aug	5:00PM	Plaza Foyer	11
Dylla-Spears, R.	5-Aug	5:00PM	Plaza Foyer	12	Pabst, W.	5-Aug	5:00PM	Plaza Foyer	11
Franks, G.V.	5-Aug	5:00PM	Plaza Foyer	12	Partyka, J.	5-Aug	5:00PM	Plaza Foyer	12
Gajek, M.	5-Aug	5:00PM	Plaza Foyer	12	Pedzich, Z.	5-Aug	5:00PM	Plaza Foyer	12
Gonzalez, J.	5-Aug	5:00PM	Plaza Foyer	13	Pottebaum, A.	5-Aug	5:00PM	Plaza Foyer	11
Gregorova, E.	5-Aug	5:00PM	Plaza Foyer	12	Regiani, I.	5-Aug	5:00PM	Plaza Foyer	12
Hinklin, T.R.	5-Aug	5:00PM	Plaza Foyer	11	Rhee, Y.	5-Aug	5:00PM	Plaza Foyer	13
Hotza, D.	5-Aug	5:00PM	Plaza Foyer	13	Rocha, J.	5-Aug	5:00PM	Plaza Foyer	12
Kandasamy, S.	5-Aug	5:00PM	Plaza Foyer	12	Sanad, M.M.	5-Aug	5:00PM	Plaza Foyer	12
Katagiri, N.	5-Aug	5:00PM	Plaza Foyer	12	Schreiner, T.	5-Aug	5:00PM	Plaza Foyer	12
Katayama, M.	5-Aug	5:00PM	Plaza Foyer	12	Shih, S.	5-Aug	5:00PM	Plaza Foyer	11, 12
Kawaminami, S.	5-Aug	5:00PM	Plaza Foyer	12	Taguchi, H.	5-Aug	5:00PM	Plaza Foyer	12
King, S.	5-Aug	5:00PM	Plaza Foyer	11	Taylor, N.J.	5-Aug	5:00PM	Plaza Foyer	13
Kini, M.K.	5-Aug	5:00PM	Plaza Foyer	12	Vaseem, M.	5-Aug	5:00PM	Plaza Foyer	13
Kobayashi, K.	5-Aug	5:00PM	Plaza Foyer	13	Wang, S.	5-Aug	5:00PM	Plaza Foyer	12
Kolesky, D.B.	5-Aug	5:00PM	Plaza Foyer	12	Watjen, A.	5-Aug	5:00PM	Plaza Foyer	13
Kothanda Ramachandran, D.	5-Aug	5:00PM	Plaza Foyer	12	Zhu, C.	5-Aug	5:00PM	Plaza Foyer	12



## Monday, August 5, 2013

### Plenary Session I

Room: Pavilion Ballroom

Session Chair: Gary Messing, Pennsylvania State University

**8:00 AM**

#### Opening Remarks

Gary Messing, Penn State University; Jennifer Lewis, Harvard University

**8:10 AM**

#### (ICCP-001-2013) Innovations Through Processing of Ceramics and Ceramic Composites

L. Gauckler\*, ETH Zurich, Switzerland

### Microfluidic Assembly and Novel Particle Fabrication

Room: Pavilion Ballroom

Session Chairs: Andre Studart, ETH Zurich; Kazumi Kato, National Institute of Advanced Industrial Science and Technology

**9:00 AM**

#### (ICCP-006-2013) From Colloids to Capsules: Novel Pathways to Ceramic Microcapsules (Invited)

M. Maas\*, T. Bollhorst, P. Kaempfe, K. Rezwan, University of Bremen, Germany

**9:30 AM**

#### (ICCP-007-2013) Monodisperse Ceramic Particles and Capsules from Double Emulsion Drops (Invited)

C. Martinez\*, C. Ye, Purdue University, USA; P. Colombo, Università di Padova, Italy

**10:00 AM**

Break

**10:15 AM**

#### (ICCP-008-2013) Functional Ceramic Colloidosomes made by Microfluidics

J. Sander\*, A. R. Studart, ETH Zurich, Switzerland

**10:30 AM**

#### (ICCP-009-2013) Bioinspired Composites with Extreme Mechanical Gradients

R. Libanori\*, R. M. Erb, A. Reiser, H. Le Ferrand, M. J. Sueess, R. Spolenak, A. R. Studart, ETH Zurich, Switzerland

**10:45 AM**

#### (ICCP-010-2013) Self-assembly Process to Construct 3D Superlattice Ceramics

K. Koumoto, C. Wan, F. Dang\*, Nagoya University, Japan

**11:00 AM**

#### (ICCP-011-2013) Synthesis and Assembly of Nano-cube BaTiO<sub>3</sub> Single Crystals through Tailored Liquid (Invited)

K. Kato\*, K. Mimura, F. Dang, National Institute of Advanced Industrial Science and Technology, Japan; H. Imai, Keio University, Japan; S. Wada, The University of Yamanashi, Japan; M. Osada, H. Haneda, National Institute for Materials Science, Japan; M. Kuwabara, Kyushu University, Japan

**11:30 AM**

#### (ICCP-012-2013) Chemically Grown Nanoparticles, Nanowires and Nanocomposites: Processing, Applications and Devices (Invited)

S. Mathur\*, University of Cologne, Germany

### Synthesis and Processing of Thin Films and Coatings

Room: Broadway I - II

Session Chairs: Matthew Seabaugh, NexTech Materials, Ltd.; Wataru Sakamoto, Nagoya University

**9:00 AM**

#### (ICCP-013-2013) Application of Water-soluble Titanium Complexes for Synthesis of Titanium Dioxide with Controlled Structures (Invited)

M. Kobayashi\*, H. Kato, M. Kakihana, Tohoku University, Japan

**9:30 AM**

#### (ICCP-014-2013) Chemical Solution Processing and Properties of Niobate-Based Lead-Free Piezoelectric Thin Films for Thin-Film Actuators (Invited)

W. Sakamoto\*, Y. Nakashima, N. Kondo, T. Matsuda, Nagoya University, Japan; B. Lee, T. Iijima, National Institute Advanced Industrial Science and Technology (AIST), Japan; M. Moriya, T. Yogo, Nagoya University, Japan

**10:00 AM**

Break

**10:15 AM**

#### (ICCP-015-2013) Highly Adherent Ceramic Coating on Metal Surface by Electrophoretic Deposition

S. De, L. Besra\*, S. Bhattacharjee, B. P. Singh, Institute of Minerals & Materials Technology (IMMT), India; T. K. Rout, Tata Steel Ltd, India; D. K. Sengupta, Institute of Minerals & Materials Technology (IMMT), India

**10:30 AM**

#### (ICCP-016-2013) Preparation of Porous Ceria Coating for Resistive Oxygen Sensor

C. Chen\*, K. Chang, Feng Chia University, Taiwan; S. Shih, National Taiwan University of Science and Technology, Taiwan; S. Wang, Southern Taiwan University, Taiwan

**10:45 AM**

#### (ICCP-017-2013) Protective Coatings for Metals in High Temperature Power Systems

M. Seabaugh\*, N. J. Kidner, S. Ibanez, K. Smith, K. M. Chenault, L. B. Thrun, S. L. Swartz, NexTech Materials, Ltd., USA

**11:00 AM**

#### (ICCP-018-2013) Zirconia Coating on Stainless Steel with Integrated Oxides Layer by Growing Integration Layer [GIL] Method (Invited)

M. Yoshimura\*, K. Chen, Y. Lin, C. Hwang, National Cheng Kung University, Taiwan

**11:30 AM**

#### (ICCP-019-2013) Nanosheet-processes for High Performance Electrodes of Energy Storage Devices (Invited)

M. Miyayama\*, S. Sugata, S. Suzuki, The University of Tokyo, Japan

### Processing of Porous Ceramics

Room: Broadway III - IV

Session Chairs: Sylvain Deville, CNRS; Paolo Colombo, University of Padova

**9:00 AM**

#### (ICCP-020-2013) Control of Microstructure of Porous Ceramic Materials (Invited)

G. V. Franks\*, University of Melbourne, Australia

**9:30 AM**

#### (ICCP-021-2013) Ice Templating, Freeze-casting: Been there, done that. Now what ? (Invited)

S. Deville\*, CNRS, France

**10:00 AM**

Break

10:15 AM

**(ICCPs-022-2013) Influence of Freezing Conditions on the Internal Structure of Directionally Freeze-Cast Porous Ceramics**

S. Miller\*, K. Faber, Northwestern University, USA; X. Xiao, Argonne National Laboratory, USA

10:30 AM

**(ICCPs-023-2013) A Novel Method for Manufacturing Porous Ceramics**

S. Gupta\*, M. F. Faisal, T. Hammann, R. Johnson, University of North Dakota, USA

10:45 AM

**(ICCPs-024-2013) Magnetic Freeze Casting inspired by Nature**

M. M. Porter\*, M. A. Meyers, J. McKittrick, University of California, San Diego, USA

11:00 AM

**(ICCPs-025-2013) Hierarchically Porous Ceramic Monolith via Sol-gel with Phase Separation in Aqueous Systems (Invited)**

K. Nakanishi\*, Kyoto University, Japan

11:30 AM

**(ICCPs-026-2013) Structural Characterization of Heat-treated Fullerene Nanowhiskers (Invited)**

K. Miyazawa\*, National Institute for Materials Science, Japan; R. Kano, Yokohama City University, Japan; T. Konno, C. Hirata, T. Wakahara, J. Tang, National Institute for Materials Science, Japan; M. Tachibana, Yokohama City University, Japan

**Plenary Session 2**

Room: Pavilion Ballroom

Session Chair: Jennifer Lewis, Harvard University

12:55 PM

**(ICCPs-002-2013) Colloids with Directional Interactions**

D. J. Pine\*, New York University, USA

**Anisotropic Particles and 3D Assemblies**

Room: Pavilion Ballroom

Session Chairs: Fabrice Rossignol, CNRS; Edson Leite, CNPq

1:45 PM

**(ICCPs-027-2013) Hydrothermal Processing and Dispersion of Nanometer-sized Ceria Crystals with Organic Modification**

K. Kobayashi\*, Y. Miura, M. Haneda, M. Ozawa, Nagoya Institute of Technology, Japan

2:00 PM

**(ICCPs-028-2013) Structure of Colloidal Suspensions: What to Expect from Numerical Simulations (Invited)**

A. Videcoq\*, A. Tomilov, SPCTS, ENSCI, CNRS, France; D. Bochicchio, Università di Genova, Italy; F. Rossignol, C. Pagnoux, T. Chartier, SPCTS, ENSCI, CNRS, France; R. Ferrando, Università di Genova, Italy; T. Ala-Nissilä, Aalto University, Finland

2:30 PM

**(ICCPs-029-2013) Ceramics Assembly: From Model Colloids to 3D Architectures (Invited)**

J. A. Lewis\*, Harvard University, USA

3:00 PM

**(ICCPs-030-2013) Highly Selective and Sensitive Multifunctional Biosensors Fabricated by Coupling Ink-jet Printing, EISA and Click Chemistry**

F. Rossignol\*, O. De Los Cobos, M. Lejeune, M. Colas, CNRS, France; F. Lalloue, H. Akil, University of Limoges, France; C. Carrion, CNRS, France; P. Faugeras, Ester Technopole, France; C. Boissiere, C. Sanchez, X. Cattoen, M. Wong Chi Man, J. Durand, CNRS, France

3:15 PM

**(ICCPs-031-2013) Zirconia Nanocrystals Self-Assembled in Superlattices Directed by the Solvent-Organic Capping Interaction**

C. J. Dalmaschio, A. F. Moura, D. G. Nephew, E. R. Leite\*, Federal University of Sao Carlos, Brazil

3:30 PM

Break

**Non-aqueous Ceramic Dispersions**

Room: Pavilion Ballroom

Session Chairs: Keith DeCarlo, Blasch Precision Ceramics; George Franks, University of Melbourne

3:45 PM

**(ICCPs-044-2013) Electrostatic Dispersion of Sulfosilane Modified Particles in Rubber Fluids**

N. Bell\*, T. J. Boyle, Sandia National Laboratories, USA

4:00 PM

**(ICCPs-045-2013) Evaluation of DLVO Predictions for Non-aqueous Stability of Selected Colloids Using Rheology**

W. M. Carty\*, Alfred University, USA; K. DeCarlo, Blasch Precision Ceramics, USA

4:15 PM

**(ICCPs-046-2013) An Exact DLVO Solution for Various Powders in Non-aqueous Mediums**

K. DeCarlo\*, Blasch Precision Ceramics, USA; W. M. Carty, Alfred University, USA

4:30 PM

**(ICCPs-047-2013) Development of High-performance Si<sub>3</sub>N<sub>4</sub> Ceramics Using Low-cost Raw Si<sub>3</sub>N<sub>4</sub> Powders (Invited)**

J. Tatami\*, Yokohama National University, Japan

5:00 PM

**(ICCPs-048-2013) Controlling Stability of Functional Nanoparticles in Organic Solvents and Polymeric Materials: From Homogeneous Dispersion to Nano-scale Alignment**

M. Iijima\*, H. Kamiya, Tokyo University of Agriculture and Technology, Japan

**Novel Characterization Tools for Ceramic Microstructures**

Room: Broadway I - II

Session Chairs: Peter Voorhees, Northwestern University; Linnéa Andersson, Oregon State University

1:45 PM

**(ICCPs-032-2013) Evaluating Pore Space in Macroporous Ceramics with Water-based Porosimetry**

L. M. Andersson\*, Oregon State University, USA; P. Larsson, Innventia AB, Sweden; L. Wågberg, Royal Institute of Technology, Sweden; L. Bergström, Stockholm University, Sweden

2:00 PM

**(ICCPs-033-2013) 4D Evolution of Material Microstructure (Invited)**

P. Voorhees\*, J. Gibbs, A. Johnson, Northwestern University, USA; C. Park, K. Thornton, University of Michigan, USA; J. Fife, Paul Scherrer Institute, Switzerland; D. Dunand, Northwestern University, USA; K. Chen-Wiegart, Brookhaven National Laboratory, USA

2:30 PM

**(ICCPs-034-2013) Nondestructive Evaluation of Environmental Barrier Coatings for Ceramic Matrix Composites**

B. T. Richards\*, H. Wadley, University of Virginia, USA; P. A. Howell, NASA Langley Research Center, USA

2:45 PM

**(ICCPs-035-2013) Direct Observation of Sintering, Coalescence and Thermal Stability of CeO<sub>2</sub> by in situ ETEM**

M. L. Cordeiro\*, Federal University of São Carlos, Brazil; P. A. Crozier, Arizona State University, USA; E. R. Leite, Federal University of São Carlos, Brazil

3:00 PM

**(ICCPs-036-2013) An Evaluation of Non-destructive Real-time Analysis of Bulk Ceramics Using Raman Spectroscopy and Polarised Light Microscopy with a High Temperature Hot Stage**

B. Taylor\*, S. Blackburn, Birmingham University, United Kingdom; S. T. Welch, Rolls Royce PLC, United Kingdom

**3:15 PM****(ICCP-037-2013) In Situ Time-resolved Synchrotron Radiation Studies of Oxide Ceramics during Ultrafast Microwave Heating**

S. Vaucher\*, K. Ishizaki, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland; M. Stir, University of Berne, Switzerland; A. Cervellino, Paul Scherrer Institute, Switzerland; J. Catalá-Civera, Universidad Politécnica de Valencia, Spain; R. Nicula, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

**3:30 PM****Break****Additive Fabrication of Mesoscale Ceramic Components**

Room: Broadway I - II

Session Chairs: James Adair, Penn State University; Francis Cambier, BCRC

**3:45 PM****(ICCP-049-2013) Shaping of Oxide Ceramics by Laser Assisted Process: Additive Technology (SLM) and Machining in the Green State**

E. Juste, F. Petit, C. Ott, V. Lardot, F. J. Cambier\*, BCRC (EMRA), Belgium

**4:00 PM****(ICCP-050-2013) Powder-based Additive Manufacturing of Ceramic Parts (Invited)**

J. Günster\*, C. Gomes, BAM, Germany; T. Muehler, J. G. Heinrich, TU Clausthal, Germany

**4:30 PM****(ICCP-051-2013) Design and Manufacture of Mesoscale Components and Devices (Invited)**

J. H. Adair\*, M. I. Frecker, G. A. Lesieutre, B. L. Babcox, R. L. Staveky, Penn State University, USA

**5:00 PM****(ICCP-052-2013) 3D Printing of Interdigitated Li-Ion Microbattery Architectures**

T. Wei\*, Harvard, USA; K. Sun, University of Illinois, USA; B. Ahn, Harvard, USA; J. Seo, Korea Advanced Institute of Science and Technology, Democratic People's Republic of Korea; S. J. Dillon, University of Illinois, USA; J. A. Lewis, Harvard, USA

**Microstructure Tailoring of Ceramics**

Room: Broadway III - IV

Session Chairs: Florian Bouville, LSFC/ Saint Gobain CREE; Veena Tikare, Sandia National Laboratories

**1:45 PM****(ICCP-038-2013) Fabrication of Highly  $\langle 001 \rangle_c$  Textured PMN-PT Ceramics by Low-field Dynamic Magnetic Alignment and Templated Grain Growth**

S. F. Poterala\*, Channel Technologies Group, USA; R. J. Meyer, G. L. Messing, Pennsylvania State University, USA

**2:00 PM****(ICCP-039-2013) Texture Developing and some Properties of Feeble Magnetic Ceramics by Colloidal Processing in a Strong Magnetic Field (Invited)**

Y. Sakka\*, NIMS, Japan

**2:30 PM****(ICCP-040-2013) A Review of Meso-scale Simulation Capabilities of Microstructural Evolution in Ceramics (Invited)**

V. Tikare\*, Sandia National Laboratories, USA

**3:00 PM****(ICCP-041-2013) Control of Lamellar Structure with Different Orientated Layers by Using Electric and Magnetic Fields**

T. S. Suzuki\*, T. Uchikoshi, Y. Sakka, National Institute for Materials Science, Japan

**3:15 PM****(ICCP-042-2013) Phase-Field Simulation of Plate-like Grain Growth during Sintering of Alumina**

K. Shinagawa\*, S. Maki, Kagawa University, Japan; K. Yokota, Kagawa Industrial Technology Center, Japan

**3:30 PM****(ICCP-043-2013) Strong and Tough Ceramics by Grain Orientation Control and Interface Design**

F. Bouville\*, Laboratoire de Synthèse et Fonctionnalisation des Céramiques/ Saint Gobain CREE, France; E. Maire, INSA de Lyon MATEIS, France; S. Deville, Laboratoire de Synthèse et Fonctionnalisation des Céramiques/ Saint Gobain CREE, France

**3:45 PM****Break****Synthesis and Processing of Electrical Ceramics**

Room: Broadway III - IV

Session Chair: Stephen Poterala, Channel Technologies Group

**4:00 PM****(ICCP-055-2013) Ceramic Processing and Evaluation of (Na,K)NbO<sub>3</sub>-based Lead-free Piezoelectrics (Invited)**

K. Kakimoto\* Nagoya Institute of Technology, Japan

**4:30 PM****(ICCP-125-2013) Nanostructured Oxide Ceramics for Efficient Thermoelectric Energy Conversion (Invited)**

M. Ohtaki\* Kyushu University, Japan

**Poster Session**

Room: Plaza Foyer

**5:00 PM****(ICCP-P001-2013) Influence of Oxygen Potential and Time on Grain Growth of Cr<sub>2</sub>O<sub>3</sub>-doped UO<sub>2</sub> Pellets**

J. Oh\*, J. Yang, D. Kim, J. Kim, Y. Rhee, K. Kim, Korea Atomic Energy Research Institute, Republic of Korea

**(ICCP-P002-2013) Formulation of Pickering Emulsions Using Natural Phyllosilicates**

G. Lecomte-Nana\*, D. Kpogbemabou, ENSCI - GEMH, France; A. Aimable, M. Bienia, CNRS - SPCTS, France; C. Peyratout, ENSCI - GEMH, France; F. Rossignol, CNRS - SPCTS, France

**(ICCP-P003-2013) Evolved Gas Analysis during the Sintering of Barium Titanate**

S. J. Lombardo\*, M. Moss, University of Missouri, USA

**(ICCP-P004-2013) Porous Alumina and Mullite-based Ceramics prepared with Starch or Wheat Flour: Processing and Characterization**

W. Pabst\*, E. Gregorova, T. Uhlírova, M. Vesely, Institute of Glass and Ceramics (ICT Prague), Czech Republic; M. Camerucci, M. Talou, Instituto de Investigaciones en Ciencia y Tecnología de Materiales (INTEMA), Argentina

**(ICCP-P007-2013) Modeling Supported Experimental Demonstration of Thermo-chemical Expansion**

T. R. Hinklin\*, C. Lewinsohn, Ceramtec Inc., USA

**(ICCP-P008-2013) Design and Synthesis of Spray Pyrolyzed Bioactive Glass**

S. Shih\*, National Taiwan University of Science and Technology, Taiwan; C. Chen, Feng Chia University, Taiwan; Y. Chou, National Taiwan University of Science and Technology, Taiwan

**(ICCP-P009-2013) Structure Property Relationships in Nano-Porous low-k a-SiC:H Dielectric Materials**

S. King\*, Intel Corporation, USA

**(ICCP-P010-2013) Scalable Production Methods for the Controlled Synthesis of TiO<sub>2</sub>-Based Photocatalysts**

A. Pottebaum\*, N. Taylor, R. M. Laine, University of Michigan, USA

**(ICCPs-P011-2013) Investigation and Optimization of Aqueous, High-energy Ball-milling for Producing Fine-grained, Transparent Ceramic Materials**

T. Schreiner\*, H. vom Stein, E. M. Pfaff, Institute for Materials Applications in Mechanical Engineering, Germany

**(ICCPs-P012-2013) Mechanochemical Approaches to Tailoring Nanostructure for Digitally Fabricated Ceramics**

L. Deiner\*, New York City College of Technology (CUNY), USA; M. Rottmayer, B. Eigenbrodt, T. Jenkins, T. Reitz, The Air Force Research Laboratory, USA

**(ICCPs-P013-2013) Developing MgO Support and Dip Coating CGO Slurry for Oxygen Transport Membrane**

D. Kothanda Ramachandran\*, K. Kwok, M. Søgaard, Technical University of Denmark, Denmark; F. Clemens, EMPA, Swiss Federal Laboratories for Materials Science and Technology, Switzerland; A. Kaiser, Technical University of Denmark, Denmark

**(ICCPs-P014-2013) Stabilization of Ceria and Zirconia Polishing Slurries above Isoelectric Point Using Anionic Surfactants**

R. Dylla-Spears\*, L. Wong, P. Miller, M. Feit, W. Steele, T. Suratwala, Lawrence Livermore National Laboratory, USA

**(ICCPs-P015-2013) Colloidal Processing of Difficult-to-Densify Ceramics**

S. Leo\*, University of Melbourne, Australia; C. Tallon, Defence Materials Technology Centre, Australia; G. V. Franks, University of Melbourne, Australia

**(ICCPs-P016-2013) Colloidal Processing of Yttria Powders Composed of Mono-dispersed Spheres**

X. Li\*, J. Li, X. Chen, W. Zeng, J. He, S. Liu, X. Sun, Northeastern University, China

**(ICCPs-P017-2013) Evolution of Grain Boundaries during Initial Stage Sintering of Polycrystalline Zirconia Spheres**

M. K. Kini\*, A. H. Chokshi, Indian Institute of Science, India

**(ICCPs-P018-2013) Novel Yttria Doped Mullite Particles with Increased Properties for Sintering**

J. Rocha\*, S. Sugita, J. Martínez, University of Guanajuato, Mexico; K. Uematsu, S. Tanaka, Nagaoka University of Technology, Japan

**(ICCPs-P020-2013) Effect of Zr<sup>4+</sup> Ion Substitution on the Crystal Structure, Microstructure, Mechanical and Electrical Properties of Porous Cordierite Ceramics**

M. M. Sanad\*, M. M. Rashad, E. A. Abdel-Aal, Central Metallurgical R & D Institute, Egypt; K. W. Powers, Central Metallurgical R & D Institute, USA

**(ICCPs-P022-2013) Evolved Gas Analysis during the Sintering of Ultra-high Temperature Ceramics Based on Zirconium Diboride**

S. J. Lombardo\*, M. Moss, University of Missouri, USA

**(ICCPs-P023-2013) Magnetic Softening of  $\alpha''$ -Fe<sub>16</sub>N<sub>2</sub> Applying Low Temperature Nitridation Method**

Y. Masubuchi\*, H. Sato, T. Motohashi, S. Kikkawa, Hokkaido University, Japan

**(ICCPs-P025-2013) Ceramization of Silicone-based Composites**

Z. Pedzich\*, AGH-University of Science and Technology, Poland; R. Anyszka, D. M. Bielinski, Lodz University of Technology, Poland

**(ICCPs-P026-2013) Auto-Granulation Behavior of Fine Cohesive Powders**

N. Ku\*, F. Nicodemi, R. A. Haber, Rutgers University, USA; M. Ghadiri, University of Leeds, United Kingdom; M. J. Murtagh, P. Oram, Corning Inc., USA

**(ICCPs-P027-2013) Colloidal Processing and Complex Shaping of Ultra High Temperature Ceramics**

G. V. Franks\*, C. Tallon, S. Leo, University of Melbourne, Australia

**(ICCPs-P029-2013) Crystal Growth of SrTiO<sub>3</sub> by Flame-Fusion Method and Preparation of its Raw Material**

S. Kawaminami\*, Nagoya institute of technology, Japan; Y. Kameda, Shinkosha Co., Ltd., Japan; N. Adachi, T. Ota, Nagoya institute of technology, Japan

**(ICCPs-P030-2013) Hydrothermal Synthesis of Pt-loaded Allophane Nanoparticles**

S. Arakawa\*, Y. Matsuura, F. Iyoda, S. Hayashi, M. Okamoto, Toyota Technological Institute, Japan; H. Hayashi, Tsuchiya Co., Ltd., Japan

**(ICCPs-P031-2013) The Microstructure and Mechanical Properties of Fast Firing Glazes within the System CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-TiO<sub>2</sub>**

M. Gajek\*, J. Lis, J. Partyka, AGH University of Science and Technology, Poland

**(ICCPs-P032-2013) Direct Ink Writing of Bio-Inspired, Highly Toughened Hybrid Materials**

C. Zhu\*, W. Floyd, E. Duoss, J. Kuntz, C. Spadaccini, Lawrence Livermore National Lab, USA

**(ICCPs-P033-2013) The Connectedness of Porous Media: Pore Space Topology**

L. M. Andersson\*, A. L. Herring, G. C. Iltis, D. Wildenschild, Oregon State University, USA

**(ICCPs-P034-2013) Glycothermal Synthesis of Alkali Aluminates**

N. Bell\*, G. L. Brennecke, P. Lu, M. A. Rodriguez, Sandia National Laboratories, USA

**(ICCPs-P035-2013) Synthesis of Various Morphological Powders for Strontium Titanate by Spray Pyrolysis**

W. Tzeng, S. Shih\*, National Taiwan University of Science and Technology, Taiwan

**(ICCPs-P036-2013) Morphology Control of Two-dimensional SnS Nanosheets Using SnO and S as Precursor**

S. Wang\*, B. Huang, Southern Taiwan University of Science and Technology, Taiwan; C. Chen, Feng Chia University, Taiwan; S. Shih, National Taiwan University of Science and Technology, Taiwan

**(ICCPs-P037-2013) Shaping of Mullite Green Foams by Protein Thermogelling Process**

M. Sandoval, M. A. Camerucci\*, INTEMA, Argentina

**(ICCPs-P038-2013) Sintering and Characterization of Starch Consolidated Porous Mullite Bodies**

M. H. Talou, M. A. Camerucci\*, Research Institute for Materials Science and Technology (INTEMA), CONICET/UNMdP, Argentina

**(ICCPs-P039-2013) Synthesis and Evaluation of Ferrite-Silica Aerogel Nanocomposite**

N. Katagiri\*, N. Adachi, T. Ota, Nagoya Institute of Technology, Japan

**(ICCPs-P040-2013) Crystal Orientation and Sintering of Cordierite Ceramics prepared from Natural Raw Materials**

M. Katayama\*, J. Nakakuki, Y. Kobayashi, Aichi institute of technology, Japan

**(ICCPs-P041-2013) Differences in Pyrocarbon Matrices made by FB-CVI with Organic Precursors**

I. Regiani\*, J. S. dos Santos, Institute of Technology in Aeronautics, Brazil

**(ICCPs-P042-2013) Preparation of Composite Fe-Ni Thin Sheets with Nano-sized Lithium-Copper Ferrites by Using Self-propagating High Temperature Synthesis and Electroforming**

Y. Choi\*, Dankook University, Republic of Korea; M. Kim, J. Lee, Korea Institute of Materials Science, Republic of Korea

**(ICCPs-P043-2013) Morphology and Properties of TiN Thin Films Deposited onto a  $\beta$ -type Ti Alloy Substrate by Reactive Magnetron Sputtering**

J. Lee, Y. Choi\*, S. Hong, Chungnam National University, Republic of Korea

**(ICCPs-P044-2013) Particle Size Control of Murdochite-type Ni<sub>3</sub>MnO<sub>8</sub>**

H. Taguchi\*, Okayama University, Japan; S. Tahara, Okayama University, Japan; K. Hirota, Doshisha University, Japan

**(ICCPs-P046-2013) Clay Extrusion Process Modelling and Performance Assessment of Vacuum Extruders Using Computational Fluid Dynamics**

S. Kandasamy\*, A. Asthana, A. Young, Sheffield Hallam University, United Kingdom

**(ICCPs-P047-2013) Cellular Alumina Ceramics prepared via Biological Foaming with Yeast**

E. Gregorova\*, T. Uhlírova, W. Pabst, Institute of Chemical Technology, Prague (ICT Prague), Czech Republic

**(ICCPs-P048-2013) Direct-Write Assembly of Copper Oxide and Copper Architectures**

D. B. Kolesky\*, Harvard University, USA; C. Zhu, Lawrence Livermore National Laboratory, USA; J. J. Adams, University of Illinois at Urbana-Champaign, USA; K. Sullivan, E. B. Duoss, Lawrence Livermore National Laboratory, USA; H. Yang, J. Bernhard, University of Illinois at Urbana-Champaign, USA; C. Spadaccini, Lawrence Livermore National Laboratory, USA; J. A. Lewis, Harvard University, USA

**(ICCPs-P049-2013) Modification of Major Surface Parameters of the Ceramic Glaze by the Selective Selection of Grain Size of Raw Materials**

J. Partyka\*, AGH University of Science and Technology, Poland; M. Gajek, AGH University of Science and Technology, Poland; J. Lis, AGH University of Science and Technology, Poland

**(ICCP5-P050-2013) Theoretical and Experimental Investigation of the Role of the Electric Current in the Spark Plasma Processing of a Copper Powder**

E. Alexandrova\*, I. Alexandra, E. Grigoryev, E. Olevsky, Moscow Engineering Physics Institute, Russian Federation

**(ICCP5-P051-2013) Synthesis of Nanostructured Zinc Oxide by means of FAST/ SPS**

B. Bohne, J. Gonzalez\*, O. Guillon, Friedrich Schiller University Jena, Germany

**(ICCP5-P052-2013) Effect of Reduction of Thickness on Microstructure and Technological Properties of Porcelain Tiles**

A. L. Silva, UFSC, Brazil; A. M. Bernardin, UNESC, Brazil; M. Dal Bó, D. Hotza\*, UFSC, Brazil

**(ICCP5-P053-2013) The Search for a Scalable Nano- $\alpha$ -Al<sub>2</sub>O<sub>3</sub> Production Process**

N. J. Taylor\*, A. J. Pottebaum, R. M. Laine, University of Michigan, USA

**(ICCP5-P054-2013) All Layers Inkjet-printed Field Effect Transistors using p-CuO Nanoparticles Based Ink-formulation**

M. Vaseem\*, A. Hong, H. Kim, Y. Hahn, Chonbuk National University, Republic of Korea

**(ICCP5-P055-2013) Fabrication of U3O8-added UO2 Pellet for Compaction and Sintering Behavior Simulation**

D. Kim, J. Yang, Korea Atomic Energy Research Institute, Republic of Korea; S. Park, Pohang University of Science and Technology, Republic of Korea; J. Oh, J. Kim, Y. Rhee\*, K. Kim, Korea Atomic Energy Research Institute, Republic of Korea

**(ICCP5-P056-2013) Direct Inkjet Printing of Aqueous Ceramic Suspensions**

A. Watjen\*, M. Kramer, P. Gingter, R. Telle, RWTH Aachen University, Germany

**(ICCP5-P057-2013) Raman Scattering and Optical Properties of Cerium Dioxide and Related Complex Oxides via Solution Process**

M. Amimoto, Nagoya Institute of Technology, Japan; M. Ozawa, K. Kobayashi\*, Nagoya University, Japan; M. Haneda, Nagoya Institute of Technology, Japan

**Tuesday, August 6, 2013****Plenary Session 3**

Room: Pavilion Ballroom

Session Chair: Lennart Bergstrom, Stockholm University

**8:10 AM**

**(ICCP5-003-2013) Bioinspired Techniques for Mesoscale Hierarchical Assembly of Inorganic Crystals**

H. Imai\*, Keio University, Japan

**Bioinspired Ceramic and Composite Architectures**

Room: Pavilion Ballroom

Session Chairs: Sanjay Mathur, University of Cologne; Anne Leriche, University of Valenciennes

**9:00 AM**

**(ICCP5-056-2013) Bioinspired Composites made through Colloidal Directed Assembly (Invited)**

A. R. Studart\*, ETH Zurich, Switzerland

**9:30 AM**

**(ICCP5-057-2013) Processing of Biomimetic Calcium Phosphate based Ceramic Bone Substitutes (Invited)**

A. L. Leriche\*, J. Hornez, F. Bouchart, E. Meurice, M. Descamps, University of Valenciennes, France; D. Hautcoeur, V. Lardot, BCRC (EMRA), Belgium; M. Gonon, University of Mons, Belgium; F. J. Cambier, BCRC (EMRA), Belgium

