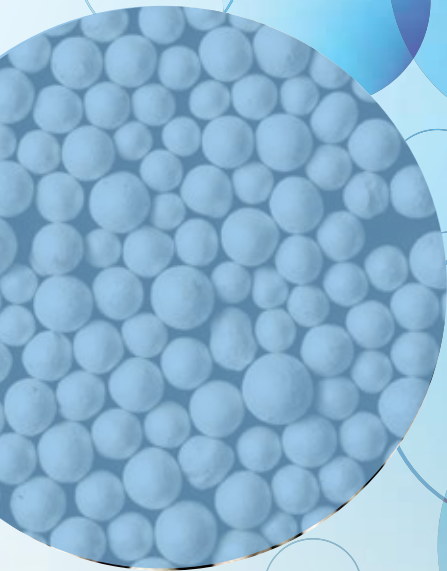


# CONFERENCE PROGRAM



INTERNATIONAL CONFERENCE ON

# SINTERING 2017

*Latest Advances in Science and Technology of Sintering and Microstructure Evolution*

November 12-16, 2017

HYATT REGENCY MISSION BAY SPA AND MARINA | SAN DIEGO, CALIFORNIA

[ceramics.org/sintering2017](http://ceramics.org/sintering2017)

# Optimizing my Sintering Process



OPTIMIZED SINTERING  
PROCESS BY USING  
**KINETIC SIMULATION**



DETERMINATION OF  
**INFLUENCING  
PROPERTIES**

THERMOGRAVIMETRY:  
**BINDER BURNOUT**

DILATOMETRY:  
**THERMAL SHRINKAGE  
AND EXPANSION**

LASER FLASH:  
**THERMAL  
CONDUCTIVITY**

DIFFERENTIAL SCANNING  
CALORIMETRY:  
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# WELCOME

Welcome to the International Conference Sintering 2017 – an international conference and a world-wide forum on sintering science and technology. This will be the eighth meeting in a series that started in 1995 as a continuation of a famous cycle of conferences on Sintering and Related Phenomena organized in US in the middle of 20th century. The previous seven conferences have been held in Pennsylvania State University, USA (1995, 1999, 2003); Grenoble, France (2005); La Jolla, USA (2008); Jeju, Korea (2011); and Dresden, Germany (2014).

The technical program will cover all aspects of sintering of all classes of powder materials through invited and contributed talks, poster presentations, and special programs designed to facilitate discussion among participants. Sintering 2017 will continue and extend the tradition of being an outstanding opportunity to meet, exchange ideas and knowledge, and enjoy the interaction with like-minded sintering experts. We thank our sponsors and the support of the ACerS San Diego chapter.

The American Ceramic Society thanks you for participating in this meeting. If you need assistance, there will be ACerS staff to help you. We hope you have a stimulating and memorable experience in San Diego.

## Conference Co-Chairs



**Rajendra K. Bordia**  
Clemson University, USA



**Eugene A. Olevsky**  
San Diego State University, USA



**Didier Bouvard**  
University of Grenoble, France



**Suk-Joong. L. Kang**  
Korea Institute of Ceramic Engineering and Technology, Korea  
Advanced Institute of Science and Technology, Republic of Korea



**Bernd Kieback**  
Fraunhofer Institute for Manufacturing Technology and Advanced Materials, Germany

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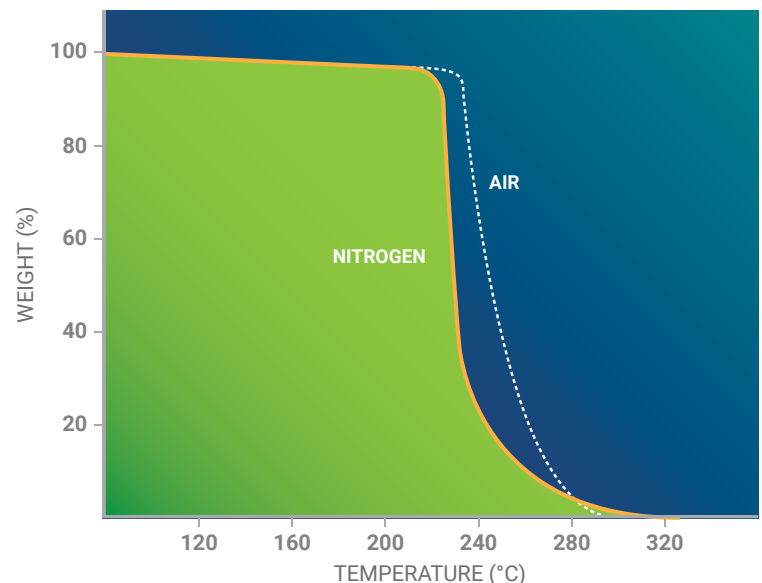
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# OPENING REMARKS AND PLENARY SESSION

## Plenary session I

Monday, November 13 | 8:30 – 9:45 a.m. | Bayview II/III



**Martin Harmer**, Lehigh University,  
Bethlehem, Pennsylvania, USA

Title: ***Know your boundaries***

## Plenary session II

Tuesday, November 14 | 8:30 – 9:30 a.m. | Bayview II/III



**Bernd Kieback**, Fraunhofer-Institut  
für Fertigungstechnik und Ange-  
wandte Materialforschung IFAM,  
Institutsteil Dresden, Germany

Title: ***Contact formation and densi-  
fication during early stages of spark  
plasma sintering of metal powders***

## Plenary session III

Wednesday, November 15 | 8:30 – 9:30 a.m. | Bayview II/III



**Didier Bouvard**, University Grenoble  
Alpes, France

Title: ***Investigating the sintering of  
multilayer components with  
advanced experimental and  
modelling tools***

## SPECIAL EVENTS

### Poster session set-up

Monday, November 13 | 10 a.m. – 4 p.m.  
Regatta Pavilion

### Welcome and poster session reception

(Co-sponsored by the ACerS San Diego section )

Monday, November 13 | 5:30 – 7:30 p.m.  
Bayview II/III

### San Diego by Land and Sea Tour

(included with full regular conference registration) Box  
lunch available for purchase prior to tour.

Tuesday, November 14 | 1 – 5:30 p.m.

### Dinner

Wednesday, November 15 | 7 – 9 p.m.  
Bayview I, II & III

### San Diego Section Sponsored

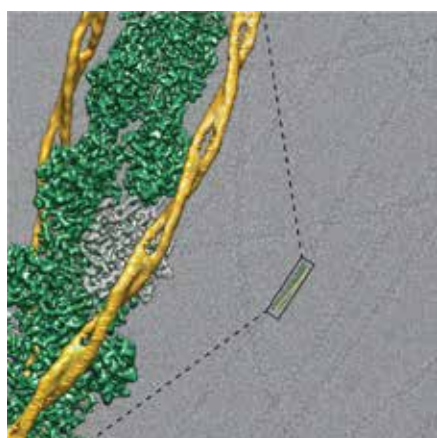
(Tabletop exhibitors)

- Accu-Tech Laser Processing
- AEM, Inc.
- Ferro Corporation
- Materion Ceramics
- Oasis Materials Company LP

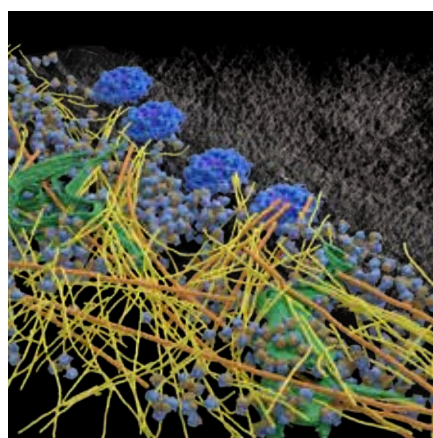


# Discover Life-changing Answers Faster

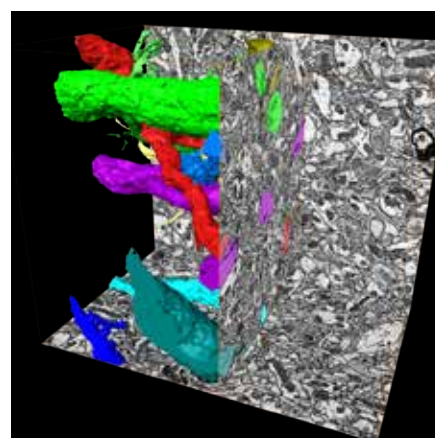
Three workflows to reveal the ultimate structural details



Courtesy of Julian von der Ecken and Prof. Dr. Stefan Raunser, Max Planck Institute of Molecular Physiology, Dortmund, Germany.



Courtesy of Dr. J. Mahamid, Max Planck Institute of Biochemistry, Martinsried, Germany.



Courtesy of Dr. P. Laserstein, Max Planck Institute, Brain Research, Germany.

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# SCHEDULE AT A GLANCE

## SUNDAY, NOVEMBER 12, 2017

Registration	5 p.m. – 7 p.m.	Bayview Foyer
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## MONDAY, NOVEMBER 13, 2017

Registration	7:30 a.m. – 6 p.m.	Bayview Foyer
Plenary session I	8:30 a.m. – 10 a.m.	Bayview II/III
Concurrent technical sessions	10 a.m. – 5 p.m.	Bayview III, Mission I, II & III
Poster session set-up	10 a.m. – 4 p.m.	Regatta Pavilion
Lunch	12:20 p.m. – 1:50 p.m.	Regatta Pavilion & Bayview Terrace
Welcome and poster session reception (Co-sponsored by the ACerS San Diego section)	5:30 p.m. – 7:30 p.m.	Regatta Pavilion

## TUESDAY, NOVEMBER 14, 2017

Registration	7:30 a.m. – 12 p.m.	Bayview Foyer
Plenary session II	8:30 a.m. – 10 a.m.	Bayview II/III
Concurrent technical sessions	10 a.m. – 12:20 p.m.	Bayview I, Mission I, II & III
San Diego by Land and Sea Tour (included with full regular conference registration) Box lunch available for purchase prior to tour.	1 p.m. – 5:30 p.m.	

## WEDNESDAY, NOVEMBER 15, 2017

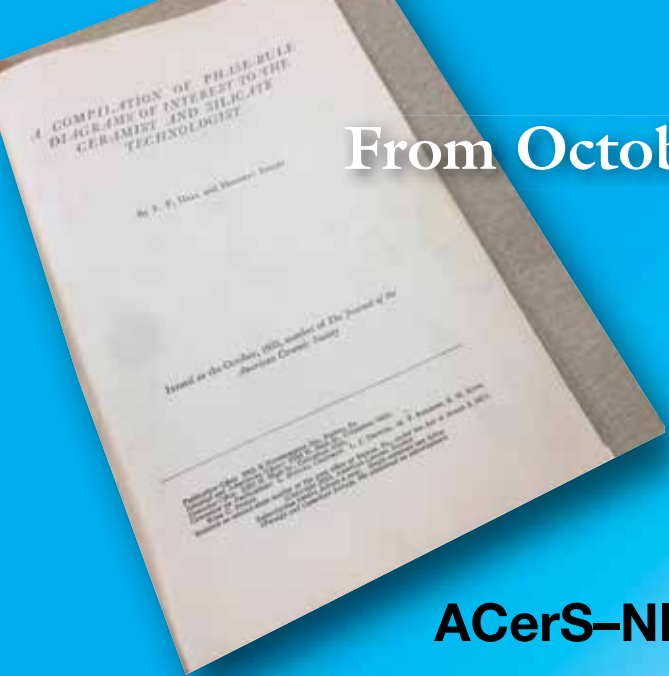
Registration	7:30 a.m. – 6 p.m.	Bayview Foyer
Plenary session III	8:30 a.m. – 10 a.m.	Bayview II/III
Concurrent technical sessions	10 a.m. – 5 p.m.	Bayview I, Mission I, II & III
Lunch	12:20 p.m. – 1:50 p.m.	Regatta Pavilion & Bayview Terrace
Dinner	7 p.m. – 9 p.m.	Bayview I, II & III

## THURSDAY, NOVEMBER 16, 2017

Registration	7:30 a.m. – 12 p.m.	Bayview Foyer
Concurrent technical sessions	8:30 a.m. – 12 p.m.	Bayview I, Mission I, II & III, Palm

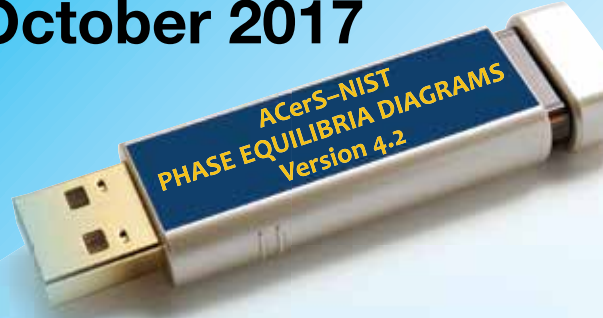






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Organized by the ACerS Electronics and Basic Science Divisions

January 17 – 19, 2018 | DoubleTree by Hilton Orlando at Sea World Conference Hotel | Orlando, Fla., USA

Electronics Materials and Applications is now the  
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Scan for EAM 18 updates.

The 2018 meeting has expanded programming, and is organized by  
ACerS Electronics and Basic Science Divisions.



Oral Presenters

Name	Date	Time	Room	Page Number	Name	Date	Time	Room	Page Number
<b>A</b>									
Abdeljawad, F.	14-Nov	11:20AM	Mission II	11	Giuntini, D.	13-Nov	2:40PM	Mission I	7
Adamek, G.	13-Nov	4:00PM	Bayview III	6	Giuntini, D.	13-Nov	3:40PM	Bayview III	6
Akinc, M.	13-Nov	10:40AM	Bayview II	4	Gordo, E.	13-Nov	4:00PM	Mission III	8
Aminirastabi, H.	16-Nov	10:10AM	Bayview I	16	Gorynski, C.	15-Nov	11:40AM	Bayview II	12
Anderson, I.E.	14-Nov	10:00AM	Mission III	11	Graeve, O.	13-Nov	11:20AM	Bayview II	4
Araujo, H.E.	16-Nov	10:50AM	Bayview I	16	Grimley, C.	14-Nov	10:40AM	Mission II	11
Araujo, H.E.	16-Nov	11:10AM	Mission I	17	Guillon, O.	15-Nov	2:00PM	Bayview III	14
Atre, S.V.	15-Nov	2:00PM	Mission III	15	Guo, J.	15-Nov	11:20AM	Mission II	13
<b>B</b>					<b>H</b>				
Bakshi, S.R.	14-Nov	10:20AM	Mission II	11	Haines, C.	13-Nov	10:40AM	Bayview I	4
Bakshi, S.R.	15-Nov	11:20AM	Mission III	13	Hara, S.	16-Nov	10:10AM	Mission I	17
Bakshi, S.R.	15-Nov	12:00PM	Bayview III	13	Harmer, M.P.	13-Nov	8:45AM	Bayview I	4
Bayode, B.L.	16-Nov	10:50AM	Mission I	17	Hayun, S.	15-Nov	2:00PM	Bayview I	14
Behler, K.D.	13-Nov	10:40AM	Mission II	5	Heian, E.M.	15-Nov	2:00PM	Mission I	15
Bjork, R.	15-Nov	2:00PM	Bayview II	14	Henniche, A.	13-Nov	4:20PM	Bayview III	7
Blaine, D.C.	13-Nov	3:00PM	Mission III	7	Hoffmann, M.J.	15-Nov	10:00AM	Bayview I	12
Blaine, D.C.	13-Nov	4:20PM	Bayview II	6	Hryha, E.	15-Nov	11:00AM	Mission III	13
Bordia, R.	16-Nov	9:30AM	Mission III	18	Hunt, A.	13-Nov	11:00AM	Bayview III	4
Borukhovich, E.	14-Nov	10:40AM	Mission I	11	Hunt, A.	15-Nov	3:00PM	Bayview III	14
Bose, A.	14-Nov	10:40AM	Mission III	11	<b>J</b>				
Bouvard, D.	15-Nov	8:30AM	Bayview I	12	Janssen, R.	13-Nov	2:00PM	Bayview III	6
Bouvard, D.	15-Nov	2:40PM	Mission III	16	Janssen, R.	13-Nov	4:20PM	Bayview I	6
Brennan, R.E.	13-Nov	2:40PM	Bayview I	6	Janssen, R.	15-Nov	5:00PM	Bayview II	14
<b>C</b>					Janssen, R.	16-Nov	10:30AM	Mission II	16
Camacho Montes, H.	13-Nov	11:40AM	Mission II	5	Jauffres, D.	13-Nov	2:00PM	Mission I	7
Camacho Montes, H.	15-Nov	12:00PM	Mission II	13	Jayaram, V.	13-Nov	10:00AM	Mission II	5
Campos, M.	13-Nov	4:20PM	Mission III	8	Jha, S.K.	15-Nov	3:00PM	Mission I	15
Campos, M.	14-Nov	10:40AM	Bayview III	10	Johnson, J.L.	13-Nov	3:40PM	Mission III	8
Carazzone, R.	15-Nov	3:00PM	Mission III	16	<b>K</b>				
Carvalho, S.G.	16-Nov	8:30AM	Palm Ballroom	17	Kang, S.L.	13-Nov	11:00AM	Mission III	5
Castro, R.H.	14-Nov	10:00AM	Mission I	11	Kang, S.L.	14-Nov	11:00AM	Bayview II	10
Cetinkaya, Z.	13-Nov	12:00PM	Bayview III	4	Kaplan, W.D.	14-Nov	10:40AM	Bayview II	10
Chaim, R.	13-Nov	3:40PM	Bayview I	6	Kieback, B.	14-Nov	8:30AM	Bayview I	10
Chaix, J.	13-Nov	3:40PM	Mission II	7	Kim, Y.	15-Nov	10:00AM	Bayview III	12
Charalambous, H.	13-Nov	4:40PM	Bayview I	6	Kiribiyik, F.	16-Nov	10:10AM	Palm Ballroom	17
Chateau-Cornu, J.	16-Nov	9:10AM	Mission III	18	Kodera, Y.	15-Nov	3:40PM	Bayview I	14
Cramer, C.L.	16-Nov	9:30AM	Bayview I	16	Kok, D.	15-Nov	4:00PM	Bayview I	14
<b>D</b>					Konyashin, I.	16-Nov	8:30AM	Mission II	16
Dancer, C.E.	15-Nov	10:40AM	Bayview II	12	<b>L</b>				
Danninger, H.	15-Nov	10:00AM	Mission III	13	Langa, T.G.	16-Nov	9:30AM	Palm Ballroom	17
Dawes, M.J.	16-Nov	10:30AM	Bayview I	16	Lebrun, J.	14-Nov	12:00PM	Bayview I	10
Delannay, F.	14-Nov	11:00AM	Mission I	11	Lee, C.S.	15-Nov	2:00PM	Mission II	15
Delannay, F.	14-Nov	11:20AM	Mission I	11	Lee, G.	14-Nov	11:20AM	Bayview I	10
Diegeler, A.	15-Nov	3:40PM	Mission I	15	Lee, J.	14-Nov	10:00AM	Bayview III	10
Diwald, O.	13-Nov	2:40PM	Bayview III	6	Liang, B.	13-Nov	3:00PM	Mission II	7
Diwald, O.	15-Nov	4:00PM	Mission II	15	Liu, J.	15-Nov	4:00PM	Mission I	15
Douse, E.	16-Nov	10:10AM	Mission II	16	Liu, X.	14-Nov	11:20AM	Bayview II	10
Drazin, J.	13-Nov	10:40AM	Bayview III	4	Liu, Y.	13-Nov	2:40PM	Bayview II	6
Ducoulombier, A.	15-Nov	11:20AM	Mission I	13	Locci, A.M.	13-Nov	4:40PM	Mission I	7
<b>E</b>					Lu, K.	13-Nov	11:40AM	Mission III	5
Engelke, L.	13-Nov	4:20PM	Mission I	7	Luo, J.	13-Nov	2:00PM	Bayview I	5
Erauw, J.	15-Nov	12:00PM	Bayview I	12	<b>M</b>				
Estournes, C.	14-Nov	10:40AM	Bayview I	10	Maca, K.	15-Nov	4:00PM	Bayview III	15
<b>F</b>					Mackert, V.	14-Nov	10:00AM	Mission II	11
Fang, Z.Z.	13-Nov	12:00PM	Mission III	5	Manchili, S.	14-Nov	11:20AM	Bayview III	11
Feigelson, B.N.	13-Nov	3:00PM	Bayview III	6	Maniere, C.	15-Nov	3:40PM	Bayview II	14
Frueh, T.	15-Nov	11:00AM	Mission I	13	Maniere, C.	16-Nov	9:10AM	Mission II	16
Fry, A.L.	15-Nov	10:40AM	Bayview III	12	Marchionna, S.	16-Nov	9:30AM	Mission I	17
Fuierer, P.	15-Nov	4:20PM	Bayview III	15	Maximenko, A.	13-Nov	11:40AM	Bayview I	4
Furlan, K.	13-Nov	2:20PM	Bayview II	6	McInerney, T.A.	16-Nov	9:10AM	Mission I	17
<b>G</b>					Mecartney, M.	15-Nov	10:40AM	Mission II	13
Galusek, D.	15-Nov	10:00AM	Bayview II	12	Messing, G.L.	15-Nov	2:40PM	Bayview III	14
Garay, J.E.	15-Nov	10:40AM	Bayview I	12	Mieller, B.	16-Nov	8:30AM	Mission III	18
German, R.M.	13-Nov	10:00AM	Mission III	5	Misiolek, W.Z.	14-Nov	10:20AM	Mission III	11
German, R.M.	15-Nov	10:40AM	Mission I	13	Missiaen, J.	13-Nov	10:40AM	Mission I	5
					Missiaen, J.	15-Nov	10:40AM	Mission III	13
					Mitic, V.	14-Nov	11:40AM	Mission I	11





## Poster Presenters

<u>Name</u>	<u>Date</u>	<u>Time</u>	<u>Room</u>	<u>Page Number</u>	<u>Name</u>	<u>Date</u>	<u>Time</u>	<u>Room</u>	<u>Page Number</u>
					<b>B</b>				
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Bouvard, D.	13-Nov	5:30PM	Regatta Pavilion	9	Mitic, V.	13-Nov	5:30PM	Regatta Pavilion	9
					<b>C</b>				
Camacho Montes, H.	13-Nov	5:30PM	Regatta Pavilion	8, 9	Na, Y.	13-Nov	5:30PM	Regatta Pavilion	9
Chateau-Cornu, J.	13-Nov	5:30PM	Regatta Pavilion	9	Nefedova, E.	13-Nov	5:30PM	Regatta Pavilion	9
Christiansen, C.D.	13-Nov	5:30PM	Regatta Pavilion	9					
					<b>D</b>				
dos Reis, S.L.	13-Nov	5:30PM	Regatta Pavilion	9	Pereira, G.J.	13-Nov	5:30PM	Regatta Pavilion	8
					<b>F</b>				
Fuda, K.	13-Nov	5:30PM	Regatta Pavilion	8	Post, E.	13-Nov	5:30PM	Regatta Pavilion	9
					<b>G</b>				
Gao, L.	13-Nov	5:30PM	Regatta Pavilion	8	Rubinkovskiy, N.A.	13-Nov	5:30PM	Regatta Pavilion	8
Grosso, R.L.	13-Nov	5:30PM	Regatta Pavilion	8					
Guillemet-Fritsch, S.	13-Nov	5:30PM	Regatta Pavilion	9	<b>S</b>				
					<b>H</b>				
Hijji, H.H.	13-Nov	5:30PM	Regatta Pavilion	8	Salem Cherif, S.	13-Nov	5:30PM	Regatta Pavilion	9
Huang, Y.	13-Nov	5:30PM	Regatta Pavilion	9	Sarma, H.	13-Nov	5:30PM	Regatta Pavilion	9
					<b>J</b>				
Jung, T.	13-Nov	5:30PM	Regatta Pavilion	9	Senos, A.M.	13-Nov	5:30PM	Regatta Pavilion	9
					<b>K</b>				
Kanchika, S.	13-Nov	5:30PM	Regatta Pavilion	9	Shoulders, W.	13-Nov	5:30PM	Regatta Pavilion	10
Kang, M.	13-Nov	5:30PM	Regatta Pavilion	9	Silva, R.S.	13-Nov	5:30PM	Regatta Pavilion	8
Kim, S.	13-Nov	5:30PM	Regatta Pavilion	8	Song, X.	13-Nov	5:30PM	Regatta Pavilion	8
					<b>L</b>				
Lange, A.	13-Nov	5:30PM	Regatta Pavilion	9					
Liebenberg, D.	13-Nov	5:30PM	Regatta Pavilion	8	<b>T</b>				
Liu, J.	13-Nov	5:30PM	Regatta Pavilion	8	Trapp, J.	13-Nov	5:30PM	Regatta Pavilion	8
Liu, Y.	13-Nov	5:30PM	Regatta Pavilion	9					
Luster, A.M.	13-Nov	5:30PM	Regatta Pavilion	9	<b>V</b>				
					<b>Y</b>				
					<b>Z</b>				
					Valdivieso, F.	13-Nov	5:30PM	Regatta Pavilion	8
					Yamada, K.	13-Nov	5:30PM	Regatta Pavilion	8
					Yu, J.	13-Nov	5:30PM	Regatta Pavilion	10
					Zhang, P.	13-Nov	5:30PM	Regatta Pavilion	8

## Monday, November 13, 2017

### Plenary Session I

Room: Bayview I

Session Chairs: Rajendra Bordia, Clemson University; Eugene Olevsky, San Diego State University

**8:30 AM**

#### Opening Remarks

**8:45 AM**

#### (SINT-PL-001-2017) Know your boundaries (Invited)

M. P. Harmer\*<sup>1</sup>

1. Lehigh University, Materials Science and Engineering, USA

**9:45 AM**

#### Break

### Spark-plasma and Flash Sintering I

Room: Bayview I

Session Chair: Eugene Olevsky, San Diego State University

**10:00 AM**

#### (SINT-001-2017) The mechanisms of field assisted sintering/spark plasma sintering of metallic materials on a microscopic scale (Invited)

J. Trapp\*<sup>1</sup>; M. Nöthe<sup>1</sup>; B. Kieback<sup>2</sup>; A. Semenov<sup>3</sup>; O. Eberhardt<sup>3</sup>; T. Wallmersperger<sup>3</sup>

1. Technische Universität Dresden, Materials Science, Germany
2. Fraunhofer IFAM, Germany
3. Technische Universität Dresden, Institute of Solid Mechanics, Germany

**10:40 AM**

#### (SINT-002-2017) Two-step, field assisted sintering (Invited)

C. Haines\*<sup>1</sup>; D. G. Martin<sup>1</sup>; K. C. Mills<sup>1</sup>

1. US Army ARDEC, Armaments Analysis & Manufacturing Directorate, USA

**11:20 AM**

#### (SINT-003-2017) Spark plasma sintering microscopic mechanisms of metallic systems: Experiments and simulations

J. Monchoux\*<sup>1</sup>; Z. Trzaska<sup>1</sup>; C. Collard<sup>1</sup>; A. Couret<sup>1</sup>; G. Bonnefont<sup>2</sup>; G. Fantozzi<sup>2</sup>

1. CEMES-CNRS UPR 8011, France
2. MATEIS-INSA Lyon, France

**11:40 AM**

#### (SINT-004-2017) Multiscale modeling of spark plasma copper powder sintering in multi-step loading regime (Invited)

A. Maximenko\*<sup>1</sup>; E. Olevsky<sup>2</sup>

1. Institute for Problems in Materials Science NAS Ukraine, Ukraine
2. San Diego State University, USA

### Microstructural Evolution in Sintering Processes I

Room: Bayview II

Session Chairs: Klaus van Benthem, University of California, Davis; Diletta Giuntini, Hamburg University of Technology (TUHH)

**10:00 AM**

#### (SINT-005-2017) Sintering pure metals: Are there really indications for defect-activated sintering? (Invited)

T. E. Staab\*<sup>1</sup>

1. University Wuerzburg, Dep. of Chemistry - LCTM, Germany

**10:40 AM**

#### (SINT-006-2017) Pressureless sintering of Mo-Si-B alloys with iron additions

M. Akinc\*<sup>1</sup>; G. Ouyang<sup>1</sup>; P. Ray<sup>2</sup>; M. J. Kramer<sup>2</sup>

1. Iowa State University, Materials Science & Engineering and Chemical & Biological Engineering, USA
2. Ames Laboratory, USA

**11:00 AM**

#### (SINT-007-2017) Densification behavior and microstructure of $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$ fabricated by spark plasma sintering

Y. Zeng\*<sup>1</sup>; X. Xiong<sup>1</sup>; Y. Wang<sup>1</sup>; Z. Chen<sup>1</sup>; W. Sun<sup>1</sup>

1. State Key Laboratory of Powder Metallurgy, China

**11:20 AM**

#### (SINT-008-2017) Phase stability and nano-domain formation in alkaline-earth hexaborides (Invited)

O. Graeve\*<sup>1</sup>; J. Cahill<sup>1</sup>; M. Alberga<sup>2</sup>; D. Edwards<sup>2</sup>; S. T. Misture<sup>2</sup>; V. Vasquez<sup>3</sup>

1. University of California, San Diego, Mechanical and Aerospace Engineering, USA
2. Alfred University, Kazuo Inamori School of Engineering, USA
3. University of Nevada, Reno, Chemical and Materials Engineering, USA

### Sintering of Nanostructured Materials I

Room: Bayview III

Session Chairs: Jean-Marie Lebrun, Saint-Gobain; Rasmus Bjørk, Technical University of Denmark

**10:00 AM**

#### (SINT-009-2017) Sintering densification of in situ synthesized WC-Co based composite powders (Invited)

X. Song\*<sup>1</sup>; X. Liu<sup>1</sup>; H. Wang<sup>1</sup>; X. Liu<sup>1</sup>; Z. Nie<sup>1</sup>

1. Beijing University of Technology, College of Materials Science and Engineering, China

**10:40 AM**

#### (SINT-010-2017) Cold isostatic pressing and low temperature sintering of nanograined ceramics

J. Drazin\*<sup>2</sup>; E. Gorzkowski<sup>1</sup>

1. US Naval Research Laboratory, USA
2. ASEE Postdoc at US Naval Research Laboratory, USA

**11:00 AM**

#### (SINT-011-2017) Sintered ceramic wire coatings as electrical insulation for high-field superconducting magnets

M. White<sup>1</sup>; R. Nesbit<sup>1</sup>; A. Hunt\*<sup>1</sup>

1. nGimat LLC, USA

**11:20 AM**

#### (SINT-012-2017) Dilatometry analysis of the sintering process of gadolinia-doped ceria

E. N. Muccillo\*<sup>1</sup>; R. M. Batista<sup>1</sup>

1. Energy and Nuclear Research Institute, Brazil

**11:40 AM**

#### (SINT-013-2017) Nanostructured $Li,La_3Zr_2O_{12}$ solid electrolytes with improved sinterability

J. M. Weller\*<sup>1</sup>; C. K. Chan<sup>1</sup>

1. Arizona State University, Materials Science and Engineering, USA

**12:00 PM**

#### (SINT-014-2017) Optimization of sintering temperature for electrospun red mud based nanofibers

Z. Cetinkaya\*<sup>1</sup>; V. Kalem<sup>1</sup>

1. Selcuk University, Metallurgy and Materials Engineering, Turkey

### Fundamental Aspects of Sintering I

Room: Mission I

Session Chairs: Francis Delannay, Université catholique de Louvain; David Jauffres, Univ. Grenoble Alpes

**10:00 AM**

#### (SINT-015-2017) Effect of geometry and green density on the anisotropic sintering shrinkage of axisymmetric iron parts (Invited)

A. Molinari\*<sup>1</sup>

1. University of Trento, Industrial Engineering, Italy

**10:40 AM****(SINT-016-2017) Effect of contact alignment on anisotropic shrinkage during sintering: Discrete element simulations, analytical models and experiments**B. Hugonnet<sup>2</sup>; J. Missiaen<sup>\*1</sup>; C. Martin<sup>3</sup>; C. Rado<sup>2</sup>

1. University of Grenoble Alpes, Grenoble INP, France
2. CEA, Liten, France
3. University of Grenoble Alpes, CNRS, France

**11:00 AM****(SINT-017-2017) Multi-scale anisotropy phenomena during sintering of iron powders**E. Torresani<sup>\*2</sup>; D. Giuntini<sup>1</sup>; C. Zhu<sup>3</sup>; T. Harrington<sup>3</sup>; K. S. Vecchio<sup>3</sup>; A. Molinari<sup>2</sup>; R. Bordia<sup>4</sup>; E. Olefsky<sup>1</sup>

1. San Diego State University, Dept. Mechanical Engineering, USA
2. University of Trento, Department of Industrial Engineering, Italy
3. University of California, San Diego, USA
4. Clemson University, USA

**11:20 AM****(SINT-018-2017) Kinetics modelling and optimization of sintering processes**E. Moukhina<sup>\*1</sup>; G. Kaiser<sup>1</sup>; D. Rapp<sup>1</sup>

1. NETZSCH Geraetebau GmbH, Germany

**11:40 AM****(SINT-019-2017) Application and sintering of porous metal fiber materials (Invited)**H. Tang<sup>\*1</sup>; J. Wang<sup>1</sup>; A. Li<sup>1</sup>; J. Ma<sup>1</sup>

1. State Key Laboratory of Porous Metal Materials, China

**Stress-assisted Sintering I**

Room: Mission II

Session Chairs: Hyo Tae Kim, Korea Institute of Ceramic Engineering and Technology (KICET); Xialu Wei, San Diego State University; Michael Hoffmann, Karlsruhe Institute of Technology

**10:00 AM****(SINT-020-2017) Reactive densification of ZrC and TiC (Invited)**V. Jayaram<sup>\*1</sup>

1. Indian Institute of Science, Materials Engineering, India

**10:40 AM****(SINT-021-2017) Effect of temperature, pressure, time, and sintering aid content on the densification and microstructure of hot-pressed boron suboxide**K. D. Behler<sup>\*1</sup>; J. LaSalvia<sup>2</sup>; C. J. Marvel<sup>3</sup>; M. C. Golt<sup>4</sup>; S. D. Walck<sup>4</sup>; M. P. Harmer<sup>2</sup>

1. U.S. Army Research Lab (SURVICE Engineering), Ceramics and Transparent Materials Branch, USA
2. U.S. Army Research Lab, Ceramics and Transparent Materials Branch, USA
3. Lehigh University, Department of Materials Science and Engineering, Center for Advanced Materials and Nanotechnology, USA
4. U.S. Army Research Lab, Coatings Corrosion and Engineered Polymers Branch, USA

**11:00 AM****(SINT-022-2017) Ultra high pressure sintering and synthesis of transparent polycrystalline cubic silicon nitride**N. Nishiyama<sup>\*1</sup>; R. Ishikawa<sup>2</sup>; H. Ohfuji<sup>3</sup>; H. Marquardt<sup>4</sup>; T. Taniguchi<sup>5</sup>; B. Kim<sup>5</sup>; A. Masuno<sup>6</sup>; Y. Ikuhara<sup>2</sup>; F. Wakai<sup>1</sup>; T. Irifune<sup>2</sup>

1. Tokyo Institute of Technology, Japan
2. University of Tokyo, Japan
3. Ehime University, Japan
4. University of Bayreuth, Germany
5. National Institute for Materials Science (NIMS), Japan
6. Hirotsuki University, Japan

**11:20 AM****(SINT-023-2017) Effect of sintering additives on microstructure and properties of hot-pressed nitride composites**T. Tabares Medina<sup>\*1</sup>; A. Maître<sup>1</sup>; N. Pradeilles<sup>1</sup>; M. Joinet<sup>2</sup>; P. Ponnard<sup>2</sup>

1. European Centre of Ceramic, SPCTS, France
2. Thales Electron Devices, France

**11:40 AM****(SINT-024-2017) Comparison of the constitutive laws for stress-assisted densification powder compact**H. Camacho Montes<sup>\*1</sup>; A. Garcia Reyes<sup>2</sup>; R. Bordia<sup>3</sup>

1. Universidad Autonoma de Ciudad Juarez, Fisica y Matematicas, Mexico
2. InterCeramic Technological Center, Mexico
3. Clemson University, USA

**12:00 PM****(SINT-025-2017) Grain growth studies and electrical response of stress assisted sintered BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub>**A. M. Senos<sup>\*1</sup>; M. Fernandes<sup>1</sup>; N. Selvaraj<sup>1</sup>; J. Abrantes<sup>2</sup>; J. Noudem<sup>3</sup>; O. Guillon<sup>4</sup>; P. Vilarinho<sup>1</sup>

1. University of Aveiro/ CICECO, Materials and Ceramic Engineering, Portugal
2. Instituto Politécnico de Viana do Castelo, UIDM, ESTG, Portugal
3. Université de Caen Basse-Normandie, CRISMAT, France
4. Forschungszentrum Jülich GmbH Institut für Energie und Klimaforschung, IEK-1: Werkstoffsynthese und Herstellungsverfahren, Germany

**Randall German Honorary Symposium I**

Room: Mission III

Session Chairs: Kathy Lu, Virginia Tech; José Manuel Torralba, Universidad Carlos III de Madrid

**10:00 AM****(SINT-026-2017) Statistics and stories on sintering (Invited)**R. M. German<sup>\*1</sup>

1. San Diego State University, Dept of Engineering, USA

**10:40 AM****(SINT-027-2017) Ceramics sintering and shaping using the electrical field assisted sintering method (Invited)**T. B. Holland<sup>1</sup>; H. Wang<sup>2</sup>; A. K. Mukherjee<sup>\*3</sup>

1. Colorado State University, Mechanical Engineering, USA
2. Purdue University, Materials Science and Engineering, USA
3. University of California, Davis, Materials Science and Engineering, USA

**11:00 AM****(SINT-028-2017) Strategies and practices for suppressing abnormal grain growth during liquid phase sintering (Invited)**S. L. Kang<sup>\*1</sup>

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

**11:20 AM****(SINT-029-2017) Challenges and further developments in modeling of sintering (Invited)**E. Olefsky<sup>\*1</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA

**11:40 AM****(SINT-030-2017) Sintering behaviors of micron- and submicron-sized ZnO features (Invited)**K. Lu<sup>\*1</sup>; H. Ju<sup>1</sup>

1. Virginia Tech, USA

**12:00 PM****(SINT-031-2017) Coarsening and densification during sintering of nano-sized tungsten and tungsten carbide powder (Invited)**Z. Z. Fang<sup>\*1</sup>

1. University of Utah, Metallurgical Engineering, USA

**Spark-plasma and Flash Sintering II**

Room: Bayview I

Session Chairs: Jan Raethel, Fraunhofer IKTS; Martha Mecartney, University of California, Irvine

**2:00 PM****(SINT-032-2017) Understanding and controlling flash sintering: A case study of ZnO (Invited)**J. Luo<sup>\*1</sup>

1. University of California, San Diego, USA

**2:40 PM****(SINT-033-2017) Manipulation of ceramic materials to promote field-enhanced sintering**R. E. Brennan\*; V. L. Blair<sup>1</sup>; M. Kornecki<sup>1</sup>; N. Ku<sup>1</sup>; S. Raju<sup>1</sup>; F. Kellogg<sup>1</sup>

1. US Army Research Laboratory, USA

**3:00 PM****(SINT-034-2017) Net shape flash spark plasma sintering of metal and ceramic powders**E. Olevsky\*; G. Lee<sup>1</sup>; C. Maniere<sup>1</sup>; J. McKittrick<sup>2</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA
2. University of California, San Diego, USA

**3:20 PM****Break****3:40 PM****(SINT-035-2017) Flash sintering of oxide nano-particles: A percolation phenomenon and particle surface softening as a universal behavior (Invited)**R. Chaim\*; G. Chevallier<sup>2</sup>; A. Weibel<sup>2</sup>; C. Estournes<sup>2</sup>

1. Technion - Israel Institute of Technology, Materials Science and Engineering, Israel
2. Université de Toulouse, Université Paul-Sabatier, CIRIMAT, CNRS INPT UPS, France

**4:20 PM****(SINT-036-2017) Onset, temperature gradient effects and defects in flash sintering**R. Janssen\*; J. Pereira da Silva<sup>1</sup>; H. Al-Qureshi<sup>2</sup>; F. Keil<sup>3</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. University Federal de Santa Catarina, Brazil
3. TUHH, Germany

**4:40 PM****(SINT-037-2017) In situ study of lattice expansion during flash sintering**H. Charalambous\*; S. K. Jha<sup>1</sup>; T. Tsakalakos<sup>1</sup>

1. Rutgers University, Materials Science and Engineering, USA

**Sintering of Multi-material and Multi-layer Systems I**

Room: Bayview II

Session Chairs: Vikram Jayaram, Indian Institute of Science; Hector Camacho Montes, Universidad Autonoma de Ciudad Juarez

**2:00 PM****(SINT-038-2017) Simulation and experimental validation of deformation and stress for co-sintered ceramic laminated composites**S. E. van Kempen\*; N. Giang<sup>1</sup>; R. Hammerbacher<sup>3</sup>; A. Bezold<sup>2</sup>; C. Broeckmann<sup>1</sup>; A. Roosen<sup>3</sup>; F. Lange<sup>4</sup>

1. RWTH Aachen University, Institute of Applied Powder Metallurgy and Ceramics at RWTH Aachen University e.V., Germany
2. RWTH Aachen University, Institute for Materials Applications in Mechanical Engineering, Germany
3. University of Erlangen-Nuremberg, Department of Materials Science, Chair of Glass and Ceramics, Germany
4. Siemens AG, Germany

**2:20 PM****(SINT-039-2017) Low-Temperature Mullite Formation in Coatings deposited by Atomic Layer Deposition for High-Temperature Applications**K. Furlan\*; T. Krekeler<sup>4</sup>; M. Ritter<sup>3</sup>; R. Blick<sup>2</sup>; G. A. Schneider<sup>1</sup>; K. Nielsch<sup>5</sup>; R. Zierold<sup>2</sup>; R. Janssen<sup>1</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. University of Hamburg, Institute of Nanostructures and Solid State Physics, Germany
3. TU Hamburg-Harburg, BE Electron Microscopy, Germany
4. TU Hamburg-Harburg, BE Electron Microscopy, Germany
5. IWF Dresden, Germany

**2:40 PM****(SINT-041-2017) Sintering of high entropy alloys (Invited)**

Y. Liu\*

1. Central South University, State Key Laboratory of Powder Metallurgy, China

**3:20 PM****Break****3:40 PM****(SINT-042-2017) Processing of Ti<sub>6</sub>Al<sub>4</sub>V compacts with graded porosity for biomedical applications**J. L. Cabezas-Villa<sup>3</sup>; D. Bouvard<sup>2</sup>; L. Olmos\*; J. Lemus-Ruiz<sup>3</sup>

1. Universidad Michoacana de San Nicolás Michoacana, INICIT, Mexico
2. Université Grenoble Alpes, CNRS, Grenoble, SIMAP, France
3. Universidad Michoacana de San Nicolas de Hidalgo, IIMM, Mexico

**4:00 PM****(SINT-043-2017) Sintering and processing of a novel titanium matrix composite**C. Zhou\*; Y. Liu<sup>1</sup>; B. Liu<sup>1</sup>

1. Central South University, State Key Lab of Powder Metallurgy, China

**4:20 PM****(SINT-182-2017) Fabrication of carbon fibre reinforced MAX-phase (Ti<sub>2</sub>AlC) composite**J. H. Nel<sup>1</sup>; D. C. Blaine\*; L. Sigalas<sup>2</sup>

1. Stellenbosch University, Mechanical and Mechatronic Engineering, South Africa
2. University of the Witwatersrand, School of Chemical and Metallurgical Engineering, South Africa

**4:40 PM****(SINT-181-2017) The sintering behavior of percolated metal-ceramic composites**A. S. Patnaik\*; J. Antalek<sup>1</sup>; J. Rawson<sup>1</sup>

1. Morgan Advanced Materials, Technology, USA

**Sintering of Nanostructured Materials II**

Room: Bayview III

Session Chairs: Gary Messing, Penn State University; Xiaoyan Song, Beijing University of Technology

**2:00 PM****(SINT-044-2017) Ordered pore structures at elevated temperatures (Invited)**R. Janssen\*; K. Furlan<sup>1</sup>; R. Pasquarelli<sup>1</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany

**2:40 PM****(SINT-045-2017) Thin films of surface water in nanocrystalline MgO: Influence on particle coarsening, coalescence and grain morphology evolution**O. Diwald\*; D. Thomele<sup>1</sup>; A. Gheisi<sup>2</sup>; J. Bernardi<sup>3</sup>

1. University of Salzburg, Chemistry and Physics of Materials, Austria
2. Friedrich-Alexander-Universität Erlangen Nürnberg, Germany
3. Vienna University of Technology, Austria

**3:00 PM****(SINT-046-2017) Restrained nanosintering to produce designed nanostructured solids**B. N. Feigelson\*; J. A. Wollmershauser<sup>1</sup>; K. Manandhar<sup>1</sup>

1. US Naval Research Laboratory, USA

**3:20 PM****Break****3:40 PM****(SINT-047-2017) Ceramic/organic supercrystalline nanocomposites with exceptional isotropic mechanical properties**D. Giuntini\*; B. Domenech<sup>1</sup>; B. Bor<sup>1</sup>; D. Benke<sup>1</sup>; G. Schneider<sup>1</sup>

1. Hamburg University of Technology (TUHH), Institute of Advanced Ceramics, Germany

**4:00 PM****(SINT-048-2017) Preparation of nanohydroxyapatite based scaffolds fabricated using magnesium as a porogen**

G. Adamek\*

1. Poznan University of Technology, Institute of Materials Science and Engineering, Poland



**4:20 PM****(SINT-049-2017) Effect of SiC incorporation on microstructure and mechanical properties of hot-pressed Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub> ceramics with eutectic composition**A. Henniche\*<sup>1</sup>; J. Ouyang<sup>1</sup>; Y. Ma<sup>2</sup>; Z. Wang<sup>1</sup>; Y. Wang<sup>1</sup>; Z. Liu<sup>1</sup>

1. Harbin Institute of Technology, School of Materials Science and Engineering, China

**4:40 PM****(SINT-050-2017) Influence of sintering temperature on micromorphology and microstructure of electrospun mullite nanofibers with different contents of SiO<sub>2</sub>**J. Wang\*<sup>1</sup>; X. Song<sup>1</sup>; W. Liu<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

**Modeling and Simulation of Sintering at Multiple Scales I**

Room: Mission I

Session Chair: Rajendra Bordia, Clemson University

**2:00 PM****(SINT-051-2017) Influence of initial packing, pore formers and sintering on mechanical properties of porous ceramics: Discrete element method simulations (Invited)**D. Jauffres\*<sup>1</sup>; C. Martin<sup>1</sup>; R. Bordia<sup>2</sup>

1. University of Grenoble Alpes, SIMAP, France
2. Clemson University, Materials Science and Engineering, USA

**2:40 PM****(SINT-052-2017) In-situ de-agglomeration during powder consolidation**D. Giuntini\*<sup>1</sup>; A. Maximenko<sup>2</sup>; E. Olevsky<sup>2</sup>

1. Hamburg University of Technology (TUHH), Institute of Advanced Ceramics, Germany
2. San Diego State University, USA
3. NAS Ukraine, Ukraine

**3:00 PM****(SINT-053-2017) FE simulation of temperature distribution, densification and grain growth during field assisted sintering of 316L stainless steel**S. Sista\*<sup>1</sup>; M. Hajeck<sup>1</sup>; A. Kaletsch<sup>1</sup>; C. Broeckmann<sup>1</sup>

1. RWTH Aachen University, IWM, Germany

**3:20 PM****Break****3:40 PM****(SINT-054-2017) Microscopic sintering forces acting among particles during sintering by grain boundary/surface diffusion (Invited)**F. Wakai\*<sup>1</sup>; S. Kanchika<sup>1</sup>; G. Okuma<sup>1</sup>; N. Nishiyama<sup>1</sup>

1. Tokyo Institute of Technology, Laboratory for Materials and Structures, Japan

**4:20 PM****(SINT-055-2017) Pattern formation during current sintering: Simulation models**L. Engelke\*<sup>1</sup>; M. Jongmanns<sup>1</sup>; C. Gorynski<sup>2</sup>; M. Winterer<sup>2</sup>; D. E. Wolf<sup>1</sup>

1. University of Duisburg-Essen, Physics, Germany
2. University of Duisburg-Essen, Nanoparticle Process Technology, Faculty of Engineering, Germany

**4:40 PM****(SINT-056-2017) Modeling of electric current assisted sintering for porous-graded materials design**A. M. Locci\*<sup>1</sup>; R. Orrù<sup>1</sup>; G. Cao<sup>1</sup>

1. Università degli Studi di Cagliari, Italy

**Novel Sintering Processes I**

Room: Mission II

Session Chairs: Olivier Guillon, Forschungszentrum Juelich; Stewart Silling, Sandia National Laboratories

**2:00 PM****(SINT-057-2017) Sintering by microwave heating for ceramics: New applications and recent developments (Invited)**F. Valdivieso\*<sup>1</sup>

1. Mines SE, France

**2:40 PM****(SINT-058-2017) Ultra-rapid microwave sintering of pure and Y<sub>2</sub>O<sub>3</sub>-doped MgAl<sub>2</sub>O<sub>4</sub>**S. Egorov<sup>1</sup>; A. Ereemeev<sup>1</sup>; V. Kholoptsev<sup>1</sup>; K. Rybakov\*<sup>1</sup>; A. Sorokin<sup>1</sup>; Y. Bykov<sup>1</sup>

1. Institute of Applied Physics, Russian Academy of Sciences, Russian Federation

**3:00 PM****(SINT-059-2017) Mechanical properties of 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> composite ceramics prepared via conventional and microwave sintering**B. Liang\*<sup>1</sup>; C. Fang<sup>1</sup>; Y. Ai<sup>1</sup>; W. He<sup>2</sup>; W. Chen<sup>1</sup>; T. Chen<sup>2</sup>; C. Liu<sup>1</sup>

1. Nanchang Hangkong University, School of Materials Science and Engineering, China
2. Nanchang Hangkong University, Instrumental Analysis Center, China

**3:20 PM****Break****3:40 PM****(SINT-060-2017) Microwave sintering of Al<sub>2</sub>O<sub>3</sub> base ceramics (Invited)**J. Chaix\*<sup>1</sup>; J. Croquesel<sup>1</sup>; D. Bouvard<sup>1</sup>; S. Saunier<sup>2</sup>; C. Carry<sup>1</sup>

1. University of Grenoble Alpes, SIMAP, France
2. Ecole des Mines de Saint-Etienne, France

**4:20 PM****(SINT-061-2017) Effect of the absorbed microwave power density on the sintering of 3YSZ ceramics**I. Plotnikov<sup>1</sup>; S. Egorov<sup>1</sup>; A. Ereemeev<sup>1</sup>; V. Kholoptsev<sup>1</sup>; K. Rybakov\*<sup>1</sup>; A. Sorokin<sup>1</sup>; Y. Bykov<sup>1</sup>

1. Institute of Applied Physics, Russian Academy of Sciences, Russian Federation

**Randall German Honorary Symposium II**

Room: Mission III

Session Chairs: Animesh Bose, Desktop Metal; Volker Pottler, Karlsruhe Institute of Technology

**2:00 PM****(SINT-062-2017) Novel multi-use liquid phase for PM components (Invited)**J. Torralba\*<sup>1</sup>; M. Campos<sup>1</sup>; A. Galán-Salazar<sup>1</sup>

1. Universidad Carlos III de Madrid, Materials Science and Engineering and Chemical Engineering, Spain

**2:20 PM****(SINT-063-2017) Novel materials for micro powder injection molding (Invited)**V. Pottler\*<sup>1</sup>

1. Karlsruhe Institute of Technology, Institute for Materials Research, Germany

**2:40 PM****(SINT-064-2017) Influence of the green microstructure on sintering shrinkage of metallic powders (Invited)**A. Molinari\*<sup>1</sup>

1. University of Trento, Industrial Engineering, Italy

**3:00 PM****(SINT-065-2017) Densification pathways – does the journey matter? (Invited)**D. C. Blaine\*<sup>1</sup>

1. Stellenbosch University, Mechanical and Mechatronic Engineering, South Africa

**3:20 PM****Break**

**3:40 PM****(SINT-066-2017) A history of liquid phase sintering in space (Invited)**J. L. Johnson\*<sup>1</sup>

1. Elmet Technologies LLC, USA

**4:00 PM****(SINT-067-2017) Improving liquid-phase sintering of iron based Ti(C,N) cermets (Invited)**E. Gordo\*<sup>1</sup>; M. Dios<sup>1</sup>; P. Alvaredo<sup>1</sup>; Z. Gonzalez<sup>2</sup>; B. Ferrari<sup>2</sup>

1. University Carlos III of Madrid, Materiales Science and Engineering, Spain
2. Institute of Ceramics and Glass, CSIC, Spain

**4:20 PM****(SINT-068-2017) Enhancing properties through liquid phase sintering (Invited)**M. Campos\*<sup>1</sup>; J. Torralba<sup>1</sup>; A. Galán-Salazar<sup>1</sup>; E. Bernardo<sup>2</sup>; R. De Oro Calderon<sup>3</sup>

1. Carlos III University of Madrid, Materials Science & Engineering, Spain
2. Aleac Met Sinterizadas AMES, R&D Department, Spain
3. Technical University Wien, Institute of Chemical Technologies and Analytics, Austria

**4:40 PM****Coming together in celebration of the sintering career of Professor Randall German****Poster Session**

Room: Regatta Pavilion

**5:30 PM****(SINT-P001-2017) Chemical reactions between natural chalcopyrite and copper in SPS sintering and the thermoelectric properties of the compacts**K. Fuda\*<sup>1</sup>

1. Akita University, Applied Chemistry, Japan

**(SINT-P002-2017) Microwave sintering of nuclear fuels: Specific development of a microwave device for UO<sub>2</sub> pellets**F. Valdivieso\*<sup>1</sup>; J. Croquesel<sup>2</sup>; S. Pillon<sup>2</sup>; S. Saunier<sup>1</sup>; C. Meunier<sup>1</sup>

1. Mines SE, France
2. CEA MARCOULE, France

**(SINT-P003-2017) Mechanical forces in the microscopic powder contact during field assisted sintering**J. Trapp\*<sup>1</sup>; B. Kieback<sup>2</sup>

1. Technische Universität Dresden, Materials Science, Germany
2. Fraunhofer IFAM, Germany

**(SINT-P004-2017) Determination of the optimal temperature of spark-plasma sintering of ALON ceramics by using calculation of thermal fields in a graphite mold**N. A. Rubinkovskiy\*<sup>1</sup>; E. G. Grigoryev<sup>1</sup>; A. G. Zholnin<sup>1</sup>; M. G. Isaenkova<sup>2</sup>; A. V. Samohin<sup>3</sup>

1. National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Key Laboratory for Electromagnetic Field Assisted Processing of Novel Materials, Russian Federation
2. National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Department of Physical Problems of Materials Science, Russian Federation
3. Russian Academy of Sciences, A. Baikov Institute of Metallurgy and Materials Science, Russian Federation

**(SINT-P005-2017) Forming micro components from ceramics materials by using FAST process**H. H. Hijji\*<sup>1</sup>; Y. Qin<sup>1</sup>

1. University of Strathclyde, Design, Manufacture and Engineering Management, United Kingdom

**(SINT-P006-2017) The effect of dislocations on flash behaviors in SrTiO<sub>3</sub> single crystals**K. Yamada\*<sup>1</sup>; H. Yoshida<sup>2</sup>; T. Tokunaga<sup>1</sup>; T. Yamamoto<sup>1</sup>

1. Nagoya University, Department of Materials Design Innovation Engineering, Japan
2. National Institute for Materials Science (NIMS), Japan

**(SINT-P007-2017) Unmasking the PECS process in a typical Dr. Sinter™ apparatus**D. Liebenberg\*<sup>1</sup>; Y. Liu<sup>1</sup>

1. Clemson University, Physics and Astronomy, USA

**(SINT-P008-2017) Laser sintering of persistent luminescent (Sr,Ca)Al<sub>2</sub>O<sub>4</sub>:Eu<sup>2+</sup>RE<sup>3+</sup> ceramics**N. R. Souza<sup>2</sup>; D. C. Silva<sup>2</sup>; D. V. Sampaio<sup>2</sup>; C. Kucera<sup>3</sup>; J. Ballato<sup>3</sup>; A. Trofimov<sup>4</sup>; L. G. Jacobsohn<sup>4</sup>; R. S. Silva\*<sup>1</sup>

1. Federal University of Sergipe / Clemson University, Brazil
2. Federal University of Sergipe, Brazil
3. Clemson University, Center for Optical Materials Science and Engineering Technologies, USA
4. Clemson University, USA

**(SINT-P009-2017) Electrical study of Li<sub>3x</sub>La<sub>(2/3-x)</sub>TiO<sub>3</sub> ceramics sintered by laser and spark plasma techniques**K. A. Barbosa<sup>2</sup>; L. M. Jesus<sup>2</sup>; Y. G. Santos<sup>2</sup>; J. S. Pereira<sup>2</sup>; F. Z. Guerrero<sup>2</sup>; Y. R. Barcelay<sup>2</sup>; J. A. Rivera<sup>2</sup>; A. Freitas<sup>2</sup>; J. C. Sales<sup>2</sup>; J. A. Moreira<sup>2</sup>; Y. L. Ruiz<sup>2</sup>; R. S. Silva\*<sup>1</sup>

1. Federal University of Sergipe / Clemson University, Brazil
2. Federal University of Sergipe, Brazil
3. Federal University of Amazonas, Brazil
4. University of Porto, Portugal

**(SINT-P010-2017) Ultrafine nanocrystalline scandia stabilized zirconia consolidated by deformable punch spark plasma sintering**R. L. Grosso\*<sup>1</sup>; D. N. Mucbe<sup>2</sup>; E. N. Muccillo<sup>1</sup>; R. H. Castro<sup>2</sup>

1. Energy and Nuclear Research Institute, Brazil
2. University of California - Davis, Department of Materials Science and Engineering & NEAT ORU, USA

**(SINT-P011-2017) Effect of CNT reinforcement on thermal diffusivity of Cu and Cu-CNT composites using the spark plasma sintering**S. Kim\*<sup>1</sup>; H. Kwon<sup>1</sup>

1. Korea Institute of Industrial Technology, Advanced Process and Materials R&D Group, Republic of Korea

**(SINT-P012-2017) Microstructure and mechanical properties of aligned electrospun mullite nanofiber yarns obtained at different temperatures**X. Song\*<sup>1</sup>; W. Liu<sup>1</sup>; Y. Ma<sup>1</sup>; J. Wang<sup>1</sup>; S. Xu<sup>1</sup>; Q. Cai<sup>1</sup>; S. Tang<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

**(SINT-P013-2017) Compaction and sintering of mixed nano-micro YSZ powder combinations**J. Liu\*<sup>1</sup>; L. Gao<sup>1</sup>

1. Shanghai Jiao Tong University, China

**(SINT-P014-2017) Investigation of sintering conditions of Mxene-ceramic composites**P. Zhang\*<sup>1</sup>; L. Gao<sup>1</sup>

1. Shanghai Jiao Tong University, China

**(SINT-P015-2017) Sintering and sintering resistance in nanostructure functional materials**L. Gao\*<sup>1</sup>; P. Zhang<sup>1</sup>

1. Shanghai Jiao Tong University, China

**(SINT-P016-2017) A brief sintering analysis on sintering of pure and tin-doped magnesium aluminate spinel using differential scanning calorimeter**G. J. Pereira\*<sup>1</sup>; R. H. Castro<sup>2</sup>; D. N. Mucbe<sup>2</sup>; G. Douglas<sup>3</sup>

1. Centro Universitário da FEI, Departamento de Engenharia de Materiais, Brazil
2. University of California, Davis, Department of Materials Science and Engineering & NEAT ORU, USA
3. University of Sao Paulo, Department of Metallurgical and Materials Engineering, Brazil

**(SINT-P017-2017) High resolution load assisted dilatometry to measure radial and axial strain rate**H. Camacho Montes\*<sup>1</sup>; L. H. Rascón Madrigal<sup>1</sup>; H. M. Loya Caraveo<sup>2</sup>; A. Delgado Salido<sup>2</sup>; A. García Reyes<sup>3</sup>; I. M. Ontiveros Muñoz<sup>2</sup>; R. Bordia<sup>4</sup>

1. Universidad Autónoma de Ciudad Juárez, Física y Matemáticas, Mexico
2. Universidad Autónoma de Ciudad Juárez, Eléctrica y Computación, Mexico
3. Interceramic Technological Center, Mexico
4. Clemson University, Materials Science and Engineering, USA
5. Productos Químicos A.R., Mexico

**(SINT-P018-2017) Synthesis and characterization of La-doped bismuth titanate by spark plasma sintering for enhancement of its ferroelectric properties**J. R. Leyva Mendoza<sup>1</sup>; G. Hernández Cuevas<sup>1</sup>; S. D. Villalobos Mendoza<sup>1</sup>; Y. Espinosa-Almeyda<sup>1</sup>; H. Camacho Montes\*<sup>2</sup>; C. A. Rodríguez González<sup>2</sup>; P. E. García Casillas<sup>1</sup>; O. Raymond Herrera<sup>2</sup>; S. Díaz de la Torre<sup>3</sup>

1. Universidad Autónoma de Ciudad Juárez, Física y Matemáticas, Mexico
2. Universidad Nacional Autónoma de México, Mexico
3. Instituto Politécnico Nacional, Mexico

**(SINT-P019-2017) Synthesis and characterization of niobium doped bismuth titanate**

G. Hernández Cuevas<sup>1</sup>; J. R. Leyva Mendoza<sup>1</sup>; Y. Espinosa-Almeyda<sup>1</sup>; J. R. Farias Mancilla<sup>1</sup>; J. F. Hernandez Paz<sup>1</sup>; H. Camacho Montes<sup>1</sup>; S. Díaz de la Torre<sup>2</sup>; O. Raymond Herrera<sup>3</sup>

1. Universidad Autónoma de Ciudad Juárez, Física y Matemáticas, Mexico
2. Instituto Politécnico Nacional, Mexico
3. Universidad Nacional Autónoma de México, Mexico

**(SINT-P020-2017) Solidification microstructure and mechanical properties of Au80Sn20 solder joints under different welding process**

Y. Huang<sup>\*1</sup>; W. Liu<sup>1</sup>; S. Tang<sup>1</sup>

1. State Key Laboratory of Powder Metallurgy, Central South University, China

**(SINT-P021-2017) Electrical study of multi-light sintered inkjet printed copper lines on flexible substrate**

M. Kang<sup>\*1</sup>; Y. Son<sup>1</sup>; C. Lee<sup>1</sup>

1. Hanyang University, Materials Science & Chemical Engineering, Republic of Korea

**(SINT-P022-2017) FLASH sintered KNN – structural and microstructural studies**

R. Serrazina<sup>1</sup>; P. Vilarinho<sup>1</sup>; A. M. Senos<sup>\*1</sup>

1. University of Aveiro/ CICECO, Materials and Ceramic Engineering, Portugal

**(SINT-P023-2017) Selective laser sintering of WC powders sputter coated with stainless steel**

A. M. Senos<sup>\*1</sup>; A. Cavaleiro<sup>1</sup>; C. Fernandes<sup>1</sup>; A. Farinha<sup>3</sup>; E. Boillat<sup>4</sup>; M. Vieira<sup>3</sup>

1. University of Aveiro/ CICECO, Materials and Ceramic Engineering, Portugal
2. INEGI, Instituto de Ciência e Inovação em Engenharia Mecânica e Engenharia Industrial, Portugal
3. University of Coimbra, Portugal
4. EPFL, Switzerland

**(SINT-P024-2017) Sintering of WC - (Cu, Fe, Cr, Ni) nanocomposites**

J. P. Cardoso<sup>1</sup>; J. B. Puga<sup>1</sup>; A. F. Rocha<sup>1</sup>; C. Fernandes<sup>1</sup>; A. M. Senos<sup>\*1</sup>

1. University of Aveiro/ CICECO, Materials and Ceramic Engineering, Portugal

**(SINT-P025-2017) Effect of additives and sintering condition on grain growth of UO<sub>2</sub> pellet**

Y. Na<sup>\*1</sup>; K. Lim<sup>1</sup>; M. Choi<sup>1</sup>; T. Jung<sup>1</sup>; S. Lee<sup>1</sup>

1. KEPCO Nuclear Fuel, Nuclear fuel development section, Republic of Korea

**(SINT-P026-2017) Self-sizing ceramic powders**

H. Sarma<sup>\*1</sup>

1. Corning Incorporated, USA

**(SINT-P027-2017)  $\beta$ -SiAlON-BN ceramic composites obtained by spark-plasma sintering**

E. Nefedova<sup>\*1</sup>; E. G. Grigoryev<sup>1</sup>; K. Smirnov<sup>2</sup>

1. National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Russian Federation
2. Institute of Structural Macrokinetics and Materials Science Russian Academy of Sciences (ISMAN RAS), Russian Federation

**(SINT-P028-2017) Sintering behavior and mechanical properties of Ti<sub>6</sub>Al<sub>4</sub>V compacts over a broad range of porosity**

J. L. Cabezas-Villa<sup>2</sup>; D. Bouvard<sup>\*3</sup>; L. Olmos<sup>1</sup>; J. Lemus-Ruiz<sup>2</sup>

1. Universidad Michoacana de San Nicolás Michoacana, INICIT, Mexico
2. Universidad Michoacana de San Nicolás de Hidalgo, IIMM, Mexico
3. Université Grenoble Alpes, CNRS, Grenoble INP, SIMAP, France

**(SINT-P029-2017) Correlation between anisothermal sintering shrinkage and structural activity of cold compacted iron parts**

S. Baselli<sup>\*1</sup>; A. Molinari<sup>1</sup>

1. University of Trento, Industrial Engineering, Italy

**(SINT-P030-2017) Multistep method application within ceramics sintering consolidation**

V. Mitic<sup>\*1</sup>; Z. B. Vosika<sup>2</sup>; V. Paunovic<sup>2</sup>; L. Kocic<sup>2</sup>; G. Lazovic<sup>3</sup>

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. Faculty of Electronic Engineering, Serbia
3. Faculty of Mechanical Engineering, Serbia