**10:00 AM**

**Break**

**Assembly of Ceramic Membranes**

Room: Pavilion Ballroom

Session Chairs: Adam Stevenson, Laboratoire de Synthèse et Fonctionnalisation des Céramiques (LSFC); Jennifer Andrew, University of Florida

**10:15 AM**

**(ICCP5-068-2013) Tailored Zeolite Monoliths for Optimized Gas Separation by Freeze-casting**

L. Bergstrom\*, A. Ojuva, F. Akhtar, Stockholm University, Sweden; A. P. Tomsia, Lawrence Berkeley National Laboratory, USA

**10:30 AM**

**(ICCP5-069-2013) Manufacturing of Dense Ceramic Membranes (Invited)**

P. V. Hendriksen\*, S. Foghmoes, A. Kaiser, Technical University of Denmark, Denmark

**11:00 AM**

**(ICCP5-070-2013) Highly Porous Reaction Bonded Silicon Nitride Foams; Foam Strength and Reaction Bonding Parameters**

A. Alem\*, M. Pugh, R. Drew, Concordia University, Canada

**11:15 AM**

**(ICCP5-071-2013) Sintering and Microstructure Development in Mixed Ionic-Electronic Conductors for Gas Separation Membranes**

A. J. Stevenson\*, S. Richaud, E. Nonnet, M. Arnold, C. Guizard, Laboratoire de Synthèse et Fonctionnalisation des Céramiques (LSFC), France

**11:30 AM**

**(ICCP5-072-2013) Multifunctional Nanofibers: New Methods for Synthesizing Composites on a Fiber (Invited)**

J. S. Andrew\*, University of Florida, USA

**12:00 PM**

**(ICCP5-126-2013) Ice-templated Hybrid Materials (Invited)**

U. Wegst\*, Dartmouth College, USA

**Synthesis and Processing of Biomaterials**

Room: Broadway I - II

Session Chairs: Steven Mullens, Flemish Institute for Technological Research; Kiyoshi Itatani, Sophia University

**9:00 AM**

**(ICCP5-058-2013) Preparation of Porous Spherical Calcium Phosphate Agglomerates prepared by Spray Pyrolysis and Their Application to Biocement (Invited)**

K. Itatani\*, T. Umeda, Sophia University, Japan; I. J. Davies, Curtin University, Australia; Y. Musha, Toho University, Japan

**9:30 AM**

**(ICCP5-059-2013) Ceramic Processing for Health Care Materials and Green Chemistry Applications (Invited)**

S. Mullens\*, J. Van Noyen, J. Lefevre, V. Ozhukil Kollath, M. Gysen, Flemish Institute for Technological Research, Belgium

**10:00 AM**

**Break**

**Processing and Characterization of Structural Ceramics**

Room: Broadway I - II

Session Chairs: William Fahrenholtz, Missouri University of S & T; Dongliang Jiang, Shanghai Institute of Ceramics

**10:15 AM**

**(ICCP5-073-2013) Carbon Depletion near Surfaces in SiCO and SiCN Polymer-Derived Ceramics**

H. Kleebe\*, TU Darmstadt, Germany

**10:30 AM****(ICCP5-074-2013) Properties of Silicon Carbide Ceramics from Aqueous Gelcasting and Pressureless Sintering (Invited)**

D. Jiang\*, Shanghai Institute of Ceramics, China

**11:00 AM****(ICCP5-075-2013) Processing for Improved Thermal Conductivity of Zirconium Diboride**

G. Harrington, J. Loneragan, W. G. Fahrenholtz\*, G. Hilmas, Missouri University of S &amp; T, USA

**11:15 AM****(ICCP5-076-2013) Processing and Testing of High-temperature Structural Ceramic Foams and Fiber-reinforced Ceramics**

J. Stiglich\*, B. Williams, Ultramet, USA

**11:30 AM****(ICCP5-077-2013) Ultrahigh Temperature Ceramics for Thermal Protection Systems, Propulsion and Energy: From Densification to Thermo-mechanical Properties (Invited)**

D. Sciti\*, S. Guicciardi, L. Silvestroni, National Research Council, Italy

## Processing and Characterization of Optical Materials

Room: Broadway III - IV

Session Chairs: Vincent Garnier, Insa de Lyon - Université de Lyon; Yiquan Wu, Alfred University

**9:00 AM****(ICCP5-060-2013) Europium doped Yttrium Aluminum Garnet Transparent Ceramics (Invited)**

Y. Wu\*, S. Chen, Y. Yang, Alfred University, USA

**9:30 AM****(ICCP5-061-2013) Processing Effects on Sintering of Transparent Al<sub>2</sub>O<sub>3</sub> and MgAl<sub>2</sub>O<sub>4</sub> Ceramics by FAST/SPS (Invited)**

V. Garnier\*, L. Lallemand, Insa de Lyon - Université de Lyon, France; N. Roussel, Université de Toulouse, France; G. Bonnefont, N. Brach, G. Fantozzi, Insa de Lyon - Université de Lyon, France; J. Chane, B. Durand, S. Guillemet-Fritsch, Université de Toulouse, France; S. Trombert, L. Bonneau, Baikowski, France

**10:00 AM**

Break

**10:15 AM****(ICCP5-063-2013) Pressureless Sintered Transparent Yttria Alumina Garnet Multilayer Structures via Tape Casting and Lamination**

M. Beck\*, University of Erlangen-Nuremberg, Germany; Y. Menke, Schott AG, Germany; A. Roosen, University of Erlangen-Nuremberg, Germany

**10:30 AM****(ICCP5-065-2013) Polycrystalline Alumina Ceramics Doped with Nanoparticles for Increased Transparency**

M. Trunec\*, K. Maca, Brno University of Technology, Czech Republic

**10:45 AM****(ICCP5-066-2013) Effects of Post-HIPing Thermal Annealing on Properties of Garnet Ceramic Scintillators**

Y. Wang\*, J. Baldoni, Radiation Monitoring Devices, Inc., USA; W. H. Rhodes, C. Brecher, ALEM Associates, USA; K. Shah, Radiation Monitoring Devices, Inc., USA

**11:00 AM****(ICCP5-067-2013) Effect of Surface Chemical Structure of Ag Nanoparticles on Supporting Behavior into TiO<sub>2</sub> Porous Films and their Optical Properties**

H. Miyazaki\*, M. Iijima, Tokyo University of Agriculture and Technology, Japan; M. Ihara, Tokyo Institute of Technology, Japan; H. Kamiya, Tokyo University of Agriculture and Technology, Japan

**11:15 AM****(ICCP5-053-2013) Preparation of Emitter Materials for Photovoltaic Electric Generation (Invited)**

K. Kakegawa\* H. Kubo, H. Tamagawa, N. Uekawa, T. Kojima, Chiba University, Japan

**11:45 AM****(ICCP5-054-2013) Soft Chemical Process for Functional Oxynitrides (Invited)**

S. Kikkawa\* Hokkaido University, Japan

## Wednesday, August 7, 2013

### Plenary Session 4

Room: Pavilion Ballroom

Session Chair: Kunihito Koumoto, Nagoya University

**8:10 AM****(ICCP5-004-2013) Roll-to-Roll Processing of Functional Materials and Devices**

J. Watkins\*, University of Massachusetts, USA

### Flow and Assembly of Dense Suspensions

Room: Pavilion Ballroom

Session Chairs: Michael Hoffmann, University of Karlsruhe; Kathy Lu, Virginia Tech

**9:00 AM****(ICCP5-078-2013) Colloidal Processing of Alumina (Invited)**

M. J. Hoffmann\*, J. Reinshagen, R. Oberacker, University of Karlsruhe, Germany

**9:30 AM****(ICCP5-079-2013) Effects of Confinement on Structure, Dynamics, and Transport of Attractive Colloidal Suspensions (Invited)**

R. Pandey, M. Spannuth, J. Conrad\*, University of Houston, USA

**10:00 AM**

Break

**10:15 AM****(ICCP5-080-2013) Direct Observation of Particle Motion in Ceramic Paste with High Solid Concentration under Low Shear Stress**

S. Tanaka\*, Y. Nagasawa, Y. Takahashi, Z. Kato, K. Uematsu, Nagaoka University of Technology, Japan

**10:30 AM****(ICCP5-081-2013) Effect of Polymer Chain Length on the Rate of Phase Migration**

T. Avery\*, MAST Carbon, United Kingdom; S. Blackburn, University of Birmingham, United Kingdom; S. Tennison, MAST Carbon, United Kingdom; N. Rowson, University of Birmingham, United Kingdom

**10:45 AM****(ICCP5-082-2013) Feedstock Properties and Mold-filling Simulations for Powder Injection-molded Silicon Carbide**

R. E. Chinn\*, National Energy Technology Laboratory, USA; K. H. Kate, S. V. Atre, Oregon State University, USA; V. P. Onbattuvelli, Intel Corp., USA; R. K. Enneti, Global Tungsten Powders, USA

**11:00 AM****(ICCP5-083-2013) Development of a Nanoparticle-based Surface Templating Approach (Invited)**

K. Lu\*, Virginia Tech, USA

**11:30 AM****(ICCP5-084-2013) Aqueous Co-dispersion of LSM-YSZ for Directional Freeze-Casting**

A. Lichtner\*, University of Washington, USA; D. Jauffrès, University of Grenoble, France; J. Villanova, European Synchrotron Radiation Facility, France; D. Roussel, F. Charlot, C. L. Martin, University of Grenoble, France; R. Bordia, University of Washington, USA

**Flexible Electronics**

Room: Broadway I - II

Session Chairs: Lorraine Francis, University of Minnesota; Andreas Roosen, University of Erlangen-Nuremberg

**9:00 AM****(ICCP-086-2013) Self-Powered Flexible Electronic Systems (Invited)**

K. Lee\*, KAIST, Republic of Korea

**9:30 AM****(ICCP-087-2013) Printing of Colloidal Dispersions for Flexible Electronics (Invited)**

A. Mahajan, H. Zhang, S. Lim, A. Ramm, C. Frisbie, L. Francis\*, University of Minnesota, USA; B. Ahn, B. Walker, J. Lewis, Harvard University, USA

**10:00 AM****Break****10:15 AM****(ICCP-088-2013) Fabrication of Ceramic Thin Films on Plastics: A Versatile Route Utilizing Sol-Gel and Transfer Techniques**

H. Kozuka\*, T. Fukui, M. Takahashi, H. Uchiyama, S. Tsuboi, Kansai University, Japan

**10:30 AM****(ICCP-089-2013) Influence of Polymer Nature on Bioactive Glass-based Soft Coatings for Biomedical Applications obtained by EPD**

S. Cabanas-Polo\*, Institute of Biomaterials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany; J. A. Roether, Institute of Polymer Materials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany; A. R. Boccaccini, Institute of Biomaterials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany

**10:45 AM****(ICCP-090-2013) Manufacture of Particulate Structures in the Micrometer Range via Coating and Printing Techniques (Invited)**

A. Roosen\*, University of Erlangen-Nuremberg, Germany

**11:15 AM****(ICCP-091-2013) Low Temperature Densification of Electrolytes by Using Controlled Tubular Electrode Support Shrinkage (Invited)**

T. Yamaguchi\*, T. Suzuki, H. Sumi, Y. Fujishiro, National Institute of Advanced Industrial Science and Technology (AIST), Japan

**Densification of Ceramics**

Room: Broadway III - IV

Session Chairs: Ricardo Castro, University of California, Davis; Ingo Schmidt, Fraunhofer IWM

**9:00 AM****(ICCP-092-2013) Nanoscale MgAl<sub>2</sub>O<sub>4</sub> Particles Sintering: Surface Chemistry and Agglomerations Effects (Invited)**

R. H. Castro\*, J. Rufner, University of California, Davis, USA

**9:30 AM****(ICCP-093-2013) Modelling and Simulation of Organic Binder Burnout in Ceramic Processing (Invited)**

I. Schmidt\*, T. Kraft, H. Riedel, Fraunhofer IWM, Germany; J. Svoboda, Academy of Sciences of the Czech Republic, Czech Republic

**10:00 AM****Break****10:15 AM****(ICCP-094-2013) Binder Removal by Diffusion Control: Concentration Distribution and Minimum Time Heating Cycles**

S. J. Lombardo\*, University of Missouri, USA

**10:30 AM****(ICCP-095-2013) Effects of Forming on the Sintering Kinetics and Microstructural Evolution of 99.8% Pure Specialty Alumina Powders**

I. O. Ozer, L. Kupp\*, Penn State University, USA; C. Compson, E. Koep, Almatix, Inc., USA; M. Spreij, Almatix, Inc., Germany; G. L. Messing, Penn State University, USA

**10:45 AM****(ICCP-096-2013) A Unified View of Grain Boundaries in Sintered Alumina**

W. M. Carty\*, Alfred University, USA; T. Lam, National Institute of Standards and Technology, USA

**11:00 AM****(ICCP-097-2013) Microstructural Characterization and Control of Perovskites (Invited)**

M. Baeurer\*, W. Rheinheimer, M. J. Hoffmann, Karlsruhe Institute of Technology, Germany

**11:30 AM****(ICCP-098-2013) Master Sintering Curve Theory applied to Sintering of Layered Ceramic Composites**

V. Pouchly\*, D. Drdlik, K. Maca, CEITEC BUT, Brno University of Technology, Czech Republic; H. Hadraba, Z. Chlup, CEITEC IPM, Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Czech Republic; J. Cihlar, CEITEC BUT, Brno University of Technology, Czech Republic

**11:45 AM****(ICCP-099-2013) Space-charge Contribution to Densification in Perovskite Ceramics**

F. Lemke\*, J. Hötzer, M. Bäurer, M. J. Hoffmann, B. Nestler, Karlsruhe Institute of Technology, Germany

**Plenary Session 5**

Room: Pavilion Ballroom

Session Chair: Gary Messing, Pennsylvania State University

**12:55 PM****(ICCP-005-2013) Discrete Element Simulation: Modeling Sintering at the Particle Length Scale**

Z. Yan, University of Grenoble / CNRS, France; O. Guillon, Universität Jena, Germany; R. K. Bordia, University of Washington, USA; C. L. Martin\*, University of Grenoble / CNRS, France

**Shaping/Assembly of Ceramics**

Room: Pavilion Ballroom

Session Chairs: William Carty, Alfred University; Wolfgang Sigmund, UF

**1:45 PM****(ICCP-100-2013) Synthesis and Processing of Ceramic Nanofibers (Invited)**

W. Sigmund\*, UF, USA

**2:15 PM****(ICCP-101-2013) Electrospinning of Ceramic Nanofibers from Pre-ceramic Polymers**

A. Guo, M. Roso, M. Modesti, P. Colombo\*, University of Padova, Italy

**2:30 PM****(ICCP-102-2013) Micro Geometric Patterning of Titania Polygon Tablets for Terahertz Wave Control by Materials Tectonics Processing (Invited)**

S. Kirihara\*, Osaka University, Japan

**3:00 PM****(ICCP-103-2013) Roll your own: Nanocomposite Capacitor**

E. Yi\*, J. Furgal, R. M. Laine, University of Michigan, USA

**3:15 PM****(ICCP-104-2013) Influence of Feedstock Preparation on Ceramic Core Formation Using a Development Carrier System**

T. Standing\*, University of Birmingham, United Kingdom; P. Wilson, Rolls-Royce Plc., United Kingdom; S. Blackburn, University of Birmingham, United Kingdom

**3:30 PM****(ICCPs-105-2013) Gelcasting of Oxide Ceramics by a Single Additive (Invited)**

S. Wang\*, S. Shimai, Y. Sun, Y. Yang, M. Dong, Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

**4:00 PM****(ICCPs-106-2013) Graded Materials via a Novel Light Directed Electrophoretic Deposition Technique**

A. J. Pascall\*, F. Qian, Lawrence Livermore National Laboratory, USA; G. Wang, Y. Li, University of California, USA; J. Kuntz, Lawrence Livermore National Laboratory, USA

**4:15 PM****(ICCPs-107-2013) Hollow and Solid Pressure Slip Casting of Coarse Grained Oxide Ceramics**

S. Schaffoener\*, C. Aneziris, TU Bergakademie Freiberg, Germany

**4:30 PM****(ICCPs-108-2013) Processing of Textural Clay based Materials**

G. Lecomte-Nana\*, A. Mokrani, O. Mongenovo, N. Tessier-Doyen, K. Bousois, H. Goure-Doubi, ENSCI - GEMH, France

**Synthesis and Processing of Functional Ceramics**

Room: Broadway I - II

Session Chairs: Nelson Bell, Sandia National Laboratories; D. Hotza, UFSC

**1:45 PM****(ICCPs-109-2013) Solution Processed Nanostructured Materials for Application in Energy Storage (Invited)**

S. Tolbert\*, I. E. Rauda, V. Augustyn, C. Kang, B. Dunn, UCLA, USA

**2:15 PM****(ICCPs-127-2013) Colloidal Interactions at Advancing Ice/Water Interfaces (Invited)**

S. Dillon\*, University of Illinois, Urbana-Champaign, USA

**2:45 PM****(ICCPs-111-2013) Phase Development and Densification of NaSiCON Ceramics**

N. Bell\*, C. Edney, P. Lu, M. A. Rodriguez, E. D. Spoeke, Sandia National Laboratories, USA

**3:00 PM****(ICCPs-112-2013) Image Analysis of the Porous Yttria-Stabilized Zirconia (YSZ) Structure for the Impregnated Electrode of Solid Oxide Fuel Cell (SOFC)**

C. Ni\*, M. Cassidy, J. Irvine, University of St Andrews, United Kingdom

**3:15 PM****(ICCPs-114-2013) Innovative Processing to Enhance Nuclear Fuel with Oxide Additives**

R. M. Leckie\*, E. P. Luther, A. T. Nelson, K. J. McClellan, Los Alamos National Lab, USA

**3:30 PM****(ICCPs-115-2013) Electrolyte-Supported Solid Oxide Fuel Cell Processed by Aqueous Tape Casting and Constrained Calendering**

V. Moreno, UFSC, Brazil; N. Travitzky, P. Greil, University of Erlangen-Nuremberg, Germany; D. Hotza\*, UFSC, Brazil

**Field Assisted Densification of Ceramics**

Room: Broadway III - IV

Session Chairs: Bala Vaidhyanathan, Loughborough University; Leszek Chlubny, AGH University of Science and Technology

**1:45 PM****(ICCPs-116-2013) Flash Sintering of Functional Materials (Invited)**

B. Vaidhyanathan\*, S. Ghosh, Loughborough University, United Kingdom

**2:15 PM****(ICCPs-117-2013) Electric Field and Current effect on Liquid Phase Sintering Mechanism**

J. Gonzalez\*, O. Guillon, Friedrich Schiller University Jena, Germany

**2:30 PM****(ICCPs-118-2013) Electrical Energy Storage Properties of Barium Strontium Titanate Ceramics Prepared by Spark Plasma Sintering (Invited)**

Y. Wu\*, Y. Huang, N. Wang, X. Chen, Zhejiang University, China

**3:00 PM****(ICCPs-119-2013) Using of Pulsed Electric Discharges to the Preparation and Consolidation of Powder Composites**

E. Grigoryev\*, M. S. Yurlova, E. V. Krikun, Moscow Engineering Physics Institute, Russian Federation; E. A. Olevsky, San Diego State University, USA; O. N. Sizonenko, Institute of Pulse Processes and Technologies at National Academy of Sciences, Ukraine; Y. Lin, San Diego State University, USA

**3:15 PM****(ICCPs-120-2013) Ceramic Sintering and Shaping Using the Spark Plasma Sintering Method**

A. K. Mukherjee\*, University of California, USA

**3:30 PM****(ICCPs-122-2013) Fabrication and Mechanical Properties of Nano-SiC/CNT Composites Sintered by SPS**

B. Lanfant\*, Y. Leconte, M. Pinault, M. Mayne-L'hermite, N. Herlin-Boime, CEA Saclay, France; G. Bonnefont, S. De Bernardi, G. Fantozzi, V. Garnier, Y. Jorand, INSA Lyon, France; T. Pham, S. Le Gallet, F. Bernard, Université de Bourgogne, France

**3:45 PM****(ICCPs-123-2013) Densification and Phase Evolution of SHS Derived Ti<sub>2</sub>AlC Powders in Pressureless Sintering and Hot Pressing Processes**

L. Chlubny\*, J. Lis, AGH University of Science and Technology, Poland

**4:00 PM****(ICCPs-124-2013) Densification Behavior of MnO-doped UO<sub>2</sub> during Low Temperature Sintering**

Y. Rhee\*, I. Nam, D. Kim, J. Oh, J. Kim, K. Kim, J. Yang, Y. Koo, Korea Atomic Energy Research Institute, Republic of Korea



Monday, August 5, 2013

### Plenary Session I

Room: Pavilion Ballroom

Session Chair: Gary Messing, Pennsylvania State University

8:10 AM

#### (ICCP-001-2013) Innovations Through Processing of Ceramics and Ceramic Composites

L. Gauckler\*, ETH Zurich, Switzerland

Examples are presented where novel processing is the key for new materials' property profiles and new applications are enabled. New avenues for further research are outlined in these areas. The first example reports on the colloidal processing of micrometer thin tin oxide gas sensors on micro hot plates by microfluidic techniques. They are integrated in microelectronics and operate as electronic noses. Then processes are illustrated for ultra-thin ceramic films and foils enabling micro solid oxide fuel cells for battery replacements with hitherto unrivaled energy densities for portable electronics. The third example reports on the tailoring of Janus-type nanoparticle surfaces in colloids leading to particle foams, emulsions and colloidosomes. With their complex porous microstructures they have potentials for many new applications. The fourth example is inspired by the microstructure of nacre. New layered polymer/ceramic hybrid composites were developed. The resulting ceramic/polymer composite foils combine high tensile strength and high ductility. Finally, rapid prototyping of ceramic teeth restorations and implants from pre-sintered blanks revolutionized dental restoration practice in the past decade. This example demonstrates how rigorous combination of well-known ceramic technologies with modern shape recognition techniques and computer-controlled machining can open up new applications for ceramics.

#### Microfluidic Assembly and Novel Particle Fabrication

Room: Pavilion Ballroom

Session Chairs: Andre Studart, ETH Zurich; Kazumi Kato, National Institute of Advanced Industrial Science and Technology

9:00 AM

#### (ICCP-006-2013) From Colloids to Capsules: Novel Pathways to Ceramic Microcapsules (Invited)

M. Maas\*, T. Bollhorst, P. Kaempfe, K. Rezwani, University of Bremen, Germany

The fabrication of microcapsules for the encapsulation of drugs and other active agents is an important field in pharmaceutical, nutritional and cosmetic research. A variety of strategies and materials exist for the generation of capsules for diverse applications. A specific type of microcapsule, the colloidosome, consists of a shell assembled from nanoparticles. Colloidosomes are a versatile and promising platform, which is distinguished by the tunability of properties like porosity, wall strength and size based on a multitude of available nanoparticles as building blocks. In recent work, we developed a biocompatible processing route for stable submicron colloidosomes that utilizes special interface interactions between lipophilic surfactants and nanoparticles that are dispersed in water. Another approach for the preparation of capsules with inorganic shells is the mineralization of coacervate droplets. This bio-inspired processing route is based on the demixing of lyophilic polymer-ion complexes in the form of colloidal droplets. Using this method, depending on the initial composition of the coacervate droplets, diverse containers for active molecules can be produced, such as calcium carbonate capsules and magnetite/polymer microspheres.

9:30 AM

#### (ICCP-007-2013) Monodisperse Ceramic Particles and Capsules from Double Emulsion Drops (Invited)

C. Martinez\*, C. Ye, Purdue University, USA; P. Colombo, Università di Padova, Italy

In this talk, I'll highlight recent efforts in my group to develop a robust technique to fabricate ceramic core-shell structures from monodisperse single and double emulsions generated using microcapillary microfluidic devices. These devices consist of a coaxial arrangement of tapered cylindrical glass capillaries inside a square capillary. In this device, the innermost fluid is pumped through the smaller tapered capillary tube, while the middle and outer fluids are pumped in opposite directions through the outer coaxial region. In our system the middle fluid, which is the shell of the capsules, is composed of a poly(methylsilsesquioxane) resin (the ceramic precursor), dissolved in a mixture of 1 cSt polydimethylsiloxane (PDMS) oil and Zr-acetylacetonate (crosslinker). The inner fluid is an aqueous solution of Zr-acetylacetonate and the outer fluid contains a mixture of water and glycerol. Monodisperse crosslinked drops with sizes ranging from 30 microns to 200 microns, and different shell thicknesses were generated by varying the fluid flow rates and the device geometry. Silica glass capsules were obtained by drying, and then pyrolyzing at 1100 °C the crosslinked drops. Capsules with porous shells were also fabricated by changing the composition of the middle fluid. Finally, I'll show how to tailor the capsule's porosity by osmotically regulating the flow of water in and out of the double emulsions.

10:15 AM

#### (ICCP-008-2013) Functional Ceramic Colloidosomes made by Microfluidics

J. Sander\*, A. R. Studart, ETH Zurich, Switzerland

Encapsulation and release from microcapsules are of great interest for many applications. Capsules exhibiting nanoparticle shells, known as colloidosomes, are particularly interesting due to their tunable permeability, shell thickness and size. In this work, we show how ceramic colloidosomes can be assembled and functionalized for guided transport and triggered release by adding active particles into the shell and inside the capsules. Colloidosomes are assembled from double emulsion templates made in glass capillary microfluidic devices, which allow for highly flexibility and efficient loading of encapsulants. To effectively disperse them in the middle phase of the emulsion template, the surface chemistry of ceramic nanoparticles is adjusted through the surface adsorption of surfactants. The selection of surface active molecules that form an interfacial film at the oil-water interface is found to be crucial for the effective stabilization of the double emulsion templates. Using this approach, we make colloidosomes from many ceramic materials such as silica, Al<sub>2</sub>O<sub>3</sub>-Fe<sub>3</sub>O<sub>4</sub> or tricalcium phosphate-clay mixtures. The addition of cargo-loaded particles inside such capsules opens several possibilities for the controlled release of cargo with well-defined spatial temporal patterns, including one-time release, switchable on-off release or co-release of different substances.

10:30 AM

#### (ICCP-009-2013) Bioinspired Composites with Extreme Mechanical Gradients

R. Libanori\*, R. M. Erb, A. Reiser, H. Le Ferrand, M. J. Sueess, R. Spolenak, A. R. Studart, ETH Zurich, Switzerland

Ceramic-polymer heterogeneous composites with locally tuned mechanical properties are commonly found in biological structures that couple surfaces with different elastic moduli. In contrast, the integration of synthetic materials with different mechanical properties often results in high stress concentration at interfaces, leading to premature interfacial failure. Here, we show that composite films exhibiting elastic moduli spanning 4 orders of magnitude can be produced through the hierarchical reinforcement of a soft organic matrix with polymeric crystals, laponite nanoplatelets and ceramic microplatelets. In-

dividual films with distinct elastic moduli were assembled into multi-layered structures to produce graded composites with a gradual compliant-to-stiff transition. The local elastic modulus on the top surface of the composite can approach that of the hardest biological ceramics (e.g. enamel) while still being as soft as skin on the bottom surface. Our ability to locally tune the stiffness of composites within such a broad range opens new possibilities for the design of functional materials for mechanically demanding applications. This potential is illustrated by assembling composites with a graded architecture that effectively protects brittle electronic devices deposited on stretchable polymeric substrates. Such concept may be exploited for the creation of more durable ceramic-polymer joining technologies.

**10:45 AM**

### **(ICCP5-010-2013) Self-assembly Process to Construct 3D Superlattice Ceramics**

K. Koumoto, C. Wan, F. Dang\*, Nagoya University, Japan

3D superlattice ceramics with nanocube grains of La-SrTiO<sub>3</sub> (STO) and Nb-STO 2DEG grain boundaries would generate high thermoelectric performance. In order to construct 3D superlattice hydrothermal synthesis of La-STO nanocubes was carried out first, and then thin layer of Nb-STO was coated on their surfaces. The obtained nanocubes were further dispersed in a solution and self-assembled to form a particulate film on a silicon substrate. The film was densified without significant grain growth by SPS treatment under a reducing atmosphere. The resulting film demonstrated a thermoelectric figure of merit, ZT, at room temperature which is higher than that of a single crystal with similar composition.

**11:00 AM**

### **(ICCP5-011-2013) Synthesis and Assembly of Nano-cube BaTiO<sub>3</sub> Single Crystals through Tailored Liquid (Invited)**

K. Kato\*, K. Mimura, F. Dang, National Institute of Advanced Industrial Science and Technology, Japan; H. Imai, Keio University, Japan; S. Wada, The University of Yamanashi, Japan; M. Osada, H. Haneda, National Institute for Materials Science, Japan; M. Kuwabara, Kyushu University, Japan

Nanocrystals with anisotropic shapes are expected as building blocks for high dimension hierarchical structures because of their potentials toward future functional devices. BaTiO<sub>3</sub> nanocubes were synthesized by the hydrothermal method using aqueous sources of metal ions and amphiphilic organics such as oleic acid. Oleic acid was adsorbed at the (100) surface of nuclei via the hydrophilic group to suppress the forward growth. Resultantly, the {100} dominant perovskite nanocubes were developed, which had a uniform size in 15 nm, sharp edges and coherent lattice fringes. The hydrophobic group determined the stability of nanocubes in non-polar solvents such as toluene and the derivatives. Based on the highly dispersing property, the uniform size and the volatility of solvents, the nanocubes were arranged into orderly structures on substrates according to the capillary force assisted self-assembly mechanism. The additional shear force applied by withdrawing substrates at extremely low speeds enabled the assembly to enlarge over the surface of substrates. The highly-ordered regions were shaped like islands with sizes in tens of micrometers. The nanocubes attached each other in face to face so that the assemblies had a simple cubic symmetry. The local piezoresponse behavior of the assemblies was evaluated by PFM. The ferroelectricity stemmed from constituent BaTiO<sub>3</sub> nanocubes.

**11:30 AM**

### **(ICCP5-012-2013) Chemically Grown Nanoparticles, Nanowires and Nanocomposites: Processing, Applications and Devices (Invited)**

S. Mathur\*, University of Cologne, Germany

Chemical nano-sciences enabling controllable manipulation of matter at molecular length scale have become fundamental generators for innovations in materials processing. The successful synthesis, modification and assembly of nanobuilding units such as nanocrystals, -

wires and -tubes of different materials have demonstrated the importance of chemical influence in materials synthesis, and have generated great expectations for the future. Inorganic nanostructures inherit promises for substantial improvements in materials engineering mainly due to improved physical and mechanical properties resulting from the reduction of microstructural features by two to three orders of magnitude, when compared to current engineering materials. This talk will present how chemically grown nanoparticles, nanowires and nanocomposites of different metal oxides open up new vistas of material properties, which can be transformed into advanced material technologies. The examples will include application of superparamagnetic iron oxide nanoparticles for drug delivery applications, molecule-based synthesis of nanowires and development of single-nanowire based devices.

## **Synthesis and Processing of Thin Films and Coatings**

Room: Broadway I - II

Session Chairs: Matthew Seabaugh, NexTech Materials, Ltd.; Wataru Sakamoto, Nagoya University

**9:00 AM**

### **(ICCP5-013-2013) Application of Water-soluble Titanium Complexes for Synthesis of Titanium Dioxide with Controlled Structures (Invited)**

M. Kobayashi\*, H. Kato, M. Kakihana, Tohoku University, Japan

In the synthesis of titanium dioxide, titanium chloride, sulfate or alkoxide is often employed as a practical titanium source. However, they can be actually used only under limited experimental conditions, and one must suffer from the difficulty of their handling because of their toxicity and instability. We have developed a series of water-soluble titanium complexes, which do not undergo a hydrolysis under ambient conditions and in a wide range of pH. Of particular importance is that they can be used as a titanium source in aqueous solution process, which led to success in selective synthesis of titania polymorphs by a hydrothermal treatment of the complexes. Only by changing ligand and/or additive, anatase, rutile, brookite, and TiO<sub>2</sub>(B), could be readily obtained each individually as a single-phase material. Moreover, morphologies of titania polymorph particles could be highly controlled by the hydrothermal treatment of the complexes in the presence of given simple molecules. We also found that the obtained titania polymorphs with well-controlled structures exhibited high performance as a photocatalyst, dielectric substance and so on. In our presentation, we will show our results on selective synthesis and morphological control of titania polymorphs using a series of water-soluble titanium complexes and their characters.

**9:30 AM**

### **(ICCP5-014-2013) Chemical Solution Processing and Properties of Niobate-Based Lead-Free Piezoelectric Thin Films for Thin-Film Actuators (Invited)**

W. Sakamoto\*, Y. Nakashima, N. Kondo, T. Matsuda, Nagoya University, Japan; B. Lee, T. Iijima, National Institute Advanced Industrial Science and Technology (AIST), Japan; M. Moriya, T. Yogo, Nagoya University, Japan

Recently, increased demand for piezoelectric thin film processing for micro-electromechanical system device development and the development of Pb-free ferroelectric materials is expected to produce a wide variety of eco-friendly thin-film sensor, actuator and energy harvester devices. Among various Pb-free ferroelectric oxide materials, (K,Na)NbO<sub>3</sub> (KNN) is a promising candidate material because of its excellent piezoelectric properties. In this study, to achieve piezoelectric Pb-free KNN thin films with desired electrical properties, modified KNN thin films were synthesized through a chemical solution deposition using tailored metal-organic precursor solutions. Furthermore, appropriate functional element (such as Mn) doping effect on electrical properties of the KNN-based thin film was also studied. Perovskite single-phase thin films were fabricated by optimizing the chemical composition and heating condition of the pre-

cursor films. Leakage current and ferroelectric properties were remarkably improved by controlling the amount of Mn doping for Nb site in KNN. Mechanism of properties improvement including valence state of the doped Mn and field-induced strain behaviours including effective-d33 values of the synthesized thin films on substrates are discussed for the implementation of the KNN-based thin films in piezoelectric applications.