**(SINT-P031-2017) Post-sintering of a HfO<sub>2</sub> ceramic**

E. Post<sup>\*1</sup>; D. Rapp<sup>1</sup>

1. NETZSCH Geraetebau GmbH, Germany

**(SINT-P032-2017) Effect of sintering conditions on the transparent AION during two-stage reaction sintering**

T. Jung<sup>\*1</sup>; D. Kim<sup>1</sup>; M. Shin<sup>1</sup>; G. Lee<sup>1</sup>

1. SungKyunkwan University, Advanced Materials Science & Engineering, Republic of Korea

**(SINT-P033-2017) Modeling of compaction mechanisms during hot isostatic pressing of a 316L steel**

J. Chateau-Cornu<sup>\*1</sup>; Y. Kchaou<sup>1</sup>; L. Minier<sup>1</sup>; M. Ardigo-Besnard<sup>1</sup>; F. Bernard<sup>1</sup>

1. University of Burgundy, Carnot Interdisciplinary Laboratory of Burgundy, France

**(SINT-P034-2017) Anisotropic shrinkage of a single pore in simulation of viscous sintering by finite element method**

S. Kanchika<sup>\*1</sup>; F. Wakai<sup>1</sup>

1. Tokyo Institute of Technology, Laboratory for Materials & Structures, Japan

**(SINT-P035-2017) Sintering analysis of LSGM by construction of the master sintering curve**

S. L. dos Reis<sup>\*1</sup>; R. M. Batista<sup>1</sup>; E. N. Muccillo<sup>1</sup>; R. Muccillo<sup>1</sup>

1. Energy and Nuclear Research Institute, Brazil

**(SINT-P036-2017) Grain growth analysis of sintered alpha alumina**

A. M. Luster<sup>\*1</sup>

1. Rutgers University, Materials Science and Engineering, USA

**(SINT-P037-2017) Microstructure and phase evolution of direct laser sintering Ti/Al lightweight gradient material**

Y. Liu<sup>\*1</sup>; C. Zhang<sup>1</sup>; Y. Ma<sup>1</sup>; C. Liu<sup>1</sup>; S. Tang<sup>1</sup>; W. Liu<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

**(SINT-P038-2017) Effects of magnetic field on the interfacial microstructure and shear strength of Sn-3.5 wt%Ag-(0-0.1 wt%Fe) during sintering process**

S. Tang<sup>1</sup>; Y. Huang<sup>\*1</sup>; B. Chen<sup>1</sup>; W. Liu<sup>1</sup>; Y. Ma<sup>1</sup>

1. State Key Laboratory of Powder Metallurgy, China

**(SINT-P039-2017) Grain evolution during free-sintering of nanoparticles**

A. Lange<sup>\*1</sup>; A. Samanta<sup>1</sup>; T. Olson<sup>1</sup>; S. Elhadji<sup>1</sup>

1. Lawrence Livermore National Laboratory, USA

**(SINT-P040-2017) Spark plasma sintering of pure titanium powders**

S. Guillet-Fritsch<sup>\*1</sup>; G. Motsi<sup>2</sup>; P. A. Olubambi<sup>3</sup>; C. Estournes<sup>1</sup>

1. CNRS, Material Science, France
2. Université Paul Sabatier, Material Science, France
3. University of Johannesburg, Metallurgy and Chemical Engineering, South Africa

**(SINT-P041-2017) Spark plasma sintering of ZnO nanoparticles for thermoelectric applications**

S. Guillet-Fritsch<sup>\*1</sup>; P. M. Radingoana<sup>2</sup>; P. A. Olubambi<sup>3</sup>; C. Estournes<sup>1</sup>

1. CNRS, Material Science, France
2. Université de Toulouse, Material Science, France
3. University of Johannesburg, Metallurgy and Chemical Engineering, South Africa

**(SINT-P042-2017) Recycling of thermoplastic waste in the industry**

S. Salem Cherif<sup>\*1</sup>

1. University de Tizi-ouzou, Algeria

**(SINT-P043-2017) Freeze-casting to create micro-channels in La<sub>0.66</sub>Ca<sub>0.33-x</sub>Sr<sub>x</sub>Mn<sub>1.05</sub>O<sub>3</sub> ceramics**

C. D. Christiansen<sup>\*1</sup>; K. Nielsen<sup>1</sup>; R. Bordia<sup>2</sup>; R. Bjørk<sup>1</sup>

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark
2. Clemson University, Department of Materials Science and Engineering, USA

**(SINT-P044-2017) Synergistic reinforcement of tungsten carbide and carbon nanotubes for improving the thermal shock resistance of ultra-high temperature ceramic**

S. Mathiyalagan<sup>\*1</sup>; B. Mukherjee<sup>1</sup>; A. K. Keshri<sup>1</sup>

1. Indian Institute of Technology Patna, Materials Science and Engineering, India

**(SINT-P045-2017) Thermal and mechanical properties of SKD61 base tool steel alloys manufactured by selective laser melting process**J. Yu\*; J. Yun<sup>1</sup>; J. Choe<sup>1</sup>; S. Yang<sup>1</sup>; D. Yang<sup>1</sup>; Y. Kim<sup>1</sup>; C. Lee<sup>2</sup>

1. Korea Institute of Materials Science, Powder Technology Research, Republic of Korea
2. Korea Institute of Mechanics and Materials, Republic of Korea

**(SINT-P046-2017) On the stoichiometry and sintering behavior of boron suboxide**W. Shoulders\*; H. Payne<sup>2</sup>; K. D. Behler<sup>1</sup>; J. LaSalvia<sup>1</sup>; L. Vargas<sup>1</sup>

1. US Army Research Laboratory, USA
2. Pennsylvania State University, USA

**Tuesday, November 14, 2017****Plenary Session II**

Room: Bayview I

Session Chair: Didier Bouvard, Univ. Grenoble Alpes

**8:30 AM****(SINT-PL-002-2017) Contact formation and densification during early stages of spark plasma sintering of metal powders (Invited)**B. Kieback\*; J. Trapp<sup>1</sup>; T. Wallmersperger<sup>2</sup>; A. Semenov<sup>2</sup>; O. Eberhardt<sup>2</sup>; M. Nöthe<sup>1</sup>

1. Technische Universität Dresden, Institute of Materials Science, Germany
2. Technische Universität Dresden, Chair Mechanics of Multifunctional Structures, Germany

**9:30 AM****Break****Spark-plasma and Flash Sintering III**

Room: Bayview I

Session Chairs: Javier Garay, University of California, San Diego; François Valdivieso, Mines SE

**10:00 AM****(SINT-069-2017) FAST/Flash: A combination of FAST/SPS and flash sintering (Invited)**J. Raethel\*<sup>1</sup>

1. Fraunhofer IKTS, Sintering & Characterization, Germany

**10:40 AM****(SINT-070-2017) Spark plasma sintering of titanium based alloys: From finite element modeling to the fabrication of near net shape (NNS) specimens (Invited)**D. Martins<sup>1</sup>; U. Kus<sup>1</sup>; C. Maniere<sup>1</sup>; G. Chevallier<sup>1</sup>; P. Sallot<sup>2</sup>; K. Mocellin<sup>3</sup>; M. Bellet<sup>3</sup>; D. Delagnes<sup>4</sup>; J. Huez<sup>2</sup>; C. Estournes\*<sup>1</sup>

1. CIRIMAT, UPS/CHIMIE, France
2. Safran Tech, France
3. CEMEF, France
4. ICA Mines Albi, France
5. CIRIMAT, ENSIACET, France

**11:20 AM****(SINT-071-2017) Intrinsic electric current effects on densification behavior of fine zirconium nitride powders**G. Lee\*<sup>1</sup>; C. Maniere<sup>1</sup>; J. McKittrick<sup>1</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Mechanical Engineering, USA
2. University of California, San Diego, Mechanical and Aerospace Engineering, USA

**11:40 AM****(SINT-072-2017) Large scale direct current based spark plasma/field assisted sintering: Uniformity and densification kinetics**L. S. Walker\*<sup>1</sup>

1. Thermal Technology, USA

**12:00 PM****(SINT-073-2017) Traveling flash-sintering**J. Lebrun\*<sup>1</sup>; E. Sortino<sup>2</sup>; A. Sansone<sup>2</sup>; R. Raj<sup>3</sup>

1. Saint-Gobain, Innovative Materials, USA
2. Lucideon, United Kingdom
3. University of Colorado, USA

**Microstructural Evolution in Sintering Processes II**

Room: Bayview II

Session Chair: TBA

**10:00 AM****(SINT-074-2017) Electric field induced equilibrium grain boundary configurations in perovskite ceramics (Invited)**K. van Benthem\*<sup>1</sup>

1. University of California, Davis, Materials Science and Engineering, USA

**10:40 AM****(SINT-075-2017) Solute-drag versus solute-acceleration of grain boundary mobility**W. D. Kaplan\*<sup>1</sup>

1. Technion - Israel Institute of Technology, Dept. of Materials Science and Engineering, Israel

**11:00 AM****(SINT-076-2017) Boundary Structural Transition and Grain Growth Behavior during Sintering**S. L. Kang\*<sup>1</sup>

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

**11:20 AM****(SINT-077-2017) Effect of nanopowder addition on grain growth behavior of ultracoarse cemented carbides**X. Liu\*<sup>1</sup>; X. Song<sup>1</sup>; H. Wang<sup>1</sup>; C. Hou<sup>1</sup>; Z. Nie<sup>1</sup>

1. Beijing University of Technology, College of Materials Science and Technology, China

**11:40 AM****(SINT-078-2017) Liquid-like grain boundary complex ion and sub-eutectic activated sintering in CuO-doped TiO<sub>2</sub>**J. Nie\*<sup>1</sup>; J. M. Chan<sup>1</sup>; M. Qin<sup>1</sup>; N. Zhou<sup>1</sup>; J. Luo<sup>1</sup>

1. University of California, San Diego, USA

**12:00 PM****(SINT-079-2017) Prediction of microstructure evolution of solid oxide fuel cell electrodes during sintering through Kinetic Monte Carlo (KMC) simulations**Z. Yan\*<sup>1</sup>; S. Hara<sup>2</sup>; Y. Kim<sup>1</sup>; N. Shikazono<sup>1</sup>

1. University of Tokyo, Industrial Institute of Science, Japan
2. Chiba Institute of Technology, Department of Mechanical Engineering, Faculty of Engineering, Japan

**Sintering of Nanostructured Materials III**

Room: Bayview III

Session Chairs: Ricardo Castro, University of California, Davis; Caroline Lee, Hanyang University

**10:00 AM****(SINT-080-2017) Sintering property of bimodal metal nanopowder mixtures with various size ratios (Invited)**J. Lee\*<sup>1</sup>; J. Song<sup>1</sup>; E. Hong<sup>1</sup>

1. Hanyang University, Materials Engineering, Republic of Korea

**10:40 AM****(SINT-081-2017) Microstructural evolution in a four group element ODS ferritic steel (Y-Al-Ti-Zr) consolidated by SPS**E. Macia<sup>1</sup>; J. Cornide<sup>1</sup>; A. Garcia-Junceda<sup>2</sup>; M. Campos\*<sup>1</sup>; J. Torralba<sup>1</sup>

1. Universidad Carlos III de Madrid, Department of Materials, Spain
2. IMDEA Materials, Spain

**11:00 AM****(SINT-082-2017) Microstructural changes on Co-9Al-9W alloy produced by SPS and Ti and Ta additions**M. Carton Cordero<sup>1</sup>; M. Campos<sup>2</sup>; J. Torralba\*<sup>2</sup>

1. IMDEA Materials, Spain
2. Carlos III University, Materials Science and Engineering, Spain



**11:20 AM****(SINT-083-2017) Effect of nano-powder addition on sintering of water atomized iron powder**S. Manchilli<sup>\*1</sup>; J. Wendel<sup>1</sup>; A. Zehri<sup>2</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>; J. Liu<sup>2</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden
2. Chalmers University of Technology, Department of Microtechnology and Nanoscience, Sweden

**11:40 AM****(SINT-084-2017) Reaction spark plasma sintering for ceramic matrix nanocomposites (Invited)**A. V. Ragulya<sup>\*1</sup>; V. V. Skorokhod<sup>2</sup>

1. Frantsevich Institute for Problems in Materials Science, Dpt. Nanostructured Ceramics and Nanocomposites, Ukraine
2. Frantsevich Institute for Problems in Materials Science, Director Office, Ukraine

**Fundamental Aspects of Sintering II**

Room: Mission I

Session Chairs: Alberto Molinari, University of Trento; Charles Maniere, San Diego State University

**10:00 AM****(SINT-085-2017) The role of thermodynamics in controlling sintering and grain growth of ceramics (Invited)**R. H. Castro<sup>\*1</sup>

1. University of California, Davis, Materials Science & Engineering, USA

**10:40 AM****(SINT-086-2017) Full field study of sagging during sintering: A phase field and Navier-Stokes based approach**E. Borukhovich<sup>\*1</sup>; R. Babu<sup>1</sup>; D. Chasoglou<sup>2</sup>; J. Odqvist<sup>1</sup>

1. KTH Royal Institute of Technology, Enheten Struktur, Sweden
2. Höganäs AB | Global Development, Sweden

**11:00 AM****(SINT-087-2017) Variation with strain rate of the contribution of pore surface diffusion to bulk sintering viscosity accounting for evolving coordination**F. Delannay<sup>\*1</sup>; L. Brassart<sup>2</sup>

1. Université catholique de Louvain, Institute of Mechanics, Materials and Civil Engineering, Belgium
2. Monash University, Department of Materials Science and Engineering, Australia

**11:20 AM****(SINT-088-2017) Quasi-static model for the dependence of shear sintering viscosity on dihedral angle, grain boundary friction, relative density and grain coordination**F. Delannay<sup>\*1</sup>; L. Brassart<sup>2</sup>

1. Université catholique de Louvain, Institute of Mechanics, Materials, and Civil Engineering, Belgium
2. Monash University, Department of Materials Science and Engineering, Australia

**11:40 AM****(SINT-089-2017) Fractal correction of Coble's sintering model (Invited)**V. Mitic<sup>\*1</sup>; L. Kocić<sup>2</sup>; V. Paunovic<sup>2</sup>; Z. Nikolic<sup>2</sup>

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. Faculty of Electronic Engineering, Serbia

**Novel Sintering Processes II**

Room: Mission II

Session Chairs: Kirill Rybakov, Institute of Applied Physics, Russian Academy of Sciences; Jean-Michel Missiaen, Univ. Grenoble Alpes

**10:00 AM****(SINT-090-2017) Reactive laser sintering of electrophoretically deposited nanoparticulate films**V. Mackert<sup>\*1</sup>; C. Notthoff<sup>1</sup>; J. Gebauer<sup>1</sup>; M. Winterer<sup>1</sup>

1. University of Duisburg-Essen, Nanoparticle Process Technology, Germany

**10:20 AM****(SINT-091-2017) Reactive spark plasma sintering of ZrB<sub>2</sub> and TiB<sub>2</sub> from elemental mixtures**S. R. Bakshi<sup>\*1</sup>; K. NS<sup>1</sup>; M. BS<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

**10:40 AM****(SINT-092-2017) Electric-field assisted bonding of layered YSZ/ alumina for improvement in thermal barrier coat adhesion**C. Grimley<sup>\*1</sup>; J. Schwartz<sup>1</sup>; A. Prette<sup>2</sup>

1. North Carolina State University, Materials Science and Engineering, USA
2. Lucideon, United Kingdom

**11:00 AM****(SINT-093-2017) The effect of a small cation off-stoichiometry on flash sintering for SrTiO<sub>3</sub> and BaTiO<sub>3</sub>**T. Yamamoto<sup>\*1</sup>; T. Yoshino<sup>1</sup>; H. Yoshida<sup>2</sup>; T. Tokunaga<sup>1</sup>

1. Nagoya University, Department of Materials Design Innovation Engineering, Japan
2. National Institute for Materials Science (NIMS), Japan

**11:20 AM****(SINT-094-2017) A mesoscopic treatment of sintering in ceramics: Application to direct write additive manufacturing**F. Abdeljawad<sup>\*1</sup>; D. Bolintineanu<sup>1</sup>; A. W. Cook<sup>1</sup>; H. J. Brown-Shaklee<sup>1</sup>; D. Kammler<sup>1</sup>

1. Sandia National Laboratories, USA

**11:40 AM****(SINT-095-2017) Spark plasma sintering of structure-tailored ultra-high temperature components: First step to being a complex shape**X. Wei<sup>\*1</sup>; O. Izhvanov<sup>2</sup>; C. A. Back<sup>2</sup>; C. D. Haines<sup>3</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, USA
2. General Atomics, USA
3. US Army ARDEC, USA

**12:00 PM****(SINT-096-2017) Producing functional gradient material by high-voltage consolidation of powders**E. G. Grigoryev<sup>1</sup>; A. V. Yudin<sup>1</sup>; E. Olevsky<sup>\*2</sup>

1. NRCU MEPhI, Key Laboratory for Electromagnetic Field Assisted Materials Processing of Novel Materials, Russian Federation
2. San Diego State University, USA

**Randall German Honorary Symposium III**

Room: Mission III

Session Chairs: Kathy Lu, Virginia Tech; Hideshi Miura, Kyushu University

**10:00 AM****(SINT-097-2017) Developing grain aligned Alnico type-8 permanent magnets in near-final shape by novel compression molding and sintering treatments (Invited)**I. E. Anderson<sup>\*1</sup>

1. Ames Laboratory (USDOE), Division of Materials Sciences and Engineering, USA

**10:20 AM****(SINT-098-2017) Engineering challenges in additive manufacturing of metal components fabricated by selective laser melting (SLM) (Invited)**A. Z. Bunsch<sup>1</sup>; A. P. Ventura<sup>2</sup>; R. Cunningham<sup>3</sup>; W. Z. Misiolek<sup>\*2</sup>

1. AGH University of Science and Technology, Poland
2. Lehigh University, Materials Science & Engineering, USA
3. Carnegie Mellon University, Materials Science & Engineering, USA

**10:40 AM****(SINT-099-2017) Review of some non-conventional rapid hot consolidation techniques**A. Bose<sup>\*1</sup>; R. Sadangi<sup>2</sup>

1. Desktop Metal, USA
2. Armament R&D Engineering Center, USA

**11:00 AM****(SINT-100-2017) Development of super-high performance injection molded Ti alloy compacts for aerospace application**H. Miura\*<sup>1</sup>

1. Kyushu University, Research Center for Steel, Japan

**11:20 AM****(SINT-101-2017) Ni-based superalloy sintered engineered porous layer for direct surface cooling**E. M. White\*<sup>1</sup>; A. J. Heidloff<sup>2</sup>; D. Byrd<sup>1</sup>; R. Anderson<sup>1</sup>; I. E. Anderson<sup>1</sup>

1. Ames Laboratory of USDOE, Materials Sciences & Engineering, USA
2. Praxair, Inc., USA

**Wednesday, November 15, 2017****Plenary Session III**

Room: Bayview I

Session Chair: Suk-Joong Kang, KAIST

**8:30 AM****(SINT-PL-003-2017) Investigating the sintering of multilayer components with advanced experimental and modelling tools (Invited)**D. Bouvard\*<sup>1</sup>

1. University of Grenoble Alpes, SIMAP, France

**9:30 AM****Break****Spark-plasma and Flash Sintering IV**

Room: Bayview I

Session Chairs: Claude Estournes, CIRIMAT

**10:00 AM****(SINT-102-2017) Grain growth in oxides in the presence of an electric field (Invited)**W. Rheinheimer<sup>1</sup>; J. H. Preusser<sup>1</sup>; S. Karras<sup>3</sup>; J. P. Parras<sup>2</sup>; R. A. De Souza<sup>2</sup>; R. Garcia<sup>2</sup>; M. J. Hoffmann\*<sup>1</sup>

1. Karlsruhe Institute of Technology, Institute for Applied Materials, Germany
2. RWTH Aachen University, Institute of Physical Chemistry, Germany
3. Purdue University, School of Materials Engineering, USA

**10:40 AM****(SINT-103-2017) Integration of nanoscale phases: Leveraging kinetics to densify functional nano-composites (Invited)**Y. Kodera<sup>1</sup>; A. Volodchenkov<sup>1</sup>; K. Chan<sup>1</sup>; J. E. Garay\*<sup>1</sup>

1. University of California, San Diego, Dept. of Mechanical and Aerospace Engrg., USA

**11:20 AM****(SINT-104-2017) Densification of FeO and FeS with pulsed electric current sintering**M. Nanko\*<sup>1</sup>; K. Ueda<sup>1</sup>; N. Yamaguchi<sup>1</sup>

1. Nagaoka University of Technology, Department of Mechanical Engineering, Japan

**11:40 AM****(SINT-105-2017) Effect of austenite fraction on the microstructure and properties of duplex stainless steels consolidated by field-assisted sintering**A. García-Junceda<sup>1</sup>; C. D. Rivera<sup>2</sup>; V. G. Torralba<sup>3</sup>; M. Campos<sup>2</sup>; J. Torralba\*<sup>2</sup>

1. IMDEA Materials Institute, Spain
2. Universidad Carlos III, Spain
3. Universidad Rey Juan Carlos, Spain

**12:00 PM****(SINT-106-2017) Sintering kinetics of titanium silicon carbide MAX phase densified by field assisted sintering**J. Erauw\*<sup>1</sup>; V. Dupont<sup>1</sup>; M. Demuyne<sup>1</sup>; P. Aubry<sup>1</sup>; V. Lardot<sup>1</sup>; F. J. Cambier<sup>1</sup>

1. Belgian Ceramic Research Centre, Belgium

**Microstructural Evolution in Sintering Processes III**

Room: Bayview II

Session Chairs: Wayne Kaplan, Technion - Israel Institute of Technology; Torsten Staab, University Wuerzburg

**10:00 AM****(SINT-107-2017) Two stage sintering – does it work for alumina? (Invited)**K. Drdlikova<sup>1</sup>; D. Drdlik<sup>1</sup>; R. Klement<sup>2</sup>; K. Maca<sup>1</sup>; D. Galusek\*<sup>2</sup>

1. Central European Institute of Technology, Czech Republic
2. Alexander Dubcek University of Trencin, Centre for Functional and Surface Functionalized Glasses, Slovakia

**10:40 AM****(SINT-108-2017) Sintering of pure, doped, and graded magnesium diboride for optimised superconducting properties**C. E. Dancer\*<sup>1</sup>

1. University of Warwick, Warwick Manufacturing Group, United Kingdom

**11:00 AM****(SINT-109-2017) The effects of particle grading on the sintering behavior, microstructures and mechanical properties of solid-state-sintered SiC ceramics**H. Wu\*<sup>1</sup>; Z. Huang<sup>1</sup>

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, State Key Laboratory of High Performance Ceramics and Superfine Microstructure, China

**11:20 AM****(SINT-110-2017) Densification and grain growth of transparent polycrystalline alumina by two-step pulsed electric current sintering**H. H. Nguyen\*<sup>1</sup>; M. Nanko<sup>1</sup>

1. Nagaoka University of Technology, Japan

**11:40 AM****(SINT-111-2017) Pattern formation during current sintering: Experiments**C. Gorynski\*<sup>2</sup>; L. Engelke<sup>1</sup>; M. Jongmanns<sup>1</sup>; D. E. Wolf<sup>1</sup>; M. Winterer<sup>2</sup>

1. University of Duisburg-Essen, Computational and Statistical Physics, Germany
2. University of Duisburg-Essen, Nanoparticle Process Technology, Germany

**12:00 PM****(SINT-112-2017) Al/P/Yb-doped active optical fibers produced using a modified powder sintering method**V. Velmiskin\*<sup>1</sup>; O. Egorova<sup>1</sup>; L. Iskhakova<sup>1</sup>; S. Semjonov<sup>1</sup>

1. FORC RAS, Russian Federation

**Emerging Topics in Sintering and Micro Development I**

Room: Bayview III

Session Chairs: Jianhua Tong, Clemson University; Deborah Blaine, Stellenbosch University

**10:00 AM****(SINT-113-2017) Relationship between grain growth and thermal conductivity in liquid-phase sintered silicon carbide ceramics (Invited)**Y. Kim\*<sup>1</sup>; T. Cho<sup>1</sup>; Y. Kim<sup>1</sup>

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea

**10:40 AM****(SINT-114-2017) Sintering of glass frit**A. L. Fry\*<sup>1</sup>; H. Lee<sup>1</sup>; W. Carty<sup>1</sup>

1. Alfred University, Ceramic Engineering and Materials Science, USA

**11:00 AM****(SINT-115-2017) Sintering of a zirconia green body investigated by dilatometry and laser flash analysis**E. Post\*<sup>1</sup>

1. NETZSCH Geraetebau GmbH, Germany

11:20 AM

**(SINT-116-2017) Development of SiC nanopowder coated with a very thin additives and its utilization to low temperature SPS**K. Shimoda<sup>\*</sup>; H. Murakami<sup>1</sup>

1. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan

11:40 AM

**(SINT-117-2017) Inverse Hall-Petch behavior of nanocrystalline oxide ceramics fabricated by high-pressure SPS**M. Sokol<sup>\*</sup>; B. Ratzker<sup>1</sup>; S. Kalabukhov<sup>1</sup>; N. Frage<sup>1</sup>

1. Ben-Gurion University of the Negev, Materials Engineering, Israel

12:00 PM

**(SINT-118-2017) Graphene nanoplatelet and carbon nanotube reinforced titanium diboride composites by reactive spark plasma sintering**S. R. Bakshi<sup>\*</sup>; K. NS<sup>1</sup>; M. BS<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

**Fundamental Aspects of Sintering III**

Room: Mission I

Session Chairs: Jian Luo, UCSD; Aravind Mohanram, Mott Corporation

10:00 AM

**(SINT-119-2017) Sintering behavior of compacted water-atomized iron powder: Effect of initial green state**J. Wendel<sup>\*</sup>; S. Manchili<sup>1</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden

10:20 AM

**(SINT-120-2017) Effect of surface chemistry and processing conditions on densification of carbonyl iron powder**J. Wendel<sup>\*</sup>; R. Shvab<sup>1</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden

10:40 AM

**(SINT-121-2017) Gravitational role on dimensional control in liquid phase sintering**R. M. German<sup>\*</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Engineering, USA

11:00 AM

**(SINT-122-2017) Powder chemistry effects on grain boundaries and sintering of Bayer alumina**T. Frueh<sup>\*</sup>; E. R. Kupp<sup>1</sup>; C. Compson<sup>2</sup>; J. Atria<sup>2</sup>; G. L. Messing<sup>1</sup>

1. Pennsylvania State University, USA
2. Almatix, Inc., USA

11:20 AM

**(SINT-123-2017) Experimental study of electromagnetic field impact on surface diffusion in zinc oxide ceramic**A. Ducoulombier<sup>\*</sup>; F. Valdivieso<sup>1</sup>; J. Bruchon<sup>1</sup>; P. Ganster<sup>1</sup>; S. Saunier<sup>1</sup>; C. Meunier<sup>1</sup>

1. Mines Saint Etienne, SMS/MPE, France

11:40 AM

**(SINT-124-2017) Evolution of uniaxial viscosity and microstructure during field-assisted sintering of rare earth doped ceria**M. Mutter<sup>\*</sup>; C. Cao<sup>1</sup>; R. Mücke<sup>1</sup>; O. Guillon<sup>1</sup>

1. Forschungszentrum Jülich GmbH, IEK-1, Germany

**Sintering of Multi-material and Multi-layer Systems II**

Room: Mission II

Session Chairs: Chris Haines, US Army ARDEC; Zuoren Nie

10:00 AM

**(SINT-125-2017) Synthesis of multiphase Nb/SiCN ceramic composites by reaction of niobium with polysilazanes (Invited)**G. Motz<sup>\*</sup>; M. Seifert<sup>1</sup>

1. University of Bayreuth, Ceramic Materials Engineering, Germany

10:40 AM

**(SINT-126-2017) Electric field flash sintering of multiphase ceramics (Invited)**M. Mecartney<sup>\*</sup>; D. Kok<sup>1</sup>; Y. Yang<sup>1</sup>; R. Raj<sup>2</sup>

1. University of California, Irvine, Chem. Engr. & Materials Science, USA
2. University of Colorado, Boulder, USA

11:20 AM

**(SINT-127-2017) Utilizing cold sintering process for the fabrication of ceramics and ceramic-based composites**J. Guo<sup>\*</sup>; H. Guo<sup>1</sup>; A. Baker<sup>1</sup>; S. Funahashi<sup>2</sup>; M. Lanagan<sup>1</sup>; C. Randall<sup>1</sup>

1. Pennsylvania State University, Department of Materials Science & Engineering, USA
2. Murata Mfg. Co., Ltd., Japan

11:40 AM

**(SINT-128-2017) Sintering of titanium carbonitride reinforced alumina tool ceramics**M. Szutkowska<sup>\*</sup>; B. Smuk<sup>1</sup>; P. Putyra<sup>1</sup>; M. Podsiadlo<sup>1</sup>

1. The Institute of Advanced Manufacturing Technology, Poland

12:00 PM

**(SINT-129-2017) Heterogeneity effects during sintering for tile clay traditional materials**H. Camacho Montes<sup>\*</sup>; C. I. Villa Garcia<sup>2</sup>; A. Garcia Reyes<sup>2</sup>; R. Bordia<sup>3</sup>

1. Universidad Autonoma de Ciudad Juarez, Fisica y Matematicas, Mexico
2. InterCeramic Technological Center, Mexico
3. Clemson University, USA

**Chemical Interactions during Sintering**

Room: Mission III

Session Chair: Zhigang Fang, University of Utah

10:00 AM

**(SINT-130-2017) Reactions during sintering of metallic compacts that involve interstitial elements (Invited)**H. Danninger<sup>\*</sup>; R. De Oro Calderon<sup>1</sup>; C. Gierl-Mayer<sup>1</sup>

1. Technische Universität Wien, Institut für Chemische Technologien und Analytik, Austria

10:40 AM

**(SINT-131-2017) Quantitative analysis of the effect of refractory particles on the sintering of Cu: From preventing swelling to hindering shrinkage**A. Papillon<sup>2</sup>; J. Missiaen<sup>\*</sup>; J. Chaix<sup>5</sup>; S. Roure<sup>3</sup>; H. Schellekens<sup>4</sup>

1. University of Grenoble Alpes, Grenoble INP, France
2. Schneider Electric, Site Technopole, France
3. Schneider Electric, 38EQI, France
4. Schneider Electric, Usine 38V, France
5. University of Grenoble Alpes, CNRS, France

11:00 AM

**(SINT-132-2017) Effect of the sintering parameters and process atmosphere on the thermodynamics and kinetics of the sintering of prealloyed water-atomised PM steels**E. Hryha<sup>\*</sup>

1. Chalmers University of Technology, Materials and Manufacturing Technology, Sweden

11:20 AM

**(SINT-133-2017) Effect of C/Ti ratio on densification, microstructure and properties of TiC<sub>x</sub> prepared by reactive spark plasma sintering**S. R. Bakshi<sup>\*</sup>; V. Kombamuthu<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

**11:40 AM****(SINT-134-2017) Effect of boriding on mechanical, thermal and oxidation properties of TZM alloy prepared by spark plasma sintering process**B. Yavas\*<sup>1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgical and Materials Engineering, Turkey

**Spark-plasma and Flash Sintering V**

Room: Bayview I

Session Chairs: Rachman Chaim, Technion - Israel Institute of Technology; Raymond Brennan, US Army Research Laboratory

**2:00 PM****(SINT-135-2017) The effect of lithium doping on the sintering and grain growth of non-stoichiometric magnesium aluminate spinel (Invited)**S. Hayun\*<sup>1</sup>

1. Ben-Gurion University of the Negev, Materials Engineering, Israel

**2:40 PM****(SINT-136-2017) Effect of electric current on high temperature flow behavior of 8Y-ZrO<sub>2</sub>**K. Morita\*<sup>1</sup>; H. Yoshida<sup>1</sup>; B. Kim<sup>1</sup>; K. Hiraga<sup>1</sup>; Y. Sakka<sup>1</sup>

1. National Institute for Materials Science (NIMS), Japan

**3:00 PM****(SINT-137-2017) Rapid rate sintering of highly transparent AION ceramics**Y. Shan<sup>1</sup>; X. Wei\*<sup>2</sup>; X. Sun<sup>1</sup>; E. Olefsky<sup>2</sup>; J. Xu<sup>1</sup>; Q. Qin<sup>3</sup>

1. Dalian Maritime University, Department of Materials Science and Engineering, China
2. San Diego State University, College of Engineering, USA
3. Australian National University, Research School of Engineering, Australia

**3:20 PM****Break****3:40 PM****(SINT-138-2017) Processing and properties of transparent anisotropic ceramics using applied currents and pressures**Y. Kodera\*<sup>1</sup>; E. Penilla<sup>1</sup>; A. Wieg<sup>1</sup>; J. E. Garay<sup>1</sup>

1. University of California, San Diego, Dept. of Mechanical and Aerospace Engrg., USA

**4:00 PM****(SINT-139-2017) Flash sintering of a three-phase alumina, spinel, and yttria-stabilized zirconia composite**D. Kok\*<sup>1</sup>; S. K. Jha<sup>2</sup>; D. Yadav<sup>2</sup>; E. Sortino<sup>2</sup>; R. Raj<sup>2</sup>; M. Mecartney<sup>1</sup>

1. University of California, Irvine, Chemical Engineering and Material Science, USA
2. University of Colorado at Boulder, Mechanical Engineering, USA

**4:20 PM****(SINT-140-2017) Spark plasma sintering of SiC fiber-reinforced SiC composite**M. Ohyanagi\*<sup>1</sup>; K. Shirai<sup>1</sup>; R. Ozaki<sup>1</sup>

1. Ryukoku University, Japan

**Modeling and Simulation of Sintering at Multiple Scales II**

Room: Bayview II

Session Chairs: Bernd Kieback, Technische Universität Dresden / Fraunhofer IFAM; Fumihiro Wakai, Tokyo Institute of Technology

**2:00 PM****(SINT-141-2017) Modeling a material from packing, through sintering and to the final microstructural properties (Invited)**R. Bjørk\*<sup>1</sup>; K. Nielsen<sup>1</sup>

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

**2:40 PM****(SINT-142-2017) Modelling sintering by cellular Automata-Monte Carlo approach**X. Wang\*<sup>1</sup>; A. Atkinson<sup>1</sup>

1. Imperial College London, Dept of Materials, United Kingdom

**3:00 PM****(SINT-143-2017) A thermodynamically consistent phase-field framework for solid-state sintering**Q. Yang\*<sup>1</sup>; C. Liu<sup>2</sup>; J. Shen<sup>2</sup>; L. Chen<sup>1</sup>

1. Pennsylvania State University, Materials Science and Engineering, USA
2. Pennsylvania State University, Mathematics, USA
3. Purdue University, Mathematics, USA

**3:20 PM****Break****3:40 PM****(SINT-144-2017) Electromagnetic thermal mechanical modeling of microwave sintering: From simple to complex shapes (Invited)**C. Maniere\*<sup>1</sup>; T. Zahrah<sup>1</sup>; E. Olefsky<sup>1</sup>

1. San Diego State University, Engineering, USA
2. Matsys Inc., USA

**4:20 PM****(SINT-145-2017) Peridynamic modeling of sintering (Invited)**S. A. Silling\*<sup>1</sup>; V. Tikare<sup>1</sup>; B. G. Van Bloemen Waanders<sup>2</sup>; D. Z. Turner<sup>1</sup>

1. Sandia National Laboratories, Multiscale Science Department, 1444, USA
2. Sandia National Laboratories, USA

**5:00 PM****(SINT-146-2017) Discrete Element Sinter Modeling of Two-Phase Structures**R. Janssen\*<sup>1</sup>; R. Besler<sup>1</sup>; M. Marcel Rossetti da Silva<sup>1</sup>; K. Furlan<sup>1</sup>; C. Ohmstedt<sup>2</sup>; S. Heinrich<sup>2</sup>; M. Dosta<sup>2</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. TU Hamburg-Harburg, Inst. of Solids Process Eng. & Particle Technology, Germany

**Novel Sintering Processes III**

Room: Bayview III

Session Chairs: Manshi Ohyanagi, Ryukoku University; Johannes Trapp, Technische Universität Dresden

**2:00 PM****(SINT-147-2017) From field-assisted sintering to water-assisted sintering: FAST/SPS as a tool to understand and enable ultra-low temperature sintering (Invited)**O. Guillon\*<sup>1</sup>

1. Forschungszentrum Juelich, Germany

**2:40 PM****(SINT-148-2017) Room temperature sintering of alkali halide salts**G. L. Messing\*<sup>1</sup>; E. R. Kupp<sup>1</sup>; J. Anderson<sup>1</sup>; N. Pulati<sup>1</sup>

1. Pennsylvania State University, USA

**3:00 PM****(SINT-149-2017) Advances in flame assisted flash sintering**A. Hunt\*<sup>1</sup>

1. nGimat LLC, USA

**3:20 PM****Break****3:40 PM****(SINT-150-2017) Non-conventional sintering routes of a sodium-ion conducting NASICON material for battery application**J. Pereira da Silva\*<sup>1</sup>; A. Laptev<sup>1</sup>; J. Gonzalez-Julian<sup>1</sup>; Q. Ma<sup>1</sup>; F. Tietz<sup>1</sup>; M. Bram<sup>1</sup>; O. Guillon<sup>1</sup>

1. Forschungszentrum Juelich, Germany



**4:00 PM****(SINT-151-2017) Fast sintering of advanced oxide ceramics by conventional heating**K. Maca\*; D. Salamon<sup>1</sup>; V. Pouchly<sup>1</sup>; V. Prajzler<sup>1</sup>

1. Brno University of Technology, Czech Republic

**4:20 PM****(SINT-152-2017) Aerosol deposition: Mechanical sintering of ceramics, one particle at a time**P. Fuierer\*<sup>1</sup>

1. New Mexico Tech, Materials Engineering, USA

**4:40 PM****(SINT-153-2017) Doping effect on flash-sintering behavior in nanocrystalline yttria**H. Yoshida\*<sup>1</sup>; K. Morita<sup>1</sup>; B. Kim<sup>1</sup>; T. Yamamoto<sup>2</sup>

1. National Institute for Materials Science (NIMS), Japan
2. Nagoya University, Japan

**In situ Measurements and Analysis of Sintering**

Room: Mission I

Session Chairs: Jean-Marc Chaix, Univ. Grenoble Alpes, CNRS, Grenoble INP; Dusan Galusek, Alexander Dubcek University of Trencin

**2:00 PM****(SINT-154-2017) Temperature mapping in SPS using binder evaporation**E. M. Heian\*<sup>1</sup>; I. Pikus<sup>2</sup>

1. Gentherm Inc., USA
2. Tungsten Parts Wyoming, USA

**2:20 PM****(SINT-155-2017) Flash sintering of B<sub>2</sub>C: A time resolved energy dispersive x-ray diffraction study with an ultrahigh energy synchrotron probe**I. Savkliyildiz\*<sup>1</sup>; K. Akdogan<sup>2</sup>; H. Bicer<sup>3</sup>; T. Ozdemir<sup>2</sup>; C. Haines<sup>4</sup>; Z. Zhong<sup>5</sup>; T. Tsakalakos<sup>2</sup>

1. Selcuk University, Metallurgical and Materials Engineering, Turkey
2. Rutgers University, Materials Science and Engineering Department, USA
3. Dumlupinar University, Materials Science and Engineering Department, Turkey
4. United States Army ARDEC, ARDEC, USA
5. Brookhaven National Laboratory, USA

**2:40 PM****(SINT-156-2017) Abnormal unit cell expansion during flash sintering: A time-resolved in-situ EDXRD study with an ultrahigh energy synchrotron probe**I. Savkliyildiz\*<sup>1</sup>; K. Akdogan<sup>2</sup>; H. Bicer<sup>3</sup>; T. Ozdemir<sup>2</sup>; Z. Zhong<sup>4</sup>; T. Tsakalakos<sup>2</sup>

1. Selcuk University, Metallurgical and Materials Engineering, Turkey
2. Rutgers University, Materials Science and Engineering, USA
3. Dumlupinar University, Materials Science and Engineering Department, Turkey
4. Brookhaven National Laboratory, USA

**3:00 PM****(SINT-157-2017) Understanding the mechanism of flash sintering with in situ EDXRD experiments**S. K. Jha\*<sup>1</sup>; H. Charalambous<sup>1</sup>; T. Tsakalakos<sup>1</sup>

1. Rutgers University, MSE, USA

**3:20 PM****Break****3:40 PM****(SINT-158-2017) Using thermo-optical-measurement technique (TOM) to characterize sintering and melting behavior of ceramic and glass parts under atmospheric control**A. Diegeler\*<sup>1</sup>

1. Fraunhofer ISC, Germany

**4:00 PM****(SINT-159-2017) The temperature and electrical conductivity related to flash-sintering cubic zirconia**J. Liu\*<sup>1</sup>; Y. Wang<sup>2</sup>; D. Liu<sup>1</sup>; L. An<sup>3</sup>

1. Southwest Jiaotong University, School of Mechanics and Engineering, China
2. Northwestern Polytechnical University, School of Materials Science and Engineering, China
3. University of Central Florida, Department of Materials Science and Engineering, USA

**4:20 PM****(SINT-160-2017) Experimental characterization of sintering mechanism using optical dilatometer and push rod dilatometer: A comparative study**K. K. Telkicherla\*<sup>1</sup>; V. N. Nurni<sup>2</sup>; H. Ahmed<sup>1</sup>; C. Andersson<sup>1</sup>

1. Lulea University of Technology, Civil, Environmental and Natural Resources, Sweden
2. Indian Institute of Technology Bombay, Metallurgical Engineering and Material Science, India

**Sintering of Electronic Materials**

Room: Mission II

Session Chairs: Herbert Danninger, Technische Universität Wien; Monica Campos, Carlos III University of Madrid

**2:00 PM****(SINT-161-2017) Flash light sintered pattern on flexible substrate using 1-Octanethiol-coated copper nano-ink via inkjet printing technology (Invited)**C. S. Lee\*<sup>1</sup>; Y. Son<sup>1</sup>; M. Kang<sup>1</sup>

1. Hanyang University, Republic of Korea

**2:40 PM****(SINT-162-2017) Enhancement of lead-free NBBT piezoelectric ceramics via employing nanopowders and texturing technique (Invited)**D. Zhang\*<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

**3:20 PM****(SINT-163-2017) Silver sintering: An emerging technology to substitute soldered connections in the electronic industry**A. Steindamm\*<sup>1</sup>

1. SMT Maschinen- und Vertriebs GmbH & Co. KG, Project Management, Germany

**3:40 PM****Break****4:00 PM****(SINT-164-2017) The impact of intergranular regions on defect formation and electronic reducibility in nanocrystalline TiO<sub>2</sub> and In<sub>2</sub>O<sub>3</sub>**O. Diwald\*<sup>1</sup>; M. Elser<sup>2</sup>; N. Siedl<sup>2</sup>; D. Thomele<sup>1</sup>; J. Bernard<sup>3</sup>

1. University of Salzburg, Chemistry and Physics of Materials, Austria
2. Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany
3. Vienna University of Technology, Austria

**4:20 PM****(SINT-165-2017) Additive-engineered reactive sintering for high-performance protonic ceramics with well-designed microstructures**S. Mu<sup>1</sup>; Y. Hong<sup>1</sup>; Z. Zhao<sup>1</sup>; F. Peng<sup>1</sup>; J. Tong\*<sup>1</sup>

1. Clemson University, Materials Science and Engineering, USA

**Sintering Phenomena in Additive Manufacturing**

Room: Mission III

Session Chairs: Sundar Atre, University of Louisville; Tatsuki Ohji, National Institute of Advanced Industrial Science and Technology (AIST)

**2:00 PM****(SINT-166-2017) Microstructures, properties and applications of laser sintered metal powders (Invited)**S. V. Atre\*<sup>1</sup>

1. University of Louisville, Mechanical Engineering, USA

**2:40 PM****(SINT-167-2017) Hybrid structures processed by joining dense EBM-prepared lattices and partially sintered powder**O. Liashenko<sup>1</sup>; D. Bouvard<sup>\*</sup>; R. Dendievel<sup>1</sup>

1. University of Grenoble Alpes, SIMAP, France

**3:00 PM****(SINT-168-2017) Distortion and cracking during the densification of 3D printed green bodies**Z. C. Cordero<sup>1</sup>; R. Carazzone<sup>\*</sup>; M. Bonar<sup>1</sup>