**10:15 AM**

**(ICCP-015-2013) Highly Adherent Ceramic Coating on Metal Surface by Electrophoretic Deposition**

S. De, L. Besra\*, S. Bhattacharjee, B. P. Singh, Institute of Minerals & Materials Technology (IMMT), India; T. K. Rout, Tata Steel Ltd, India; D. K. Sengupta, Institute of Minerals & Materials Technology (IMMT), India

Ceramic coating on metal is generally non-adherent resulting in delamination of the deposits during drying and sintering because of the large difference in their thermal expansion coefficients. In this paper, we report a simple and novel strategy based on Electrophoretic deposition at 100 V for 30 sec from a stable TiO<sub>2</sub> suspension of 0.5 wt% in ethanol in presence of methyl hydrogen silicon fluid (KF-99) to obtain crack-free coating of TiO<sub>2</sub> on steel that can withstand a temperature of 500 oC and exhibit high resistance to corrosion. The presence of KF-99 filled and bridged the microcracks developed during drying and sintering leading to increased adhesion. Adhesion was tested using scotch adhesion test and corrosion test was performed using salt spray test in 3.5 wt% NaCl solution as well as by electrochemical measurements. Adhesion and corrosion resistance was improved further by dipping the coated sample in KF-99 immediately after deposition followed by drying and sintering. The time to initiate pitting increased from 24 h for coating in absence of KF-99 to 1200 hrs in presence of KF -99. The improved corrosion resistance was due to increase in hydrophobicity of the coating (water contact angle: 100 -105 o) in presence of KF-99 which prevented wetting by the corrosive liquid. The coating delaminated above a temperature of 500 oC because of removal of organic component KF-99.

**10:30 AM**

**(ICCP-016-2013) Preparation of Porous Ceria Coating for Resistive Oxygen Sensor**

C. Chen\*, K. Chang, Feng Chia University, Taiwan; S. Shih, National Taiwan University of Science and Technology, Taiwan; S. Wang, Southern Taiwan University, Taiwan

Oxygen sensors play important roles in determining the oxygen content in the exhaust gas, optimizing combustion, among others. Resistive oxygen sensors made of an n-type semiconductor, doped ceria (CeO<sub>2</sub>), have recently received much attention due to the relatively compact and simple structures. In the present study, alumina substrate was first coated with electrodes by screen printing. Porous ceria films were then coated on the electrodes using the mixture of two undoped ceria powders synthesized by spray-pyrolysis and precipitation, respectively. The ratio of the two powders was varied to result in ceria-films with different sintered porosity. The resistive oxygen sensors exhibit a 3-D interconnecting pore structure which can be optimized by the powder ratio, revealing better sensing properties. The optimum porous structure shows >1.8 times faster dynamic response to the change in oxygen partial pressure at 1073 and 1123 K, comparing to the one without optimization. Detailed processing, optimization of compositions and responses to variation of oxygen partial pressure will be given. The mechanisms for the formations of the powders and the porous coatings are also delineated.

**10:45 AM**

**(ICCP-017-2013) Protective Coatings for Metals in High Temperature Power Systems**

M. Seabaugh\*, N. J. Kidner, S. Ibanez, K. Smith, K. M. Chenault, L. B. Thrun, S. L. Swartz, NexTech Materials, Ltd., USA

Cost is a key consideration for advanced power technologies, whether high efficiency combined cycle combustion systems, advanced high temperature batteries or solid oxide fuel cells (SOFCs). As systems are designed to operate more efficiently at high operating temperatures, the demands on metal components can be severe. To maintain affordable system costs, developers have substituted lower cost stainless steel for nickel superalloys, which demands affordable and scalable high temperature coatings. Coating processes are presented that use the free energy of in-situ coating /substrate reactions to produce overlay and diffusion coatings. Aerosol and dip coating methods can be used to produce protective coatings comparable to those achieved by processes requiring much larger capital investments. In this presentation, two coating examples will be discussed. For SOFC interconnects, manganese cobaltite (MCO) oxide coated ferritic stainless steel has been demonstrated. Aluminide-based diffusion coatings have been developed for the seal area where coatings with low chemical reactivity are necessary. Processing of these coatings has been tailored to be compatible with the MCO coating to allow for co-processing. In high process-intensity chemical reactors, aluminide coatings on protect austenitic and high temperature alloys against oxidation and other corrosion reactions.

**11:00 AM**

**(ICCP-018-2013) Zirconia Coating on Stainless Steel with Integrated Oxides Layer by Growing Integration Layer [GIL] Method (Invited)**

M. Yoshimura\*, K. Chen, Y. Lin, C. Hwang, National Cheng Kung University, Taiwan

Zirconia coating on stainless steel (SS) is important in various applications like thermal barrier, anti-corrosion, energy, solid oxide fuel cell, bio-medical, etc. In those applications, the adhesion is the most serious problem. We have succeeded well adhered zirconia coating on SS304 with a novel "Growing Integration Layer" [GIL] concept, where grown oxide film(s) from SS304 would prepare intermediate layer(s) between zirconia and SS substrate. The intermediate oxide layer of FeCr<sub>2</sub>O<sub>4</sub> -rich with 50-400 nm thickness could be fabricated by the anodization of SS plate in a H<sub>2</sub>SO<sub>4</sub> + HNO<sub>3</sub> solution at 70 C by a square wave pulses (E =1.2 V, E = 0.8 V, and 0.3 Sec interval). Pure and CeO<sub>2</sub>-doped Zirconia films were fabricated on those GIL substrates by Dip-Coating from precursor solutions, where Polyvinyl Alcohol (PVA) was used as Complex- and Gel-formation agent. After calcined at 300 C in air zirconia coating films with 100-500 nm thick were obtained. Improved adhesion by scratch tests and increased corrosion potentials by 0.1-0.5 Volts were confirmed by Tafel-plot. The effects of anodization condition and Dip-Coating have been optimized.

**11:30 AM**

**(ICCP-019-2013) Nanosheet-processes for High Performance Electrodes of Energy Storage Devices (Invited)**

M. Miyayama\*, S. Sugata, S. Suzuki, The University of Tokyo, Japan

The development of secure, cost-effective energy storage devices with high energy densities and power densities is an urgent subject to solve the energy problems and expand green technologies. Lithium-ion batteries and protonic electrochemical capacitors are widely studied for the purpose, but various problems are still left on electrode materials. A large surface area, effective diffusion paths for mobile ions and a high electronic conductivity are necessary for the electrodes to achieve complete and rapid redox reactions of active materials. Inorganic nanosheets are very thin crystalline sheets with nanometer-level thickness, delaminated from layer-structured compounds. Electrodes prepared using nanosheet-restacked particles or thin films by

## Abstracts

electrophoretic deposition meet the above structural requirements for the high performance electrodes. In the talk, our recent results are reported on fabrication of nanosheet-based electrodes and their electrode properties for Li-ion and proton batteries. MnO<sub>2</sub>-based nanosheet electrodes exhibited high charge/discharge rates and Hx(Ni,Co,Mn)O<sub>2</sub> electrodes exhibited large and stable capacities through redox reactions by protons in aqueous electrolyte solutions. Mechanisms and effects of microstructure for the improved properties are also discussed.

### Processing of Porous Ceramics

Room: Broadway III - IV

Session Chairs: Sylvain Deville, CNRS; Paolo Colombo, University of Padova

**9:00 AM**

#### (ICCP-020-2013) Control of Microstructure of Porous Ceramic Materials (Invited)

G. V. Franks\*, University of Melbourne, Australia

Porous ceramic materials can be produced by several colloidal processing methods. These include, sacrificial template, particle stabilized foams, freeze casting and partial sintering. Each of these techniques is suitable to produce porous ceramics with different morphology (size, shape and alignment) of pores. Multiscale porous ceramics can be produced by combining two or more of these methods. The techniques developed are suitable for oxide as well as non-oxide ceramics depending on the formulation. X-ray computed tomography is a good technique to characterise the 3D microstructure of the materials. The tomography data can be manipulated into suitable virtual representations for prediction of thermo and mechanical properties with appropriate computer models.

**9:30 AM**

#### (ICCP-021-2013) Ice Templating, Freeze-casting: Been there, done that. Now what? (Invited)

S. Deville\*, CNRS, France

Ice templating is able to do much more than macroporous, cellular materials. The underlying phenomenon –the freezing of colloids– is ubiquitous, at a unique intersection of a variety of fields and domains, from materials science to physics, chemistry, biology, food engineering, and mathematics. In this talk, I shall try to answer the following questions: what did we achieve so far? What have we learned? Where can we get further inspiration? And more importantly, what can we use this process and associated knowledge for? I will walk through the seemingly divergent domains in which the occurrence of freezing colloids can benefit from the work on ice templating, or which may provide additional understanding or inspiration for further development in materials science. This talk does not intend to be extensive, but rather to illustrate the richness of this phenomenon and the obvious benefits of a pluridisciplinary approach. I shall briefly review how the research around these ideas has evolved starting from the simple idea of processing macroporous, single phase materials before expanding to more complex composite materials, structures, and architectures. Based on these results and ideas, I will describe how other fields could benefit from this approach and vice versa, from physics and self-assembly to ice physics and geophysics, chemistry, life and health science, and finally materials science.

**10:15 AM**

#### (ICCP-022-2013) Influence of Freezing Conditions on the Internal Structure of Directionally Freeze-Cast Porous Ceramics

S. Miller\*, K. Faber, Northwestern University, USA; X. Xiao, Argonne National Laboratory, USA

Freeze casting is a method of fabricating directionally porous ceramics that promises a high degree of control over structure via alterations in the fabrication process. By changing freezing conditions, the

pore network, the internal structure of the porous construct, can be modified. This study investigates the effect of freezing temperature on the pore structure of aluminum oxide directionally freeze-cast ceramics from water-based and camphene-based slurries. X-ray computed tomography was employed to attain three-dimensional representations of samples fabricated under varying conditions. These 3D stacks were computationally analyzed using MATLAB® and Amira® to quantify pore network characteristics such as pore size, specific surface area, and tortuosity. Changes in pore structure are compared to differences in processing conditions, and correlations to classical solidification theories are presented.

**10:30 AM**

#### (ICCP-023-2013) A Novel Method for Manufacturing Porous Ceramics

S. Gupta\*, M. F. Faisal, T. Hammann, R. Johnson, University of North Dakota, USA

Porous ceramics are used for numerous structural applications, for example, filtration of molten metals or of particulate from exhaust gases, radiant burners, catalyst supports, biomedical devices, kiln furniture, lightweight sandwich structures etc. The manufacturing steps required to produce porous ceramic materials are often tedious and time consuming, for example, replica technique, gel casting, hollow bead method, freeze-casting, physical and chemical foaming, among others. Due to complicated manufacturing steps, there are often high rejection rates during manufacturing of porous materials. In this presentation, we will present a novel method for manufacturing porous materials by oxidation of reactive precursors (ORP) during sintering. The authors will demonstrate that it is possible to fabricate 70-80 % porous materials by using simple manufacturing cycle, for example, heating a green body at 50°C in air until final sintering temperature. In addition, it is also possible to control pore size and geometry by adding different additives, like graphite, polyethylene (PE), among others. The authors will also demonstrate that mechanical properties of the porous materials fabricated by ORP method are comparable, if not better, to porous ceramics, for example porous TiO<sub>2</sub> scaffolds, fabricated by traditional methods.

**10:45 AM**

#### (ICCP-024-2013) Magnetic Freeze Casting inspired by Nature

M. M. Porter\*, M. A. Meyers, J. McKittrick, University of California, San Diego, USA

Magnetic freeze casting is a novel method to fabricate porous ceramic scaffolds with a hierarchy of architectural alignment in multiple directions. A weak rotating magnetic field applied normal to the ice growth direction in a uniaxial freezing apparatus allowed the manipulation of magnetic nanoparticles to create different pore structures with long-range order in directions parallel and perpendicular to the freezing direction. Porous scaffolds consisting of different host ceramics (hydroxyapatite (HA), ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, or TiO<sub>2</sub>) mixed with varying concentrations (0-9 wt%) of Fe<sub>3</sub>O<sub>4</sub> nanoparticles were fabricated by freezing under three different conditions: (1) no magnetic field, (2) a static magnetic field of 0.12 T, or (3) a rotating magnetic field of 0.12 T at 0.05 rpm. The HA, ZrO<sub>2</sub>, and Al<sub>2</sub>O<sub>3</sub> scaffolds showed biphasic material properties. The TiO<sub>2</sub> scaffolds showed homogeneous distributions of Fe<sub>3</sub>O<sub>4</sub>, aligned pore channels, and enhanced mechanical properties parallel to the magnetic field, normal to the freezing direction.

**11:00 AM**

#### (ICCP-025-2013) Hierarchically Porous Ceramic Monolith via Sol-gel with Phase Separation in Aqueous Systems (Invited)

K. Nakanishi\*, Kyoto University, Japan

Monolithic amorphous and crystalline oxides having hierarchical macro/mesopores have long been exploited in the systems of silica-based oxides, titania, zirconia as well as organosilica-based organic-inorganic hybrids. The method has recently been extended to oxides

that contain lower valency cations, e.g. alumina, iron oxides, nickel oxides and copper oxide. In addition, not a few phosphate systems containing calcium, aluminum or zirconium have also been exploited to give monolithic ceramics with well-defined macro/mesopores. These polycrystalline ceramics systems can be prepared from metal-salt precursors in water-based solvents. The gelation of the system is usually controlled by pH swing using acid scavenging reagents such as propylene oxide. For example, pure alumina monolith can be prepared by hydrolyzing aluminum chloride in the presence of poly(ethylene oxide) and propylene oxide. Relatively quick gel formation (typically less than 30 min) gives well defined macroporous monoliths. After drying, the gels can be converted into crystalline monolith composed of various phases of alumina. An addition of second and third component to the pure alumina system is also simple, by adopting corresponding chlorides as precursors. Examples include Ma-Al spinel, Y-Al garnet and Mg-Al-Si cordierite.

**11:30 AM**

**(ICCP-026-2013) Structural Characterization of Heat-treated Fullerene Nanowhiskers (Invited)**

K. Miyazawa\*, National Institute for Materials Science, Japan; R. Kano, Yokohama City University, Japan; T. Konno, C. Hirata, T. Wakahara, J. Tang, National Institute for Materials Science, Japan; M. Tachibana, Yokohama City University, Japan

In 2001, C60 nanowhiskers (C60NWs) were discovered in a colloidal solution of PZT containing a small amount of C60. The C60NWs with a single crystal structure can be synthesized by a simple method called "liquid-liquid interfacial precipitation method (LLIP method)". In 2004, C70 nanotubes, C70 nanowhiskers with a tubular structure, were found to be synthesized by using the LLIP method, followed by the synthesis of C60-C70 two-component nanotubes and C60 nanotubes (C60NTs). Those fullerene nanofibers exhibit semiconducting properties of n-type and their application for field-effect transistors have been investigated. On the other hand, C60NWs and C60NTs can be turned into amorphous carbon nanofibers by heat treatment at high temperatures up to 3273 K. Those glassy carbon nanofibers become electrically conductive and can be used as the tips for electron emission. Moreover, the C60NWs heat-treated at 1173 K acquire the adsorption properties of H<sub>2</sub>O molecules. This affinity for water of the heat-treated C60NWs is very interesting in that carbon fibers are usually hydrophobic. In this presentation, we will show the structural change of C60NWs by heat-treatments at various temperatures by use of Raman spectroscopy and high-resolution transmission electron microscopy, and discusses the origin of the unique surface properties of the heat-treated C60NWs.

**Plenary Session 2**

Room: Pavilion Ballroom

Session Chair: Jennifer Lewis, Harvard University

**12:55 PM**

**(ICCP-002-2013) Colloids with Directional Interactions**

D. J. Pine\*, New York University, USA

We have developed new kinds of colloidal particles with either geometrical or chemical patches that give rise to directional interactions. These interactions allow colloids to interact with each other more like atoms, which in turn are used to build up structures that are not possible with isotropic interactions. These directional interactions are being developed to make self-replicating colloidal motifs and new colloidal crystals.

**Anisotropic Particles and 3D Assemblies**

Room: Pavilion Ballroom

Session Chairs: Fabrice Rossignol, CNRS; Edson Leite, CNPq

**1:45 PM**

**(ICCP-027-2013) Hydrothermal Processing and Dispersion of Nanometer-sized Ceria Crystals with Organic Modification**

K. Kobayashi\*, Y. Miura, M. Haneda, M. Ozawa, Nagoya Institute of Technology, Japan

Recently, morphology-controlled nanometer-sized inorganic materials such as nanocrystals and quantum dots have attracted strong attention due to their highly functional properties. Morphological tuning of nanoparticles has been an important subject in the field of advanced materials. From the viewpoint of industrial manufacturing, hydrothermal processing is one of the promising routes for nanoparticle synthesis due to its simplicity and relatively low environmental load. Although hydrothermal synthesis of nanoparticles has deeply been investigated by many researchers, their growth mechanism has not been understood in detail yet. In this study, the growth scheme of ceria (CeO<sub>2</sub>) nanoparticles with organic modification in a hydrothermal process has been investigated through Raman and IR spectroscopy, XRD measurement, and TEM observation. Ceria is a suitable material for model study, because it has the simplest cubic crystal structure and exhibits a moderate growth rate under a hydrothermal condition. Effects of hydrothermal temperature, processing time, concentration of a stabilizing agent, and pH of the solution were investigated. Morphologies of nanoparticles varied from nanocubic structure of single crystal to coalescent aggregates in accordance with the parameter control. The obtained samples were then dispersed in a non-polar solvent and aqueous solution for the assembly study.

**2:00 PM**

**(ICCP-028-2013) Structure of Colloidal Suspensions: What to Expect from Numerical Simulations (Invited)**

A. Videcoq\*, A. Tomilov, SPCTS, ENSCI, CNRS, France; D. Bochicchio, Universita di Genova, Italy; F. Rossignol, C. Pagnoux, T. Chartier, SPCTS, ENSCI, CNRS, France; R. Ferrando, Universita di Genova, Italy; T. Ala-Nissila, Aalto University, Finland

Many ceramic shaping processes are using the liquid route. Here, controlling the structure of colloidal suspensions is crucial in order to reliably adapt the suspension rheology to the process and to get the desired microstructure in the final part. We believe that numerical simulations, which take into account the discrete nature of the colloids, may be of help in this goal. We have recently developed codes based on two techniques: Brownian dynamics (BD) and the hybrid stochastic rotation dynamics – molecular dynamics (SRD-MD). There main difference is that in the first one we neglect the hydrodynamic interactions (HIs) between the colloids, while, as we have shown, the second one is able to properly include the essential features of the HIs. The BD technique will be first described and we will show how it can be employed to control the suspension stability and the suspension structure (particle ordering, compactness of the aggregate network, percolation properties, etc.), especially in the case of heteroaggregation (i.e. aggregation between oppositely charged colloids). Then, we will explain how the hybrid SRD-MD technique is designed, and we will show what are the effects of the HIs on the aggregation process and by consequence on the suspension structure.

**2:30 PM**

**(ICCP-029-2013) Ceramics Assembly: From Model Colloids to 3D Architectures (Invited)**

J. A. Lewis\*, Harvard University, USA

This talk will highlight our recent efforts to create and assemble novel ceramic building blocks and 3D architectures. First, we will describe a new pathway for producing shape and chemically anisotropic colloids. Bare and gold-coated silica rods have been produced in both

tip- and side-coated Janus motifs. We have explored their self- and electric field-induced assembly and observed myriad structures, including clusters, chains, bilayers, and 3D oriented templates. We have also recently created concentrated copper- and lithium-based oxide inks for patterning 3D interpenetrating architectures for micro-energetic and micro-battery applications.

**3:00 PM**

### **(ICCP-030-2013) Highly Selective and Sensitive Multifunctional Biosensors Fabricated by Coupling Ink-jet Printing, EISA and Click Chemistry**

F. Rossignol\*, O. De Los Cobos, M. Lejeune, M. Colas, CNRS, France; F. Lalloue, H. Akil, University of Limoges, France; C. Carrion, CNRS, France; P. Faugas, Ester Technopole, France; C. Boissiere, C. Sanchez, X. Cattoen, M. Wong Chi Man, J. Durand, CNRS, France

Bundles of optical fibers can be selectively multifunctionalized to serve as smart endoscopic devices for recognition of tumoral biomarkers in membrane cells. The device fabrication is performed by combining ink-jet printing process (IJP), evaporation induced self-assembly (EISA) and click chemistry. More specifically, mesoporous silica microdots are first precisely deposited at the tip of optical fibers by IJP, the structuring of the porosity being achieved following an EISA strategy. Then click chemistry is used to anchor bio-receptors onto the mesoporous silica surface. Those bio-receptors are labeled with specific fluorophores that are chosen so that Fluorescence Resonance Energy Transfer (FRET) detection is made possible when antigens and antibodies interact. Fluorescence excitation and FRET signal read-out are performed directly through the optical fibers. We demonstrate here that ink-jet printing is an excellent technique to realize highly sensitive and selective immunosensors when combining it to EISA and click chemistry. The use of different bio-receptors for multi-detection immunosensors is very promising for a complete in vivo diagnosis in the near future. In addition, the same approach can be performed to implement photodynamic therapy (PDT) paving the way to smart "theranostic" endoscopes.

**3:15 PM**

### **(ICCP-031-2013) Zirconia Nanocrystals Self-Assembled in Superlattices Directed by the Solvent-Organic Capping Interaction**

C. J. Dalmaschio, A. F. Moura, D. G. Nephew, E. R. Leite\*, Federal University of Sao Carlos, Brazil

Close-packed arrays of ZrO<sub>2</sub> nanocrystals (NCs) have been self-assembled from colloidal solution using dip-coating process. The benzyl alcohol route was used to obtain narrow controlled size NCs (5.56 nm). Then the capping layer was replaced by oleate using solvothermal treatment. The oleate solubility was explored in chloroform, hexane and toluene to prepare thin films of NCs using a dip coating process. From TEM images the final structures show that increasing the solvent polarity an improved self-assembled is possible to prepare mono and multilayer superlattices, during solvent evaporation in short time. The entangled organic chain in the NCs surface redress the faceted NCs improving the assembly quality, approaching the NCs assemble to hard sphere model resulting in a FCC closed packed structure. From SAXS data the lattice parameter found in the superlattices was 11.25 nm. Molecular dynamics simulations with soft potentials give support to the conclusion that hexane interacts with the organic capping, increasing the apparent radius of each NCs and stabilizing the colloidal suspension, whereas chloroform is partially removed from the capping during the aggregation process, forming an array of nanoparticles.

## **Non-aqueous Ceramic Dispersions**

Room: Pavilion Ballroom

Session Chairs: Keith DeCarlo, Blasch Precision Ceramics; George Franks, University of Melbourne

**3:45 PM**

### **(ICCP-044-2013) Electrostatic Dispersion of Sulfosilane Modified Particles in Rubber Fluids**

N. Bell\*, T. J. Boyle, Sandia National Laboratories, USA

The fundamental mechanisms of particle dispersion in low polarity elastomers are poorly understood. In polymer composites, particle dispersion is commonly interpreted through examination of wetting interactions between a silane modified surface, and the elastomer. However, electrostatic forces can be generated in low polarity fluids, leading to very long range repulsion. This study examines the formation of surface potential between sulfosilane modified particles under low polarity fluid conditions. In this work, an oligomeric proxy to isoprene is used to create a low viscosity solution in which colloidal stability of sulfosilane particles can be examined. Measurements of zeta potential for three sulfosilanes suggest that increasing the Sulfur atoms content of the sulfosilane modifying the particle surface correlates with an increasing magnitude of negatively charged zeta potential. Measurements using acoustic spectroscopy as well as light scattering techniques were used in for characterization, and show qualitative trends that concur. These results give new insight into the commonly applied vulcanization of particle reinforced composite materials. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

**4:00 PM**

### **(ICCP-045-2013) Evaluation of DLVO Predictions for Non-aqueous Stability of Selected Colloids Using Rheology**

W. M. Carty\*, Alfred University, USA; K. DeCarlo, Blasch Precision Ceramics, USA

DLVO theory was used to calculate the total interaction potential of 10 different colloids in 11 different suspending mediums to predict electrostatic stability. The rheology of each suspension was measured to assess the DLVO predictions of electrostatic stability and measure the required thermal boundary for dispersion. The mechanism of dispersion was hypothesized to be able to be determined by comparison of the specific viscosity of the suspension at a shear rate of 1.0s<sup>-1</sup> with the shear thinning exponent. It was determined that all suspensions with the exception of those prepared with heptane, octanoic acid, and poly(ethylene glycol) all operated with an electrostatic dispersion mechanism with no other mechanisms operating in a significant capacity. An algorithm to predict electrostatic stability was created by using viscosity as a metric for the total interaction potentials calculated by DLVO theory. These results indicate that DLVO accurately predicts colloidal stability in non-aqueous mediums.

**4:15 PM**

### **(ICCP-046-2013) An Exact DLVO Solution for Various Powders in Non-aqueous Mediums**

K. DeCarlo\*, Blasch Precision Ceramics, USA; W. M. Carty, Alfred University, USA

Non-retarded Hamaker constants and double-layer potentials were calculated for 10 different colloids in 11 different suspension mediums and were then used to calculate the total interaction potential of particle-medium pairs via DLVO theory. Colloids were selected based on crystal structure and bonding and included diamond, SiC, Cu and Co (both metallic particles and oxide particles), SiO<sub>2</sub> (crystalline and amorphous), TiO<sub>2</sub> (anatase and rutile), and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. The mediums were selected based on polarity and functional group and included methanol, ethanol, isopropanol, 2-butanol, acetone, 2-butanone, toluene, heptane, octanoic acid, poly(ethylene glycol), and water. The

non-retarded Hamaker constants for each colloid/medium suspension combination were calculated using the Lifshitz equation with the Ninham-Parsegian dielectric dispersion oscillator model. The double-layer interaction potential, and correspondingly, the predicted suspension stability, were calculated using an exact solution of the Poisson-Boltzmann differential equation.

**4:30 PM**

**(ICCP-047-2013) Development of High-performance  $\text{Si}_3\text{N}_4$  Ceramics Using Low-cost Raw  $\text{Si}_3\text{N}_4$  Powders (Invited)**

J. Tatami\*, Yokohama National University, Japan

$\text{Si}_3\text{N}_4$  ceramics have excellent mechanical properties. They have been applied to structural components, such as bearing balls and substrates. Generally,  $\text{Si}_3\text{N}_4$  ceramics are fabricated by powder processing and the production cost strongly depends on the raw powder. Direct nitridation of silicon is one of the techniques to synthesize  $\text{Si}_3\text{N}_4$  powders, which is known as low-cost powders. It has been difficult to fabricate high-strength  $\text{Si}_3\text{N}_4$  ceramics using the direct nitridation  $\text{Si}_3\text{N}_4$  powders due to the large fracture origin. Infra-red microscopy is a powerful tool to observe the internal structure of ceramics. As a result of internal structure observation, the size of an inhomogeneous region in the  $\text{Si}_3\text{N}_4$  ceramics and a green body fabricated from the direct nitridation powders was very similar to that of the granule. In order to reduce the flaw size, we prepared micro-granules, of which granule size is about 10  $\mu\text{m}$ , prepared by spray drying technique. The bending strength of the  $\text{Si}_3\text{N}_4$  ceramics fabricated using the micro-granules was about 1 GPa, which is as high as that of conventional  $\text{Si}_3\text{N}_4$  ceramics from high-cost powders, because of smaller fracture origin and more homogeneous internal structure by using the micro-granules.

**5:00 PM**

**(ICCP-048-2013) Controlling Stability of Functional Nanoparticles in Organic Solvents and Polymeric Materials: From Homogeneous Dispersion to Nano-scale Alignment**

M. Iijima\*, H. Kamiya, Tokyo University of Agriculture and Technology, Japan

The control of dispersion and aggregation behavior of nanoparticles is one of the essential processes to improve the final properties of composite materials. Since there are various species of solvents which are used during processing composite materials, understanding the strategy to control the dispersion/aggregation phenomena of nanoparticles in the desired media will be a powerful tool. In this study, a processing protocol to homogeneously disperse functional nanoparticles in various organic solvents and polymers as well as forming nano-scale alignments in these organic matrixes will be focused. Modifying nanoparticles by an anionic surfactant which is designed to have organic chain branched into hydrophilic PEG chain and hydrophobic alkyl chain was useful to homogeneously disperse nanoparticles in various species of organic solvents and polymers. Furthermore, a processing route to fabricate nano-scale alignments of these functional nanoparticles was designed by using polyamide nanofibers as templates. The stability of polyamide nanofibers in organic solvents/polymers can be improved by attaching these highly stabilized nanoparticles on their surface and the nano-scale alignments of functional nanoparticles can be achieved by dispersing the fabricated polyamide nanofiber/nanoparticle composites in the organic matrixes.

**Novel Characterization Tools for Ceramic Microstructures**

Room: Broadway I - II

Session Chairs: Peter Voorhees, Northwestern University; Linnéa Andersson, Oregon State University

**1:45 PM**

**(ICCP-032-2013) Evaluating Pore Space in Macroporous Ceramics with Water-based Porosimetry**

L. M. Andersson\*, Oregon State University, USA; P. Larsson, Innventia AB, Sweden; L. Wågberg, Royal Institute of Technology, Sweden; L. Bergström, Stockholm University, Sweden

We show that the pores size distribution and throat size distribution of macroporous ceramics can be determined by water-based porosimetry (WBP). We corroborate these findings by mercury porosimetry, and by pore and throat size distributions calculated from 3D data retrieved with X-ray micro-computed tomography (micro-CT). A combination of these two porosimetry methods offers the possibility to evaluate a very large span of throat and pore diameter sizes; from sub-micron sized pores with mercury porosimetry and up to 1200 micrometer sized pores with water-based porosimetry. The porosimetry methods are measuring the pore volume accessed at a given pressure, which involves complex interactions of distributions of pore and throat sizes as well as how they are connected, compared to X-ray micro-CT which evaluates the diameters of pores and throats independently. The sampling volume in WBP can be greatly increased compared to X-ray micro-CT and mercury porosimetry which increases the chance of finding and evaluating singularities, such as very large pores, in a macroporous material. In contrast to mercury porosimetry, water-based porosimetry is a non-destructive method that allows the liquid to be completely removed after the characterisation.

**2:00 PM**

**(ICCP-033-2013) 4D Evolution of Material Microstructure (Invited)**

P. Voorhees\*, J. Gibbs, A. Johnson, Northwestern University, USA; C. Park, K. Thornton, University of Michigan, USA; J. Fife, Paul Scherrer Institute, Switzerland; D. Dunand, Northwestern University, USA; K. Chen-Wiegart, Brookhaven National Laboratory, USA

With the advent of high-energy x-ray sources it is now possible to follow microstructural evolution in three dimensions and as a function of time (4D). The ability to observe and quantify the evolution of a microstructure provides fundamentally new insights into this complex process. This is especially true of those microstructures that have complicated interfacial morphologies, such as those found during ceramic processing. For example, we have studied the evolution of dendritic solid-liquid mixtures, which are of micron length scales and also are morphologically complex, using in situ three-dimensional (4D) x-ray tomography. In this case, the structure is quantified by the spatial variation of the interfacial curvature. The time evolution of this curvature provides new insights into the mechanisms responsible for the evolution of the structure. Different imaging techniques are required at nanometer length scales. As an example, we shall discuss measurements of the interfacial structure of nanoporous gold. We quantify the structure by measuring both the interfacial curvature and interfacial normals as a function of position and time. The interfacial normal distribution shows clearly that the structure becomes more anisotropic during coarsening.

2:30 PM

### (ICCP-034-2013) Nondestructive Evaluation of Environmental Barrier Coatings for Ceramic Matrix Composites

B. T. Richards\*, H. Wadley, University of Virginia, USA; P. A. Howell, NASA Langley Research Center, USA

The identification of reliable Nondestructive Evaluation (NDE) techniques is necessary to allow Ceramic Matrix Composites (CMCs) with Environmental Barrier Coatings (EBCs) to be used in gas turbines. Infrared Pulse Thermography, x-ray Computed Tomography (xCT), and Scanning Electron Microscopy (SEM) were used to evaluate trilayer yttrium monosilicate topcoat EBCs from initial fabrication through mock lean combustion thermal cycling to failure. Infrared Pulse Thermography provided rapid imaging with the ability to detect and monitor defects in the plane normal to the coating surface. In the EBCs imaged, these defects included delamination cracks, porosity, and oxidation which altered the thermal conductivity of the coating. xCT was used to detect and monitor growth of flaws throughout the lifetime of the coating at near-micron resolution. Drawbacks of xCT included long imaging times and a small imaging area at desirable resolution. Both NDE techniques were validated by SEM and were used to monitor flaw growth throughout the coating lifetime. These techniques are powerful research tools that are also promising candidate NDE technologies for EBC lifetime monitoring in gas turbines.

2:45 PM

### (ICCP-035-2013) Direct Observation of Sintering, Coalescence and Thermal Stability of CeO<sub>2</sub> by in situ ETEM

M. L. Cordeiro\*, Federal University of São Carlos, Brazil; P. A. Crozier, Arizona State University, USA; E. R. Leite, Federal University of São Carlos, Brazil

Bulk nanostructured materials have been at the cutting edge of materials science due to their outstanding properties. However, the ability of achieving full density and maintaining nanosized grains (< 20 nm) in sintered body is still a challenge. Most of this hindrance belongs to the difficulty in understanding the process of mass transfer in nanoscale during the sintering progress. In order to provide some insights on the nanocrystals (NC's) sintering and thermal stability, CeO<sub>2</sub> NC's, synthesized by hydrothermal in two-phase condition, were studied by in-situ environmental transmission electron microscopy. The experiments were carried out in a FEI Tecnai F20 field emission with a differentially pumped environmental cell under vacuum and O<sub>2</sub> atmosphere (1 Torr), and temperatures between 400 °C and 900 °C. The sintering process was directly observed and new features were found. The anisotropic behavior of Ostwald ripening process was identified and correlated with the evolution of the crystal to reach a minimum surface free energy (Gibbs–Wulff theorem). The size and shape stability of CeO<sub>2</sub> NC's were reached until 700 °C, in vacuum, likely due to the carbon shell from the pyrolysis, whereas at 890 °C a non-densifying behavior plays a fundamental role. At 500 °C in O<sub>2</sub> atmosphere, CeO<sub>2</sub> NC's show a dual behavior about the densification, mainly due to the unoxidized carbon trapped among NC's.