1. Rice University, Materials Science and NanoEngineering, USA

**3:20 PM****Break****3:40 PM****(SINT-169-2017) Selective laser sintering techniques for improved porous media performance (Invited)**V. Palumbo<sup>1</sup>; J. Steele<sup>1</sup>; S. Lagocki<sup>1</sup>; A. Mohanram<sup>\*</sup><sup>1</sup>

1. Mott Corporation, R&D, USA

**4:20 PM****(SINT-170-2017) Additive manufacturing of advanced ceramics and components (Invited)**T. Ohji<sup>\*</sup><sup>1</sup>

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

**Thursday, November 16, 2017****Microstructural Evolution in Sintering Processes IV**

Room: Bayview I

Session Chairs: Jai Sung Lee, Hanyang University; Shmulik Hayun, Ben-Gurion University of the Negev

**8:30 AM****(SINT-171-2017) Superior reliability via two-step sintering: Barium titanate based ceramics (Invited)**X. Wang<sup>\*</sup><sup>1</sup>

1. Tsinghua University, Materials Science and Engineering, China

**9:10 AM****(SINT-172-2017) Mechanics and evolution of microstructural topology in viscous sintering observed by X-ray microtomography**G. Okuma<sup>\*</sup><sup>1</sup>; S. Tanaka<sup>2</sup>; F. Wakai<sup>1</sup>

1. Tokyo Institute of Technology, School of Material and Chemical Technology, Japan
2. Nagaoka University of Technology, Material Design Engineering Group, Japan

**9:30 AM****(SINT-173-2017) Predicting functionally graded microstructure of ZnO sintered in a large induced thermal gradient**C. L. Cramer<sup>\*</sup><sup>1</sup>; T. B. Holland<sup>1</sup>

1. Colorado State University, Mechanical Engineering, USA

**9:50 AM****Break****10:10 AM****(SINT-174-2017) Evaluation of nanograin growth of TiO<sub>2</sub> fibers fabricated by centrifugal jet spinning**H. Aminirastabi<sup>\*</sup><sup>2</sup>; Z. Xiong<sup>2</sup>; G. Ji<sup>1</sup>; Z. Xiong<sup>2</sup>; H. Xue<sup>2</sup>

1. Xiamen University, Aerospace College, China
2. Xiamen University, Materials College, China

**10:30 AM****(SINT-175-2017) Manipulating carbon content and sintering schedule to increase the density of pressureless sintered B<sub>4</sub>C**M. J. Dawes<sup>\*</sup><sup>1</sup>

1. University of Birmingham, Chemical Engineering, United Kingdom

**10:50 AM****(SINT-176-2017) Sintering map of barium cerate based protonic conductors**H. E. Araujo<sup>\*</sup><sup>2</sup>; K. Ramos<sup>1</sup>; D. Souza<sup>1</sup>

1. Federal University of Sao Carlos, Materials Engineering Department, Brazil
2. Federal Institute of Sao Paulo, Brazil

**11:10 AM****(SINT-177-2017) Densification and microstructural development of a new varistor ceramic**M. S. Nobrega<sup>\*</sup><sup>1</sup>

1. Federal University of Rio de Janeiro, Metallurgy and Materials Science, Brazil

**Modeling and Simulation of Sintering at Multiple Scales III**

Room: Mission II

Session Chair: Rasmus Bjørk, Technical University of Denmark

**8:30 AM****(SINT-178-2017) Sintering mechanisms of functionally graded WC-Co materials (Invited)**I. Konyashin<sup>\*</sup><sup>2</sup>; B. Ries<sup>1</sup>; A. Zaitsev<sup>2</sup>; E. Levashov<sup>2</sup>

1. Element Six GmbH, Germany
2. National University of Science and Technology MISiS, Russian Federation

**9:10 AM****(SINT-179-2017) Advanced complex shapes and productivity improvements in spark plasma sintering**C. Maniere<sup>\*</sup><sup>1</sup>; G. Chevallier<sup>2</sup>; A. Weibel<sup>2</sup>; L. Durand<sup>2</sup>; E. Olevsky<sup>1</sup>; C. Estournes<sup>2</sup>

1. San Diego State University, Engineering, USA
2. CIRIMAT, France
3. CEMES, France

**9:30 AM****(SINT-180-2017) Sintering processes for dual-phase solid oxide fuel cell electrodes**C. Xia<sup>\*</sup><sup>1</sup>

1. University of Science and Technology of China, Materials Science and Engineering, China

**9:50 AM****Break****Sintering of Multi-material and Multi-layer Systems III**

Room: Mission II

Session Chair: Rolf Janssen, TU Hamburg-Harburg

**10:10 AM****(SINT-183-2017) Predicting flexural deformation in multi-layered ceramic systems utilised in investment casting moulds**E. Douse<sup>\*</sup><sup>1</sup>; S. Blackburn<sup>1</sup>

1. University of Birmingham, United Kingdom

**10:30 AM****(SINT-184-2017) Sintering of laminated oxide-oxide FRCMC with tailored internal and surface structure**R. Janssen<sup>\*</sup><sup>1</sup>; D. Blaese<sup>1</sup>; P. Guglielmi<sup>2</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. HzG, Germany

**Novel Sintering Processes IV**

Room: Palm Ballroom

Session Chair: Andrii Maximenko, Institute for problems in materials science NAS Ukraine; Thomas Tsakalakos, Rutgers University

**8:30 AM****(SINT-185-2017) Effect of pressure on electric field-assisted pressureless sintering yttria-stabilized zirconia**S. G. Carvalho<sup>\*1</sup>; E. N. Muccillo<sup>1</sup>; R. Muccillo<sup>1</sup>

1. Energy and Nuclear Research Institute, Center of Science and Technology of Materials, Brazil

**8:50 AM****(SINT-186-2017) Flash sintering of samaria-doped ceria/carbon nanotube composites**R. Muccillo<sup>\*1</sup>; A. Ferlauto<sup>2</sup>; E. N. Muccillo<sup>1</sup>

1. IPEN, Brazil
2. Federal University of Minas Gerais, Brazil

**9:10 AM****(SINT-187-2017) Innovative control systems for Flash-Sintering**A. Prette<sup>\*1</sup>

1. Lucideon, Advanced Materials and Processing, United Kingdom

**9:30 AM****(SINT-188-2017) Densification and structural transformation during spark plasma sintering of WC-Co-YSZ-cBN systems**P. A. Olubambi<sup>1</sup>; T. G. Langa<sup>\*2</sup>; T. Shabalala<sup>3</sup>; M. R. Mphahlele<sup>1</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa
3. Element Six Pty, South Africa

**9:50 AM****Break****10:10 AM****(SINT-189-2017) Microstructural evaluation of spark plasma sintered (SPS) and atmospheric plasma sprayed (APS) CYSZ / bond coat thermal barrier coatings**F. Kirbijik<sup>\*1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgy and Material Engineering Dept., Turkey

**10:30 AM****(SINT-190-2017) Structural evolution in TiAl-CNT nanocomposites during in-situ alloying and consolidation by spark plasma sintering technique**P. A. Olubambi<sup>\*1</sup>; E. Nsiah-Baafi<sup>2</sup>; A. O. Adegbenjo<sup>2</sup>; M. B. Shongwe<sup>2</sup>; M. M. Ramakokovhu<sup>2</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa

**10:50 AM****(SINT-191-2017) Effect of graphene addition on mechanical and thermal properties of ZrC-TiC composites prepared by spark plasma sintering**B. C. Ocak<sup>\*1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgical and Materials Engineering, Turkey

**11:10 AM****(SINT-192-2017) Microstructural characterization of spark plasma sintered binderless Ti-based cermets**P. A. Olubambi<sup>1</sup>; M. B. Shongwe<sup>2</sup>; M. S. Ramatja<sup>2</sup>; M. R. Mphahlele<sup>\*1</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa

**Emerging Topics in Sintering and Micro Development II**

Room: Mission I

Session Chairs: Guenter Motz, University of Bayreuth; Dou Zhang, Central South University

**8:30 AM****(SINT-193-2017) Preparation of calcium carbonate porous ceramics**S. Umemoto<sup>\*1</sup>; M. Tajika<sup>1</sup>; H. Unuma<sup>3</sup>; S. Shima<sup>2</sup>

1. Shiraishi Central Laboratories Co., Ltd., Japan
2. Shanghai Institute of Ceramics, Chinese Academy of Sciences, R&D Center For Structural Ceramics and Composite, China
3. Yamagata University, Graduate School of Science and Engineering, Japan

**8:50 AM****(SINT-194-2017) Effects of alumina incorporation by particle atomic layer deposition on the sintering of Y-TZP and YSZ for biomedical and electrochemical applications**C. Bartel<sup>2</sup>; R. J. O'Toole<sup>\*2</sup>; M. Kodas<sup>2</sup>; A. Drake<sup>2</sup>; A. Horrell<sup>2</sup>; C. Gump<sup>1</sup>; R. Hall<sup>1</sup>; C. Musgrave<sup>2</sup>; A. W. Weimer<sup>2</sup>

1. ALD NanoSolutions, Inc., USA
2. University of Colorado, Chemical and Biological Engineering, USA

**9:10 AM****(SINT-195-2017) Assessment of sintering using pyrometric products**T. A. McInerney<sup>\*1</sup>

1. The Edward Orton Jr. Ceramic Foundation, USA

**9:30 AM****(SINT-196-2017) From MAX phase to MXenes: 2D anodic materials for sodium-ion batteries**S. Marchionna<sup>\*1</sup>; F. Cernuschi<sup>1</sup>; M. Balordi<sup>1</sup>; R. Riccardo<sup>2</sup>; M. Fiore<sup>2</sup>; M. Merlini<sup>3</sup>

1. RSE - Ricerca Sistema Energetico, TGM - generation technologies and related materials, Italy
2. Milan-Bicocca University, Materials Science department, Italy
3. University of Milan, Earth science dept., Italy

**9:50 AM****Break****10:10 AM****(SINT-197-2017) Numerical simulations towards microstructural design of solid oxide fuel cell anodes**S. Hara<sup>\*1</sup>; Z. Yan<sup>2</sup>; Z. Jiao<sup>2</sup>; N. Shikazono<sup>2</sup>

1. Chiba Institute of Technology, Mechanical Engineering, Japan
2. The University of Tokyo, Japan

**10:30 AM****(SINT-198-2017) The effect of heat treatment on remanufactured H13 Steel / Stellite<sup>®</sup> 21 moulds and dies**G. L. Payne<sup>\*1</sup>; C. Cullen<sup>2</sup>; S. Fitzpatrick<sup>2</sup>; T. Konkova<sup>2</sup>; P. Xirouchakis<sup>1</sup>; W. Ion<sup>2</sup>

1. University of Strathclyde, Design, Manufacture & Engineering Management, United Kingdom
2. Advanced Forming Research Centre, Design, Manufacture & Engineering Management, United Kingdom

**10:50 AM****(SINT-199-2017) Effects of sintering parameters on spark plasma sintered Ti-Zr alloys**B. L. Bayode<sup>\*1</sup>; L. Lethabane<sup>2</sup>; P. A. Olubambi<sup>1</sup>; L. Sigalas<sup>3</sup>; S. Mxolisi<sup>2</sup>; M. Ramakokovhu<sup>2</sup>

1. Centre for Nanoengineering and Tribocorrosion, School of Mining, Metallurgical and Chemical Engineering, University of Johannesburg, South Africa
2. Tshwane University of Technology, Chemical, Metallurgical and Materials Engineering, South Africa
3. University of the Witwatersrand, Chemical and Metallurgical Engineering, South Africa

**11:10 AM****(SINT-200-2017) BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3-d</sub>: A correlation between electrical properties, processing and microstructure**H. E. Araujo<sup>\*2</sup>; K. Ramos<sup>1</sup>; D. Souza<sup>1</sup>

1. Federal University of Sao Carlos, Materials Engineering Department, Brazil
2. Federal Institute of Sao Paulo, Brazil

## Stress-assisted Sintering II

Room: Mission III

Session Chair: Eduard Hryha, Chalmers University of Technology

### 8:30 AM

#### (SINT-201-2017) Pressure-assisted sintering of $\text{Ca}_3\text{Co}_4\text{O}_9$ multilayers for thermoelectric applications

B. Mieller<sup>\*1</sup>; S. Bresch<sup>1</sup>; T. Rabe<sup>1</sup>

1. BAM Federal Institute for Materials Research and Testing, Advanced Technical Ceramics, Germany

### 8:50 AM

#### (SINT-202-2017) Comparison of hot pressing and spark plasma sintering in the densification behavior of indium tin oxide – borosilicate glass composites

T. Rudzik<sup>\*1</sup>; R. A. Gerhardt<sup>1</sup>

1. Georgia Institute of Technology, Materials Science and Engineering, USA

### 9:10 AM

#### (SINT-203-2017) Influence of the elaboration process on the microstructure and durability of hard coatings obtained from metal powders

J. Chateau-Cornu<sup>\*1</sup>; M. Ardigo-Besnard<sup>1</sup>; Y. Kchaou<sup>1</sup>; L. Minier<sup>1</sup>; F. Bernard<sup>1</sup>

1. University of Burgundy, Carnot Interdisciplinary Laboratory of Burgundy, France

### 9:30 AM

#### (SINT-204-2017) Evolution of anisotropic microstructures in hierarchical porous ceramics during sinter-forging

R. Bordia<sup>\*1</sup>; H. Shang<sup>2</sup>; A. Mohanram<sup>3</sup>; E. Olevsky<sup>4</sup>

1. Clemson University, Materials Science and Engineering, USA
2. University of Washington, USA
3. Mott Corp, USA
4. San Diego State University, Dept of Mechanical Engrg, USA

### 9:50 AM

#### Break

### 10:10 AM

#### (SINT-205-2017) Modeling pore shape evolution during sinter-forging of LSM-YSZ composites

E. Olevsky<sup>\*1</sup>; A. Maximenko<sup>1</sup>; D. Giuntini<sup>1</sup>; R. Bordia<sup>2</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA
2. Clemson University, Materials Science and Engineering, USA



Monday, November 13, 2017

### Plenary Session I

Room: Bayview I

Session Chairs: Rajendra Bordia, Clemson University;  
Eugene Olevsky, San Diego State University

8:45 AM

#### (SINT-PL-001-2017) Know your boundaries (Invited)

M. P. Harmer<sup>\*1</sup>

1. Lehigh University, Materials Science and Engineering, USA

Do you know that grain boundaries can behave in a “phase-like” manner and undergo transformations that can dramatically influence the sintering behavior and properties of a material? Do you know that grain boundaries exhibit time-temperature-transformation (TTT) characteristics like steels? Do you know how to exploit grain boundary TTT diagrams in sintering? Do you know how “two-step” sintering and “flash” sintering work? Do you know about “anti-thermal” behavior of grain growth? Do you want a glimpse into the future of data-driven scientific discovery in sintering? I don’t have all the answers, but if these sorts of questions interest you, then you may want to come to this talk.

### Spark-plasma and Flash Sintering I

Room: Bayview I

Session Chair: Eugene Olevsky, San Diego State University

10:00 AM

#### (SINT-001-2017) The mechanisms of field assisted sintering/ spark plasma sintering of metallic materials on a microscopic scale (Invited)

J. Trapp<sup>\*1</sup>; M. Nöthe<sup>1</sup>; B. Kieback<sup>2</sup>; A. Semenov<sup>3</sup>; O. Eberhardt<sup>3</sup>;  
T. Wallmersperger<sup>3</sup>

1. Technische Universität Dresden, Materials Science, Germany
2. Fraunhofer IFAM, Germany
3. Technische Universität Dresden, Institute of Solid Mechanics, Germany

The Field Assisted Sintering Technique (FAST), also known as Spark Plasma Sintering (SPS), is an innovative method with growing employment in research and industrial production. It is typically used for hard to densify materials or the fast and efficient production of fine- or even nano-crystalline materials. The method is closely related to hot pressing with the difference of heating being accomplished by means of a pulsed electric current. To improve the understanding of the process, we experimentally investigated the influence of applied mechanical pressure, particle size, temperature and oxygen content (primarily by considering a surface oxide layer of varying thickness) on the rates of densification and neck growth during initial and intermediate stage of field assisted sintering of copper. Additionally, we determined the temperature distribution in the particles, neck growth and densification numerically by using a fully coupled electro-thermo-mechanical finite element method approach, taking into account the potential presence of an oxide. The results of experiment and simulation match well. We identified typical creep mechanisms, Coble-creep for lower and dislocation-creep for higher mechanical pressure, as the relevant mechanisms for material transport leading to the densification of the disperse system.

10:40 AM

#### (SINT-002-2017) Two-step, field assisted sintering (Invited)

C. Haines<sup>\*1</sup>; D. G. Martin<sup>1</sup>; K. C. Mills<sup>1</sup>

1. US Army ARDEC, Armaments Analysis & Manufacturing Directorate, USA

Field Assisted Sintering (FAS) aka Spark Plasma Sintering (SPS) has drawn a lot of attention as a sintering technology capable of minimizing grain growth due to lower sintering temperatures and times. Two-step sintering has also been employed to limit grain growth, by sintering at temperatures as low as 40% of the typical temperature. However, to date, a majority of the research in two-step sintering has been done in a pressure-less mode. This paper will present the results of applying two-step sintering principles during the field assisted sintering process. We will show the effects of the two-step process on the kinetics of grain growth in both low and high temperature materials, as compared with baseline samples sintered without the two-step process. Implications for the development of nanostructured, bulk materials will be discussed.

11:20 AM

#### (SINT-003-2017) Spark plasma sintering microscopic mechanisms of metallic systems: Experiments and simulations

J. Monchoux<sup>\*1</sup>; Z. Trzaska<sup>1</sup>; C. Collard<sup>1</sup>; A. Couret<sup>1</sup>; G. Bonnefont<sup>2</sup>;  
G. Fantozzi<sup>2</sup>

1. CEMES-CNRS UPR 8011, France
2. MATEIS-INSA Lyon, France

SPS mechanisms have been studied in metallic systems which allow high electric currents flowing through them, thus potentially allowing new electrically-induced mechanisms, or accelerating conventional ones (e.g. plasticity, diffusion). An originality of this work was to extract transmission electron microscopy (TEM) thin foils by focused ion beam (FIB) at the necks between TiAl powder particles during densification. Deformation by dislocations and recrystallization have been observed, showing that densification occurs by classical high temperature mechanisms. Comparisons with hot-pressing showed that the SPS current had no influence on them. Then, diffusion under current has been studied in Ag-Zn. Though diffusion in this system has been previously shown to be accelerated by currents above 200 A/cm<sup>2</sup> in dedicated experiments, no influence of the current was observed here, even if the SPS current was artificially increased up to ≈1000 A/cm<sup>2</sup>. Finally, finite element simulations at the scale of the powder particle size (≈100 μm) showed that, despite a strong intensification of the current density at the necks between the powder particles, very little overheating by Joule effect occurred. This was interpreted as a very high thermal diffusivity at these length scales, which makes it impossible to stabilize high thermal differences between the necks and the centers of the particles.

11:40 AM

#### (SINT-004-2017) Multiscale modeling of spark plasma copper powder sintering in multi-step loading regime (Invited)

A. Maximenko<sup>\*1</sup>; E. Olevsky<sup>2</sup>

1. Institute for Problems in Materials Science NAS Ukraine, Ukraine
2. San Diego State University, USA

Traditionally, powder consolidation during SPS is modeled as a steady-state power-law creep. Experiments with the stepped loading of copper specimen allow verification of this assumption. Different versions of copper constitutive equations have been used for modeling of powder consolidation during multi-step spark plasma sintering. It is shown, that good agreement between experiment and theory can be obtained if constitutive equations of primary creep with corresponding strain-hardening relationships are used for the modeling of copper behavior instead of steady-state equations. High strain non-homogeneity in powder particles during SPS makes contribution of primary creep significant even for comparatively

small porosities of the specimen. Modeling of consolidation is based on direct multiscale numerical approach, where all macroscopic results for porous bodies are obtained by numerical homogenization of mesoscopic calculations for cubic packing of spherical copper particles. This approach circumvents uncertainties related with selection of proper macroscopic model for porous material.

### Microstructural Evolution in Sintering Processes I

Room: Bayview II

Session Chairs: Klaus van Benthem, University of California, Davis; Diletta Giuntini, Hamburg University of Technology (TUHH)

10:00 AM

#### (SINT-005-2017) Sintering pure metals: Are there really indications for defect-activated sintering? (Invited)

T. E. Staab\*<sup>1</sup>

1. University Wuerzburg, Dep. of Chemistry - LCTM, Germany

Defects in powder metallurgical samples may enhance diffusion processes during sintering. Therefore, samples of pure copper, nickel, carbonyl iron and steel are prepared by interrupted sintering, i.e. removing the furnace at the target temperature and rapid cooling the samples, which are then investigated by several analytical methods: X-ray diffraction, DSC and positron annihilation lifetime spectroscopy (PALS). Thus, we can follow the sintering process by changes in the microstructure. These results are compared to heavily deformed reference samples to determine the recovery temperature (0.4 of the melting temperature), i.e. annealing of dislocations. By metallography we determine an effective powder particle size, which is used to model the shrinkage by a modified two-particle model. For larger samples (30-40mm in diameter) we follow the shrinkage by thermo-optical measurement to determine influences of the pressing tools by monitoring the shrinkage at different height positions during one run.

10:40 AM

#### (SINT-006-2017) Pressureless sintering of Mo-Si-B alloys with iron additions

M. Akinc\*<sup>1</sup>; G. Ouyang<sup>1</sup>; P. Ray<sup>2</sup>; M. J. Kramer<sup>2</sup>

1. Iowa State University, Materials Science & Engineering and Chemical & Biological Engineering, USA
2. Ames Laboratory, USA

The sintering behavior of Mo-Si-B with Fe as a sintering additive was investigated. The addition of small amounts of Fe effectively enhanced the densification of Mo-Si-B at temperatures below 1900 °C. The addition of 5 at% Fe resulted in nearly full densification (97.0 % of theoretical) when sintered at 1750 °C for 2 h, while the unmodified Mo-Si-B alloy could be densified to only 66.8 % of its theoretical density under these conditions. Addition of 0.5 at% Fe and 2 at% Fe increased the degree of densification of Mo-Si-B by 15.4 % and 17.0 % respectively, and led to nearly full densification at 1900 °C and 1850 °C, respectively. A continuous, Fe containing liquid was formed at low temperatures and disappeared at high temperatures. We propose that the addition of Fe led to the formation of a transient liquid, facilitating liquid phase sintering of the powder compacts.

11:00 AM

#### (SINT-007-2017) Densification behavior and microstructure of $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$ fabricated by spark plasma sintering

Y. Zeng\*<sup>1</sup>; X. Xiong<sup>1</sup>; Y. Wang<sup>1</sup>; Z. Chen<sup>1</sup>; W. Sun<sup>1</sup>

1. State Key Laboratory of Powder Metallurgy, China

Ultra-high temperature ceramics (UHTCs) exhibit good ablation resistance for sustained use in the extreme environments experienced by hypersonic vehicles.  $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$  is a quaternary UHTC, owning ultra-high temperature melting point and good

oxidation resistance. The densification behavior and microstructure of  $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$  ceramic fabricated by a spark plasma sintering process were systematically investigated. The sintering was conducted at temperatures ranging from 1700° to 1900°C in vacuum under several applied loads (20, 50, 80 MPa). The heating rate and the soaking time varied from 50° to 400°C/min and from 0 to 20 min, respectively. The microstructure was observed by scanning electron microscopy and electron backscattered diffraction. Results show that the density of ceramic increases with increase in sintering temperature, loads, heating rate and holding time, and there is a strong dependence of grain size of  $Zr_{0.8}Ti_{0.2}C_{0.74}B_{0.26}$  on the sintering temperature, heating rate, and holding time.

11:20 AM

#### (SINT-008-2017) Phase stability and nano-domain formation in alkaline-earth hexaborides (Invited)

O. Graeve\*<sup>1</sup>; J. Cahill<sup>1</sup>; M. Alberga<sup>2</sup>; D. Edwards<sup>2</sup>; S. T. Misture<sup>2</sup>; V. Vasquez<sup>3</sup>

1. University of California, San Diego, Mechanical and Aerospace Engineering, USA
2. Alfred University, Kazuo Inamori School of Engineering, USA
3. University of Nevada, Reno, Chemical and Materials Engineering, USA

We present the behavior of multiple solid solutions within ternary  $(Ba_xCa_{1-x})B_6$  and  $(Ba_xSr_{1-x})B_6$  compounds and demonstrate that nano-domain formation is preferred over uniform solid solutions under certain processing conditions. This phase separation has been observed from detailed analyses of the shapes of the peaks in X-ray diffraction data, where peak splitting and asymmetry are the result of multiple solid solutions with lattice parameters differing by up to 1.4%. High-resolution transmission electron microscopy confirms the presence of these nano-domains, which are about 2-3 nm in size and reveals varying degrees of lattice misalignment. We also present X-ray diffraction analysis of  $(Ba_xCa_{1-x})B_6$  powders calcined from 1273 to 1973 K and document the enhancement in sample homogeneity as the separated phases merge into a uniform solid solution. As subsequent calcinations at lower temperatures do not result in a re-separation of phases, the nano-domains are deemed metastable. The greatest degree of phase separation is observed in the  $(Ba_xCa_{1-x})B_6$  system, which corresponds to the largest difference in cation radii (0.161 vs. 0.134 nm for  $Ba^{2+}$  and  $Ca^{2+}$ , respectively). Analysis of the chemical reactions that occur during synthesis suggests that the decomposition of the metal precursors (nitrates and carbonates) to metal oxides may cause selective  $MB_6$  phase formation in mixed-cation hexaborides.

### Sintering of Nanostructured Materials I

Room: Bayview III

Session Chairs: Jean-Marie Lebrun, Saint-Gobain; Rasmus Bjørk, Technical University of Denmark

10:00 AM

#### (SINT-009-2017) Sintering densification of in situ synthesized WC-Co based composite powders (Invited)

X. Song\*<sup>1</sup>; X. Liu<sup>1</sup>; H. Wang<sup>1</sup>; X. Liu<sup>1</sup>; Z. Nie<sup>1</sup>

1. Beijing University of Technology, College of Materials Science and Engineering, China

In the presentation our work concerning developments of ultra-fine and nanocrystalline WC-Co cemented carbides with both high fracture toughness and strength will be introduced. The WC-Co based composite powders with super-ultrafine nanostructure were firstly synthesized by the in-situ reactions. The dense ultrafine and nanocrystalline cemented carbide bulk materials were prepared by sinter-HIP and spark plasma sintering, respectively, using the composite powders as starting materials. The phases evolution behavior and effects on mechanical properties of the sintered bulk materials were studied, particularly for the liquid-state sintering of composite powders containing grain growth inhibitors (GGI). The

factors of optimization of grain growth inhibitors, WC/Co phase boundaries and second phase particles in WC grains were proposed to achieve high fracture strength of the sintered cemented carbides. At a constant composition, the strengthening and toughening effects can be obtained by optimized scale matching of WC-Co composite powder and GGI particles combined by suitable sintering processing.

**10:40 AM**

**(SINT-010-2017) Cold isostatic pressing and low temperature sintering of nanograined ceramics**

J. Drazin<sup>\*2</sup>; E. Gorzkowski<sup>1</sup>

1. US Naval Research Laboratory, USA
2. ASEE Postdoc at US Naval Research Laboratory, USA

Nanograined ceramic oxide powders can be produced or acquired via multiple routes. However, to date, there does not exist a universal way of processing and sintering these powders to retain the nanograined structure without the application of large external pressures. Therefore, in this work, novel low pressure shaping coupled with high pressure cold isostatic pressing (CIP) (dry processing and without binders) was employed to shape and consolidate the nanograined ceramic oxide powders to high green densities (>55%) prior to sintering. With these high green densities, the pellets were sintered at 100s of °C lower temperatures than conventional firing while retaining the nanograined structure without application of an external pressure. Final grain sizes of sub-35nm can be achieved without any chemical binders as well. This approach opens up a new avenue for nanoparticle sintering that can be transitioned to industrial applications.

**11:00 AM**

**(SINT-011-2017) Sintered ceramic wire coatings as electrical insulation for high-field superconducting magnets**

M. White<sup>1</sup>; R. Nesbit<sup>1</sup>; A. Hunt<sup>\*1</sup>

1. nGimat LLC, USA

Green ceramic coatings were sintered to ~15 μm thick electrical insulation for the superconducting wires of Bi2212 and Nb3Sn accelerator magnets. The insulation must tolerate coil winding, reaction heat treatment to about 890°C, be non-detrimental to wire properties, allow full oxygenation of the superconductor, withstand voltages up to several kV during quench, be sufficiently radiation resistant to maintain isolation for long magnet life, provide good transverse heat transfer, and be economical. Issues with the prior aluminosilicate fiber braid insulation included: 1) too thick, reducing fill factor and field strength, 2) reaction with the wire, lowering critical current by 15-20%, 3) poor transverse heat transfer due to low thermal conductivity, 4) poor radiation tolerance of the epoxy impregnation. The new sintered insulation coatings: 1) were highly radiation-tolerant due to being all-ceramic, 2) were tightly adhered to the wire during heat treatment, 3) allowed oxygen access to the wire, 4) did not affect superconductor properties, 5) provided adequate breakdown strength, while being ~10x thinner than resin-filled ceramic fiber braid, 6) had thermal conductivity at 4.2K 7-10 times higher than resin-filled ceramic fiber, providing a 7-8x increase in transverse heat conduction, and therefore greater protection against quench, and also 7) were speedily and economically applied.

**11:20 AM**

**(SINT-012-2017) Dilatometry analysis of the sintering process of gadolinia-doped ceria**

E. N. Muccillo<sup>\*1</sup>; R. M. Batista<sup>1</sup>

1. Energy and Nuclear Research Institute, Brazil

The sintering process of nanostructured (specific surface area ~ 200 m<sup>2</sup>.g<sup>-1</sup>) 10 mol% gadolinia-doped ceria (10GDC) was investigated by dilatometry using the Arrhenius method and the master sintering curve (MSC) approaches. Dilatometry experiments were carried out for 3, 6, 10 and 12 °C.min<sup>-1</sup> heating rates in a vertical

push-rod type dilatometer. Refinement of these methods was accomplished by taking into account the weight loss of the nanostructured powder compacts. The MSC was validated by isothermal and non-isothermal sintering experiments. Good agreement was found for results obtained by the Arrhenius method and construction of the MSC. Combined analysis by these methods allows for choosing the best density range for construction of the MSC and to predict the density of conventionally sintered nanostructured 10GDC compacts.

**11:40 AM**

**(SINT-013-2017) Nanostructured Li<sub>7</sub>La<sub>3</sub>Zr<sub>2</sub>O<sub>12</sub> solid electrolytes with improved sinterability**

J. M. Weller<sup>\*1</sup>; C. K. Chan<sup>1</sup>

1. Arizona State University, Materials Science and Engineering, USA

Lithium lanthanum zirconate (Li<sub>7</sub>La<sub>3</sub>Zr<sub>2</sub>O<sub>12</sub>, "LLZO") is a highly promising, Li-ion conducting ceramic at the forefront of efforts to realize all-solid-state lithium-ion batteries with drastically improved safety, stability, and volumetric energy capacity. Much effort has been expended to characterize and prevent the deleterious formation and propagation of lithium dendrites through the electrolyte. Control over the grain size, density, and other critical factors is required to prevent device failure due to the growth of these dendrites during battery cycling. One approach is to increase the grain size to minimize the grain boundary area at which lithium tends to aggregate and propagate through the material. In this work, a different approach is taken, focusing on utilizing nanoparticles of LLZO to confer nanocrystalline grains with the goal of preventing growth of dendrites via the relatively higher mechanical resistance to crack propagation of small grains. In this presentation, the optimized synthesis of powders, consolidation, and sintering conditions to realize ceramics with well-controlled nanosized crystalline domains will be discussed. Our studies show that nanostructured LLZO may sinter at lower temperatures than bulk particles, which could mitigate Li loss, which is known to lead to deleterious effects such as secondary phase formation and decreased ionic conductivity.

**12:00 PM**

**(SINT-014-2017) Optimization of sintering temperature for electrospun red mud based nanofibers**

Z. Cetinkaya<sup>\*1</sup>; V. Kalem<sup>1</sup>

1. Selcuk University, Metallurgy and Materials Engineering, Turkey

Electrospinning is a technique production for inorganic nanofibers using their precursor solutions. The availability of many inorganic material precursors has led to the fabrication of inorganic nanofibers using electrospinning. Sintering is the final step for the formation of fiber structure. Besides, sintering parameters such as temperature and time have an influence on the fiber form and phase structure. In this study, we have utilized the electrospinning process to fabricate red mud based nanofibers. Nanofibers were dried at 70 for a period of 12 hours under vacuum condition and then, sintering process was performed at 600-800 for 6 hours. During the sintering process, the organics belonged to polymer groups and other volatiles were removed. Microstructural and compositional analyses of nanofibers have been carried out using scanning electron microscopy (SEM) and X-ray diffraction (XRD). Experimental results indicated that the sintering process at 650 resulted in the uniform nanofibers with average diameter of 45-60 nm.



## Fundamental Aspects of Sintering I

Room: Mission I

Session Chairs: Francis Delannay, Université catholique de Louvain;  
David Jauffres, Univ. Grenoble Alpes

10:00 AM

### (SINT-015-2017) Effect of geometry and green density on the anisotropic sintering shrinkage of axisymmetric iron parts (Invited)

A. Molinari\*<sup>1</sup>

1. University of Trento, Industrial Engineering, Italy

Sintering shrinkage of cold compacted axisymmetric parts is anisotropic. Not only the shrinkage of height (parallel to the compaction direction) is different from that of the diameters (in the compaction plane), but even the dimensional changes of the inner and of the outer diameters significantly differ. This behaviour has been investigated on iron rings with different geometry (height to wall thickness ratio) and green density in the 6.5-7.3 g/cm<sup>3</sup> range. Such a behaviour was related to the effect of green density on the sintering shrinkage, and to the effect of wall thickness on the axial gradient of the compaction force and, in turn, of green density. The correlation with the deformation of the powder particles in the green specimen and, through it, with the multiaxial stress field during uniaxial compaction was also analysed.

10:40 AM

### (SINT-016-2017) Effect of contact alignment on anisotropic shrinkage during sintering: Discrete element simulations, analytical models and experiments

B. Hugonnet<sup>2</sup>; J. Missiaen\*<sup>1</sup>; C. Martin<sup>3</sup>; C. Rado<sup>2</sup>

1. University of Grenoble Alpes, Grenoble INP, France

2. CEA, Liten, France

3. University of Grenoble Alpes, CNRS, France

Hexagonal ferrites or NdFeB magnetic materials are used as permanent magnets and they are usually processed by the powder metallurgy route. Alignment of magnetic moments is obtained during powder forming by applying a magnetic field, which maximizes the saturation magnetization of the final materials. Preferential orientation of particle contacts also results from this forming step. This may contribute to the anisotropic shrinkage observed during sintering of these materials. This point is addressed through discrete element simulations and analytical models of sintering for particle packings with an anisotropic distribution of the contact number per unit length. This initial anisotropic distribution is due to a preferential alignment of particle contacts along the vertical axis of the powder compact. The discrete element simulations and the analytical model lead to comparable results on the shrinkage anisotropy. These results are compared with experiments on the shrinkage anisotropy and on the microstructure of NdFeB sintered materials. The 3D distribution of grain boundary orientation is determined by image analysis combined with a stereological reconstruction. The contribution of contact alignment to anisotropic shrinkage of these materials is discussed in reference with the models, together with other possible sources of anisotropy.

11:00 AM

### (SINT-017-2017) Multi-scale anisotropy phenomena during sintering of iron powders

E. Torresani\*<sup>2</sup>; D. Giuntini<sup>1</sup>; C. Zhu<sup>3</sup>; T. Harrington<sup>3</sup>; K. S. Vecchio<sup>3</sup>;  
A. Molinari<sup>2</sup>; R. Bordia<sup>4</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Dept. Mechanical Engineering, USA

2. University of Trento, Department of Industrial Engineering, Italy

3. University of California, San Diego, USA

4. Clemson University, USA

The shrinkage anisotropy that occurs during sintering of metallic powders is correlated with the deformations introduced into the

material during the pre-sintering uniaxial cold compaction. The anisotropic porous structures, together with the inhomogeneous plastic deformation concentrated at the inter-particle contacts were analyzed for the case of iron powder. These non-uniformities are considered to be the cause of the occurrence of anisotropic dislocation pipe diffusion mechanisms, and thus of the undesired shape distortion during shrinkage. These considerations were used to develop and experimentally assess a general micromechanical model for shrinkage anisotropy during sintering of metallic powders. The proposed model describes the shrinkage rates in the compaction and transverse directions, as functions of both structural and geometrical activities of the samples. Dislocation densities can be estimated from the model equations, provided the availability of dilatometry and image analysis data. The reliability and applicability of the developed modeling framework is verified by comparing the calculated dislocation densities with the results of nanoindentation and electron backscattered diffraction derived lattice rotations.

11:20 AM

### (SINT-018-2017) Kinetics modelling and optimization of sintering processes

E. Moukhina\*<sup>1</sup>; G. Kaiser<sup>1</sup>; D. Rapp<sup>1</sup>

1. NETZSCH Geraetebau GmbH, Germany

Production of different ceramics like porcelain or technical ceramics require high firing temperatures depending on different parameters like quality of the raw materials, their percentage composition, particle sizes, particle distributions, atmospheres or temperature profiles. High production temperatures determine the energy costs, and therefore the optimization of the firing cycles is very important for the production process. Ceramic sintering usually accompanies the changing of the volume and linear sizes of the sample, which could be measured, analyzed and used for the process optimization. The current work contains the examples of typical solution steps for sintering optimization of different materials like porcelain or technical ceramics, in order to get the highest quality during the lowest costs. The examples of a standard porcelain green body were investigated, by means of simultaneous thermal analysis and dilatometry. The measured data were analyzed in the new NETZSCH software for kinetic analysis Kinetics Neo and the kinetic model of each individual reaction step in the sintering process was found. After that the software allows user to calculate the length change profiles for almost any temperature-time-progression or calculate the temperature profile for desired sintering profile. This saves a lot of time and effort compared to the classical way of trial-and-error.

11:40 AM

### (SINT-019-2017) Application and sintering of porous metal fiber materials (Invited)

H. Tang\*<sup>1</sup>; J. Wang<sup>1</sup>; A. Li<sup>1</sup>; J. Ma<sup>1</sup>

1. State Key Laboratory of Porous Metal Materials, China

Porous metal fiber materials are a class of very important materials combination of structure and function. They possess an array of attractive properties, including low density, high specific strength, high specific stiffness, high specific surface area, high permeability, excellent thermal conductivity. Therefore, they have found wide industry applications, such as filtration or separation, sound absorption, damping or vibration reduction, heat transfer, electromagnetic shielding, surface combustion, carriers of catalyst, and so on. Up to date, sintering remains to be the only commercially viable method for the production of porous metal fiber materials. The stainless steel fiber with diameter of 28 μm and the copper fiber with diameter of 90 μm were used as raw material. The sintering behavior of two kinds of metal fibers has been studied systematically for the first time, assisted with synchrotron radiation experiments to understand the evolution of the sintered joints. The simplified sintering model between two fibers was conducted. Based on the sintering theory of powder, the dynamics equations of sintered joints was established



under different diffusion conditions, including surface diffusion, grain boundary diffusion, volume diffusion and dislocation diffusion. At last, the sintering diagrams of two kinds of metal fibers were determined, and the forming and growing mechanisms of sintered joints were disclosed.

### Stress-assisted Sintering I

Room: Mission II

Session Chairs: Hyo Tae Kim, Korea Institute of Ceramic Engineering and Technology (KICET); Xialu Wei, San Diego State University; Michael Hoffmann, Karlsruhe Institute of Technology

10:00 AM

#### (SINT-020-2017) Reactive densification of ZrC and TiC (Invited)

V. Jayaram\*<sup>1</sup>

1. Indian Institute of Science, Materials Engineering, India

In a certain class of refractory materials, exemplified by transition metal carbides, it is possible to exploit the plasticity of the metal to densify compacts of metal-metalloids (such as Zr with C, B<sub>4</sub>C or BN) before significant reaction to the final carbide/boride takes place. The final density achievable is then dependent on the initial volume fraction of metal and the volume change upon reaction. The primary densification can be carried out at temperatures at which the metal creeps: which is usually much lower than what is required for sintering the ceramic. The above variables are sensitive to stoichiometry and the molar volumes of the reactants and therefore the final result can vary substantially in carbides for which the carbon:metal ration can vary from as little as 0.5 to 1.0 with concurrent changes in properties. The present talk will use ZrC and TiC, primarily produced in monolithic form, but also in composites, to illustrate how control experiments and modelling of densification can allow us to develop new heating-pressure schedules that permit processing of refractory carbides at low temperatures with minimal intervention of pressure.

10:40 AM

#### (SINT-021-2017) Effect of temperature, pressure, time, and sintering aid content on the densification and microstructure of hot-pressed boron suboxide

K. D. Behler\*<sup>1</sup>; J. LaSalvia<sup>2</sup>; C. J. Marvel<sup>3</sup>; M. C. Golt<sup>2</sup>; S. D. Walck<sup>4</sup>; M. P. Harmer<sup>3</sup>

1. U.S. Army Research Lab (SURVICE Engineering), Ceramics and Transparent Materials Branch, USA
2. U.S. Army Research Lab, Ceramics and Transparent Materials Branch, USA
3. Lehigh University, Department of Materials Science and Engineering, Center for Advanced Materials and Nanotechnology, USA
4. U.S. Army Research Lab, Coatings Corrosion and Engineered Polymers Branch, USA

The effect of temperature, pressure, time, and sintering aid content on the densification and microstructure of hot-pressed boron suboxide (B<sub>2</sub>O<sub>3</sub>) was investigated. Submicron-sized B<sub>2</sub>O<sub>3</sub> powders with 1 – 5 vol.% silica (SiO<sub>2</sub>) additions were hot-pressed at 1700°C – 1900°C for 1 – 4 hours with pressures between 20 MPa – 50 MPa applied at the maximum temperature. Shrinkage was monitored in-situ using an LVDT and post-processed to determine densification histories from which apparent activation energies were derived using the master sintering curve approach. SEM, EDS, and XRD were used to characterize the as-received B<sub>2</sub>O<sub>3</sub> powder and the microstructures of the consolidated samples. SiO<sub>2</sub> content, temperature, and pressure were found to have a strong effect on densification and microstructure. Full densification was achieved at lower temperatures for high SiO<sub>2</sub> contents relative to lower contents. In both cases, higher pressures resulted in higher densities. No apparent grain growth was observed at lower temperatures and SiO<sub>2</sub> contents, while abnormal grain growth was observed at higher temperatures and SiO<sub>2</sub> contents. AC-STEM was used to characterize segregation

of additives and impurities at both abnormal and normal grain boundaries. Differences in grain boundary segregant types and concentrations are believed the cause for the abnormal grain growth.

11:00 AM

#### (SINT-022-2017) Ultra high pressure sintering and synthesis of transparent polycrystalline cubic silicon nitride

N. Nishiyama\*<sup>1</sup>; R. Ishikawa<sup>2</sup>; H. Ohfuji<sup>3</sup>; H. Marquardt<sup>4</sup>; T. Taniguchi<sup>5</sup>; B. Kim<sup>5</sup>; A. Masuno<sup>6</sup>; Y. Ikuhara<sup>2</sup>; F. Wakai<sup>1</sup>; T. Irifune<sup>3</sup>

1. Tokyo Institute of Technology, Japan
2. University of Tokyo, Japan
3. Ehime University, Japan
4. University of Bayreuth, Germany
5. National Institute for Materials Science (NIMS), Japan
6. Hirotsuki University, Japan

Ultra high pressure sintering at 15.6 GPa was performed using a Kawai-type multi anvil press. We used  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> powder as starting material and obtained transparent bulk polycrystalline form of cubic silicon nitride, which is a high-pressure phase in Si<sub>3</sub>N<sub>4</sub> stable under pressure above 13 GPa. The dimensions of the synthesized disks are 2 mm in diameter and 0.5 mm in thickness. Real in-line transmission (RIT) was measured at wavelength between 200 and 1600 nm. The RIT value of visible light reaches 38%. Observations by atomic resolution scanning transmission electron microscopy were performed for the grain boundaries. We observed the presence of disordered/amorphous intergranular films (IGFs) with thickness less than 1 nm including multi-grain junctions. The thin IGFs produce no triple junction pocket, which can be one of the main reasons of the optical transparency. In order to show a potential of this transparent material as a structural material, we measured hardness and estimated fracture toughness by Vickers indentation tests. The Vickers hardness determined at 9.8 N is 34.9 GPa. This material is categorized as one of the third hardest materials next to diamond and cubic boron nitride. The fracture toughness is determined to be 3.5 MPa m<sup>0.5</sup>. This material is tougher than other transparent spinel ceramics such as MgAl<sub>2</sub>O<sub>4</sub> and  $\gamma$ -AlON.