3:00 PM

### (ICCP-036-2013) An Evaluation of Non-destructive Real-time Analysis of Bulk Ceramics Using Raman Spectroscopy and Polarised Light Microscopy with a High Temperature Hot Stage

B. Taylor\*, S. Blackburn, Birmingham University, United Kingdom; S. T. Welch, Rolls Royce PLC, United Kingdom

An evaluation is given regarding the use of Raman spectroscopy by reflection, and transmitted polarised light microscopy for analysis of bulk ceramics used in passageway cores. Sacrificial ceramic cores are used to provide internal features such as cooling channels to aero engine turbine blades during investment casting. During the firing process the conversion of the main ingredient (amorphous silica) into β-cristobalite plays a significant role, as it directly affects the dimensional stability, shrinkage and leachability. Thin sections (150 μm)

produced from low density ceramics and raw powders were studied. Polarised light microscopy was shown to be favourable for analysis of crystal growth mechanisms and low temperature (≈250°C) α-β cristobalite phase transitions. Polymorph transformations were recorded in real time and provided detail on the cracking caused by cyclic heating which aids in the leaching of cores after casting. Raman spectroscopy provided a method for evaluating the level of mixing of powders, with potential use as real time analysis during flow in a production environment. The difficulties of high temperature analysis (≈1500°C) are discussed and methods to improve observations of crystallisation provided.

3:15 PM

### (ICCP-037-2013) In Situ Time-resolved Synchrotron Radiation Studies of Oxide Ceramics during Ultrafast Microwave Heating

S. Vaucher\*, K. Ishizaki, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland; M. Stir, University of Berne, Switzerland; A. Cervellino, Paul Scherrer Institute, Switzerland; J. Catalá-Civera, Universidad Politécnica de Valencia, Spain; R. Nicula, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

We report on our recent progress with the in situ characterization of microwave heating of ceramic materials at very high heating rates. Experimental methods with subsecond temporal resolution, in particular high-temperature time-resolved X-ray scattering (SAXS/WAXS) and time-resolved X-ray micro-tomography using synchrotron radiation, will be discussed in more detail, with application to microwave sintering or to the characterization of athermal effects during thermal runaway in ZnO or TiO<sub>2</sub> ceramics. Various examples of rapid nanocrystallization processes, grain growth or structural phase transitions followed in situ during microwave irradiation will be given as well.

## Additive Fabrication of Mesoscale Ceramic Components

Room: Broadway I - II

Session Chairs: James Adair, Penn State University; Francis Cambier, BCRC

3:45 PM

### (ICCP-049-2013) Shaping of Oxide Ceramics by Laser Assisted Process: Additive Technology (SLM) and Machining in the Green State

E. Juste, F. Petit, C. Ott, V. Lardot, F. J. Cambier\*, BCRC (EMRA), Belgium

Laser manufacturing allows production of complex parts. Despite its good potential, results are not convincing for ceramics because of their intrinsic properties: refractoriness and brittleness. However, it has advantages: it is a high energy contactless method, allowing high precision. Two approaches can be considered for shaping ceramics: by material subtraction and by addition. Two main drawbacks limit development of the subtractive method applied after firing: the low material removal rate and the heat affected zone, detrimental for surface quality and component integrity. Additive manufacturing by selective laser melting (SLM) of a ceramic powder is yet a process under development: parts are usually damaged and their properties are low by comparison with classical materials. Moreover, the low laser absorption of oxide ceramics in the near IR region is also a critical issue. By coating ceramic powders with carbon, the coupling with near IR lasers is significantly improved. Results obtained for laser machining of green bodies and SLM of powders will be shown: complex ceramic shapes of relative density up to 80% can be obtained by SLM and laser green machining is a very rapid technique to prepare complex cm-sized 3D parts (micro-turbines and devices, dental prostheses), fully dense after firing. The paper gives details on the process and attained properties.



**4:00 PM****(ICCP5-050-2013) Powder-based Additive Manufacturing of Ceramic Parts (Invited)**

J. Günster\*, C. Gomes, BAM, Germany; T. Muehler, J. G. Heinrich, TU Clausthal, Germany

The production of ceramic parts by an additive manufacturing process is still a challenging task. Especially in powder-based technologies, such as 3D printing or laser sintering, the feedstock material is commonly spread out as thin layers of a dried powder/granulate by a roller or a shaker system. As consequence of that, the layers are characterized mostly by a low packing rate. On the other hand, from the ceramic processing viewpoint, appreciable densities can be reached by the use of ceramic slurries. In this context, the so-called Layer-wise Slurry Deposition (LSD) process has been developed. The latest advances in the development of this technology will be reported, the microstructure of laser sintered bodies will be discussed and strategies for an improved microstructure, i.e. denser parts, during laser sintering will be introduced. Additionally, the role of additive manufacturing technologies for the processing of ceramics will be discussed, in particular related to some concepts for an improved powder spreading approach in both laser-based and 3D-printing technologies will be evaluated.

**4:30 PM****(ICCP5-051-2013) Design and Manufacture of Mesoscale Components and Devices (Invited)**

J. H. Adair\*, M. I. Frecker, G. A. Lesieutre, B. L. Babcox, R. L. Staveky, Penn State University, USA

The lost-mold, rapid infiltration forming (LM-RIF) process is a manufacturing approach that integrates CAD-CAM design principles, semi-conductor large-scale-microfabrication, and particulate processing science. Metal, ceramic and composite materials can be manufactured using LM-RIF. The use of nanoscale ceramic particulates particularly Y-TZP and alpha alumina is presented with an aim toward high strength structural applications and the design of phonon switches for aerospace applications based on ceramic – metal composite architectures. The Weibull average bend strength of 2.3 GPA has been achieved in a Y-TZP bend bar geometry in prior reports. This permits the design of reasonably complex surgical instruments with a 6 sigma certainty of no failure during surgical interventions. Examples of the LM-RIF surgical instruments will be presented as well as the results of testing by a team of surgeons. Among the thermal conduction modules presented are cellular composite materials composed of thermal elements based on high thermal conductivity metals interfaced with ceramic components. The design of such phonon switches and preliminary results will be presented. It is shown that the LM-RIF approach has the ability to produce reasonably complex components based on current 2D approaches. The prospects and preliminary prototypes for even more complex architectures based on 3D LM-RIF will be presented.

**5:00 PM****(ICCP5-052-2013) 3D Printing of Interdigitated Li-Ion Microbattery Architectures**

T. Wei\*, Harvard, USA; K. Sun, University of Illinois, USA; B. Ahn, Harvard, USA; J. Seo, Korea Advanced Institute of Science and Technology, Democratic People's Republic of Korea; S. J. Dillon, University of Illinois, USA; J. A. Lewis, Harvard, USA

Developing compact high energy density power sources will enable autonomous micro/nano devices used in next generation microelectronics. Ideal structures combine intimately positioned electrodes with short diffusional path lengths assembled in a three-dimensional structure. Emerging applications require highly integrated patterning approaches for defining the spatial location and composition of metal oxide and carbon-based electrodes as well as polymer separators and electrolytes. Typical fabrication approaches require complex lithography or etching processes, which are either costly or difficult to scale

into the third dimension. In this presentation, we describe recent efforts in fabricating 3D Li-ion microbattery with interpenetrating high-aspect ratio architectures via layer-by-layer printing. The concentrated viscoelastic inks composed of Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> or LiFePO<sub>4</sub> nanoparticles were carefully tailored for the desired rheological, drying and printing behavior. Both fabrication and electrochemical characterization of these 3D interdigitated microbattery architectures (3D-IMA) will be discussed.

**Microstructure Tailoring of Ceramics**

Room: Broadway III - IV

Session Chairs: Florian Bouville, LSFC/ Saint Gobain CREE; Veena Tikare, Sandia National Laboratories

**1:45 PM****(ICCP5-038-2013) Fabrication of Highly <001><sub>C</sub> Textured PMN-PT Ceramics by Low-field Dynamic Magnetic Alignment and Templated Grain Growth**

S. F. Poterla\*, Channel Technologies Group, USA; R. J. Meyer, G. L. Messing, Pennsylvania State University, USA

Highly <001><sub>C</sub> textured Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-PbTiO<sub>3</sub> (PMN-PT) ceramics were fabricated by a novel combination of dynamic magnetic alignment and templated grain growth (TGG). In this approach, plate-like 0.4(Na<sub>1/2</sub>Bi<sub>1/2</sub>)TiO<sub>3</sub>-0.6PbTiO<sub>3</sub> (0.4NBT-0.6PT) templates were aligned in a PMN-PT matrix via slip casting in a low field (2.2 Tesla) permanent magnet array. The resulting ceramics show a narrower grain orientation distribution (FWHM ~7°) and higher piezoelectric response (d<sub>33</sub> = 1222 pC/N) than textured ceramics made via tape casting and TGG with the same templates. This work departs from prior magnetic alignment literature in that optimal grain alignment was achieved by design of the powder system and slurry rheology rather than by utilization of ultra-high (≥10 Tesla) magnetic fields. We show that alignment kinetics in this system are strongly dependent on slurry rheology, with the best results obtained at viscosities of 0.1-1 Pa s (shear rate of 0.01 s<sup>-1</sup>) under conditions used in this study (B = 2.2 Tesla, 3 RPM sample rotation). The magnetic anisotropy of 0.4NBT-0.6PT templates used in this work is estimated at 2.3 × 10<sup>-8</sup> (SI units) based on observed alignment kinetics in this system.

**2:00 PM****(ICCP5-039-2013) Texture Developing and some Properties of Feeble Magnetic Ceramics by Colloidal Processing in a Strong Magnetic Field (Invited)**

Y. Sakka\*, NIMS, Japan

The controlled development of texture is one of the ways for effectively improving properties of ceramics. We have demonstrated a new processing of textured ceramics with a feeble magnetic susceptibility by colloidal processing in a high magnetic field and subsequent heating. The principle of the process is that a crystal with an anisotropic magnetic susceptibility will rotate to an angle minimizing the system energy when placed in a magnetic field. Some trials have been presented to fabricate highly textured ceramics using template particles, changing magnetic susceptibility of different cationic doping, rotation magnetic field, etc. This processing can be applied to fabricate many kinds of textured ceramics with non-cubic structure, such as alpha-alumina, aluminium nitride, silicon carbide, silicon nitride, MAX phase materials, ZrB<sub>2</sub>, B<sub>4</sub>C, etc. Crystalline-textured controlled laminated composites can be fabricated by two methods: one is textured layered structure materials, such as textured MAX phase ceramics, and the other is using electrophoretic deposition by varying the angle between the vectors of electric field and magnetic field. Some anisotropic properties, such as mechanical properties, thermal conductivity, oxidation resistance, etc. will be demonstrated.

2:30 PM

**(ICCPs-040-2013) A Review of Meso-scale Simulation Capabilities of Microstructural Evolution in Ceramics (Invited)**

V. Tikare\*, Sandia National Laboratories, USA

Three decades of microstructural evolution model development starting in the early 80's has resulted in models that are sufficiently mature so that they can inform engineering design of ceramic materials. In this presentation, the most widely used microstructural evolution models, namely phase-field, Monte Carlo Potts, cellular automata and discrete element, will be reviewed and their application to simulate processes such as coarsening, sintering, diffusion-controlled phase transition, evolution in thermal gradients and more will be demonstrated. Furthermore, examples of the use of simulations to design engineering components and optimize processing conditions will be presented. Finally, the limitations of these models will also be reviewed followed by a discussion of the latest trends in model development to address the simulation limitations. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

3:00 PM

**(ICCPs-041-2013) Control of Lamellar Structure with Different Orientated Layers by Using Electric and Magnetic Fields**

T. S. Suzuki\*, T. Uchikoshi, Y. Sakka, National Institute for Materials Science, Japan

Tailoring the crystallographic orientation in ceramics is one of effective ways for improving their properties, such as electrical, piezoelectric and mechanical properties. Layered structure has been proposed as an alternative for the design of structural ceramics. On the other hand, in this decade, we have reported that the successful control of the crystallographic orientation in diamagnetic ceramics, such as alumina, titania,  $\alpha$ -SiC, was achieved by a colloidal processing in a strong magnetic field. When we use the crystals with asymmetric unit cells, these crystals have anisotropic susceptibility. These ceramic particles in suspension become rotated to an angle that minimizes the system energy by a magnetic torque generated from the interaction between the anisotropic magnetic susceptibility and the applied magnetic field. Electrophoretic deposition (EPD) is a colloidal processing technique wherein ceramic bodies are directly shaped from a stable colloid suspension by a dc electric field. The suspension was consolidated by EPD in a strong magnetic field of 12T to control the lamellar structure with different orientated-layers. The direction of the electric field relative to the magnetic field was altered to control the dominant crystal faces. This processing was applied to alumina and SiC, and the lamellar microstructure was examined by EBSD analysis.

3:15 PM

**(ICCPs-042-2013) Phase-Field Simulation of Plate-like Grain Growth during Sintering of Alumina**

K. Shinagawa\*, S. Maki, Kagawa University, Japan; K. Yokota, Kagawa Industrial Technology Center, Japan

The toughness of alumina can be improved by utilizing the in-situ formation of plate-like anisotropic grains during sintering, that is, abnormal grain growth (AGG). Computer simulations of AGG may be effective to realize the conditions for obtaining the desired self-composite microstructure. In the first part of this study, sintering experiments of high-purity alumina powders were conducted to confirm the effects of powder size distribution as well as the amounts of additives. The specimens with none or small amounts of the additives showed normal grain growth regardless of the size distribution. With increasing the amounts of the additives, large plate-like grains appeared, while the matrix grains remained small. In this situation, the offset of AGG was accelerated by broadening of the size distribution. In the second part, phase-field simulations for the plate-like grain growth were implemented. The grains became angular by introduc-

ing the anisotropic grain boundary energy, and large plate-like grains were reproduced when the critical driving force of coarsening was set up. The incubation time of AGG was also observed in the case of the narrow size distribution. Although the morphology of the plate-like grains did not exactly agree with the experimental observations, a possibility of the present method as a computational tool for simulating plate-like AGG was verified.

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**(ICCPs-043-2013) Strong and Tough Ceramics by Grain Orientation Control and Interface Design**

F. Bouville\*, Laboratoire de Synthèse et Fonctionnalisation des Céramiques/ Saint Gobain CREE, France; E. Maire, INSA de Lyon MATEIS, France; S. Deville, Laboratoire de Synthèse et Fonctionnalisation des Céramiques/ Saint Gobain CREE, France

Controlling the assembly and the interface between anisotropic grains of polycrystalline material is a possible path to combine strength and toughness in polycrystalline ceramics. Inspired by the architecture of nacre, a dense alumina material comprising aligned platelets and nanoparticles have been obtained by ice templating, and presents a remarkable combination of mechanical properties. The growth of ice crystals is used to align the alumina platelets. To improve our understanding of the particle arrangement during freezing and assess the proper working conditions, a discrete element model has been developed. After ice removal, the porous samples have been sintered by SPS to obtain high relative density while restricting grain growth. Strength and toughness measurements were carried out to investigate the influence of the materials composition over the mechanical properties. Because of their unique microstructure, these materials are characterized by a unique combination of strength and toughness. The processing principles exposed here are independent of the nature of the material and can thus be applied to other systems.

### Synthesis and Processing of Electrical Ceramics

Room: Broadway III - IV

Session Chair: Stephen Poterala, Channel Technologies Group

4:00 PM

**(ICCPs-055-2013) Ceramic Processing and Evaluation of (Na,K)NbO<sub>3</sub>-based Lead-free Piezoelectrics (Invited)**

K. Kakimoto\*, Nagoya Institute of Technology, Japan

(Na,K)NbO<sub>3</sub>-based system has been considered to be one of the most attractive ceramic compositions in the research field of lead-free piezoelectric ceramics. A number of studies have been carried out to improve their densification and piezoelectric properties. However, there are still problems in the manufacturing and the reliance in the electric properties. We report the latest our success that a dense (Na,K)NbO<sub>3</sub> ceramics with enhanced piezoelectric properties could be synthesized by low-temperature atmospheric sintering. The key technology was the development of fine powders derived from a new aqueous-based citrate precursor technique, which is classified to chemical solution process. This route is now extended to engineer film and fiber structure, too. Furthermore, the critical size for inducing good electric properties are addressed for better understanding of (Na,K)NbO<sub>3</sub>-based system from a viewpoint of processing. In this study, a model experiment regarding the size effect was carried out by a centrifugal size-classification technique for the ceramic particles. The results clearly showed the distinguishing of different ferroelectric and piezoelectric properties on the basis of grain size for (Na,K)NbO<sub>3</sub>-based ceramics with different microstructures.

4:30 PM

**(ICCP-125-2013) Nanostructured Oxide Ceramics for Efficient Thermoelectric Energy Conversion (Invited)**

M. Ohtaki\*, Kyushu University, Japan

Boosted by the global environment issues and carbon dioxide mitigation problems, thermoelectric energy conversion is becoming more and more of vital importance for recuperation of decentralized waste heat energy for higher energy efficiency. Nevertheless, conventional TE materials such as Bi<sub>2</sub>Te<sub>3</sub> and PbTe based on Bi, Te, and Pb are unlikely to satisfy wide commercialization because of the toxicity, poor heat durability, and low abundance of the comprising elements, particularly for those containing Te. In terms of durability at high temperature in air, metal oxides are most attractive, which are highly durable particularly at the temperature range of > 400 °C, where all the non-oxide candidate materials will eventually be oxidized under aerobic conditions. In this paper, microstructure control of ZnO-based oxides by multinary doping and nanovoid formation will be reported. Upon the binary doping of ZnO with Al and Ga, the amount of a Ga-related impurity phase is revealed to show an excellent quantitative agreement with the decrease in the thermal diffusivity. Furthermore, multinary doping with Al, Cu, and Ga results in a substantially low thermal conductivity values just 2–3 times higher than the theoretical minimum of the thermal conductivity, showcasing an effective thermal conductivity reduction by a nanocomposite structure in bulk oxides.

**Poster Session**

Room: Plaza Foyer

**(ICCP-001-2013) Influence of Oxygen Potential and Time on Grain Growth of Cr<sub>2</sub>O<sub>3</sub>-doped UO<sub>2</sub> Pellets**

J. Oh\*, J. Yang, D. Kim, J. Kim, Y. Rhee, K. Kim, Korea Atomic Energy Research Institute, Republic of Korea

The recent development of advanced UO<sub>2</sub> pellet materials for commercial reactors is mainly focused on the large grain pellet, which can deform easily at an elevated temperature. Cr<sub>2</sub>O<sub>3</sub>-doped UO<sub>2</sub> pellet is one of the promising candidates for the high burn-up fuel in commercial LWRs. To increase the grain size effectively, it is important to control the additive content and sintering atmosphere. Relevant research on the Cr<sub>2</sub>O<sub>3</sub>-doped UO<sub>2</sub> system revealed that the doped Cr<sub>2</sub>O<sub>3</sub> formed a liquid phase under optimized oxygen potential, and those liquid phases promoted the grain growth. This study deal with the influence of parameters such as the oxygen potential of the sintering atmosphere and sintering time on grain growth of Cr<sub>2</sub>O<sub>3</sub>-doped UO<sub>2</sub> pellets. The results showed that the grain size increased until the CO<sub>2</sub> in H<sub>2</sub> reached at 1.7% and then saturated. In case of 7.4%CO<sub>2</sub> in H<sub>2</sub>, the initial grain growth rate was faster than 1.7%CO<sub>2</sub> in which Cr-O could form a eutectic liquid phases. Those result may imply that grains grow very fast when the liquid phase has been formed so that integral amount of liquid phase or its sustainment time does not affect the grain growth behavior.

**(ICCP-002-2013) Formulation of Pickering Emulsions Using Natural Phyllosilicates**

G. Lecomte-Nana\*, D. Kpogbembabou, ENSCI - GEMH, France; A. Aimable, M. Bienia, CNRS - SPCTS, France; C. Peyratout, ENSCI - GEMH, France; F. Rossignol, CNRS - SPCTS, France

The present study aimed to investigate the formulation of Pickering emulsions using palygorskyte, talc and halloysite. These clays minerals (phyllosilicates) exhibited different chemical and structural compositions. Four formulations of Oil-in-Water (O/W) and of Water-in-Oil (W/O) emulsions were tested using 5, 15 and 45 mass% (regarding water) of each phyllosilicate. Effects of nature and amount of phyllosilicates on size distribution and stability of emulsion's droplets and rheological properties of emulsions were studied. All stable emulsions were obtained when clays minerals (15 mass%) were previously mixed with water phase. Talc (T1) and palygorskyte (P1)

appear to stabilize W/O emulsion. Conversely, halloysite (H3) allows stabilization of O/W. These stable emulsions exhibited  $G' > G''$  between in the range 0.1-100 Hz, thus presenting a solid-like behavior. Moreover, talc led to Pickering emulsion (T1) with the lowest surface tension (34 mN.m<sup>-1</sup>) and highest dynamic cohesion energy ( $E_c=726$  J.m<sup>-3</sup>). This trend was in agreement with the observed stability and density of obtained Pickering emulsion (T1>P1>H3). Indeed, T1 exhibited larger amount of droplets and a wide size distribution compared to P1 and H3. The characterization of the stability of Pickering emulsions showed that emulsion droplet size distributions governed the rheological behavior of the emulsion and its stability.

**(ICCP-003-2013) Evolved Gas Analysis during the Sintering of Barium Titanate**

S. J. Lombardo\*, M. Moss, University of Missouri, USA

A combined dilatometer-mass spectrometer system (CDMS) has been developed to examine the high temperature chemistry that accompanies the sintering of ceramic materials. During the sintering of barium titanate, a number of species appear in the gas phase, and the assignment of these species to chemical compounds was made using comparisons to cracking patterns, natural isotopic abundances, and the decomposition of model compounds. Carbon dioxide is evolved in the early part of the heating cycle and into the sintering hold period at 1350°C. Just prior to the onset of sintering, a species with mass-to-charge ratio of 64, either titania or sulfur dioxide, appears in the gas phase; the species is ultimately assigned to sulfur dioxide. The origin of the species in the gas phase is discussed in terms of the synthesis scheme for barium titanate. The influence of the evolved gases on microstructural development is also examined.

**(ICCP-004-2013) Porous Alumina and Mullite-based Ceramics prepared with Starch or Wheat Flour: Processing and Characterization**

W. Pabst\*, E. Gregorova, T. Uhlirova, M. Vesely, Institute of Glass and Ceramics (ICT Prague), Czech Republic; M. Camerucci, M. Talou, Instituto de Investigaciones en Ciencia y Tecnologia de Materiales (INTEMA), Argentina

Porous oxide and silicate ceramics, like alumina and mullite-based ceramics, are widely used as furnace linings and kiln furniture. Apart from light weight and material savings, the main advantage in these refractory applications is their low thermal mass. This work is concerned with the preparation of porous alumina and mullite-based ceramics using starch or wheat flour as pore formers, with the objective to prepare porous refractories with porosities exceeding 45 %. Starch consolidation casting (SCC) into metal molds is used for shaping, i.e. when using starch it acts as a fugitive pore former and as a stiffening agent, while in the case of wheat flour the SCC process is additionally accompanied by foaming. Porosities of more than 45 % can easily be achieved with starch and more than 60 % with wheat flour. A hierarchical microstructure with the largest pores of order 100–300 microns results in the case of processing with wheat flour. The work includes rheological studies (rotational viscometry), processing optimization issues and microscopic investigation results concerning dry-green and as-fired samples. Quantitative image analysis of the pore space is performed using reliable microstructural descriptors such as the mean intercept length (surface density) and the mean curvature integral as well as Saltykov-transformed pore size distributions.

**(ICCP-007-2013) Modeling Supported Experimental Demonstration of Thermo-chemical Expansion**

T. R. Hinklin\*, C. Lewinsohn, Ceramtec Inc., USA

Many important ion transport ceramics exhibit chemical expansion during processing and use. Surface ion implanted glass uses chemical expansion to its advantage; however, traditional thermal processing of thermo-chemical expansive materials leaves the surface in tension, resulting in reduced reliability materials. Here, controlled morphology bi-layer strips of a substituted rare earth cobaltite, which exhibits

a large thermo-chemical expansion in air, are used to demonstrate and quantify the magnitude of stresses developed in thermo-chemical expansive ceramics. Analytic and numerical models supported by physical property measurement are employed to interpret and demonstrate the experimental results.

### (ICCPSP-008-2013) Design and Synthesis of Spray Pyrolyzed Bioactive Glass

S. Shih\*, National Taiwan University of Science and Technology, Taiwan; C. Chen, Feng Chia University, Taiwan; Y. Chou, National Taiwan University of Science and Technology, Taiwan

Bioactive glasses (BGs) have become one of the important bone implant materials due to their superior biocompatibilities and bioactivities. Various processes have been developed to fabricate BGs. Among them, the conventional glass process and sol-gel process are common methods for fabricating BGs. However, the conventional glass process has disadvantages of low purity and chemical inflexibility. Also, the sol-gel process has the drawbacks of discontinuous processing and long processing time, making it unsuitable for mass production. This study demonstrates a successful synthesis of BGs using a spray pyrolysis (SP) method to overcome these problems. The bioactivities of the SP synthesized BGs are correlated with the main SP processing parameters, morphologies, crystallographic structures and surface areas. Finally, the BG formation mechanisms using SP were proposed.

### (ICCPSP-009-2013) Structure Property Relationships in Nano-Porous low-k a-SiC:H Dielectric Materials

S. King\*, Intel Corporation, USA

Materials with a low dielectric constant (i.e. low-k) are increasingly replacing SiO<sub>2</sub> as insulating dielectrics in nano-electronic products in order to reduce resistance-capacitance delays in interconnect wiring. The current industry standard low-k materials are plasma deposited "carbon doped" variations of SiO<sub>2</sub> where carbon has been incorporated as terminal organic groups to disrupt the local SiO<sub>2</sub> network and introduce nano scale porosity. This reduces the k of the film, but also reduces the thermal, mechanical, and electrical properties of the nano-porous a-SiOC:H material. In this regard, similarly prepared nano-porous low-k a-SiC:H materials are of increased interest due to their potential for improved thermal-mechanical properties and resistance to environmental degradation. To fully understand the structure-property relationships in low-k a-SiC:H materials, we have utilized Fourier Transform-Infrared spectroscopy to study local structure and additional thermal, mechanical, and electrical measurements to uncover the interplay between network/terminal bonding and resulting properties. In this report, we will detail the observed structure - property relationships for low-k a-SiC:H dielectrics and specifically demonstrate that a remarkable range in dielectric constant (< 3 - > 7), Young's Modulus (< 5 - > 200 GPa), and thermal conductivity (0.09 - 4 W/mK) can be achieved.

### (ICCPSP-010-2013) Scalable Production Methods for the Controlled Synthesis of TiO<sub>2</sub>-Based Photocatalysts

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The inability of pure TiO<sub>2</sub> photocatalysts to absorb in the visible region and the tendency for recombination of electron-hole pairs in the bulk has limited the usefulness of TiO<sub>2</sub>, however, several approaches to modifying the band structure to promote visible light absorption have been identified including the introduction of metal dopants that occupy Ti<sup>4+</sup> sites and non-metal dopants that replace O<sup>2-</sup>. Previous processing methods offer limited compositional control and/or poor scalability. Here we discuss synthesis methods that afford fine compositional control and scalability. Liquid feed-flame spray pyrolysis (LF-FSP) provides ultrafine and nanosized metal-oxide powders by combustion of metalloorganic precursors dissolved in flammable solvents. Recent efforts involve nitriding photocatalyst nanopowders produced by LF-FSP using a fluidized bed reactor (FBR) at elevated temperatures under NH<sub>3</sub> flow. Results indicate the formation of

oxynitride surface phases which can be tailored in terms of N/O composition and overall oxynitride content by varying nitridation conditions. Our objective is to create novel photocatalytic materials using the fine compositional control provided by combining these scalable synthesis methods. Studies involving the effects of dopants in the bulk and modifications to the surface composition on the photocatalytic activity of TiO<sub>2</sub> nanopowders will be discussed.

### (ICCPSP-011-2013) Investigation and Optimization of Aqueous, High-energy Ball-milling for Producing Fine-grained, Transparent Ceramic Materials

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Magnesium aluminate spinel is considered as one of the most promising transparent ceramic. Reaction sintering of alumina and magnesia offers the investigation of the wide solid solution range in the binary system. One approach to manufacture transparent spinel is aqueous high-energy ball-milling of the powder mixture, followed by a suitable densification process. The rheological behavior and the influence on the particle size distribution are shown for the stoichiometric composition and two alumina-rich, non-stoichiometric compositions. The optimization of the suspensions is presented in order to achieve narrow particle size distributions that allow the desired high densification level. The viscosity is correlated to the particle size, measured by rotational viscometer and laser diffraction analyzer. Three different levels of viscosity are adjusted at 15, 60 and 120 minutes of grinding times. The lowest level of viscosity creates low particle sizes of 128 ± 1 nm (d<sub>90</sub>) after 120 minutes in a narrow distribution, whereas particle sizes of 214 ± 7 nm (d<sub>90</sub>) in a broad distribution are achieved for the high level viscosity suspension. By transmitting the results to the alumina-rich compositions it is shown that the particle size and the width of the distribution increase by increasing the alumina-content.

### (ICCPSP-012-2013) Mechanochemical Approaches to Tailoring Nanostructure for Digitally Fabricated Ceramics

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High energy wet and dry milling are frequently employed to create the inks used for digital fabrication of ceramics. This work explores the effect of typical milling protocols on the physical and chemical characteristics of NiO particles and on the microstructure of sintered NiO parts. NiO is dry milled with and without carbon additives, and wet milled in inert fluid (Vertrel XF) and in methanol. Scanning electron microscopy and Brunauer Emmet Teller measurements are used to track the particle size distributions of the milled powders. Upon sintering the wet and dry milled parts to 1100° C and 1500° C, cross-sectional scanning electron microscopy shows that the wet milled powders preserve homogeneous nanoscale particles as high as 1100° C, while the dry milled powders undergo dramatic densification and grain growth at these temperatures. In addition, the particles wet milled in methanol undergo less densification and grain growth at 1100° C as compared to the particles wet milled in inert fluid. Taken together, these results suggest that the particle size distribution differences imparted by wet versus dry milling will be the primary drivers of microstructural evolution in milled NiO inks, but that additional structural control can be exerted by the choice of milling fluid and its resultant effect on particle surface chemistry.

### (ICCPSP-013-2013) Developing MgO Support and Dip Coating CGO Slurry for Oxygen Transport Membrane

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Ceramic oxygen transport membranes (OTMs) can effectively be used to separate oxygen from air at high temperature in integrated

with a high temperature process. In this work, a thin film of  $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95-\delta}$  (CGO) layer was prepared on an extruded MgO support structure by dip coating. The optimization of the porous MgO support by extrusion and of the dip coating of the functional CGO layer on the support are described. In the thermoplastic feedstock the ceramic, polymer and pore former content were systematically varied to achieve sufficient porosity of > 35% and a suitable densification behavior for co-sintering with the CGO membrane layer. TGA and dilatometer studies were employed for the optimization of debinding and sintering cycle of the MgO support. The MgO support had a porosity of about 38% at 1300°C/ 2 hrs. A nitrogen gas flux of 6.5 ml/min  $\text{cm}^{-2}$  for  $\Delta P$  of 1 bar could be achieved at room temperature operation. The mechanical strength was characterized using a 4-point bending method and the measured Weibull strength was 59 MPa which is considered sufficient for the application. For the functional CGO layer, an ethanol based dip coating process was optimized and the coating thickness could be controlled by withdrawal speed, the solid content and the viscosity of the slurry. Finally, Scanning Electron Microscope (SEM) was used to confirm the microstructure integrity of the MgO/CGO bi-layer.

#### (ICCP-2014-2013) Stabilization of Ceria and Zirconia Polishing Slurries above Isoelectric Point Using Anionic Surfactants

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A method for stabilizing polishing slurries to prevent their agglomeration while maintaining surface activity is demonstrated. Negatively charged ceria particles in agglomeration-prone polishing slurry are size-stabilized using anionic surfactant. The stabilization technique is demonstrated using multiple surfactants and in both ceria and zirconia polishing slurries. Results suggest that the surfactant molecules, which are of like charge to the slurry particles at pH above the isoelectric point, bind either in low numbers or weakly to the slurry particles. Zeta potential measurements reveal that negatively charged surfactant slightly increases the surface potential of the ceria under conditions where the ceria is negatively charged. Changes to pH and ionic strength are shown to disrupt the surfactant's ability to size-stabilize the ceria. Stabilized polishing slurry removes material at a rate comparable to that of unstabilized slurry and leads to improved work piece surface quality after polishing. Size-stabilizing colloids by this method may prove valuable for colloidal systems where surface functionality is important, such as those used in catalysis, ceramics processing, and polishing. \*Work performed under the auspices of U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-ABS-624452

#### (ICCP-2015-2013) Colloidal Processing of Difficult-to-Densify Ceramics

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Ultra-high-temperature ceramics (UHTCs) are a class of materials with supreme strength and hardness, extremely high melting point, and excellent chemical stability under extreme environments. Materials within this category are ZrB<sub>2</sub>, B<sub>4</sub>C, SiC and many others, which are generally difficult to densify due to their strong covalent bonding and low diffusion coefficient. Colloidal processing has been considered as a promising approach to fabricate UHTCs into dense, complex-shaped objects with reduced flaws. Compared to the conventional hot pressing, colloidal processing offers improvement in shape capability and product reliability. Colloidal dispersion of ceramic powder in both aqueous and non-aqueous media has been considered. The non-aqueous colloidal processing is the chosen approach due to the difficulty of dispersing powder in aqueous media. Using slip casting, the colloidal stable, non-aqueous suspensions can be processed into efficiently packed green bodies with relatively high green density (63%). When pressureless sintered at 2100°C for 1 hour without any sintering additive, a final sintered density as high as 92%

can be achieved. This result is significantly higher than those obtained by aqueous colloidal processing or uniaxial dry pressing. The ability to process non-aqueous suspensions also opens up the possibility for complex-shape forming using other techniques, such as gel-casting or freeze casting.

#### (ICCP-2016-2013) Colloidal Processing of Yttria Powders Composed of Mono-dispersed Spheres

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Colloidal processing method has been adopted in this work for the fabrication of transparent yttria ceramics using powders composed of mono-dispersed spherical particles. The starting powders were prepared by a urea-based homogeneous precipitation method. The precursor powder has a narrow range of particle size distribution and the particles are of spherical morphology. Aqueous suspensions were prepared by dispersing the yttria powders in deionized water with the addition of a polyelectrolyte dispersant of polycarboxylate. The characteristics of Y<sub>2</sub>O<sub>3</sub> suspensions were evaluated by the adsorption isothermal, sedimentation, and rheological measurements. It is shown that a saturation plateau level was reached when the addition of dispersant was 0.5 wt% on the basis of dry yttria powder. However, the rheological characterization of the suspension revealed that a minimum addition of 2.0 wt% of dispersant was required to get a suspension with the lowest viscosity. Under such condition, the suspensions showed Newtonian fluid behavior. The effects of the possible reaction between the dissolved Y<sup>3+</sup> ions and the added dispersant on the dispersion of yttria suspensions were discussed. The suspensions of various solids loading were subsequently consolidated into green compacts via centrifugation. The sintering behaviour and microstructure of the sintered ceramics were characterized.

#### (ICCP-2017-2013) Evolution of Grain Boundaries during Initial Stage Sintering of Polycrystalline Zirconia Spheres

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Fundamental studies on initial stage sintering of single crystal spheres suggest an increase in neck area (growth of solid – solid interface), rotation of particles and consumption of smaller particles by coarsening. While most of the studies have been carried out on single crystal spheres, neck growth and coarsening in polycrystalline particles has not been studied extensively. In the polycrystalline case, multiple grain boundaries may form in the neck region as the neck grows. In addition, there will be grain boundaries in individual polycrystalline particles that terminate at the particle surface making an angle to the plane of the neck formed. These can be additional sources of mass to be plated at the neck region. In the present study, evolution of microstructure during initial stage sintering in a compact of polycrystalline micron sized yttria stabilized cubic zirconia spheres is characterized by electron backscattered diffraction (EBSD). In spite of possible pinning by pores, grain boundary migration is observed to be an important phenomenon in initial stage sintering.

#### (ICCP-2018-2013) Novel Yttria Doped Mullite Particles with Increased Properties for Sintering

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Develop novel yttria doped mullite particles with better sintering properties, such as: high density at lower sintering temperatures, fine microstructure and without glassy phase between grain boundaries. Usually in the studies of mullite, yttria has been employed as sintering additive to reduce the temperature of densification. However, since it is difficult to homogeneously distribute the additive, some microstructural defects are generated, such as: non uniform microstructure and big amounts of glassy phase between boundaries. So, yttria doped mullite precursor (nano-composite type core and shell) has been synthesized via Homogeneous Precipitation. The particles consisted in fibrous pseudo-bohemite outer coatings and SiO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> nanoparticles as inner cores. After heating, fine mullite particles were

obtained at 1300°C, showing sizes below 0.5µm. The green bodies were obtained by standard slip casting. Comparative results of commercial and precipitated mullite are presented to show the advantages of these novel particles. The 1.5% of Y<sub>2</sub>O<sub>3</sub>-mullite system gives the best results: sintering temperature 1450°C (being 200°C lower than the undoped samples), relative density around 99%, equiaxed grain microstructure <1µm, and no glassy phase detected. Also, it was found that the use of smaller quantities of dopant such as 0.5% wt. can produce high density ceramics at 1550°C.

### (ICCPs-P020-2013) Effect of Zr<sup>4+</sup> Ion Substitution on the Crystal Structure, Microstructure, Mechanical and Electrical Properties of Porous Cordierite Ceramics

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Zirconium doped cordierite ceramics 2MgO.2Al<sub>2</sub>O<sub>3</sub>.Si(1-X)ZrXO<sub>2</sub> have been synthesized via co-precipitation method using MgCl<sub>2</sub>.6H<sub>2</sub>O, NaAlO<sub>2</sub>, Na<sub>2</sub>SiO<sub>3</sub>.5H<sub>2</sub>O and ZrOCl<sub>2</sub>.8H<sub>2</sub>O as starting materials. XRD, FT-IR and SEM techniques were employed to study the effect of zirconium doping on the crystalline structure and microstructure of each sample. XRD results revealed that α-cordierite phase is predominated with high Zr<sup>4+</sup> contents (x=0.10 and 0.15), whereas ZrO<sub>2</sub> and ZrSiO<sub>4</sub> were predominated as impurity secondary phases with low Zr<sup>4+</sup> content (x=0.05). The density measurements of sintered cordierite samples showed that the densification behavior improved from 30.4 % to maximum value of 44.6 % of the theoretical density (2.6 g/cm<sup>3</sup>) at 10 wt% of Zr. However, microhardness of sintered samples enhanced from 7.1 up to 7.5 GPa with raising Zr<sup>4+</sup> dose (x=0 to 0.15). On the other hand, the gradual substitution of Zr for Si led to the suppression the electrical resistivity (ρ) from 16.6 to 2.8 x 10<sup>9</sup> Ω.cm from x= 0.0 to 0.15, respectively. In addition, dielectric properties investigation indicated that the dielectric constant (ε) of pure cordierite is decreased with Zr<sup>4+</sup> substitution. The maximum dielectric constant (ε) of Zr-doped cordierite was equal 17.7 at 1.5 MHz and 98.6 at 1.5 GHz with x= 0.15.

### (ICCPs-P022-2013) Evolved Gas Analysis during the Sintering of Ultra-high Temperature Ceramics Based on Zirconium Diboride

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A combined dilatometer-mass spectrometer system (CDMS) has been used to characterize the gases evolved during the heating of zirconium diboride-based compositions for ultra-high temperature applications. The pure materials zirconium-diboride, boron carbide, silicon carbide, and mixtures thereof, including added carbon from a phenolic resin, were examined. Throughout the heating ramp and into the hold cycle, a number of species appear in the gas phase. The assignment of these species to chemical compounds was made using cracking patterns, natural isotopic abundances, and the decomposition of model compounds. The main species observed in the gas phase over a wide range of temperatures is carbon dioxide. The decomposition of phenolic resin leads to gaseous carbon-containing species being evolved later in the heating cycle. The sources of evolved carbon dioxide and other gaseous species are discussed in terms of impurities, surface oxide layers, and reduction chemistry.

### (ICCPs-P023-2013) Magnetic Softening of α''-Fe<sub>16</sub>N<sub>2</sub> Applying Low Temperature Nitridation Method

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Ferromagnetic α''-Fe<sub>16</sub>N<sub>2</sub> with larger magnetization than α-Fe needs to be managed its magnetic coercivity to realize its application. In this study, preparation of Co doped α''-Fe<sub>16</sub>N<sub>2</sub> was investigated through low temperature nitridation method starting from fine powders of (Fe,Co)<sub>3</sub>O<sub>4</sub> obtained in BA solution. Reduction of magnetic coercivity to 30 mT was observed in the nitrated products containing 3 at% cobalt than those values observed on the product without Co-doping keeping the larger magnetization of 217 emu·g<sup>-1</sup>. The decrease in magnetic coercivity might be achieved by reduction of magnetostric-

tion effect resulting from Co substitution of Fe in "α''-(Fe,Co)<sub>16</sub>N<sub>2</sub>" lattice.

### (ICCPs-P025-2013) Ceramization of Silicone-based Composites

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Ceramizable silicone rubber based composites are commonly used in cable industry as a covering material for copper wire. In the presence of flames and elevated temperature they are able to create ceramic shield maintaining the integrity of electric circuit up to 180 min. When temperature is increasing polymer matrix is degrading and mineral particles dispersed in silicone rubber matrix stick together creating stiff durable and insulating, porous ceramic skin. In the paper the influence of a type of ceramic additives on ceramization process was presented. Ceramization of the composites was discussed from the point of view of their mechanical properties and microstructure of ceramic phase after heat treatment, determined by porosimetry and SEM. The influence of various mineral fillers: titania (anatase), mica (phlogopite), calcia, calcium carbonate, aluminium hydroxide, kaolin on vulcanization, mechanical properties and ceramization of silicone rubber-based composites was studied. Kinetics of vulcanization, mechanical strength (TS) and elongation at break (Eb) of the materials were determined and discussed in terms of their flammability (OI), smoke intensity, morphology and strength after ceramization.

### (ICCPs-P026-2013) Auto-Granulation Behavior of Fine Cohesive Powders

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The handling and processing of fine powders are often accompanied by difficulties due to the cohesive nature of the individual particles. Fine powders with a particle size less than several microns are generally cohesive and tend to form aggregates on mixing and agitation due to the van der Waals forces. This can lead to the formation of large granules within the powder bed, changing the behavior of the bulk powder and potentially causing problems in later processing. In this study, fine ceramic powders are vibrated under controlled conditions in order to induce auto-granulation within the powder bed. The size of the granules formed in this manner is affected by the balance between adhesive energy of the particles and the disruptive energy of vibration. Imaging of the vibrating powder bed during the auto-granulation provides insight into the mechanisms of granule growth. The bulk cohesion as characterized by shear cell testing is used to describe auto-granulation tendency for a number of powders. The connection between the auto-granulation behavior of the powder and the rheological behavior of the paste processed from these powders is also investigated.

### (ICCPs-P027-2013) Colloidal Processing and Complex Shaping of Ultra High Temperature Ceramics

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The colloidal processing technique enables the formation of uniform and dense green bodies. These green bodies are then able to be sintered to high density without the aid of pressure. High volume fraction/low viscosity suspensions are produced in non-aqueous solvent with the use of an appropriate dispersant. Slip casting, freeze casting or gelcasting can be used to produce shaped green bodies with green density near the maximum packing limit. The dense green body is sintered in inert atmosphere to produce nearly fully dense Ultra High Temperature Ceramic components without the need for application of pressure. Shaped objects such as simulated leading edges are shown to withstand temperature in excess of 3000 C for up to 3 minutes. Surface oxidation is observed, but the components do not melt or fracture.

**(ICCPSP029-2013) Crystal Growth of SrTiO<sub>3</sub> by Flame-Fusion Method and Preparation of its Raw Material**

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SrTiO<sub>3</sub> single crystal has been grown by Flame-Fusion Method (FFM) and used widely for epitaxial film growth. The raw material for FFM must be pure, fine and flowable powder. Since there is no suitable commercial material for FFM now, we tried to prepare the raw material by solid state reaction. It is important in FFM to control the dropping rate of raw material, in other words to stabilize the change of dropping amount with the passage of time. It is known that a compressibility index considerably affects the dropping rate of raw material in FFM. The compressibility index is calculated by loose and tapped density of powder, and controlled by the granulation of powder. Thus the optimal granulation process was determined by some parameters such as mixing process, dispersant concentration, amount of water, and firing condition. SrTiO<sub>3</sub> single crystal grown by FFM using prepared material had excellent properties. The purity was more than 99.99% by ICP analysis, and the crystalline quality evaluated by X-ray rocking curve was equal to or higher than that of conventional SrTiO<sub>3</sub> powder. Substrates made of the obtained SrTiO<sub>3</sub> crystal had molecular layer steps and terraces structure on the surface.

**(ICCPSP030-2013) Hydrothermal Synthesis of Pt-loaded Allophane Nanoparticles**

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Allophane is a short-range order clay mineral which occurs in some soils derived from volcanic ejecta. The chemical composition is expressed as (1-2)SiO<sub>2</sub>·Al<sub>2</sub>O<sub>3</sub>·(5-6)H<sub>2</sub>O. The primary particles of the allophane are hollow spherules with an outer diameter of 3.5-5.0 nm and perforations with a diameter of ~0.3 nm; therefore, the specific surface area of allophane is as high as ~900 m<sup>2</sup>/g. The allophane with such a peculiar nanostructure is promising as a novel catalytic support. In the present study, Pt-loaded allophane nanoparticles with [Si]/[Al]=0.55, 0.75, 1.0 were successfully prepared through a hydrothermal process combined with selective deposition of platinum. The precursors formed by mixing the aqueous solutions of Na<sub>4</sub>SiO<sub>4</sub> and AlCl<sub>3</sub>·6H<sub>2</sub>O are hydrothermally treated at 100 °C for 48 h in an aqueous solution of H<sub>2</sub>[PtCl<sub>6</sub>](H<sub>2</sub>O)<sub>6</sub>. The aqueous dispersion of MPA (3-mercaptopropionic acid)-capped Pt nanoparticles was also used as the Pt source for comparison. XRD patterns of the powders which were autoclaved, followed by heat-treatment at 250 °C in air showed the reflections from platinum in addition to the allophane. Addition of platinum component to the allophane resulted in new peaks at 1300-1500 cm<sup>-1</sup> in the IR spectra. XPS results showed 75% of platinum atoms were present as Pt metal and 25% of those were PtO<sub>x</sub>. TEM observation showed Pt nanoparticles were well-dispersed on the allophane.

**(ICCPSP031-2013) The Microstructure and Mechanical Properties of Fast Firing Glazes within the System CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-TiO<sub>2</sub>**

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This paper presents the results of researches on frits and glass-ceramic glazes for floor tiles based on compositions located in the primary crystallization field of diopside within the system CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-TiO<sub>2</sub>. The results of investigations on the frits crystallization abilities, stability of the crystallizing phase under conditions of single-stage and fast firing cycle (time below 60 minutes) depending on their chemical composition. The influence of the chemical composition on crystalline phases developed in the glass-ceramic glazes, mechanical parameters and microstructure has been examined. The strength tests proved increased mechanical resistance

of crystalline glazes. The obtained glazes are characterized by high microhardness in range 6~7 GPa, as well as the increased wear resistance measured by the loss of weight below 100 mg / 55cm<sup>2</sup> (PN-EN ISO 10545-7). This is a significant increase of these parameters comparing to non-crystalline glazes, where microhardness values are in range between 5~6 GPa and the wear resistance values are in range from 120 to 200 mg. Starting glasses (frits) and glazes belonging to the system CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-TiO<sub>2</sub> were examined by DTA, XRD and SEM techniques.

**(ICCPSP032-2013) Direct Ink Writing of Bio-Inspired, Highly Toughened Hybrid Materials**

C. Zhu\*, W. Floyd, E. Duoss, J. Kuntz, C. Spadaccini, Lawrence Livermore National Lab, USA

Here, we report on our recent efforts to fabricate innovative laminated composites with a distinctive micro-architecture inspired by the mantis shrimp. We employ direct ink writing to create ceramic helicoidal lattice structures that are infiltrated with polymer. We demonstrate that this design concept can be applied to conventional ceramic and polymeric materials, such as alumina and poly(methyl methacrylate) (PMMA) to create composites that display exceptional strength and toughness. Selected materials properties were evaluated through appropriate ASTM tests and laminated composites theory. Structure post-damage behavior was also investigated. Significant improvement in the mechanical performance of the bio-inspired structure was observed over the baseline structure. Moreover, this additive manufacturing approach is flexible and can be readily extended to other material combinations and bio-inspired designs. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. IM release number: LLNL-ABS-618012

**(ICCPSP033-2013) The Connectedness of Porous Media: Pore Space Topology**

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Porous media are three-dimensional (3D) structures which often consist of a complex network of pores connected by throats. Traditionally the pore space is characterized by its pore and throat size distributions and sometimes its mean connectivity number and aspect ratio. These values, however, rarely describe the connectedness of the 3D pore space, i.e. its topology. X-ray tomography is a non-destructive imaging technique which allows to capture 3D data related to the pore-scale of heterogeneous materials. The topology of 3D pore space data, e.g. acquired by X-ray tomography, can be described by Minkowski functionals, which quantify volume, surface area, integrated mean curvature and integrated Gauss curvature. These properties can then be related to physical properties of the pore space. We have studied drainage and imbibition processes in sintered glass bead columns with synchrotron X-ray micro tomography. The pore space of the glass bead columns was altered by varying the sizes of the glass beads as well as by adding, or replacing the glass beads, with fractured glass beads. These variations yielded glass bead columns with varying porosity, pore and throat size distributions and pore space curvatures. We describe and quantify the topology of the pore space with the Minkowski functionals and relate these variations in the pore space to the levels of trapped non-wetting phase after a drainage and imbibition procedure.

**(ICCPSP034-2013) Glycothermal Synthesis of Alkali Aluminates**

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Electrical energy storage devices require ion conducting membranes for stable operation, and ceramics such as beta alumina have been used for several systems of sodium ion batteries, as well as sensor devices. To date, there has not been a direct synthesis method for beta alumina particles. Glycothermal reactions show the formation of oxide materials under more benign conditions than hydrothermal

methods, and allow for control over particle shape and size. The formation of alpha alumina is well established under glycothermal conditions, and in this work, the development of phases of alkali aluminates (Li, Na, K) are studied and examined for the formation of novel phases and discrete particles. Morphology and phase development are characterized by SEM, XRD, and HRTEM. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

### (ICCPs-P035-2013) Synthesis of Various Morphological Powders for Strontium Titanate by Spray Pyrolysis

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Recently strontium titanate (SrTiO<sub>3</sub>) has been attracted attention for red phosphor materials because of their superior properties of thermal stability and light emission intensity, respectively. These properties are mainly determined by morphologies. Spray pyrolysis (SP) is one of the common candidates to synthesize various morphologies (e.g. hollow, porous and solid). Although hollow and porous SrTiO<sub>3</sub> particles have been reported, no solid particle has been synthesized due to rapid SP calcination rate. This study prepared various morphological particles using three precursors of (i) strontium nitrate (SrN), titanium isopropoxide (TTIP) and nitric acid. (ii) SrN, TTIP and citric acid. (iii) strontium acetate, TTIP and acetic acid. Crystallographic structures, surface morphologies, geometries and surface area values of these particles were characterized using X-ray diffraction, scanning electron microscopy, transmission electron microscopy and the nitrogen absorption method, respectively. Finally, SP formation mechanisms of hollow, porous and solid particles have been proposed in this study.

### (ICCPs-P036-2013) Morphology Control of Two-dimensional SnS Nanosheets Using SnO and S as Precursor

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Various two-dimensional tin sulfide nanosheets have been successfully synthesized by thermal decomposition method in high boiling point organic solvents. The precursors prepared by dissolving SnO and S powder in oleic acid and oleyamine respectively and synthesized by controlling different reactive temperature and time. The phase, microstructure and morphology of the products were characterized by X-ray diffractometer, scanning electron microscopy and transmission electron microscopy. The results reveal that reaction temperature influences the growth rate of SnS crystal and change the shape and size of the SnS nanosheet. The reaction time change the crystalline phases of tin sulfides. At 10 minutes, the product is amorphous and some triangle SnS<sub>2</sub> crystals. When the time increases to 60 minutes, the SnS phase gradual crystallized and aggregated to 3D rosa-like SnS microflowers.

### (ICCPs-P037-2013) Shaping of Mullite Green Foams by Protein Thermogelling Process

M. Sandoval, M. A. Camerucci\*, INTEMA, Argentina

The direct thermal consolidation by gelling of aqueous ceramic suspensions foamed with globular proteins is an innovative colloidal processing, non-contaminant, used to fabricate porous ceramics. Stable aqueous mullite-albumin (5, 10, 15vol.%) suspensions were foamed by mechanical stirring at 2300 rpm, 10min at room temperature (conventional route, Rc). In order to increase the suspension viscosity, decrease the foam destabilization before gelling process and minimize the particle segregation, the adding of methylcellulose (2wt.%) as an additional binder on the foaming of the suspensions was evaluated (MC route, RcMC). Rheological behavior of suspensions, foamed and without foamed, as a function of temperature was also analyzed. Mullite green disks were prepared by pouring the

foamed suspensions into warm metallic molds, heating at 80°C, 2h and drying at 50°C, 24h. Green foams were characterized by relative density and porosity measurements, and microstructural analysis by SEM on the fracture surface. Cell size distributions and cell morphology were analyzed in relation to wet foam properties. Disks prepared by Rc achieved the highest porosities (72%) and lowest relative densities (0.28). The porous homogeneity and size (85-130µm) depended on the protein content. Disks prepared by RcMC showed microstructures more homogeneous independently of the albumin amount with smaller cells (30-45µm) and denser struts.

### (ICCPs-P038-2013) Sintering and Characterization of Starch Consolidated Porous Mullite Bodies

M. H. Talou, M. A. Camerucci\*, Research Institute for Materials Science and Technology (INTEMA), CONICET/UNMdP, Argentina

Among direct consolidation methods involving gelation, the starch consolidation casting (SCC) is a non-contaminating low-cost technique in which the starch acts as a consolidator/binder of the ceramic suspension and as a pore former at high temperature. In this work, the processing and microstructures of porous mullite bodies prepared by modifying SCC were studied. The alternative processing route called Mixing Route (MR) was proposed to prevent particles segregation, and thus, to develop porous bodies with homogeneous microstructures. Stable aqueous mullite/cassava starch suspensions (0.25 starch volume fraction of 40 vol% total solids) were prepared by mixing and homogenization. The total starch content was a mixture of ungelatinized/gelatinized native starch in a 9:1 ratio. Mullite porous bodies were obtained by heating (80°C) the aqueous mullite/starch suspensions into metallic molds, burning out treatment (650°C) and sintering at 1400, 1500 and 1650°C. The phase evolution and microstructures were studied by XRD analysis, Hg-porosimetry and SEM. The porosity values (total, open and close) were obtained by water immersion method. The close porosity increased and the total porosity decreased by increasing sintering temperature. At 1650°C, homogeneous porous microstructures (42%) of mullite were obtained. The cavity (10-20µm) and throat sizes (3.3-5.4µm) did not change with the sintering temperature.

### (ICCPs-P039-2013) Synthesis and Evaluation of Ferrite-Silica Aerogel Nanocomposite

N. Katagiri\*, N. Adachi, T. Ota, Nagoya Institute of Technology, Japan

Magnetic properties of ferrite-silica aerogel nanocomposite were investigated from the point of view of new porous magnetic materials. The ferrite-silica porous materials are expected for the application of electromagnetic absorber and magnetic sensor etc. The ferrite-silica aerogel nanocomposite was prepared by sol-gel process and supercritical drying process. The ferrite nanoparticles were prepared by the coprecipitation method and were added to tetramethyl orthosilicate (TMOS) in the range of 0 to 20mol%. The ferrite-silica wet gel was prepared from TMOS containing ferrite nanoparticles, ethanol, deionized water and NH<sub>3</sub> aq. as catalyst, and aged for 10h at 50°C in the ethanol. Then, a hydrophobing treatment was performed for 24h at 110°C in a hexamethyldisilazane/toluene solution. Finally, the ferrite-silica wet gel was dried in the supercritical carbon dioxide fluid. The synthesized ferrite-silica aerogel nanocomposite was evaluated by bulk density, porosity, specific surface area, pore size distribution, SEM and TEM observation, EDS analysis, magnetic permeability and magnetization measurement by VSM. For the preparation of ferrite-silica aerogel nanocomposite, the supercritical drying process was effective method and was expected to have great advantages for low temperature synthesis technique.

### (ICCPs-P040-2013) Crystal Orientation and Sintering of Cordierite Ceramics prepared from Natural Raw Materials

M. Katayama\*, J. Nakakuki, Y. Kobayashi, Aichi institute of technology, Japan

Many researchers reported that platy structure of kaolinite mainly contribute to the crystal orientation of cordierite. Talc has the similar platy shape and crystal structure with kaolinite for raw materials with



it. We supposed that the platy shape of talc powder has to contribute to the crystal orientation of cordierite. However, it has never been referred the effect of shape anisotropy of talc on crystal orientation of cordierite. In this study, cordierite ceramics were prepared from several kind of kaolin and talc powders having various particle size distribution with nanometer-sized alumina sol. Powder mixtures were uniaxially pressed, followed by cold isostatic pressing to make a thin compact. Crystal orientation of talc or cordierite were evaluated with X-ray diffraction. The *c* axis of talc powder larger than 3 $\mu\text{m}$  aligned vertical to the pressed plane of green compact because of its large anisotropic platy shape. The crystal orientation of cordierite on the pressed plane increased with an increase in mean particle size of talc powders. High correlation was observed between orientation of talc powders and cordierite crystals. Dense cordierite ceramics were prepared using fine kaolin and talc powders below 5 $\mu\text{m}$ . At an optimum combination of kaolin and talc powders, the relative density higher than 95% was attained at a high crystal orientation of 85% [ $I_{110} / (I_{110} + I_{002})$ ].

**(ICCPs-P041-2013) Differences in Pyrocarbon Matrices made by FB-CVI with Organic Precursors**

I. Regiani\*, J. S. dos Santos, Institute of Technology in Aeronautics, Brazil

Carbon/carbon composites are useful in thermal and mechanical equipment. This composite is usually made by pyrolysis of polymers or CVD technique, both techniques are time consuming. This work shows some results of the use of film boiling chemical vapor infiltration, FB-CVI, a fast densification method to make carbon/carbon composite with different precursors. Carbon matrices were made using three different carbon precursors, hexane, ethanol and vegetable oil. All densification experiments were done in the same temperature, but ended in different densification stages to study the densification front behavior. Hexane and ethanol showed the same the deposition rate, but vegetable oil showed the highest, Archimedes densities were different for all, and vegetable oil is the most porous samples. Samples were characterized by DRX, MEV, RAMAN and polarized optical microscopy. The pyrocarbon type deposited were also different for the three precursors, vegetable oil deposited an isotropic carbon while ethanol and hexane deposited laminar ones. The conclusion is that different types of precursors results in different pyrocarbon and different structures of matrix.

**(ICCPs-P042-2013) Preparation of Composite Fe-Ni Thin Sheets with Nano-sized Lithium-Copper Ferrites by Using Self-propagating High Temperature Synthesis and Electroforming**

Y. Choi\*, Dankook University, Republic of Korea; M. Kim, J. Lee, Korea Institute of Materials Science, Republic of Korea

Composite nickel sheets with nano-sized lithium copper ferrite particles for electromagnetic shielding materials were prepared by electroforming in the modified nickel-ion sulfate bath with lithium copper ferrite particles. The lithium copper ferrites were synthesized by self-propagating high temperature synthesis and mechanical milling. Nano-sized ferrites were classified by ultrasonic-floating sedimentation method. Neutron diffractometry revealed that the final ferrites were  $\text{Li}_{0.39}\text{Cu}_{0.61}\text{Fe}_2\text{O}_4$  which crystal structure and lattice parameter were Fd3m and 0.8338 nm, respectively. Microstructure observation and chemical analysis by transmission electron microscopy and energy dispersive spectroscopy showed that nano-sized ferrite particles with less than about 40 nm exist in the thin composite iron-nickel sheet. Maximum magnetization (Ms), residual magnetization (Mr) and coercive force (iHc) were 7.72 Wb/m<sup>2</sup>/kg, 0.86 m<sup>3</sup>/kg, and 1288 A/m, respectively. The complex permeability decreases with the increase of frequency, and real value of it ( $\mu'$ ) has the peak value at about 0.8 GHz.

**(ICCPs-P043-2013) Morphology and Properties of TiN Thin Films Deposited onto a  $\beta$ -type Ti Alloy Substrate by Reactive Magnetron Sputtering**

J. Lee, Y. Choi\*, S. Hong, Chungnam National University, Republic of Korea

The morphology and properties of Titanium Nitride (TiN) thin film deposited on  $\beta$ -type Ti-15Mo-3Nb-3Al-0.2Si alloy plates by RF magnetron sputtering method were studied. The preferred orientation of TiN thin films changed from (111) to (200) as the nitrogen flow rate increased due to the effect of the kinetic energy of the bombarding particles. The coating thickness was found to decrease with increasing nitrogen concentration, which also favors (200) orientation with increasing nitrogen flow rate. With increase of nitrogen flow, the morphology of the TiN thin films changed from characteristic pyramidal shaped grains to columnar-shaped grains. The roughness analysis of the coating shows that the average roughness of the coating decreased with increasing nitrogen flow rate. The increase of hardness with increasing nitrogen flow rate is attributed to the decrease in grain size.

**(ICCPs-P044-2013) Particle Size Control of Murdochite-type  $\text{Ni}_6\text{MnO}_8$**

H. Taguchi\*, Okayama University, Japan; S. Tahara, Okayama University, Japan; K. Hirota, Doshisha University, Japan

Murdochite-type  $\text{Ni}_6\text{MnO}_8$  has high-catalytic activity because both  $\text{Ni}^{2+}$  and  $\text{Mn}^{4+}$  ions coexist in this compound. In the present study, we investigated the conditions that enable the particle size of  $\text{Ni}_6\text{MnO}_8$  to be controlled to improve their catalytic performance in hydrocarbon oxidation. Powders of nickel acetate tetrahydrate and manganese acetate tetrahydrate were weighed in a stoichiometric ratio and dissolved in distilled water. Oxalic acid dehydrate (OA) was added to induce precipitation, which resulted in a  $\text{Ni}_6\text{MnO}_8/\text{OA}$  molar ratio of 1/1.5, 1/2, 1/2.5, or 1/3. The precipitates were calcined at 350 °C, 400 °C, or 500 °C for 6 h in a flow of argon. XRD measurements indicated that the samples calcined in a flow of argon consisted of Ni and  $(\text{Ni}_{1-x}\text{Mn}_x)\text{O}$ . DTA-TG measurements of the calcined samples indicated that an exothermic reaction occurred at temperatures greater than 280 °C, and that the weight of the samples increased by 20.6% in the temperature range from 280 to 560 °C. From the XRD results, the calcined samples were transformed into murdochite-type structures by heating at 600 °C for 6 h in air. Both crystallite size ( $D_{440}$ ) and the average particle size ( $P_n$ ) increased with the calcined temperature.

**(ICCPs-P046-2013) Clay Extrusion Process Modelling and Performance Assessment of Vacuum Extruders Using Computational Fluid Dynamics**

S. Kandasamy\*, A. Asthana, A. Young, Sheffield Hallam University, United Kingdom

Clay extruders have been widely used in the ceramic industry for manufacturing brick, tiles and pipes. The design of extruder components, key for the performance of the system and quality of products, needs further research and development. The use of numerical modelling and simulation for developing the design of extruder components was very limited due to the complex rheology and flow physics of clay and geometry of the components involved. This paper presents CFD modelling of vacuum type de-airing extruders used in the process of stiff extrusion for making bricks. A 3-dimensional unsteady state, laminar flow CFD model was developed to predict the extrusion pressure and flow velocity of clay at different sections of auger/barrel chamber and die. Power consumption was also predicted for specified extrusion rates. The dependence of these performance characteristics on various designs of extruder components, moisture content of raw material and process parameters was determined. The simulation results were in good agreement with the theoretical design suggestions used by the manufacturers of extruders. The results were further validated by experimental runs on a scaled extruder. The similarities observed in the simulation and experimen-

tal suggestions, implies that the CFD modelling could be a useful tool for developing energy efficient extruder systems in future.

### **(ICCPs-P047-2013) Cellular Alumina Ceramics prepared via Biological Foaming with Yeast**

E. Gregorova\*, T. Uhlírova, W. Pabst, Institute of Chemical Technology, Prague (ICT Prague), Czech Republic

Porous and cellular ceramics have a wide range of applications, based either on the mere presence of pores (possibly closed) or on the possibility of fluid flow through open pores, i.e. the presence of an interconnected pore network. There is a strong demand in the industry to supplement the commonly used replication methods, which result in weak ceramic foams with hollow struts, by alternative techniques in which the problem of hollow struts is circumvented. Biological foaming with yeast is a promising candidate for achieving this goal. In this work, cellular alumina ceramics with a porosity higher than 70 % is prepared from 70 wt.% alumina slurries containing sugar (glucose, fructose, sucrose), starch (potato, corn, rice) and yeast. After casting into nonporous molds, gas evolution starts in situ. Subsequent heating stabilizes the foam. This work investigates how the kinetics of the process and the microstructure of the final product (porosity and pore size) can be controlled by changing the types and amounts of sugar, starch and yeast. Other process control parameters, such as temperature, are considered as well. The resulting alumina ceramics have porosities of 70–84 %, pore cavities (cells) with mean sizes in the range 500–1500 microns and do not exhibit hollow struts. Depending on the system, porosity and pore size gradients can be introduced or avoided, and pores at the wall can be open or closed.

### **(ICCPs-P048-2013) Direct-Write Assembly of Copper Oxide and Copper Architectures**

D. B. Kolesky\*, Harvard University, USA; C. Zhu, Lawrence Livermore National Laboratory, USA; J. J. Adams, University of Illinois at Urbana-Champaign, USA; K. Sullivan, E. B. Duoss, Lawrence Livermore National Laboratory, USA; H. Yang, J. Bernhard, University of Illinois at Urbana-Champaign, USA; C. Spadaccini, Lawrence Livermore National Laboratory, USA; J. A. Lewis, Harvard University, USA

One-, two-, and three-dimensional copper oxide (CuO) and copper (Cu) architectures with microscale feature sizes have been fabricated by direct writing of a concentrated CuO nanoparticle ink. These structures may find potential application in the assembly of micro-energetics, optical metamaterials, printed electronics, photovoltaics, and 3D microbatteries. For example, we are patterning micro-energetic materials in which the printed CuO (oxidizer) is infilled with aluminum (fuel) by electrophoretic, chemical vapor, or electroless deposition. These designer architectures will allow us to establish relationships between microstructure and key performance properties, including propagation velocity and energy density. In another example, we reduce the printed CuO structures by heat treatment in forming gas or photonic annealing to create patterned copper electrodes with an electrical resistivity as low as  $5 \times 10^{-5}$  ohm-cm. By printing split ring Cu resonators on dielectric substrates, we have created frequency selective surfaces (FSS) in the X-band (8-12 GHz frequencies). Our research represents an important step towards fabricating multimaterial composites with intelligent design and predictable performance, which may be readily extended to other multiphase functional materials.

### **(ICCPs-P049-2013) Modification of Major Surface Parameters of the Ceramic Glaze by the Selective Selection of Grain Size of Raw Materials**

J. Partyka\*, AGH University of Science and Technology, Poland; M. Gajek, AGH University of Science and Technology, Poland; J. Lis, AGH University of Science and Technology, Poland

Majority of esthetic and functional parameters of surface of ceramic wares strongly depend on quality of the ceramic glazes. Good quality of glaze surface means high glossy (mattness), high whiteness (proper colour) and smoothness, also absence of surface defects as:

pinholes, holes, crazing, devitrification, surface crystallization, roughness etc. Commonly, improvement of glaze parameters is achieved through change of glaze molar composition or correction of firing condition. The presented results of research demonstrate that there is a possibility for significant improvement of glaze surface parameters through the selective milling and selection of the grain size of group of raw materials. Proper milling and selection of grain size of quartz, feldspar, zircon silicate and the rest raw materials lead to change of meltability, viscosity and surface tension in high temperature and finally improvement of the surface parameters.

### **(ICCPs-P050-2013) Theoretical and Experimental Investigation of the Role of the Electric Current in the Spark Plasma Processing of a Copper Powder**

E. Alexandrova\*, I. Alexandra, E. Grigoryev, E. Olevsky, Moscow Engineering Physics Institute, Russian Federation

The purpose of the present work is the experimental and theoretical analyses of the role of the electric current in the SPS processing of a copper powder. A theoretical model is put forward based on the hypothesis of the deformation softening of the particle interfaces due to the intense localized Joule heating of the inter-particle contact areas. The comparative experiments are conducted using the SPS system Labox-625 (Sinter Land, Japan) in two modes: (1) a conductive (current-assisted) mode, when the electrical current is directly passing through the powder compact; and (2) an isolative mode, when the electrical current does not pass through the powder specimen and is transmitted through the graphite tooling only (this mode is achieved by using insulating inserts). A multi-scale model is applied to the simulation of the both above-mentioned spark plasma processing modes with current-assisted and current-isolative experimental conditions. The model predictions are compared with the observed experimental outcomes. Based on the developed model, the shrinkage kinetics is compared for the considered current-assisted and current-isolative conditions to verify the validity of the inter-particle area softening hypothesis.

### **(ICCPs-P051-2013) Synthesis of Nanostructured Zinc Oxide by means of FAST/ SPS**

B. Bohne, J. Gonzalez\*, O. Guillon, Friedrich Schiller University Jena, Germany

The retention of nanocrystallinity in dense ceramic materials is still a challenge, even with the application of stress-assisted methods like Field Assisted Sintering Technique / Spark Plasma Sintering. Interestingly, the combined effect of high heating rates and the presence of bound water seems to significantly promote densification of nano zinc oxide. In contrast, the sintering behaviour of coarser powders is not affected by these conditions. Hence, dense nano-grained ZnO could be synthesized at a temperature of only 400 °C. Electrical and mechanical properties at a given density are affected by the processing parameters and microstructure. HR TEM investigations complete this study and comparison with MgO reveal that only a small temperature frame exists in which densification mechanism is changed.

### **(ICCPs-P052-2013) Effect of Reduction of Thickness on Microstructure and Technological Properties of Porcelain Tiles**

A. L. Silva, UFSC, Brazil; A. M. Bernardin, UNESC, Brazil; M. Dal Bó, D. Hotza\*, UFSC, Brazil

Porcelain tiles with reduced thickness have a smaller mass to be fired, reducing therefore energy consumption during firing and saving raw materials. However, the relationship between ceramic tiles with reduced thickness and their microstructure and technical properties is unclear. The present work deals with the study of the microstructure and properties of porcelain tiles due to the reduction of their thickness. Five thicknesses of porcelain tiles, 1.5, 2.5, 3.5, 4.5 and 5.5 mm were used in this study. A compacting pressure of 39.2 MPa was used and the maximum firing temperatures were 1180, 1200 and 1220°C. The results showed that the thickness and the maximum firing temperature of the porcelain tiles have significant influence on the mi-

costructure and physical properties like firing shrinkage, loss on ignition, water absorption, bulk density after firing and mechanical strength.

**(ICCP5-P053-2013) The Search for a Scalable Nano- $\alpha$ - $\text{Al}_2\text{O}_3$  Production Process**

N. J. Taylor\*, A. J. Pottebaum, R. M. Laine, University of Michigan, USA

In the field of ultrafine metal oxide powder synthesis, a common ambition has been the production of nano- $\alpha$ - $\text{Al}_2\text{O}_3$ . A number of processes have produced nano- $\alpha$ - $\text{Al}_2\text{O}_3$ , but for a number of reasons, no scalable process for the production of true unaggregated < 100 nm  $\alpha$ - $\text{Al}_2\text{O}_3$  has been found. Previously, we reported nano- $\alpha$ - $\text{Al}_2\text{O}_3$  production through solid-feed flame spray pyrolysis (SF-FSP). In SF-FSP, a dispersion of metal oxide nanopowders in an alcohol solvent is aerosolized in  $\text{O}_2$ , combusted, and the resulting product stream rapidly quenched. Starting with transition- $\text{Al}_2\text{O}_3$  (t- $\text{Al}_2\text{O}_3$ ) nanoparticles, the rapid heat treatment transforms some particles to unaggregated single crystal  $\alpha$ - $\text{Al}_2\text{O}_3$  nanoparticles. In scaling SF-FSP production of nano- $\alpha$ - $\text{Al}_2\text{O}_3$ , we find the formation of micron-sized, polycrystalline  $\alpha$ - $\text{Al}_2\text{O}_3$  particles with < 100 nm domains. Through liquid-feed flame spray pyrolysis (LF-FSP), the rapid combustion and quenching of alcohol solutions of metalloorganic precursors, we also find similar nanocrystalline  $\alpha$ - $\text{Al}_2\text{O}_3$  particles in a single step from a hydrolyzed  $\text{Al}(\text{CH}_3\text{CH}_2\text{O})_3\text{N}$  precursor. These particles are reduced to nanometer size primary particles by low-energy ball milling. Chemically aided milling (CAM) further enhances the milling process, with specific surface areas increasing from 5  $\text{m}^2/\text{g}$  to 22  $\text{m}^2/\text{g}$ . Both flame spray processes coupled with CAM proved to be a viable route to produce larger quantities of nano- $\alpha$ - $\text{Al}_2\text{O}_3$ .

**(ICCP5-P054-2013) All Layers Inkjet-printed Field Effect Transistors using p-CuO Nanoparticles Based Ink-formulation**

M. Vaseem\*, A. Hong, H. Kim, Y. Hahn, Chonbuk National University, Republic of Korea

Copper oxide (CuO) nanoparticles (NPs) having 5-8 nm in diameter were synthesized by a simple solution process. The as-synthesized NPs showed highly crystalline monoclinic phase of CuO with having bandgap of ~1.75 eV. The CuO NPs were further formulated as an ink using mixed solvents of water, ethanol, isopropanol and diethylene glycol for inkjet printing of CuO field effect transistors (FETs). The ink-jetting behavior of the as-formulated ink samples showed that the CuO concentration and digitally-controlled number of over-printing are important factors for optimizing the uniformity and thickness of printed films with smooth edge definition. In this report, we present the first results of inkjet printed CuO (channel) and Cu (source/drain) based field effect transistors (FET) on Si/SiO<sub>2</sub> (gate material) substrate. As-printed devices show p-type conductivity with high carrier mobility, which can be applied further for various applications.

**(ICCP5-P055-2013) Fabrication of U3O8-added UO2 Pellet for Compaction and Sintering Behavior Simulation**

D. Kim, J. Yang, Korea Atomic Energy Research Institute, Republic of Korea; S. Park, Pohang University of Science and Technology, Republic of Korea; J. Oh, J. Kim, Y. Rhee\*, K. Kim, Korea Atomic Energy Research Institute, Republic of Korea

In the conventional fabrication process of UO<sub>2</sub> nuclear fuel pellet, 8-10 wt% U<sub>3</sub>O<sub>8</sub> added UO<sub>2</sub> powder mixture has been typically used. The addition of U<sub>3</sub>O<sub>8</sub> powder could lead to the density drop of UO<sub>2</sub> pellet. On the other hand, from the recycling the scrap of UO<sub>2</sub> pellet, the fabrication process can be possible to make more economically. To make higher efficiency of the fuel pellet fabrication process, it is necessary to predict and characterize the change of UO<sub>2</sub> compaction and sintering behavior due to the U<sub>3</sub>O<sub>8</sub> addition. In this work, U<sub>3</sub>O<sub>8</sub>-added UO<sub>2</sub> pellets were fabricated to obtain the empirical data for the compaction and sintering behavior simulation. The compacting and densification behavior of UO<sub>2</sub>-U<sub>3</sub>O<sub>8</sub> mixture was investigated using the uniaxial single acting press and dilatometer. And

then, based on the empirical data, the compaction and sintering behavior simulation was performed.

**(ICCP5-P056-2013) Direct Inkjet Printing of Aqueous Ceramic Suspensions**

A. Watjen\*, M. Kramer, P. Gingter, R. Telle, RWTH Aachen University, Germany

Direct Inkjet Printing (DIP) enables additive manufacturing of complex three-dimensional geometries from ceramic suspensions with high solids content. Even multilayered ceramics for structural and functional components can be realized. The compatibility between the ink and the print head is of utmost importance. Zirconia (3Y-TZP) and alumina suspensions were processed in an agitator bead mill to reduce the grain size < 1  $\mu\text{m}$  to avoid clogging of print head nozzles. Significant parameters are viscosity, surface tension, solids content and a controlled drying process of each layer. Diverse additives were used to ensure adjustment to the printing system and consequently the formation of uniform droplets. To build up multilayered specimens different print heads for each suspension were used consecutively. Subsequently, the fracture surfaces and the microstructures of the sintered specimens were analyzed using SEM with special attention to the interfaces.

**(ICCP5-P057-2013) Raman Scattering and Optical Properties of Cerium Dioxide and Related Complex Oxides via Solution Process**

M. Amimoto, Nagoya Institute of Technology, Japan; M. Ozawa, K. Kobayashi\*, Nagoya University, Japan; M. Haneda, Nagoya Institute of Technology, Japan

CeO<sub>2</sub> and Mixed metal oxides in the system of ZrO<sub>2</sub>-CeO<sub>2</sub> are useful for various applications such as ceramics, catalyst, and pigments etc. Electronic band structures, which govern the transport and optical properties, depend on the mixed state of cations in the same fluorite-type structure. These are assessed by UV-Vis absorption and fluorescence spectroscopy. Also, resulting from these optical properties, new pigments containing CeO<sub>2</sub> (yellow) have recently been proposed by several researchers. In the present work, we synthesized the nanometer-sized CeO<sub>2</sub> and related solid solution compounds by solution process, and characterized with Raman spectra, UV-Vis diffuse reflectance, and fluorescence spectroscopy. We discussed the relationship of oxygen defects and these properties in CeO<sub>2</sub>-related compounds.

## Tuesday, August 6, 2013

### Plenary Session 3

Room: Pavilion Ballroom

Session Chair: Lennart Bergstrom, Stockholm University

#### 8:10 AM

**(ICCP5-003-2013) Bioinspired Techniques for Mesoscale Hierarchical Assembly of Inorganic Crystals**

H. Imai\*, Keio University, Japan

Biomaterials have structures that are completely different from those of artificial simple crystalline materials. We can observe mesoscale hierarchical architectures consisting of oriented nanocrystals in various biomaterials. The fascinating structural design of the inorganic framework with organic molecules has attracted the interest of many researchers in a broad range of science and technology fields. In this presentation, several strategies based on self-organized growth in artificial solution systems are proposed for bioinspired syntheses of mesoscale hierarchical architectures of inorganic crystals. The superstructures consisting of oriented micrometric and/or nanometric units were fabricated with carbonate, sulfate, phosphate, and oxide crystals in aqueous systems with soluble and insoluble organic species. Since several biomaterialization events take place through a

precursor phase, the transformation of a metastable phase also provides an effective synthesis route to the construction of hierarchical morphologies. We report that specific hierarchical architectures of various metal oxides consisting of oriented nanocrystals were obtained through topotactic transformation of a precursor phase. The superstructures similar to the biological architectures would serve as an excellent base for the further development of functional materials in various application fields.

### **Bioinspired Ceramic and Composite Architectures**

Room: Pavilion Ballroom

Session Chairs: Sanjay Mathur, University of Cologne; Anne Leriche, University of Valenciennes

**9:00 AM**

#### **(ICCP5-056-2013) Bioinspired Composites made through Colloidal Directed Assembly (Invited)**

A. R. Studart\*, ETH Zurich, Switzerland

Biological composites like seashells and teeth are made of soft constituents and stiff reinforcing building blocks assembled into unique hierarchical architectures. Because of their ubiquitous microstructures, natural materials often display unusual combinations of functional properties. In this talk, I will show that colloidal routes typically used in advanced ceramic processing can potentially be exploited to create artificial materials that replicate some of the architectures and design principles of biological composites. To demonstrate this potential, I will present a directed assembly route that enables alignment of reinforcing ceramic particles in a fluid using very low magnetic fields. Particles aligned in specific orientations can be fixed in place by consolidating the suspending fluid through drying or polymerization reactions to result in advanced composites with tailored bioinspired microstructures. Inspired by the structure of mollusk shells and plant seedpods, we show that synthetic composites can exhibit unusual out-of-plane stiffness, wear resistance and shape-changing effects, depending on the consolidated reinforcement architecture. This indicates that the microstructural design of composites guided by the optimized architectures found in nature is a powerful way to create ceramic-reinforced polymers with unprecedented functional properties using a limited selection of building blocks.

**9:30 AM**

#### **(ICCP5-057-2013) Processing of Biomimetic Calcium Phosphate based Ceramic Bone Substitutes (Invited)**

A. L. Leriche\*, J. Hornez, F. Bouchart, E. Meurice, M. Descamps, University of Valenciennes, France; D. Hautcoeur, V. Lardot, BCRC (EMRA), Belgium; M. Gonon, University of Mons, Belgium; F. J. Cambier, BCRC (EMRA), Belgium

Calcium phosphate ceramics like hydroxyapatite (HA) or  $\beta$ -tricalcium phosphate (TCP) are largely used as bone substitutes because of their chemical composition close to bone mineral phase. Today research is focused on the development of biomimetic structures. Results are compared from two bone substitute processes: (1) freeze drying of TCP suspensions; (2) impregnation by both TCP and HA slurries of a polymeric skeleton made of bonded organic PMMA beads. The obtained porous architectures are compared according to cell colonization ability along with their mechanical properties. The freeze-dried porous material presents channel like micron sized pores whereas the material prepared from the impregnation technique is macroporous and characterized by spherical pores, the size of which depend on bead diameter and binding process parameters. An additional microporosity can also be obtained by adding a microporogenic agent allowing, besides the cell colonization, loading of the implant with anti-bacterial and host biological agents. Dense, microporous and macroporous spherical granules with diameters in the range of 300  $\mu\text{m}$  to 3 mm are also prepared by impregnating a porous  $\text{CaCO}_3$  skeleton with HA slurry, heating to remove  $\text{CO}_2$  and dissolution of the residual CaO. The drug and phage releasing kinet-

ics from these various as-prepared porous structures are compared as a function of time and ceramic porosity.

### **Assembly of Ceramic Membranes**

Room: Pavilion Ballroom

Session Chairs: Adam Stevenson, Laboratoire de Synthèse et Fonctionnalisation des Céramiques (LSFC); Jennifer Andrew, University of Florida

**10:15 AM**

#### **(ICCP5-068-2013) Tailored Zeolite Monoliths for Optimized Gas Separation by Freeze-casting**

L. Bergstrom\*, A. Ojuva, F. Akhtar, Stockholm University, Sweden; A. P. Tomsia, Lawrence Berkley national Laboratory, USA

We demonstrate how high-performance adsorbents for gas separation can be shaped by freeze-casting into structures with good volume efficiency, short cycle times and a high working capacity. Structured zeolite 13X monoliths with a laminated structure and hierarchical macro-/microporosity were prepared by freeze-casting colloidally stable aqueous suspensions. Slow directional freezing of the suspensions led to the formation of well-defined and thin lamellar pores and pore walls while fast freezing resulted in more cylindrical pores. The wall thickness, which varied between 8 and 35 micrometers, increased with increasing solids loading of the suspension. Thermal treatment at 1035K of the freeze-cast bodies containing between 9 and 17 wt% of bentonite resulted in mechanically stable monoliths. The monoliths displayed a carbon dioxide uptake capacity of 4-5 mmol/g and an uptake kinetics characterized by a very fast initial uptake where more than 50% of the maximum uptake was reached within 15 seconds. Freeze-cast laminated zeolite monoliths could be used to improve the volumetric efficiency and reduce the cycle time, of importance in e.g., biogas upgrading and  $\text{CO}_2$  separation from flue gas.

**10:30 AM**

#### **(ICCP5-069-2013) Manufacturing of Dense Ceramic Membranes (Invited)**

P. V. Hendriksen\*, S. Foghmoes, A. Kaiser, Technical University of Denmark, Denmark

A dense layer of a mixed conducting ceramic may be used as a gas separation membrane. Mixed proton/electronic conductors may be used for hydrogen separation and mixed oxide ion/electronic conductors for oxygen separation. The advantages of the membrane processes compared to other separation techniques is a very high selectivity and low energy consumption. To maximise performance the membrane should be as thin as possible pointing to designs of a thin (1-20 micron) dense layer on a porous support structure. Overall geometry may be planar or tubular and catalysts are typically needed on both surfaces to enhance the gas-to solid exchange reactions. Preferably the support is a cheap strong material whereas the membrane material has to be chosen maximising the ambipolar conductivity. Hence, gas separation membrane devices are complex multilayer devices. The paper discusses manufacturing routes based on co-firing and the needed tools to ensure a strong component which matches the requirement of maintaining one dense and one highly porous material after firing. Examples are given on routes to achieve a dense membrane layer on different types of support. Both ceria and La-transition metal perovskite membrane materials will be discussed. For the ceria membranes the studied supports are either ceria, Ni/YSZ-cermet or a metal. For the perovskite membranes the studied supports are the membrane material itself and MgO.

11:00 AM

**(ICCP5-070-2013) Highly Porous Reaction Bonded Silicon Nitride Foams; Foam Strength and Reaction Bonding Parameters**

A. Alem\*, M. Pugh, R. Drew, Concordia University, Canada

Recently, many technological applications have been found in which porous ceramics have been utilized due to their unique characteristics. Although silicon nitride ceramics have many remarkable potentials and properties, silicon nitride foams have not received sufficient attention. In this study highly porous silicon nitride foams with open-cell structures have been fabricated via a sacrificial template technique. In this newly designed fabrication procedure, gel-casting and reaction bonding processes were combined in order to make a homogeneous reaction bonded silicon nitride foam without the normal issues of working with Si<sub>3</sub>N<sub>4</sub> materials namely the high sintering temperature, the large linear shrinkage, costly post-sintering machining steps, and poor rheological behaviors. The fabricated foam has a controlled level of porosity which varies up to 87 vol% with a highly interconnected network. The high level of porosity in the foam and its large surface area significantly affect the reaction bonding process. It was observed that due to the large surface area of the foam, vapor-phase reactions resulted in  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> whiskers as the dominant phase but under specific conditions,  $\beta$ -Si<sub>3</sub>N<sub>4</sub> can form in a major quantity. The reaction bonding process was also optimized in terms of the nitriding conditions to get the highest strength while the pore-interconnectivity is completely maintained.

11:15 AM

**(ICCP5-071-2013) Sintering and Microstructure Development in Mixed Ionic-Electronic Conductors for Gas Separation Membranes**

A. J. Stevenson\*, S. Richaud, E. Nonnet, M. Arnold, C. Guizard, Laboratoire de Synthèse et Fonctionnalisation des Céramiques (LSFC), France

Based on dense mixed-conducting ceramics, oxygen transport membranes (OTMs) and hydrogen transport membranes (HTMs) may be efficient alternatives to cryogenic gas separation, particularly in applications where high temperature (> 500°C) gases are required. Mass transport across OTMs and HTMs is controlled by solid state diffusion, so the measured gas flux is generally inversely proportional to the membrane thickness. For this reason, asymmetric membranes, where a thin dense layer is mechanically supported by a porous support, are one of the state of the art geometries under heavy development. Saint-Gobain is involved in several projects, including the CARENA and HETMOC European consortia, aiming to develop and manufacture asymmetric ceramic membranes for gas separation at high temperature for various applications. Based on single phase mixed-conducting ceramic oxides, Ba<sub>0.5</sub>Sr<sub>0.5</sub>Co<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3- $\delta$</sub>  and Ca(Ti,Fe)O<sub>3</sub>, membrane processing will be described with a special focus on sintering. In particular, the roles of powder synthesis method, sintering atmosphere, and constrained sintering between the dense layer and the support on densification and microstructure development will be presented. Strategies to improve membrane performance through improved microstructure control will be highlighted, and problems specific to single phase membrane solutions will be discussed.

11:30 AM

**(ICCP5-072-2013) Multifunctional Nanofibers: New Methods for Synthesizing Composites on a Fiber (Invited)**

J. S. Andrew\*, University of Florida, USA

Multiferroic materials hold enormous potential for a variety of applications, including tunable microelectronics and multiphase memory. The development of novel complex oxide-based composite materials provides an opportunity to fabricate multiferroic materials with performance suitable for real-world applications. In composite multiferroics, the resultant magnetoelectric effect arises from coupling at the interface between a piezoelectric and a magnetostrictive phase. Therefore, it is desirable to assemble a composite such that the inter-

facial contact area between each phase is maximized for increased performance. Here, we present the first example of composite nanostructured building block with a Janus-type morphology for multiferric applications. This composite is composed piezoelectric BaTiO<sub>3</sub> and magnetostrictive CoFe<sub>2</sub>O<sub>4</sub> in an architecture that simultaneously provides access to the bulk and surface properties of both phases. These Janus-type fibers combine the large contact area of a core-shell fiber with the segmented ordering of a thin film, and allow for the control of both composition and surface anisotropy, providing additional degrees of freedom in the design of composite materials. The results from magnetic, dielectric, and magnetoelectric measurements will be presented along with the effects of processing on the size, crystallinity and morphology of these novel composites.

12:00 PM

**(ICCP5-126-2013) Ice-templated Hybrid Materials (Invited)**

U. Wegst\*, Dartmouth College, USA

Freeze-casting, a process that uses the solidification of a liquid carrier such as water for templating, has in recent years been discovered as a route to create highly porous hybrid materials with complex, hierarchical architectures. Freeze-casting is highly attractive for the manufacture of materials for applications that range from scaffolds for tissue engineering to structures for energy generation because it offers several advantages over other techniques. One advantage is that all classes of materials polymers, ceramics, metals and their composites can be shaped with it; another is that materials can be processed with benign, biocompatible liquid carriers; a third is that the resulting hierarchical microstructures can be carefully controlled by both the physical and chemical properties of the components used and the processing parameters such as the cooling rate; finally advantage can be taken of component self-assembly during solidification. The amount, type, size and geometry of the particles and the type of liquid carrier determine the slurry's viscosity and amount of sedimentation as well as the slurry's thermal properties and freezing behavior. In combination with the freezing front velocity and additives, they also determine pore connectivity and morphometry. The thickness and spacing of the cell walls and the size and the number of the material bridges between them can be controlled, as can be the cell wall's bulk and surface properties, and thus the materials interaction with a second phase. This is important for the manufacture of composites by infiltration or for the optimization of the interaction between scaffold and native tissue in biomedical applications. As a result, the freeze-casting process is ideally suited for the custom-designed manufacture of complex, hybrid materials with that emulate in synthetic materials multi-level hierarchical composite structures, which are thought to be the origin of the mechanical property amplification which is frequently observed in biological materials.

**Synthesis and Processing of Biomaterials**

Room: Broadway I - II

Session Chairs: Steven Mullens, Flemish Institute for Technological Research; Kiyoshi Itatani, Sophia University

9:00 AM

**(ICCP5-058-2013) Preparation of Porous Spherical Calcium Phosphate Agglomerates prepared by Spray Pyrolysis and Their Application to Biocement (Invited)**

K. Itatani\*, T. Umeda, Sophia University, Japan; I. J. Davies, Curtin University, Australia; Y. Musha, Toho University, Japan

Porous spherical tetracalcium orthophosphate (Ca<sub>4</sub>(PO<sub>4</sub>)<sub>2</sub>O: TTCP) and tricalcium orthophosphate ( $\alpha$ -Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>: TCP) agglomerates were simultaneously prepared by double-nozzle spray pyrolysis using separate two fluid nozzles. In order to achieve this, calcium phosphate solutions with Ca/P ratios of 2.00 and 1.50 were spray-pyrolyzed at 600C and then heat treated at 1250C for 10 min. The resulting powder was found to contain hydroxyapatite (Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>: HAp), together with TTCP and TCP. Moreover,

irregular shaped agglomerates were formed by undesirable sintering of the original spherical agglomerates during heat treatment. In order to decrease the proportion of HAp and to inhibit sintering, 0.25 mol dm<sup>-3</sup> glutaric acid was added to the starting solution for TTCP with the expectation of forming residual carbon following pyrolysis of the glutaric acid. Heat treatment of this modified spray-pyrolyzed powder at 1250°C for 10 min was found to result in the formation of porous spherical TTCP and TCP agglomerates. When the hardened body (setting agent: citric acid) was immersed in simulated fluid body at 37.0°C, the TTCP and TCP were converted into HAp within 14 days with the formation of porous body (porosity: 67%).

**9:30 AM**

### **(ICCP5-059-2013) Ceramic Processing for Health Care Materials and Green Chemistry Applications (Invited)**

S. Mullens\*, J. Van Noyen, J. Lefevre, V. Ozhukil Kollath, M. Gysen, Flemish Institute for Technological Research, Belgium

This presentation will provide an overview of the variety in application domains for ceramic materials and ceramic processing science, by focusing on two examples. First, material science and catalysis play an important role in the chemical industry. A growing need for flexible and versatile processes is increasing the pressure on processes, reactors and catalysts, in terms of efficiency and flexibility. Plenty of applications are emerging for porous functional components, with engineered pores sizes ranging from vacancies at the atomic level to macro pores with sizes of millimeters. A novel structured catalyst is introduced here: support structures manufactured by robocasting are coated by a thin MFI-type zeolite film and tested in a catalytic reactor for methanol-to-olefins. The macroporous architecture of these supports has a major impact on the mass and heat transfer properties and pressure drop of the final catalyst. Second, advanced ceramic processing can be used for the development of health care materials. Calcium phosphate powders are synthesized, processed and functionalized to be used as antigen or protein carriers in oral vaccination. The physico-chemical properties of the carrier material play a major role in determining the cellular uptake of the administered vaccine. The use of linker molecules or plasma activation is used to increase the protein adsorption on the carriers.

## **Processing and Characterization of Structural Ceramics**

Room: Broadway I - II

Session Chairs: William Fahrenholtz, Missouri University of S & T; Dongliang Jiang, Shanghai Institute of Ceramics

**10:15 AM**

### **(ICCP5-073-2013) Carbon Depletion near Surfaces in SiCO and SiCN Polymer-Derived Ceramics**

H. Kleebe\*, TU Darmstadt, Germany

Silicon oxycarbide and carbonitride ceramics were studied by transmission electron microscopy (TEM) upon isothermal annealing at 1300°C for 1 to 200 hours. TEM investigations in conjunction with energy-dispersive X-ray spectroscopy (EDS) analysis revealed a pronounced reduction of the local carbon content in close proximity to internal surfaces. Such small microcracks are a consequence of the polymer-to-ceramic transition and, hence, are commonly formed upon thermal annealing. A systematic study of the degradation of SiCO and SiCN was performed, in particular, with focus on the carbon depletion in the vicinity of internal surfaces. The profiles of the carbon content between surface and bulk were analyzed employing error functions to yield carbon diffusivities. Apart from the lowering of the carbon content, surface crystallization of cristobalite was also observed after an incubation period of approximately 5 hours. The presented results clearly imply that the often reported high thermal stability of such polymer-derived ceramics is limited, especially when thin film applications are anticipated.

**10:30 AM**

### **(ICCP5-074-2013) Properties of Silicon Carbide Ceramics from Aqueous Gelcasting and Pressureless Sintering (Invited)**

D. Jiang\*, Shanghai Institute of Ceramics, China

Silicon carbide ceramics has many excellent properties, such as high strength, high hardness, high resistance to corrosion etc, is a promising candidate for high temperature structural components in heat engines, heat exchangers, wear resistant components, etc. There are two conventional methods to sinter SiC named solid state sintering and liquid phase sintering. The solid state sintered SiC materials have fine-grained equiaxed microstructure, exhibit high hardness and good high-temperature mechanical properties. Liquid phase sintering (LPS), by using oxides as the sintering additives, can result in liquid phase formation at elevated temperatures and promote densification at low temperature. The obtained SiC ceramics exhibited high strength and toughness at room temperature. In the present work, aqueous gelcasting of SiC for both solid state sintering and liquid phase sintering was studied. The dispersion, slurry properties and gelation process were investigated and optimized based on powder surface characterization, slurry rheological properties measurement and microstructure observation of green samples. Subsequently, the pressureless sintering process was investigated and optimized for effective control of the microstructure evolution of SiC ceramics. The sintering mechanism and resulting mechanical properties of SiC ceramics were studied.

**11:00 AM**

### **(ICCP5-075-2013) Processing for Improved Thermal Conductivity of Zirconium Diboride**

G. Harrington, J. Lonergan, W. G. Fahrenholtz\*, G. Hilmas, Missouri University of S & T, USA

The effect of processing conditions on the thermal conductivity of zirconium diboride ceramics was examined. Densification of commercially available zirconium diboride can be accomplished by hot pressing with additions of up to 1 wt% carbon as a sintering aid. Carbon reacts with and removes oxide surface impurities from the zirconium diboride powder particles, but also results in the formation of boron carbide inclusions in the final ceramic. Dense ceramic produced by hot pressing at 1900°C had a thermal conductivity of ~80 W/m-K at 200°C. Based on phase equilibria in the Zr-B-C system, additional Zr was incorporated into the ceramics to prevent formation of boron carbide, which improved the thermal conductivity to ~85 W/m-K. To further reduce impurity content, reactive hot pressing of zirconium hydride and boron was also used to produce dense zirconium diboride. Compared to the conventional route, reactive processing is expected to reduce impurity contents and resulted in an additional increase in thermal conductivity of more than ten percent. This presentation will focus on the role of processing method on microstructure, phase development, and thermal conductivity of zirconium diboride.

**11:15 AM**

### **(ICCP5-076-2013) Processing and Testing of High-temperature Structural Ceramic Foams and Fiber-reinforced Ceramics**

J. Stiglich\*, B. Williams, Ultramet, USA

Ultramet has developed innovative processing for ultrahigh temperature structural materials including porous ceramic foams and fiber-reinforced ceramics. Highly insulating and lightweight thermal protection materials have been produced by combining an open-cell carbon or ceramic foam skeleton with an ultralow density aerogel filler material. The foam serves as an easily machinable structural reinforcement for the very low-strength aerogel insulator and defines the shape of the component. In the foam-aerogel composite structure, the aerogel exists in small discrete cells and is supported by the foam skeleton. The combined density of the composite insulator is as low as 0.1 g/cm<sup>3</sup>, and the thermal conductivity is <1 W/m-K at 2000°C. Single-piece panels up to 30" square are feasible. Using a rapid melt infil-

tration process, Ultramet also fabricates high strength fiber-reinforced ceramic matrix composite structures that can operate in various hot-gas environments at temperatures in excess of 2800°C. Processing and testing of ceramic foam and ceramic composite materials will be discussed.

**11:30 AM**

**(ICCP-077-2013) Ultrahigh Temperature Ceramics for Thermal Protection Systems, Propulsion and Energy: From Densification to Thermo-mechanical Properties (Invited)**

D. Sciti\*, S. Guicciardi, L. Silvestroni, National Research Council, Italy

Ultra high temperature ceramics (UHTC) include borides and carbides of early transition metals and are presently considered a class of promising materials for several applications, the most appealing ones being in the aerospace and energy sectors. Once densification problems are properly addressed, a crucial issue for application in severe environment is the improvement of fracture toughness and thermal shock resistance. To this purpose incorporation of SiC or C short fibers in UHTC matrices is adopted and effects on densification, microstructure and thermo-mechanical properties are studied. The present talk will show the latest developments in UHTC research at ISTE, including processing and characterization aspects of composites containing different kinds of commercially available fibers. Transmission electron microscopy is used as a fundamental technique for microstructural characterization and for a deeper comprehension of densification mechanisms and microstructure/thermo-mechanical properties relationships.

**Processing and Characterization of Optical Materials**

Room: Broadway III - IV

Session Chairs: Vincent Garnier, Insa de Lyon - Université de Lyon; Yiquan Wu, Alfred University

**9:00 AM**

**(ICCP-060-2013) Europium doped Yttrium Aluminum Garnet Transparent Ceramics (Invited)**

Y. Wu\*, S. Chen, Y. Yang, Alfred University, USA

Europium (Eu) doped yttrium aluminum garnet (YAG) transparent ceramics are prepared by using a vacuum sintering method. The valences of Eu dopant are investigated using XPS and EPR to correlate the relationships between the valences and the optical properties of Eu doped YAG transparent ceramics. Optical properties are tailored through the manipulation of the valences of Eu via annealing and reduction. The microstructures of the ceramics are characterized by electronic microscopy. Photoluminescence and radioluminescence spectra are measured to study the optical properties of the materials. Furthermore, a new gelling system has been explored to fabricate Eu doped YAG transparent ceramics through a casting processing.

**9:30 AM**

**(ICCP-061-2013) Processing Effects on Sintering of Transparent Al<sub>2</sub>O<sub>3</sub> and MgAl<sub>2</sub>O<sub>4</sub> Ceramics by FAST/SPS (Invited)**

V. Garnier\*, L. Lallemand, Insa de Lyon - Université de Lyon, France; N. Roussel, Université de Toulouse, France; G. Bonnefont, N. Brach, G. Fantozzi, Insa de Lyon - Université de Lyon, France; J. Chane, B. Durand, S. Guillemet-Fritsch, Université de Toulouse, France; S. Trombert, L. Bonneau, Baikowski, France

FAST/SPS is now commonly used to sinter ceramics rapidly while keeping a small grain size. However, the technique itself is not enough. To get transparent alumina (~100% dense ceramic with the smallest grain size) a careful control of all the processing steps is always needed. Different shaping methods leading to different green sample quality and thereafter to various transparencies will be presented. The optimization of dopants amount and their influence on the sintering mechanisms will be discussed. The crucial role of the starting powder and the slurry quality/preparation will be shown

through examples dealing with the grain size and morphology, the powder "cleaning" and the dry weight basis. Highly transparent ceramics (up to 70% Real In-line Transmission) could be obtained for small samples (20mm diameter and 1-2mm thickness). However, efforts must be made on getting larger transparent ceramics. This objective requires overcoming processing issues (homogeneous green samples with adequate size prior introduction into the sintering mould) as well as FAST/SPS issues (temperature homogeneity and dark coloration).

**10:15 AM**

**(ICCP-063-2013) Pressureless Sintered Transparent Yttria Alumina Garnet Multilayer Structures via Tape Casting and Lamination**

M. Beck\*, University of Erlangen-Nuremberg, Germany; Y. Menke, Schott AG, Germany; A. Roosen, University of Erlangen-Nuremberg, Germany

Optical transparent Yttria Alumina Garnets (YAG) are applied in various technological fields. Ce-doped YAG, e.g., is known as a fluorescence material which is used in white LEDs. The processing of the material via tape casting and subsequent lamination of tapes is an interesting method to produce thin planar and transparent Ce:YAG structures of defined thickness. Nano-scaled Y<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and CeO<sub>2</sub> powders were dispersed in an organic solvent system by using a steric dispersant. By adding binder and plasticizer to the dispersions, slurries were formed. The degassed slurries were characterized by rheological measurements. The slurries were tape cast using the Doctor-Blade-Process under various parameters. The green tapes were analysed with respect to density and thickness. Slurries of different particle loading and composition were investigated. Lamination tests of the green tapes under different conditions (i.e. time, temperature and pressure) were performed to optimize the laminate quality. The laminates were debinded and sintered in vacuum at temperatures up to 1700 °C. The sintered laminates were analysed with respect to density, microstructure, transparency, and colour coordinates. The interrelation between processing parameters and properties of the sintered material will be discussed.

**10:30 AM**

**(ICCP-065-2013) Polycrystalline Alumina Ceramics Doped with Nanoparticles for Increased Transparency**

M. Trunc\*, K. Maca, Brno University of Technology, Czech Republic

The present work is aimed at two most important goals of the current research into polycrystalline transparent alumina for ballistic and high-temperature applications. The first one includes grain size refinement in fully dense ceramics towards the nanometre range. This improves the transparency of birefringent alumina in the visible and near-infrared wavelength regions and also improves mechanical properties. The second research goal is focused on the processing of large and complex transparent bodies. We have refined the alumina microstructure by doping the fine alumina suspensions with zirconia and spinel nanoparticles. Dispersed nanoparticles reduced the grain growth during sintering by the pinning effect. In order to avoid light scattering the doping nanoparticles had to be extremely small (zirconia nanoparticles) or had to dissolve in the matrix during the later stage of sintering and act further as individual ions without causing any absorption or scattering losses (spinel nanoparticles). The in-line transparency up to 70% (at 640 nm wavelength and 0.8 mm sample thickness) was obtained for samples prepared by HIPing of presintered green bodies consolidated by gelcasting of doped alumina suspensions. The gelcasting proved to be the appropriate consolidating method for shaping large and complex transparent bodies. The examples of large transparent alumina bodies will be presented.

10:45 AM

### (ICCP-066-2013) Effects of Post-HIPing Thermal Annealing on Properties of Garnet Ceramic Scintillators

Y. Wang\*, J. Baldoni, Radiation Monitoring Devices, Inc., USA; W. H. Rhodes, C. Brecher, ALEM Associates, USA; K. Shah, Radiation Monitoring Devices, Inc., USA

Transparent optical ceramics find wide applications in the field of scintillation applications. Garnet materials have cubic structure which makes it ideal candidate for fully transparent ceramics. Different garnet ceramic such as Lu<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:Ce, Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>:Ce, etc are developed. In this study, transparent garnet ceramics were prepared by sintering followed by hot-isostatic-pressing (HIPing). The ceramics were thermally annealed after HIPing to restore from the oxygen deficiency resulted from the HIPing. The relationships between the transparency/scintillation properties of garnet ceramics and the thermal annealing conditions were discussed. The micro-structural evolution during thermal annealing was investigated. It is found that thermal annealing restore the samples from oxygen deficiency, which resulted in dramatic improvement of scintillation properties (light yield and emission). The scintillation properties increased with progressive annealing time and temperatures initially and then stabilized after reaching certain annealing time. The thermal annealing also resulted in a volume expansion and relaxation of residual pores inside the ceramics and hence a decrease in transparency.

11:00 AM

### (ICCP-067-2013) Effect of Surface Chemical Structure of Ag Nanoparticles on Supporting Behavior into TiO<sub>2</sub> Porous Films and their Optical Properties

H. Miyazaki\*, M. Iijima, Tokyo University of Agriculture and Technology, Japan; M. Ihara, Tokyo Institute of Technology, Japan; H. Kamiya, Tokyo University of Agriculture and Technology, Japan

Recently, an improvement of the light conversion efficiency of dye-sensitized solar cell (DSSC) by introducing Ag nanoparticles (NPs) which possesses localized surface plasmon resonance (LSPR) effects into the electrodes has attracted wide attention. Generally, these electrodes of DSSC are prepared by supporting Ag NPs and dye molecules in the sintered TiO<sub>2</sub> nanoparticle films formed on conductive glass substrates. Toward improving the property of DSSC by LSPR effects, it is important to control the deposition structure of Ag NPs in the TiO<sub>2</sub> nanoparticle film and the interactions with dye molecules. However, the effect of the surface chemical structure of Ag NPs on their dispersion state in solvents, their supporting behavior into TiO<sub>2</sub> films, their interactions with the dye molecules and the final cell properties are not well understood. In this study, a protocol to fix carboxyl- or amino- groups, which may interact with TiO<sub>2</sub> nanoparticles and dye molecules, on Ag NPs without forming aggregates were developed through ligand exchange process starting from hydrophobic Ag NPs. These nanoparticles were then supported into TiO<sub>2</sub> porous films with dye molecules to analyze the relationship among the surface structure of Ag NPs, their deposited structure and interaction with dye molecules for effectively improving the light conversion efficiency of DSSC.

11:15 AM

### (ICCP-053-2013) Preparation of Emitter Materials for Photovoltaic Electric Generation (Invited)

K. Kakegawa\*, H. Kubo, H. Tamagawa, N. Uekawa, T. Kojima, Chiba University, Japan

Photovoltaic (TPV) is environmentally friendly electric generation system, because waste heat or solar heat can be used. The system consists of heat source, emitter and TPV cell. TPV cell generates electricity by an application of near infrared ray. TPV cell typically has a highest efficiency at about 1500 nm of wavelength. The emitter should concentrate the emission energy into this wavelength region. Er<sup>3+</sup> has a characteristic emission around 1500 nm, which concen-

trate the emission energy in this region. Er<sub>2</sub>O<sub>3</sub> itself cannot be used as the emitter, because of its mechanical weakness and hygroscopicity. A eutectic system of Er<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>-Al<sub>2</sub>O<sub>3</sub> has been reported for the use of emitter. Because of the restriction of the eutectics, the composition was limited at the eutectic one. We have developed a method of a preparation of eutectic like structure. That is a formation of the structure from amorphous phase. It has two advantages; one is ultra-fine structure and the other is a flexibility of composition. In this study, such material having eutectic-like structure was used as the emitter of the TPV system. The performance of the system was evaluated. The amorphous material was prepared both by rapid quenching and wet method. Emitter having high performance and containing small amount of rare-earth was successfully created. Expensive rare-earth could be cut down.

11:45 AM

### (ICCP-054-2013) Soft Chemical Process for Functional Oxynitrides (Invited)

S. Kikkawa\*, Hokkaido University, Japan

Transition metal nitrides and oxynitrides are increasing their importance as the functional ceramic materials. They are extremely superior in magnetic and electric properties in comparison to their oxides. Reduction of their preparation temperature is very important to obtain these thermally metastable materials. They can be prepared by the nitridation of their oxide precursor synthesized through soft chemical route in relatively small particle size. Fine powdered oxide realizes the preparation of the nitrides and oxynitrides. The mixture of  $\alpha$ -Fe<sub>16</sub>N<sub>2</sub> with the residual  $\alpha$ -Fe showed a large magnetization value of 219 A m<sup>2</sup> kg<sup>-1</sup> among their fine powdered nitridation products of  $\alpha$ -Fe. The usage was important of the fine Fe<sub>3</sub>O<sub>4</sub> powder obtained from Fe(acac)<sub>3</sub> in benzyl alcohol. SrTaO<sub>2</sub>N ceramics showed a large dielectric constant value of  $\epsilon_r = 1.0 \times 10^4$ . The SrTaO<sub>2</sub>N fine powder was prepared by the nitridation of the amorphous oxide precursor obtained in citrate route. The sintered body attained a relative density >90% by using the fine powder with sintering aids such as SrCO<sub>3</sub>.

## Wednesday, August 7, 2013

### Plenary Session 4

Room: Pavilion Ballroom

Session Chair: Kunihiro Koumoto, Nagoya University

8:10 AM

### (ICCP-004-2013) Roll-to-Roll Processing of Functional Materials and Devices

J. Watkins\*, University of Massachusetts, USA

Roll-to-roll (R2R) technologies provide routes for continuous production of materials and devices with high throughput and low cost. We employ additive-driven self-assembly to produce well-ordered polymer/nanoparticle hybrid materials that can serve as active device layers, we use highly filled nanoparticle/polymer hybrids for applications that require tailored dielectric constant or refractive index, and we employ R2R nanoimprint lithography (NIL) for device scale patterning. Specific application examples to be discussed include the fabrication of memory devices, light emitting electrochemical cells, and large area films for light/EM management. In addition, a new process that allows direct printing of patterned crystalline metal oxide films and composites with feature sizes as small as 100 nm will be described. The fabrication schemes can be scaled in our newly constructed R2R processing facility, which includes a custom designed, precision R2R UV-assisted nanoimprint lithography system and hybrid nanostructured materials coaters.



## Flow and Assembly of Dense Suspensions

Room: Pavilion Ballroom

Session Chairs: Michael Hoffmann, University of Karlsruhe; Kathy Lu, Virginia Tech

**9:00 AM**

### (ICCP-078-2013) Colloidal Processing of Alumina (Invited)

M. J. Hoffmann\*, J. Reinshagen, R. Oberacker, University of Karlsruhe, Germany

Colloidal processing of ceramic suspensions allows the fabrication of complex shaped parts with superior mechanical and optical properties. Precondition to obtain these properties are green bodies with high packing densities and microstructures of best homogeneity, as they can be sintered at relatively low temperatures maintaining the homogeneous and fine grained microstructure. The required green body properties are solely attainable by the precise control of the interparticle forces and the consolidation procedure used. The paper describes the effect of interparticle forces and solids volume fraction on the rheological properties of alumina suspensions with defined interparticle forces. It will be demonstrated that a precise control of the interparticle forces can be achieved by well-defined amounts of salt for a given pH value. Steady shear experiments are used to indicate colloidal phase transitions of the suspensions with an increasing viscosity from a "liquid-like-" to "solid-like-behavior" depending on the interparticle forces and solids volume fraction. The colloidal phase behavior of alumina suspensions is discussed using a colloidal phase diagram including soft- and hard-sphere-behavior. Consolidation experiments reveal that the green body densities strongly increase with solids volume fraction of the starting suspensions if the DLVO-interaction potential is kept constant.

**9:30 AM**

### (ICCP-079-2013) Effects of Confinement on Structure, Dynamics, and Transport of Attractive Colloidal Suspensions (Invited)

R. Pandey, M. Spannuth, J. Conrad\*, University of Houston, USA

Ceramic processing often requires particulate suspensions to be shaped into fine features as rods, thin films, or coatings. To understand the effects of confining geometries on the structure of suspensions during processing, we use confocal microscopy to quantify the structure, dynamics, and transport properties of concentrated attractive colloidal suspensions. Our model system consists of slightly charged spheres with a controlled short-range interparticle attraction induced by non-absorbing linear polystyrene. For a range of polymer concentrations, the suspensions undergo a phase transition from a colloidal fluid of clusters to a colloidal gel as confinement increases while polymer and particle concentration are held constant. In both fluid- and solidlike attractive suspensions, effects of confinement on the structure and dynamics appear at much larger thicknesses than for hard-sphere suspensions. During flow in microchannels, the particle density in weakly attractive suspensions increases as suspensions are flowed downstream. Increasing the strength of attraction increases the resistance of the suspension to densification and to shear-induced migration. These studies provide insight into the interplay of confinement and attractions in conditions that mimic those found in processing.

**10:15 AM**

### (ICCP-080-2013) Direct Observation of Particle Motion in Ceramic Paste with High Solid Concentration under Low Shear Stress

S. Tanaka\*, Y. Nagasawa, Y. Takahashi, Z. Kato, K. Uematsu, Nagaoka University of Technology, Japan

Transparent ceramic paste with high solid concentration were prepared with fine silica grass powder (spherical shape, mean size 2.0 $\mu$ m), carboxy-methyl-cellulose (CMC) and glycerin solution. The

volume content of the powder is 50vol%. A small amount of fluorescent dye was added to the mixture to observe the internal structure with a confocal laser scanning fluorescent microscope (CLFSM) at high resolution. Rheological examination showed that all pastes had the yield stress in the flow curve. Under the gravitational field, the paste with the CMS concentration under 2% shows liquid-like behavior. It became plastic for the concentration over 3%. The paste was set between two silica grass plates under the microscope and sheared at various rates. Low shear stress was loaded to particles in paste between plates. Individual particles and their motion were clearly observed by CLFSM. The flow rate varied linearly with the distance from the plate surface in the liquid-like paste. The rate of particle motions varied irregularly and complicated motions were noted in the plastic paste. Each particle made network with around particles momentarily. The relevance for particle motion and the property of the paste will be discussed.

**10:30 AM**

### (ICCP-081-2013) Effect of Polymer Chain Length on the Rate of Phase Migration

T. Avery\*, MAST Carbon, United Kingdom; S. Blackburn, University of Birmingham, United Kingdom; S. Tennison, MAST Carbon, United Kingdom; N. Rowson, University of Birmingham, United Kingdom

Two binder systems were formulated from different grades of Polyethylene Oxide (MW 400,000 and 1,000,000), each having the same shear rheological properties, in order to determine whether polymer chain length would have an effect on the rate of phase migration during ram extrusion. Pastes were formulated from these binders so as to promote phase migration, the rate of which was measured from the gradient of the load/extension profile during ram extrusion. The rate of phase migration was found to vary significantly between the two different binder systems, despite apparently having the same flow characteristics. The increase in extrusion pressure in the lower molecular weight binder system was 0.12 MPa/mm and 0.09 MPa/mm for the higher molecular weight binder system. Models available in the literature do not appear to fully account for the observed behaviour. Work is ongoing to establish what is causing the difference between the two systems. It may be due to a secondary permeability interaction of the water phase through a PEO network or due to differences in viscoelasticity of the binders.

**10:45 AM**

### (ICCP-082-2013) Feedstock Properties and Mold-filling Simulations for Powder Injection-molded Silicon Carbide

R. E. Chinn\*, National Energy Technology Laboratory, USA; K. H. Kate, S. V. Atre, Oregon State University, USA; V. P. Onbattuvelli, Intel Corp., USA; R. K. Enneti, Global Tungsten Powders, USA

Silicon carbide, the most important non-oxide ceramic in the world, has a variety of applications in microelectronics, armor, aerospace optics, nuclear fuel cladding, textiles and many other specialties. Powder injection molding (PIM) enables rapid production of SiC and other ceramics and powder metals in complex shapes and precise sizes. In this paper, the rheological and thermal properties of SiC and a PIM binder system were analyzed as functions of temperature, pressure, powder volume fraction and other processing parameters. These properties were used with mold-filling simulation software to predict defects and set the stage for green micromachining of SiC PIM compacts.

**11:00 AM**

### (ICCP-083-2013) Development of a Nanoparticle-based Surface Templating Approach (Invited)

K. Lu\*, Virginia Tech, USA

Patterning of nanoparticle arrays represents exciting opportunities in solar cell, electronic, biomedical, chemical, and catalytic applications. In this talk, a direct nanoparticle suspension casting process will be discussed as a means for nanoparticle-based array formation. Differ-

## Abstracts

ent templates are produced by focused ion beam lithography followed by mold making using polydimethylsiloxane (PDMS) and silicone. The templated molds allow reproduction of micron-sized arrays of different features using nanoparticle suspensions. The effect of the pH value and dispersant on the maximum solids loading is examined. Different surface modifications of the PDMS molds influence the wettability of the nanoparticle suspension on them. The volume shrinkage during the transformation from nanoparticle suspensions into solid features inside the mold features is explored. The minimum diameter and the maximum aspect ratio of the features that can be obtained by this templating method are studied. This opens numerous opportunities for direct-device fabrication due to the pressureless and large surface area templating nature of the liquid-based process.

**11:30 AM**

### **(ICCP-084-2013) Aqueous Co-dispersion of LSM-YSZ for Directional Freeze-Casting**

A. Lichtner\*, University of Washington, USA; D. Jauffrès, University of Grenoble, France; J. Villanova, European Synchrotron Radiation Facility, France; D. Roussel, F. Charlot, C. L. Martin, University of Grenoble, France; R. Bordia, University of Washington, USA

Concentrated, aqueous co-dispersions of Lanthanum Strontium Manganite (LSM) and Ytria-Stabilized Zirconia (YSZ) were prepared and utilized for directional freeze-casting of Solid Oxide Fuel Cell (SOFC) cathodes. Simultaneous addition of LSM and YSZ to the slurry led to the formation of large (> 20 µm), tightly packed YSZ spheres and separation of the two particle types. By increasing the viscosity of the slurry and dispersing YSZ before LSM, a stable co-dispersion was achieved. Rheological studies were performed to determine the optimal level of dispersant for freeze-casting while still maintaining a homogenous slurry. Results from the directional freeze casting of these slurries together with microstructural analysis of the produced anisotropic, hierarchically porous structures will be presented. Homogeneous distribution of YSZ and LSM was confirmed through elemental mapping, FIB and nano-tomography reconstruction.

## **Flexible Electronics**

Room: Broadway I - II

Session Chairs: Lorraine Francis, University of Minnesota; Andreas Roosen, University of Erlangen-Nuremberg

**9:00 AM**

### **(ICCP-086-2013) Self-Powered Flexible Electronic Systems (Invited)**

K. Lee\*, KAIST, Republic of Korea

This seminar introduces three recent progresses that can extend the application of self-powered flexible inorganic electronics. The first part will introduce self-powered flexible piezoelectric energy harvesting technology. Energy harvesting technologies converting external sources (such as vibration and bio-mechanical energy) into electrical energy is recently a highly demanding issue. The high performance flexible thin film nanogenerator was fabricated by transferring the BaTiO<sub>3</sub> thin film from bulk substrates. Second, we report the nanocomposite generator (NCG) for achieving a simple, low-cost, and large area fabrication based on BaTiO<sub>3</sub> (or PZT) nanoparticles and graphitic carbons (CNT or RGO). The second part will introduce flexible electronics including memory and large scale integration (LSI). Flexible memory is an essential part of electronics for data processing, storage, and radio frequency (RF) communication. To fabricate a fully functional flexible memory, we integrated flexible single crystal silicon transistors with an amorphous titanium oxide (a-TiO<sub>2</sub>) based memristor to control the logic state of memory. Finally, the third part will discuss the flexible GaN LED for implantable biomedical applications. Inorganic III-V light emitting diodes (LEDs) have superior characteristics, such as long-term stability, high efficiency, and strong brightness. Our flexible GaN thin film LED enable

the dramatic extension of not only consumer electronic applications but also the biosensing scale. A water-resist and a biocompatible PTFE-coated flexible LED biosensor can detect PSA at a detection limit of 1 ng/mL.

**9:30 AM**

### **(ICCP-087-2013) Printing of Colloidal Dispersions for Flexible Electronics (Invited)**

A. Mahajan, H. Zhang, S. Lim, A. Ramm, C. Frisbie, L. Francis\*, University of Minnesota, USA; B. Ahn, B. Walker, J. Lewis, Harvard University, USA

Printing processes are finding increased application for manufacturing of electronic devices on flexible substrates. To meet the demands of resolution and device performance, new printing processes and improvements on traditional printing processes are needed. In this presentation, one newer process, Aerosol Jet Printing (AJP), and one traditional process, gravure printing, will be discussed. In AJP, a colloidal dispersion (i.e., ink) is aerosolized and transported by a carrier gas to a nozzle where it is focused, with the aid of a sheath gas, into narrow aerosol beam. A substrate moves beneath the beam and a printed pattern is produced. A systematic study of process parameters revealed a process window for creating narrow and tall conductive lines from silver ink. Gravure printing involves the transfer of ink from a pattern of recesses or cells in a plate or cylinder to the substrate, which comes into contact with the plate or cylinder. A key advantage of gravure printing is that it is designed for roll-to-roll processes. To boost the resolution for electronic applications, the use of photolithography to create precise patterns with controlled cell geometry and surface properties was explored. The process window and performance for gravure printing as it relates to creating conductive networks from silver inks will be discussed.

**10:15 AM**

### **(ICCP-088-2013) Fabrication of Ceramic Thin Films on Plastics: A Versatile Route Utilizing Sol-Gel and Transfer Techniques**

H. Kozuka\*, T. Fukui, M. Takahashi, H. Uchiyama, S. Tsuboi, Kansai University, Japan

Ceramic thin films on plastics are strongly demanded in flexible electronic devices and by those aiming at replacing glasses by lightweight plastics. Active functions of ceramic thin films often emerge in their crystalline states with low porosities. However, crystallization and densification are driven at high temperatures where plastics cannot survive. This is why great efforts have been made for over thirty years on low-temperature processing. Here we propose a totally different route. The technique is significant in that a "firing" step guarantees the crystallization and densification. The technique comprises (i) the deposition of a gel film on a release layer on a silicon substrate, and (ii) the transfer of the fired film onto plastics. The transfer is conducted by melting the plastics surface, which can be realized by heating the plastics/ceramic-film/release-layer/silicon stack from the silicon substrate side. Ceramic thin films thus fabricated on plastics are crack-free and transparent. Anatase thin films with high reflectivity, ITO thin films with electrical conductivity, and ZnO thin films with (002) orientation were realized on plastics. Patterned ITO thin films could also be prepared on plastics simply by using a mother silicon substrate with periodic trenches. Dual patterning with alternating ITO and ZnO ribbons was also possible.

10:30 AM

**(ICCPs-089-2013) Influence of Polymer Nature on Bioactive Glass-based Soft Coatings for Biomedical Applications obtained by EPD**

S. Cabanas-Polo\*, Institute of Biomaterials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany; J. A. Roether, Institute of Polymer Materials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany; A. R. Boccaccini, Institute of Biomaterials, Department of Materials Science and Engineering, University of Erlangen-Nuremberg, Germany

The increasing interest in the fabrication of composite coatings based on biopolymers and bioactive glasses (or ceramics) for orthopedic applications arises mainly from the potentially improved attachment that can be obtained between the rigid implant (e.g. Ti alloy) and the vascularized "softer" bone tissue. These so called soft coatings should also increase the corrosion protection of the metallic implant and avoid the high temperature sintering of pure bioceramic coatings. Electrophoretic deposition (EPD) is a colloidal processing method based on the electrically driven movement and subsequent deposition of charged particles or molecules onto a conducting substrate. The particle stability in suspension plays a key role in the production of homogeneous coatings and, given the novelty of bioactive glass-biopolymer coatings, their colloidal behavior should be carefully evaluated. In this work the effect of three different biocompatible polymers (polyacrylic acid, polyvinyl alcohol and polyvinylpyrrolidone) in the fabrication of bioactive glass-based coatings by EPD is investigated. The colloidal behavior is evaluated in terms of zeta potential to optimize the particle stability and the polymer/bioactive glass ratio in the coatings. The link between suspension stability, rate of deposition and coating microstructure for each investigated system will be highlighted.

10:45 AM

**(ICCPs-090-2013) Manufacture of Particulate Structures in the Micrometer Range via Coating and Printing Techniques (Invited)**

A. Roosen\*, University of Erlangen-Nuremberg, Germany

Printed electronics is an emerging technology in which electrically functional devices are generated by printing methods. In contrast to silicon technology and vacuum deposition techniques, printing of organic or inorganic components is a low cost process, which targets on products like FETs, RFIDs or displays on flexible polymer carriers. A very successful printing technique for the deposition of inorganic particles on substrates is screen printing, but its resolution is not low enough to print economically, e.g., field effect transistors (FET). The paper is dealing with the formation of active devices based on the deposition of functional inorganic nano-powders like ITO and ZnO. Processing issues like dispersion and stabilization of nano-sized powders to achieve homogenous slurries, pastes and inks of suitable concentration and rheological behaviour will be discussed. The paper will address the preparation of layers and structures in the micrometer range by different casting and printing techniques. To achieve functional devices, these techniques have to be combined to form multilayer structures from different materials. The characteristics of the deposited structures and their physical performance will be compared and their dependency on processing parameters will be discussed. Finally, these techniques are applied to print operational FETs and electro luminescence lamps.

11:15 AM

**(ICCPs-091-2013) Low Temperature Densification of Electrolytes by Using Controlled Tubular Electrode Support Shrinkage (Invited)**

T. Yamaguchi\*, T. Suzuki, H. Sumi, Y. Fujihiro, National Institute of Advanced Industrial Science and Technology (AIST), Japan

Solid oxide fuel cells (SOFCs) have received a great deal of attention recently, due to in part to their high energy conversion efficiency and environmental compatibility. The use of alternative high performance

electrolytes and electrodes materials means that the SOFC operation temperature can be reduced. In addition, miniaturization of the cell size and integration of multiple miniaturized cells is also considered to be one of the most effective approaches to increase the volumetric power density. Micro SOFC designs exhibit many desirable characteristics, such as increasing the net electrode area per unit volume and the durability to thermal stress caused by rapid start-up operation. Very recently, various researchers reported the excellent electrochemical performances and durability of heat-cycles for micro SOFC designs. Most of the cells are supported by a porous anode tube. Therefore the anode tubular support works as a current collector and gas diffusion layer as well as support medium of SOFC. Thus, the aim of this study is to realize the micro SOFC development with a reliable electrolyte layer on the excellent gas permeation, conductivity and mechanical strength. In this presentation, we will report low temperature preparation technologies of the electrolyte film on the tubular anode support in order to improve the anode polarization resistances.

**Densification of Ceramics**

Room: Broadway III - IV

Session Chairs: Ricardo Castro, University of California, Davis; Ingo Schmidt, Fraunhofer IWM

9:00 AM

**(ICCPs-092-2013) Nanoscale MgAl<sub>2</sub>O<sub>4</sub> Particles Sintering: Surface Chemistry and Agglomerations Effects (Invited)**

R. H. Castro\*, J. Rufner, University of California, Davis, USA

Sintering is considered a thermodynamically favorable process in which surface is eliminated by thermally activated specific mass transport mechanisms. At the nanoscale, this process is expected to show increased driving forces due to the excess energy from the enlarged surface area. However, the beneficial expectations from the nanoscale are not always observed in real processing, and this can be attributed to interfacial features that typically can be ignored for microscale sintering. Here we present data for MgAl<sub>2</sub>O<sub>4</sub> supporting and detailing the effects of interfacial chemistry and agglomeration on the densification process. We show a comparison between MgAl<sub>2</sub>O<sub>4</sub> synthesized by a polymeric precursor method and synthesized by a cleaner co-precipitation. The latter presents significantly higher densification due to less agglomeration, cleaner surface chemistry, and therefore more reactivity. The co-precipitated powders showed particle sizes below 8nm, with little agglomeration, and thereafter a fast densification under isothermal conditions up to 95% of the theoretical density in a few minutes (not pressure assisted). The polymeric-precursor powder, though showing similar initial particle sizes, had carbonaceous residues (only observed by thermogravimetry tests at high temperatures – 1000 °C) and significant agglomeration that lead to limited densification under similar sintering conditions.

9:30 AM

**(ICCPs-093-2013) Modelling and Simulation of Organic Binder Burnout in Ceramic Processing (Invited)**

I. Schmidt\*, T. Kraft, H. Riedel, Fraunhofer IWM, Germany; J. Svoboda, Academy of Sciences of the Czech Republic, Czech Republic

In the production of ceramic parts, organic additives are admixed to the powder for various reasons. Prior to sintering, these additives have to be removed from the green body which is typically done by a thermal debinding process. During this process, the binder usually undergoes thermal decomposition into gaseous reaction products which subsequently have to be transported to the surface through the pore channels. Depending on the heating rate and the green body's size and permeability, a pressure develops inside the body, and the resulting stresses in the solid skeleton can cause damage of the part. The paper presents a model which describes the chemical decomposition of organic binders, the combined Maxwell-Stefan and Knudsen diffusion and the seepage flow of multiple gaseous reaction products through a porous body as well as the implementation of the model

into a finite element framework. The formulation and implementation of Robin-type boundary conditions for the multi-component diffusion problem is addressed and results are presented for a prototype problem to demonstrate the capabilities of the model. This includes the computation of the solid skeleton stresses and a discussion of the relative importance of different transport mechanisms. The paper also presents an experimental setup that allows the determination of the parameters related to diffusion and seepage flow from a single experiment.

**10:15 AM**

**(ICCP-094-2013) Binder Removal by Diffusion Control: Concentration Distribution and Minimum Time Heating Cycles**

S. J. Lombardo\*, University of Missouri, USA

The removal of binder from green ceramic compacts when diffusion is the controlling transport mechanism is complicated due to the unsteady state nature of the process and to the highly non-linear diffusion coefficient. To circumvent these difficulties, an analytical model is developed for describing the spatial and temporal evolution of the concentration of binder and binder decomposition products within the green body during the heating cycle. The analytical solution is in terms of the decomposition reaction rate, the diffusivity, the length scale of the body, and the non-linearity in the diffusivity as a function of temperature and diffusant concentration. The analytical model is then combined with an algorithm based on variational calculus that predicts the form and duration of the minimum time heating cycle for binder removal.

**10:30 AM**

**(ICCP-095-2013) Effects of Forming on the Sintering Kinetics and Microstructural Evolution of 99.8% Pure Specialty Alumina Powders**

I. O. Ozer, L. Kupp\*, Penn State University, USA; C. Compson, E. Koep, Altmatis, Inc., USA; M. Spreij, Altmatis, Inc., Germany; G. L. Messing, Penn State University, USA

Variations within or between nominally identical grades of alumina powder may require adjustments in processing or sintering in order to achieve an equivalent result. Ideally, these variations and subsequent differences in processing behavior can be quantifiably linked to specific powder characteristics. This work aims to quantify the effects of physical and chemical differences between powders using dry pressing, slip casting and tape casting to provide consistently homogeneous, high green density compacts. These compacts minimize the effects of forming on subsequent sintering kinetics and microstructure evolution. The sintering and densification behavior of each alumina compact was characterized using TMA and microstructural analysis. Changes in activation energies for densification were determined as a function of forming technique using master sintering curves (MSC).

**10:45 AM**

**(ICCP-096-2013) A Unified View of Grain Boundaries in Sintered Alumina**

W. M. Carty\*, Alfred University, USA; T. Lam, National Institute of Standards and Technology, USA

Research on porcelain indicated that the glass phase in commercial porcelains reside on the glass formation boundary in the alkali-alumino-silicate system. It was hypothesized that the grain boundaries in sintered alumina must also reside on the glass formation boundary. 94% ( $\pm 0.5\%$ ) alumina samples were prepared with five impurity ratios of CaO:SiO<sub>2</sub> to allow grain boundary formation in the CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system to be evaluated. Samples were prepared using a unique colloidal processing technique to ensure the sample chemistry. Samples were heat-treated at 1400°C, 1600°C, and 1700°C for one hour. Approximately 450 grain boundaries were evaluated using TEM to determine chemistry and mineralogy – and average of 30 grain boundaries per composition at each temperature. The amor-

phous grain boundary chemistry was coincidental with the glass formation boundary in the system when sintered at sufficient temperature. The mineralogy observed in the grain boundaries containing crystalline precipitates correlated with the phase field intersecting the glass formation boundary. In addition, two glass forming regions were identified: a normal glass structure (silicate based) and an invert glass structure (CaO-based).

**11:00 AM**

**(ICCP-097-2013) Microstructural Characterization and Control of Perovskites (Invited)**

M. Baeurer\*, W. Rheinheimer, M. J. Hoffmann, Karlsruhe Institute of Technology, Germany

Perovskite materials are widely used in electronic components. The macroscopic properties are often governed by grain boundaries and therefore knowledge of the microstructural evolution during processing is highly important. A deeper understanding of the influence of grain boundary properties on grain growth is necessary in order to tailor the microstructure as needed for best performance. Strontium Titanate has been used as a model system to study grain growth in perovskite systems. It has been found that grain growth does not follow Arrhenius type behavior over the temperature range typical for sintering of the material. Two distinct drops in grain growth rate with raising temperature are present. The effect is reversible; with step changes in temperature grain growth rate can be lowered and raised by orders of magnitude. This counterintuitive behavior is discussed on the background of relative surface energies extracted from pore shapes, grain boundary morphology and growth studies on embedded single crystals. The grain boundary network in polycrystals is characterized on different length scales, ranging from TEM techniques at grain boundaries to non-destructive X-ray diffraction contrast tomography. It can be shown that the anisotropy in grain boundary energy is insufficient to explain the changes in microstructure observed. A combination of anisotropic properties in energy and mobility is needed.

**11:30 AM**

**(ICCP-098-2013) Master Sintering Curve Theory applied to Sintering of Layered Ceramic Composites**

V. Pouchly\*, D. Drdlik, K. Maca, CEITEC BUT, Brno University of Technology, Czech Republic; H. Hadraba, Z. Chlup, CEITEC IPM, Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Czech Republic; J. Cihlar, CEITEC BUT, Brno University of Technology, Czech Republic

The layered Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> composites with layers thickness of ca 50 micrometers were prepared by electrophoretic deposition followed by pressureless sintering. The sintering behaviour of green bodies in transversal (perpendicular to layers) and longitudinal (parallel with layers) directions were monitored using high-temperature dilatometer. The differences in sintering in both mentioned directions were described with the help of Master Sintering Curve theory. They were characterized by different sintering activation energies and related to the individual processes taking place during sintering.

**11:45 AM**

**(ICCP-099-2013) Space-charge Contribution to Densification in Perovskite Ceramics**

F. Lemke\*, J. Hötzer, M. Bäurer, M. J. Hoffmann, B. Nestler, Karlsruhe Institute of Technology, Germany

The basic mechanisms during densification of ceramics are usually obtained experimentally by studying microstructural development and sintering rate combined with analytical sintering models. Therefore the effect of changes in sintering parameters on the densification have to be studied. Defect chemical approaches to analyse densification and sintering are still not very common despite of the good availability of literature data on defect chemistry in the bulk and at the grain-boundaries (GB's) for model systems such as SrTiO<sub>3</sub> in high

temperature regions. Diffusion coefficients based on defect chemical calculations were used with Coble's sintering equations to obtain densification rates as a function of temperature, oxygen partial pressures, doping, space-charge potential and grain-size. The results were compared to experimental data from undoped SrTiO<sub>3</sub>. A strong contribution of the increased diffusion in the space-charge layer at GBs to material transport during sintering especially for fine grained materials is proposed. It can be shown that an adapted model including space-charge contributions reflects diffusion in bulk and at GBs respectively, depending on particle size and defect concentration. Because the grain size distribution is not considered in the analytical model used, additional phase-field simulations were carried out to estimate the influence on the densification rates.

## Plenary Session 5

Room: Pavilion Ballroom

Session Chair: Gary Messing, Pennsylvania State University

12:55 PM

### (ICCPs-005-2013) Discrete Element Simulation: Modeling Sintering at the Particle Length Scale

Z. Yan, University of Grenoble / CNRS, France; O. Guillon, Universität Jena, Germany; R. K. Bordia, University of Washington, USA; C. L. Martin\*, University of Grenoble / CNRS, France

This talk reviews recent advances in simulations of sintering based on the Discrete Element Method (DEM). In DEM, particles and their interactions are explicitly modeled. It offers a powerful tool for investigating links between microstructure and sintering behavior. In such simulations each particle interacts with its neighbors through appropriate contact laws. The gradual appearance of new contacts and the loss of others are included and local constraints may be simulated. This allows more realistic sintering conditions to be taken into account as compared to simulations that operate at a larger length scale. In this contribution, we present results on the evolution of defects during sintering which reveal that some geometrical constraint is necessary for a defect to grow into a crack. We demonstrate the use of DEM for simulating multilayered systems in which the appearance of defects originates mainly from the particulate nature of the material to sinter. We also examine how it is possible to model the sintering of complex microstructures for which a continuum approach would not be appropriate. Composite powders made of a sintering matrix mixed with inclusions offer such an example. The retarding consequences of inclusions are explained in terms of particle packing. The effects of inclusion size (relative to the matrix particle size), volume fraction, and packing homogeneity have been investigated.

## Shaping/Assembly of Ceramics

Room: Pavilion Ballroom

Session Chairs: William Carty, Alfred University; Wolfgang Sigmund, UF

1:45 PM

### (ICCPs-100-2013) Synthesis and Processing of Ceramic Nanofibers (Invited)

W. Sigmund\*, UF, USA

Electric fields allow to shape the size and form of liquids. Under specific conditions particles or fibers with nanometers in diameter may be achieved. The challenges in this process are the control of the sol-gel chemistry, the complex fluid behavior and the impact of processing parameters on fiber properties after heat treatment. This talk will give a review of ceramic nanofibers synthesis since it starts, the state of the art in the electrohydrodynamic processing and show selected advances in electrode materials in energy storage or harvesting.

2:15 PM

### (ICCPs-101-2013) Electrospinning of Ceramic Nanofibers from Pre-ceramic Polymers

A. Guo, M. Roso, M. Modesti, P. Colombo\*, University of Padova, Italy

Silicon oxycarbide fibers were successfully fabricated by electrospinning a mixture of commercially available polymethylsilsesquioxane (MK) or polymethylphenylsilsesquioxane (H44) pre-ceramic polymers and polyvinylpyrrolidone, followed by cross-linking and pyrolysis at 1000°C in inert atmosphere. The influence of the processing procedure (solvent selection, cross-linking catalyst, additives, voltage) on the morphology of the produced fibers was investigated. For the MK/isopropanol system, the introduction of 20 vol% N,N-dimethylformamide (DMF) enabled to decrease the diameter of the as-spun fibers from 2.82 μm to 1.72 μm. For the H44/DMF systems, the formation of beads could be avoided by adding 50 vol% chloroform. The addition of a suitable cross-linking catalyst was indispensable for maintaining the fiber shape during the high temperature thermal treatment. After pyrolysis, the resultant SiOC fibers derived from MK and H44 resins possessed uniform shape, with an average diameter of 1.05 and 1.13 μm, respectively. Doping of the fibers with metal precursors was also explored, for the development of functional ceramic nano-fiber based devices.

2:30 PM

### (ICCPs-102-2013) Micro Geometric Patterning of Titania Polygon Tablets for Terahertz Wave Control by Materials Tectonics Processing (Invited)

S. Kirihara\*, Osaka University, Japan

Materials tectonics is new concept to control energy flows from environmental field to human beings through artificial interfaces with ceramics patterns. Titania micro patterns with periodic-array or self-similar arrangements were formed for electromagnetic wave localizations in terahertz frequency ranges. Geometric arrangements of polygon tablets with anatase phase were created through liquid phase crystal depositions under micro templates fabricated by stereolithography. The terahertz waves having micrometer order wavelengths and belonging in far-infrared frequency ranges are expected to apply for analyses and detections of environmental pollutants. In the micro patterning, high viscosity slurry of photo sensitive acrylic resin was supplied on a flat substrate with 10 μm in layer thickness. Cross sectional images was exposed on the slurry surface by digital micro mirror devices with 2 μm in part accuracy. Through the layer stacking, a solid component was obtained. The micro template was heated at 75 °C for 12 hs in a water solvent of 0.05 mol/L ammonium titanium fluoride and 0.15 mol/L boric acid. The crystal phase and microstructure of the formed patterns were analyzed and observed by a X-ray diffraction and scanning electron microscopy. The terahertz wave properties were measured and simulated by time domain spectroscopy and finite difference time domain.

3:00 PM

### (ICCPs-103-2013) Roll your own: Nanocomposite Capacitor

E. Yi\*, J. Furgal, R. M. Laine, University of Michigan, USA

BaTiO<sub>3</sub> has long been investigated and used as a capacitor material due to its inherent dipole moment in the tetragonal phase. Although there are controversies on the critical size at which BaTiO<sub>3</sub> becomes cubic, sub 100 nm particles offer optimal potential for making flexible BaTiO<sub>3</sub>/polymer nanocomposite films. To date several reports describe processing BaTiO<sub>3</sub>/epoxy nanocomposite capacitors but none mention flexibility, nor have there been any attempts to fabricate rolled capacitors. In this work, we compare the dielectric constants of composite films of nano-BaTiO<sub>3</sub> filler and epoxy resin matrix. LF-FSP (liquid feed flame spray pyrolysis) derived and commercial BaTiO<sub>3</sub> are used as fillers. Flexible epoxy resin matrices were formulated using Diaminodiphenylmethane (DDM) and Octaglycidyl (OG) functionalized silica cage core (Q cages) as a matrix as several literatures show it to improve breakdown voltages.

## Abstracts

Nanocomposite films were cast at 30, 40 and 50 volume %. Dielectric constants and loss tangents at selected frequencies are reported. For the first time, we demonstrate a rolled capacitor of BaTiO<sub>3</sub>/epoxy nanocomposite film. The dielectric constants obtained were 19, 21 and 16 for 30, 40 and 50 volume % films, respectively at 100 KHz. It offers potential to replace commercially available counterparts. Furthermore, due to nanocomposite's higher energy density, less volume is necessary.

**3:15 PM**

### **(ICCPs-104-2013) Influence of Feedstock Preparation on Ceramic Core Formation Using a Development Carrier System**

T. Standing\*, University of Birmingham, United Kingdom; P. Wilson, Rolls-Royce Plc., United Kingdom; S. Blackburn, University of Birmingham, United Kingdom

The following study describes optimising the success of a development carrier system (binder) in forming complex ceramic components through ceramic injection moulding. This investigation shows how the incorporation of different levels of plasticiser in the feedstock and various solids loadings affect both feedstock properties and subsequent component fabrication, both in the 'green' and fired states. The study uses pycnometry, rheometry, mechanical testing, interferometry and SEM to characterise the material properties. The experimental data illustrates that full component fabrication was only possible when plasticiser was incorporated into the feedstock. Increasing plasticiser additions led to a higher critical solids loading, however, beyond a coating of '10 layers' of plasticiser on the powders surface, a drop in achievable loading was observed. Green component properties were affected by the levels of plasticiser, with strength and surface roughness influenced. Fired components properties were not severely affected by the binder formulation; however, specific formulations did cause surface roughness issues to present after the firing process. The study highlights that although it is important to use a carrier system with idealised properties, optimising processing conditions can also critically influence fabrication goals.

**3:30 PM**

### **(ICCPs-105-2013) Gelcasting of Oxide Ceramics by a Single Additive (Invited)**

S. Wang\*, S. Shimai, Y. Sun, Y. Yang, M. Dong, Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

A novel and simple gelling system for gelcasting of oxide ceramics was developed by using a nontoxic and water-soluble co-polymer of isobutylene and maleic anhydride (commercially called Isobam). Alumina slurry was made with deionized water, alumina powder, and a small amount of Isobam (0.3 wt%, relative to the weight of the powder). Rheological property and gelling behavior evaluation demonstrated that the gelling rate decreased with the increase of solid loading but increased with the Isobam content and this system could gel at room temperature in air. Then, the gelling system was successfully employed to gelcast zirconia ceramics, alumina translucent ceramics, YAG and yttria transparent ceramics, respectively. Further, by integrating mechanical foaming method, the gelling system was extended to prepare alumina and zirconia porous ceramics with controlled porosity and cell size. For example, alumina porous ceramics was obtained with porosity from 20% to 89% and concurrently with cell size from 60µm to 220µm, and the compressive strength was as high as 75MPa when the porosity was 60%.

**4:00 PM**

### **(ICCPs-106-2013) Graded Materials via a Novel Light Directed Electrophoretic Deposition Technique**

A. J. Pascall\*, F. Qian, Lawrence Livermore National Laboratory, USA; G. Wang, Y. Li, University of California, USA; J. Kuntz, Lawrence Livermore National Laboratory, USA

Electrophoretic deposition (EPD) is an industrially relevant process in which colloidal particles are suspended in a liquid are forced to de-

posit onto an electrode under an applied electric field. EPD has generally only allowed for the fabrication of materials with gradients in material properties normal to the electrode's surface (z-direction) due to the static nature of the electrode. Here, we present a novel EPD technique, light directed EPD, which utilizes a photoconductive electrode that can be dynamically patterned with light during the course of the deposition. This allows for the fabrication of graded deposits in the x-, y-, and z axes. We will present experimental results demonstrating the technique as well as numerical modeling of the deposition process on photoconductive electrodes. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-ABS-616512

**4:15 PM**

### **(ICCPs-107-2013) Hollow and Solid Pressure Slip Casting of Coarse Grained Oxide Ceramics**

S. Schaffoener\*, C. Aneziris, TU Bergakademie Freiberg, Germany

This contribution presents the forming of solid and hollow oxide ceramic refractory components by pressure slip casting. For that purpose a commercial pressure slip casting machine has been adapted for pumping coarse grain slips. The use of an organic additive system allowed the preparation of a stable and pumpable slip containing grains of a size of up to 3 mm and an easy demolding of crack free, dimensionally stable bodies with negligible gradients due to sedimentation. The presented pressure slip casting technique is a way to produce complicated near net geometries for modern refractory applications. Advantages of the pressure slip casting are the possibility of automation, faster production, higher densities and lower quality variations of the bodies. We will demonstrate main process factors influencing the casting of solid plate like refractory components as well as hollow crucibles with a height of 15 cm. Simple and effective measurement techniques like an adapted falling sphere viscometer and a slump flow channel are adopted to characterize the flowing behavior of coarse grain slips. The filtration behavior of the slips was optimized by the application of a compression-filtration-permeability cell. In a last step the fired solid and hollow bodies were analyzed by X-ray computer tomography to reveal possible sedimentation, gradients and cracks.

**4:30 PM**

### **(ICCPs-108-2013) Processing of Textural Clay based Materials**

G. Lecomte-Nana\*, A. Mokrani, O. Mongenovo, N. Tessier-Doyen, K. Bousois, H. Goure-Doubi, ENSCI - GEMH, France

The present work investigated the processing of textural clay based materials using tape casting together with freezing. Three model commercial raw materials were used, namely: BIP kaolin from France, ABM illite and ABM montmorillonite from Mediterranean region. Mixtures of both clays were studied; the amount of illite or montmorillonite was respectively 0, 5, 10 and 15 mass %. Tape casting was performed first, and then the as-obtained green bands were frozen into liquid nitrogen, lyophilized and then calcined at 1050°C and 1200°C. The amount of illite or montmorillonite appeared to control the cohesion of the dry products. For montmorillonite-based products, the cohesion of the dry products was satisfactorily and a macroscopic cross-linked surface texturation was observed. With illite, the texturation was difficult to control. After calcination at 1050°C, the texturation seemed well defined. Increasing the calcination temperature to 1200°C tended to increase the densification of products and the occurrence of a glassy phase was noted. Besides, the texturation of products was roughly modified. The combination of both tape casting and freezing (freeze tape casting) is a promising way to develop various clay-based and composites materials exhibiting unique microstructure organization and characteristics with potential application in the field of low energy and environmentally friendly filtration, adsorption or catalysis.

## Synthesis and Processing of Functional Ceramics

Room: Broadway I - II

Session Chairs: Nelson Bell, Sandia National Laboratories; D. Hotza, UFSC

1:45 PM

### (ICCPs-109-2013) Solution Processed Nanostructured Materials for Application in Energy Storage (Invited)

S. Tolbert\*, I. E. Rauda, V. Augustyn, C. Kang, B. Dunn, UCLA, USA

Block-copolymer templating provides a powerful method to create complex nanostructured inorganics through low-cost processing routes. Here we utilize this method for application in electrochemical energy storage. We first examine porous pseudocapacitors built from nanocrystal building-blocks. This architecture combines high surface-area, electrical connectivity, and facile electrolyte access to surface redox sites, resulting in fast redox kinetics. Composite systems can also be produced that combine electrical conductivity with kinetically-accessible redox sites. In some materials we find that pore flexibility, combined with short diffusion lengths produces a phenomena called intercalation pseudocapacitance, where traditional battery-like intercalation become kinetically facile. Such materials provide a unique opportunity to combine high energy density and high power density. Finally, for high-capacity anodes, we examine porous silicon produce by reduction of porous silica. Here, the mechanical flexibility of nanoporous materials can produce robust cycling behavior, despite the large volume increase that occurs in silicon upon alloying with lithium.

2:15 PM

### (ICCPs-127-2013) Colloidal Interactions at Advancing Ice/Water Interfaces (Invited)

S. Dillon\*, University of Illinois, Urbana-Champaign, USA

Colloidal interactions at the ice/water interface affect particle occlusion and have been the basis for ice templating processes that efficiently assemble nanoparticles. While these interactions have been well studied at the micron scale, limited experimental insights exist with regards to particle-ice interactions in nanoparticle systems with small radii of curvature. This work investigates dynamic interactions of Au nanoparticles at the crystallization front of hexagonal and cubic ice by in-situ cryogenic environmental transmission electron microscopy in the temperature range of 220 to 260 K. The magnitude of the short-range (~1 nm) interactions is quantified based on a statistical treatment of occlusion probability coupled with ex-situ adhesion force measurements and a force balance analysis. The results provide new insights into the nature of particle occlusion in ice, suggest new approaches to control such occlusion, and provide insights into ice phase evolution at different temperatures.

2:45 PM

### (ICCPs-111-2013) Phase Development and Densification of NaSICON Ceramics

N. Bell\*, C. Edney, P. Lu, M. A. Rodriguez, E. D. Spoeke, Sandia National Laboratories, USA

NaSICON ceramics are leading compositions in sodium ion conduction, yet suffer from several processing difficulties in manufacturing dense components. Phase development is affected by composition, precursors, and thermal profile, whereas defects relate to the formation of ZrO<sub>2</sub> inclusions and poorly characterized glass phases. This work examines NaSICON ceramics formed using a molecular precursor approach, and examines densification of Na<sub>1+x</sub>Zr<sub>2</sub>SixP<sub>3-x</sub>O<sub>12</sub> materials varying x = 0 to 3. The impacts of ceramic processing and chemical composition upon densification, phase purity, stability and performance are examined. Using SEM, XRD, and microstructural EDS composition mapping, new insight into complex phase behavior is found. Small variations in stoichiometry influence reactive sintering of NaSICON, impacting densification, grain growth and

phase purity of these materials. Non-crystalline phases play a critical role in these processes, reinforcing the need for detailed understanding of the powder preparation and processing treatments in the formation of components for advanced materials applications. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

3:00 PM

### (ICCPs-112-2013) Image Analysis of the Porous Yttria-Stabilized Zirconia (YSZ) Structure for the Impregnated Electrode of Solid Oxide Fuel Cell (SOFC)

C. Ni\*, M. Cassidy, J. Irvine, University of St Andrews, United Kingdom

Image analysis and quantification were performed on the porous scaffolds for SOFC cathodes using the two types of YSZ powders, Unitec 1- $\mu$ m powder with a broad particle-size distribution having two maxima at ~0.1  $\mu$ m and 0.8  $\mu$ m, and Unitec 2- $\mu$ m powder with only one at ~1  $\mu$ m. The inks from the two powders with ~50 vol.% pore-forming agent, graphite and glassy carbon, were screen-printed onto a dense 2-mm YSZ electrolyte and sintered at 1300 °C to produce the scaffold. The porous structure using Unitec 2- $\mu$ m powder shows finer YSZ grains and a higher boundary length than the 1- $\mu$ m powder. Ac impedance on symmetrical cells was used to evaluate the performance of the electrode impregnated with 35-wt.% La<sub>0.8</sub>Sr<sub>0.2</sub>FeO<sub>3</sub>. At 700 °C, the electrode from Unitec 2- $\mu$ m powder shows a polarization resistance ( $R_p$ ) of 0.21  $\Omega$  cm<sup>2</sup>, and series resistance ( $R_s$ ) of 12.31  $\Omega$  cm<sup>2</sup>, lower than the electrode from Unitec 1- $\mu$ m powder does. The quantitative study on image indicates that Unitec 2- $\mu$ m powder is better in producing architecture of high porosity or long triple phase boundary (TPB), which is attributed as the reason for the higher performance of the LSF-impregnated electrode.

3:15 PM

### (ICCPs-114-2013) Innovative Processing to Enhance Nuclear Fuel with Oxide Additives

R. M. Leckie\*, E. P. Luther, A. T. Nelson, K. J. McClellan, Los Alamos National Lab, USA

Over the past 50 years much effort has gone into using oxide additives to modify the properties and/or microstructure of UO<sub>2</sub>. Despite this long history a few areas have not been well explored. Details are scarce in the open literature on creating fine grained microstructures and on techniques to distribute the additives. Nevertheless, all indications point to altering the underlying microstructure of the UO<sub>2</sub> fuel pellet as the best way to improve the performance of UO<sub>2</sub> in reactors. This work seeks to address the above areas by examining the use of additives to restrict grain size and by exploring an unconventional method of incorporating those additives into UO<sub>2</sub> powder using metal precursors. Results using Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub> and other oxides to control microstructure in UO<sub>2</sub> and their resulting effect upon mechanical properties will be discussed. Additionally, the different processing techniques will be compared.

3:30 PM

### (ICCPs-115-2013) Electrolyte-Supported Solid Oxide Fuel Cell Processed by Aqueous Tape Casting and Constrained Calendering

V. Moreno, UFSC, Brazil; N. Travitzky, P. Greil, University of Erlangen-Nuremberg, Germany; D. Hotza\*, UFSC, Brazil

Yttria-stabilized zirconia (YSZ) electrolyte-supported SOFCs were produced combining three different techniques: (1) aqueous tape casting, (2) constrained calendering and (3) screen printing. Rheological behavior of the anode and cathode slurries was evaluated. Slurries presented viscosities between 70 and 90 mPa.s at shear rate of 100 s<sup>-1</sup>. Anode (NiO/YSZ) and cathode (LSM/YSZ) were printed on the calendered electrolyte, and showed thicknesses of ~40  $\mu$ m, ~14

$\mu\text{m}$  and  $200\ \mu\text{m}$ , respectively. Anode porosity increased after reduction, presenting a value of 53%. Electrochemical performance was evaluated in a no-chamber direct flame set-up. Cell exhibited an open circuit voltage of 0.72 V and maximum power density of 1.95 mW/cm<sup>2</sup>. No structural damage was observed after testing.

### Field Assisted Densification of Ceramics

Room: Broadway III - IV

Session Chairs: Bala Vaidhyanathan, Loughborough University; Leszek Chlubny, AGH University of Science and Technology

**1:45 PM**

#### (ICCPs-116-2013) Flash Sintering of Functional Materials (Invited)

B. Vaidhyanathan\*, S. Ghosh, Loughborough University, United Kingdom

Electroceramic devices such as varistors and capacitors are used in most of the modern day electronic appliances and constitute a multi-billion\$ market. Conventional fabrication of these devices involves high sintering temperatures and long processing time. Since sintering controls the electrical properties, it is necessary to develop simpler and less demanding processing methods. In a report on 'flash sintering' (FS) it was demonstrated that full sintering of dog-bone shaped zirconia ceramics can be achieved at 850oC in just 5 seconds rather than normally used 1450oC for few hours. This opens up the possibility of achieving significant energy savings during manufacture and the ability to produce fine grained ceramics. However the exact mechanisms by which this phenomena occur is not yet clear and the methodology is untested for the sintering of other complex functional materials. At Loughborough we investigated the feasibility of sintering nanocrystalline ZnO-varistors, BaTiO<sub>3</sub>-capacitors and CCTO dielectrics using this method along with simultaneous measurements of shrinkage, online thermal distribution mapping and atmospheric control. This allowed the fabrication of disc-shaped electroceramics using a controlled flash sintering approach and the properties of the devices are compared with conventionally sintered components. A phenomenological model is proposed to explain the FS behavior.

**2:15 PM**

#### (ICCPs-117-2013) Electric Field and Current effect on Liquid Phase Sintering Mechanism

J. Gonzalez\*, O. Guillon, Friedrich Schiller University Jena, Germany

Liquid phase sintering (LPS) of most of the ceramics materials enhances when an electric field is applied during the thermal cycle, although the mechanisms which control the sintering are still under debate. One explanation can be related with an enhancement of the particle rearrangement stage of the LPS due to a reduction of the wetting angle of the liquid phase when it is under an electric field. Field Assisted Sintering Technique/Spark Plasma Sintering and Electrical Field Assisted Sinter Forging technique are used to determine the effect of the electric field and current in LPS. Electrical insulator cell is located into the graphite die used in the Field Assisted Sintering Technique to determine the role of the electrical field during the sintering. Besides, wetting studies of the sintering additives at high temperature as a function of the applied voltage are related with the enhancement of the sinterability. A new approach on the mechanisms of techniques, which work under electric field such as Field Assisted Sintering Technique/Spark Plasma Sintering, will be presented.

**2:30 PM**

#### (ICCPs-118-2013) Electrical Energy Storage Properties of Barium Strontium Titanate Ceramics Prepared by Spark Plasma Sintering (Invited)

Y. Wu\*, Y. Huang, N. Wang, X. Chen, Zhejiang University, China

Dielectric capacitors have high power density with faster charge and discharge rate, but their energy density is typically much lower than

that of electrochemical supercapacitors. Increasing the energy storage density of dielectric capacitors is highly desired to extend their application in pulsed power system and regenerative braking system. The overall maximum energy storage in a linear dielectric is proportional to the product of the dielectric constant and the square of the dielectric breakdown strength. Barium strontium titanate ceramics have high dielectric constant, but are limited to relatively low breakdown strength. In this paper, spark plasma sintering was used to improve the breakdown strength of barium strontium titanate ceramics. Transparent and dense Ba<sub>1-x</sub>Sr<sub>x</sub>TiO<sub>3</sub> (x=0.6 and 0.7) ceramics with an average grain size of ~0.8  $\mu\text{m}$  were successfully prepared. A high dielectric breakdown strength of 230 kV/cm was obtained for Ba<sub>0.3</sub>Sr<sub>0.7</sub>TiO<sub>3</sub> ceramics prepared by spark plasma sintering. The energy storage density of spark plasma sintered Ba<sub>0.3</sub>Sr<sub>0.7</sub>TiO<sub>3</sub> ceramics is 1.13 J/cm<sup>3</sup>, which is about two times of that of the ceramics prepared by the conventional solid state method. The relationship between the microstructure and the energy storage properties will be discussed.

**3:00 PM**

#### (ICCPs-119-2013) Using of Pulsed Electric Discharges to the Preparation and Consolidation of Powder Composites

E. Grigoryev\*, M. S. Yurlova, E. V. Krikun, Moscow Engineering Physics Institute, Russian Federation; E. A. Olevsky, San Diego State University, USA; O. N. Sizonenko, Institute of Pulse Processes and Technologies at National Academy of Sciences, Ukraine; Y. Lin, San Diego State University, USA

The purposes of this study are: 1) determine the opportunities of high voltage electrical discharges in the grinding of powders and simultaneous formation of carbides in these powders; 2) use the method of spark plasma sintering to consolidation of these processed composite powders. For the powder grinding we used the high voltage electrical discharges in kerosene. For consolidation of processed powder composites we used the following of spark plasma sintering systems: Dr. Sinter SPSS -515, Labox 625 and Labox 650. Microhardness of the consolidated samples was determined by Vickers Hardness Testing using HVS-1000. The influence of high voltage electrical discharge was investigated on the regularities of morphometric characteristics and kinetics of dispersing Fe and Ti powder mixture. It was found that the electrical discharge machining produces the formation of cementite Fe<sub>3</sub>C, and such phases as TiC, FeB, Fe<sub>2</sub>Ti. After electrical discharge machining these composite powders were consolidated by spark plasma sintering. It was found that the density and microstructure of consolidated composite powder material essentially depends from electrical pulse current parameters and applied mechanical pressure. The high density and high strength of composite material reached its maximum values at certain magnitudes of applied pressure and electrical pulse current parameters.

**3:15 PM**

#### (ICCPs-120-2013) Ceramic Sintering and Shaping Using the Spark Plasma Sintering Method

A. K. Mukherjee\*, University of California, USA

The consolidation of ceramic powder particles in this investigation was achieved by electrical field assisted sintering with capability of superimposing GPa level gas pressure. The nanomaterials derived from crystallization of bulk metallic glass demonstrated extreme strength and at elevated temperatures showed superplasticity-like ductility. A three-phase alumina based nanoceramic composite demonstrated superplasticity at a lower temperature and at a higher strain rate. An alumina-carbon nanotube-niobium nanocomposite has a fracture toughness that is five times higher than that of pure alumina and an electrical conductivity that is thirteen orders of magnitude greater than that of pure nanocrystalline alumina. It also has excellent potential for use as a thermoelectric material. An alumina-spinel nanocomposite demonstrated optical transparency in the mid infrared range. A silicon carbide/silicon nitride nanocomposite produced by pyrolysis of liquid polymer precursor has pro-



duced one of the lowest creep rates in ceramics at a referred temperature of 1400 degrees C. These structural and functional properties will be discussed in the context of microstructural observations.

**3:30 PM**

**(ICCPs-122-2013) Fabrication and Mechanical Properties of Nano-SiC/CNT Composites Sintered by SPS**

B. Lanfant\*, Y. Leconte, M. Pinault, M. Mayne-L'hermite, N. Herlin-Boime, CEA Saclay, France; G. Bonnefont, S. De Bernardi, G. Fantozzi, V. Garnier, Y. Jorand, INSA Lyon, France; T. Pham, S. Le Gallet, F. Bernard, Université de Bourgogne, France

Ceramic carbides materials such as SiC are believed to be promising candidates for high temperature nuclear or aerospace applications. However, SiC brittleness has limited these applications. A reduction of grain size accompanied by a high final density seems to be the solutions for improving mechanical properties. Sintering additives are usually employed in order to achieve high densities and fine grain sizes, although they present drawbacks under irradiation. In order to avoid these problems, no sintering additives were used in this study. The counterpart of reducing the grain size is the drastic decrease of the thermal conductivity due to the phonon scattering at the grain boundary. With the aim of reducing this effect, multiwall carbon nanotubes (MWCNTs) were introduced into the SiC matrix in order to improve thermal conductivity. For this study  $\beta$ -SiC nanopowders ( $\varnothing$ : 20 nm) produced by laser pyrolysis were employed. MWCNTs (L: 400-680  $\mu$ m) were synthesized by aerosol assisted catalytic CVD. Deagglomeration of the powder has been performed in an aqueous medium under magnetic stirring. Subsequently, green bodies were prepared by slip-casting. Then Spark Plasma Sintering process was used to achieve high final densities avoiding an exaggerated grain growth. Finally, samples were subjected to mechanical characterization in order to correlate the final microstructures to the mechanical behavior.

**3:45 PM**

**(ICCPs-123-2013) Densification and Phase Evolution of SHS Derived  $Ti_2AlC$  Powders in Pressureless Sintering and Hot Pressing Processes**

L. Chlubny\*, J. Lis, AGH University of Science and Technology, Poland

$Ti_2AlC$  belongs to the interesting group of ternary nanolaminate materials, characterised by heterodesmic chemical bonding and thus

possessing unique set of properties locating them between metals and ceramics. One of the best methods of obtaining sinterable powders of this materials is Self-propagating High-temperature Synthesis (SHS), which is effective and efficient. At the basis of this method lays utilization of exothermal effect of the chemical reaction for initiation of the self-sustaining process. As a result of the SHS synthesis of elementary powders of Ti, Al and C in stoichiometric proportions, powder containing mostly  $Ti_2AlC$  and  $Ti_3AlC_2$  was obtained. This powder was examined for potential sintering possibilities both with pressureless sintering and hot-pressing techniques in various conditions. Changes in the phase compositions of the materials were observed and optimal sintering conditions were established.

**4:00 PM**

**(ICCPs-124-2013) Densification Behavior of MnO-doped  $UO_2$  during Low Temperature Sintering**

Y. Rhee\*, I. Nam, D. Kim, J. Oh, J. Kim, K. Kim, J. Yang, Y. Koo, Korea Atomic Energy Research Institute, Republic of Korea

A new sintering additive, MnO, was used to reduce the sintering temperature of  $UO_2$  in  $H_2$  atmosphere. Most of LWR fuel vendors have fabricated nuclear fuel pellets by sintering  $UO_2$  compacts at high temperatures above 1700 °C in  $H_2$  atmosphere. Over decades, a lot of attempts were made on lowering the sintering temperature of  $UO_2$ . However, those efforts were based on the improved sinterability in hyperstoichiometric  $UO_{2+x}$ , which was obtained by mixing  $U_3O_8$  with  $UO_2$  and sintering at inert or slightly oxidative atmosphere, such as Ar,  $N_2$ ,  $CO_2$ . It is known that a large degree of swelling may result from the entrapped large-sized gases such as  $CO_2$ , Ar and  $N_2$  in the sintered  $UO_2$  pellet during irradiation in a nuclear reactor. We have tried to lower the sintering temperature of  $UO_2$  under the commercially-used  $H_2$  atmosphere by using sintering additives. Among them, MnO can lower the sintering temperature down to about 1250 °C. The densification of  $UO_2$  is significantly enhanced by adding a small amount of MnO, less than 0.2 wt%. MnO-doped  $UO_2$  can be densified up to about 95 % of the theoretical density through sintering at 1250 °C in  $H_2$  atmosphere. This paper deals with preliminary results on the densification behavior and microstructural evolution of MnO-doped  $UO_2$  during low temperature sintering in  $H_2$  atmosphere.

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