11:20 AM

#### (SINT-023-2017) Effect of sintering additives on microstructure and properties of hot-pressed nitride composites

T. Tabares Medina\*<sup>1</sup>; A. Maitre<sup>1</sup>; N. Pradeilles<sup>1</sup>; M. Joinet<sup>2</sup>; P. Ponard<sup>2</sup>

1. European Centre of Ceramic, SPCTS, France
2. Thales Electron Devices, France

The aim of this work is to investigate the effect of oxide additives such as Y<sub>2</sub>O<sub>3</sub>, CaO and Al<sub>2</sub>O<sub>3</sub> in h-BN/AlN systems on their densification mechanism. Firstly, this work has been focused on the study of experimental conditions (heating rate, load, and starting composition) of the liquid phase formation. Its thermal stability and its role on the densification mechanism during hot pressing have been also studied thanks to characterization methods like SEM, TEM, dilatometry, TGA-DTA, XRD. Secondly, a particular attention needed to be paid to establish microstructural correlations between the properties of secondary phases coming from the sintering additives and the physical features of hot pressed specimens. The results indicate that 3wt.% of sintering agents is necessary to full densify h-BN/AlN composites. XRD and SEM characterizations clearly show the formation of aluminate based secondary phases which segregate at the grain boundaries. In the case of yttria doped composite, YAP (YAlO<sub>3</sub>) and YAG (Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>) phases were found. TEM observations indicate the presence of vitreous and crystallized phases whose volume fraction depend mostly of the heating rate. So, it was demonstrated that the secondary phase composition, its state of crystallization and its volume repartition have a significant effect on the thermal, electrical and mechanical properties.

11:40 AM

### (SINT-024-2017) Comparison of the constitutive laws for stress-assisted densification powder compact

H. Camacho Montes<sup>\*1</sup>; A. Garcia Reyes<sup>2</sup>; R. Bordia<sup>3</sup>

1. Universidad Autonoma de Ciudad Juarez, Fisica y Matematicas, Mexico
2. Inter ceramic Technological Center, Mexico
3. Clemson University, USA

Commonly used constitutive laws for crystalline and viscous materials have been compared to predict the densification behavior under hot-pressing and sinter-forging. Experimental results, from literature, have been used to extract the constitutive laws for amorphous and crystalline materials and used to predict behavior under a different set of loading conditions. In addition, the effect of temperature on densification for stress-assisted sintering has been investigated. It is shown that the two commonly used constitutive models for viscous sintering (Scherer and Skorohod – Olevsky) predict identical behavior for amorphous materials. For crystalline materials, there are measurable differences between the predictions of the Riedel – Svoboda and the Kuhn – Sofronis – McMeeking models. Finally, it is shown that the dependence of the normalized densification rate on temperature can be used to identify the appropriate constitutive law.

12:00 PM

### (SINT-025-2017) Grain growth studies and electrical response of stress assisted sintered $\text{BaLa}_4\text{Ti}_4\text{O}_{15}$

A. M. Senos<sup>\*1</sup>; M. Fernandes<sup>1</sup>; N. Selvaraj<sup>1</sup>; J. Abrantes<sup>2</sup>; J. Noudem<sup>3</sup>; O. Guillon<sup>4</sup>; P. Vilarinho<sup>1</sup>

1. University of Aveiro/ CICECO, Materials and Ceramic Engineering, Portugal
2. Instituto Politécnico de Viana do Castelo, UIDM, ESTG, Portugal
3. Université de Caen Basse-Normandie, CRISMAT, France
4. Forschungszentrum Jülich GmbH Institut für Energie und Klimaforschung, IEK-1: Werkstoffsynthese und Herstellungsverfahren, Germany

Stress assisted sintering (SAS) is frequently used in functional materials, either intentionally to densify at lower temperatures with compressive stresses, as in Hot pressing (HP), Hot isostatic pressing (HIP), Hot forging (HF) or, non-intentionally, as for films on rigid substrates and LTCC, where stresses are mainly generated by differential densification. Besides, mechanical stresses can be associated with electric field, as in Spark plasma sintering (SPS). Despite the fact that the role of applied stress on densification is well established, its effect on the movement of grain boundaries (GBs) for grain growth (GG) in ceramics has not been sufficiently addressed. We have been investigating this topic in functional materials, namely in  $\text{BaLa}_4\text{Ti}_4\text{O}_{15}$  (BLT), a key material for microwave applications and reported, for the first time, that the GB mobility is affected by external stresses, enhancing anisotropic GG and affecting electrical properties. In this work, BLT ceramics were densified by varied SAS techniques, HP, HIP, HF and SPS. The role of the different type of stresses and electrical field on the GG was studied by statistical analysis of the grain size distribution, separated into two populations, normal and abnormal grains. HRTEM and Impedance Spectroscopy were used to characterize the structure of the grains and GBs and to check the role of stresses on the electrical response.

## Randall German Honorary Symposium I

Room: Mission III

Session Chairs: Kathy Lu, Virginia Tech; José Manuel Torralba, Universidad Carlos III de Madrid

10:00 AM

### (SINT-026-2017) Statistics and stories on sintering (Invited)

R. M. German<sup>\*1</sup>

1. San Diego State University, Dept of Engineering, USA

Profiles of the individuals that helped shape modern conceptualizations on sintering are used to gain an overview on what were the critical steps in the evolution of sintering theory. A methodology is developed to isolate how sintering concepts progressed, showing who influenced who as a timeline is established. This critical mapping of sintering theory and its development is curious because of the diversity of backgrounds, nationalities, and even education differences. However, in a broad view it is evident large scale change from major industrial goals or global conflicts often impacted the field. This presentation will bridge from commercial developments to atomic theory to piece together a perspective on sintering theory and its ties to sintered engineering products.

10:40 AM

### (SINT-027-2017) Ceramics sintering and shaping using the electrical field assisted sintering method (Invited)

T. B. Holland<sup>1</sup>; H. Wang<sup>2</sup>; A. K. Mukherjee<sup>\*3</sup>

1. Colorado State University, Mechanical Engineering, USA
2. Purdue University, Materials Science and Engineering, USA
3. University of California, Davis, Materials Science and Engineering, USA

Ceramic powders are often produced by sol-gel method or by pyrolysis of some form of precursors. This presentation will emphasize the consolidation of such powders using the Electrical Field Assisted Sintering (EFAS) method. The role played by the external electrical field and/or the heating rate will be specifically investigated. This observation will be supported by in-situ sintering studies inside a TEM in the presence of a biased electrical voltage. One can take advantage of the EFAS process to produce functionally graded material by using asymmetric die design. Similarly, one can consolidate, as well as form to shape, ceramic components in-situ in the EFAS chamber by designing the die appropriately. Since EFAS is a fast process, grain growth can be minimized during consolidation, leading to excellent optical transmission properties in nanoceramics. Superplasticity in ceramics can be achieved at significantly lower temperatures. This investigation is supported in part by an ONR grant.

11:00 AM

### (SINT-028-2017) Strategies and practices for suppressing abnormal grain growth during liquid phase sintering (Invited)

S. L. Kang<sup>\*1</sup>

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Abnormal grain growth (AGG) often occurs during liquid phase sintering of ceramics. It commonly exhibits several characteristics: (i) AGG always in systems with faceted grains; (ii) usually more intensive AGG with particle size reduction; (iii) sometimes, introduction of an incubation period before AGG (incubated AGG); and (iv) stagnation of grain growth after impingement of abnormal grains. When a crystal grows in a liquid matrix, it is the result of serial processes of diffusion of atoms through the matrix and the reaction (atom attachment) at the solid/liquid interface. The growth rate of a faceted grain is governed by the interface reaction and the diffusion, respectively, for a driving force smaller and larger than a critical value and exhibits nonlinear growth kinetics. Based on this understanding, the mixed mechanism principle of microstructural evolution is deduced in terms of the coupling between the critical

driving force for appreciable growth of grains and the maximum driving force for the growth of the largest grain. Several strategies for suppressing AGG are deduced from the principle and are tested experimentally in carbides and oxides. The experimental results support the generality of the microstructural evolution principle for controlling the microstructure during liquid phase sintering.

**11:20 AM**

**(SINT-029-2017) Challenges and further developments in modeling of sintering (Invited)**

E. Olevsky\*<sup>1</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA

The existing challenges in modeling of sintering should be considered as incentives for further scientific endeavors, as opposed to serving as an impediment to the allowance for progress. Six possible directions of further developments in modeling of sintering are pointed out, including multi-scale modeling of sintering, development of on-line sintering damage criteria, modeling of nano-powder sintering, modeling of sintering with phase transformations or chemical reactions, modeling of field-assisted sintering, and the development of sintering optimization approaches.

**11:40 AM**

**(SINT-030-2017) Sintering behaviors of micron- and submicron-sized ZnO features (Invited)**

K. Lu\*<sup>1</sup>; H. Ju<sup>1</sup>

1. Virginia Tech, USA

Microfabrication of ceramic features has become a critical issue in realizing the miniaturization of devices. Micron-sized ZnO features attract much attention because of their unique properties and wide applications. However, limited effort has been devoted to understanding the sintering behaviors of micron-sized ZnO features. In this study, micron-sized ZnO features (ridges and rods) were prepared via centrifuge-aided micromolding and sintered at different sintering conditions. The sintering process distorts and even destructs the ZnO features due to the growth of grains and densification process. A quantitative relationship is established to describe and estimate the distortion process of the features during sintering. The difference in sintering behavior between bulk material and micron-sized feature is explained based on the corresponding sintering mechanisms and geometry factors. Effective strategies are proposed to reduce the distortion and destruction effects.

**12:00 PM**

**(SINT-031-2017) Coarsening and densification during sintering of nano-sized tungsten and tungsten carbide powder (Invited)**

Z. Z. Fang\*<sup>1</sup>

1. University of Utah, Metallurgical Engineering, USA

One of the main challenges of sintering nanosized powders is to control grain growth while achieving full densification. The mechanisms of coarsening and grain growth and densification during sintering of nanosized powder are more complex than that of sintering of coarser powders. It is critical to understand the detailed mechanistic steps in order to design processes that might lead to bulk nano materials with maximum density and minimum grain size. Using experimental data of sintering nanosized tungsten carbide and tungsten powders as well as selected data on other materials in literature, this article articulates a perspective examining the unique characteristics of nano sintering. When the density of the powder compact of nanosized particles are very low, coarsening of particles is responsible for most of the observed initial grain growth as well as densification. Because the mechanism of the initial coarsening depends on surface diffusion, surface diffusion thereby contributes to densification indirectly. Although the initial grain growth is only a small fraction of the total grain growth at the completion of the

sintering, it is the most critical part of grain growth that determines if the grain sizes of the material can be in nanoscale after sintering.

**Spark-plasma and Flash Sintering II**

Room: Bayview I

Session Chairs: Jan Raethel, Fraunhofer IKTS; Martha Mecartney, University of California, Irvine

**2:00 PM**

**(SINT-032-2017) Understanding and controlling flash sintering: A case study of ZnO (Invited)**

J. Luo\*<sup>1</sup>

1. University of California, San Diego, USA

This talk will review a series of our recent studies of flash sintering using ZnO as a model system. First, a coupled thermal and electric runaway model has been developed to forecast the onset flash temperatures [Acta Mater. 94:87 (2015)]. Second, guided by this model, we discovered a strong dependence of onset flash sintering temperature on the gas atmosphere; subsequently, ZnO specimens have been sintered to >97% densities in 30 seconds at furnace temperatures of <120 °C in flowing Ar + 5% H<sub>2</sub> [Scripta Mater. 106:26 (2015)]. Third, further studies investigate the fast densification mechanisms of flash sintering; specifically, rapid thermal annealing experiments with IR heating (without applied electric field/current) achieved similar densification and grain growth rates as flash sintering with comparable heating profiles, which demonstrated that the rapid heating profiles enable the ultrafast densification rates, at least for ZnO [Acta Mater. 125:465 (2017)]. Furthermore, various electric field/current effects on microstructural developments have been discovered and investigated. Most recently, we further demonstrated the feasibility of flash sintering of ZnO at room temperature (without any furnace/external heating!) to achieve >94% densities in seconds. Finally, another on-going research study fabricates dense nanocrystalline ceramics with controlled flash sintering.

**2:40 PM**

**(SINT-033-2017) Manipulation of ceramic materials to promote field-enhanced sintering**

R. E. Brennan\*<sup>1</sup>; V. L. Blair<sup>1</sup>; M. Kornecki<sup>1</sup>; N. Ku<sup>1</sup>; S. Raju<sup>1</sup>; F. Kellogg<sup>1</sup>

1. US Army Research Laboratory, USA

The application of magnetic, electric, microwave, and other types of external fields during sintering of ceramic materials often has a profound influence over the densification process, altering the free energy of the system and enabling densification of ceramic materials at lower temperatures and shorter times compared to conventional sintering processes. The ability to rapidly densify materials under less extreme processing conditions can allow for preservation of the nanoscale, providing the opportunity to improve mechanical properties that are vital to Army protection applications, including strength, hardness, and fracture toughness. In addition, techniques for manipulating materials to make them more responsive to specific fields can be applied to amplify these effects. For example, the addition of (a) transition metal dopants and ferromagnetic additives to ceramics that typically exhibit weak magnetic behavior, (b) electrically conductive phases that produce bulk conductivity pathways, and (c) susceptor materials that react to microwaves, can make them significantly more responsive when the respective fields are applied. Research demonstrating these effects through electric field sintering methods, single-mode microwave sintering, and thermomagnetic processing will be emphasized for developing advanced materials to meet Army needs.



3:00 PM

## (SINT-034-2017) Net shape flash spark plasma sintering of metal and ceramic powders

E. Olevsky<sup>\*1</sup>; G. Lee<sup>1</sup>; C. Maniere<sup>1</sup>; J. McKittrick<sup>2</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA
2. University of California, San Diego, USA

Flash sintering method can densify some ceramic powders very rapidly, often - in less than one second. However, this method is difficult to control and it cannot easily produce net shape parts. A different approach enabling the achievement of similar flash sintering phenomena based on the spark plasma sintering (SPS) process is developed. In this method, the electric current is concentrated in the powder sample, and an electrically insulated die is utilized to easily control the shape of the processed component. Usually, the flash sintering method is applicable to materials subjectable to a runaway of their electrical properties. The newly developed approach employing various direct/hybrid configurations is applicable to a very broad spectrum of material systems from insulative powder materials to conductive metal powders. A finite element approach has been employed to analyze the electric current, temperature and densification fields inside the samples for each considered net shape flash spark plasma sintering (NFSPS) configuration. The developed flash approach integrates the advantages of the SPS technology in terms of densification and microstructure control and improves the SPS industrial applicability by enabling net shaping and significantly reducing the operation time.

3:40 PM

## (SINT-035-2017) Flash sintering of oxide nano-particles: A percolation phenomenon and particle surface softening as a universal behavior (Invited)

R. Chaim<sup>\*1</sup>; G. Chevallier<sup>2</sup>; A. Weibel<sup>2</sup>; C. Estournes<sup>2</sup>

1. Technion - Israel Institute of Technology, Materials Science and Engineering, Israel
2. Université de Toulouse, Université Paul-Sabatier, CIRIMAT, CNRS INPT UPS, France

Percolation model was used to describe the rapid densification in ceramic nano-powder compacts subjected to flash sintering. The electrical system is composed of particles and their contact-point resistances, the latter soften/melt first due to preferred local Joule heating followed by the thermal runaway. Further local softening/melting has a hierarchical and invasive nature and propagates from the anode towards the cathode. The flash event signals the percolation threshold by invasive nature of the melt at the particle surfaces. Rapid densification is associated with local particle rearrangements due to attractive capillary forces induced by the softened/melted film at the particle surfaces. The dissipated electric power in oxide powder compacts, subjected to flash sintering, is several hundreds of  $W \times cm^{-3}$ . The energy-balance and the heat transfer considerations during the transient flash event were analyzed assuming local softening/melting at the particle contacts. These analyses are consistent with the local heating of the nano-particle surfaces to the ceramic melting temperature, or above it. The formation of the plasma by field emission of electrons also considered.

4:20 PM

## (SINT-036-2017) Onset, temperature gradient effects and defects in flash sintering

R. Janssen<sup>\*1</sup>; J. Pereira da Silva<sup>1</sup>; H. Al-Qureshi<sup>2</sup>; F. Keil<sup>3</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. University Federal de Santa Catarina, Brazil
3. TUHH, Germany

Flash sintering is a new process of sintering ceramics, discovered in 2010 by Cologna et al. at the University of Colorado. This process consists to apply an electrical field through a green body, who leads to densification at lower temperatures. This present work is directed

to develop a mathematical model which can predict the onset conditions (electrical field and temperature) for flash sintering. Our model uses an approach based on the heat equation coupled with an apparent activation energy kinetic equation for densification, leading to a system of nonlinear differential equations. Methods to analyze the onset of flash sintering via bifurcation theory are presented and correlated to experiments using different formulations, including pure Ytria-stabilized Zirconia, mixtures thereof with Barium Titanate as well as non-stoichiometric Urania. The results are in good agreement and confirm that the critical condition for the flash phenomenon is rather the temperature dependent resistivity than the sintering kinetics. In addition, in this paper results will be presented concerning the beneficial effect of temperature gradients as well as the influence and evolution of defects.

4:40 PM

## (SINT-037-2017) In situ study of lattice expansion during flash sintering

H. Charalambous<sup>\*1</sup>; S. K. Jha<sup>1</sup>; T. Tsakalakos<sup>1</sup>

1. Rutgers University, Materials Science and Engineering, USA

Flash sintering is a new technique that uses an electric field to pass current through a ceramic at a characteristic temperature in order to enhance the diffusion. The mechanism for flash sintering is under debate so the effect of electric field on crystal structure is studied to help understand how the change in crystal structure can explain this abnormal rate of sintering. The behavior of tin oxide (SnO<sub>2</sub>) under flash sintering conditions has been investigated in situ through use of energy dispersive x-ray diffraction. Due to the anisotropic nature of tetragonal SnO<sub>2</sub>, 13 diffraction peaks were tracked during the experiment. The lattice expansion for each peak was used to determine the geometry of expansion of the unit cell volume characteristic of flash sintering.

## Sintering of Multi-material and Multi-layer Systems I

Room: Bayview II

Session Chairs: Vikram Jayaram, Indian Institute of Science; Hector Camacho Montes, Universidad Autonoma de Ciudad Juarez

2:00 PM

## (SINT-038-2017) Simulation and experimental validation of deformation and stress for co-sintered ceramic laminated composites

S. E. van Kempen<sup>\*1</sup>; N. Giang<sup>1</sup>; R. Hammerbacher<sup>3</sup>; A. Bezold<sup>2</sup>; C. Broeckmann<sup>1</sup>; A. Roosen<sup>3</sup>; F. Lange<sup>4</sup>

1. RWTH Aachen University, Institute of Applied Powder Metallurgy and Ceramics at RWTH Aachen University e.V., Germany
2. RWTH Aachen University, Institute for Materials Applications in Mechanical Engineering, Germany
3. University of Erlangen-Nuremberg, Department of Materials Science, Chair of Glass and Ceramics, Germany
4. Siemens AG, Germany

Co-sintering of heterogeneous multilayer ceramics often leads to out-of-plane deformation, premature fracture, and delamination. This behavior is induced by residual stresses resulting from mismatches in thermal expansion and sinter shrinkage between adjacent layers that are mutually constrained after their initial bonding. In this work, the Skorohod-Olevsky model for viscous sintering was modified to consider anisotropic co-sintering behavior and implemented in an FE environment to predict residual stresses and laminate deformation for a variety of material combinations based on Al<sub>2</sub>O<sub>3</sub> and MgAl<sub>2</sub>O<sub>4</sub>. The shear viscosity and creep activation energy have been determined using viscosimetry experiments. Optical dilatometry experiments were used to validate the sinter shrinkage evolution of single layers and curvature of bilayers during sintering. Additionally, residual stress measurements of symmetric trilayer laminates were used to validate the simulated stresses. It has



been observed that the simulated densification and deformation/curvature behavior of monolithic materials and multilayer laminates show a discrepancy of 0.5 and 10% with experimental data, respectively. The simulated stress showed a good qualitative agreement with experiments.

### 2:20 PM

#### (SINT-039-2017) Low-Temperature Mullite Formation in Coatings deposited by Atomic Layer Deposition for High-Temperature Applications

K. Furlan<sup>\*1</sup>; T. Krekeler<sup>4</sup>; M. Ritter<sup>3</sup>; R. Blick<sup>2</sup>; G. A. Schneider<sup>1</sup>; K. Nielsch<sup>5</sup>; R. Zierold<sup>2</sup>; R. Janssen<sup>1</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. University of Hamburg, Institute of Nanostructures and Solid State Physics, Germany
3. TU Hamburg-Harburg, BE Electron Microscopy, Germany
4. TU Hamburg-Harburg, BE Electron Microscopy, Germany
5. IWF Dresden, Germany

Atomic Layer Deposition (ALD) process presents the thickness control in an Å-scale and surface self-limited reactions as its inherent advantages. In this work, an ALD low temperature super cycle was developed and thin films with varying ratio of Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub> were deposited both onto silicon wafers and into photonic crystals. The as-deposited films were amorphous, but conversion to mullite occurred already at a temperature of 1000°C, lower than the conversion temperatures found in powder processing of diphasic sol-gel route (type II) and comparable to monophasic sol-gels (type I). This is related to the high compositional mixing of the deposited Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub> films, which are homogeneous in the atomic scale. By means of such a mullite coating the structural stability of an inverse photonic crystal was increased up to 1400 °C.

### 2:40 PM

#### (SINT-041-2017) Sintering of high entropy alloys (Invited)

Y. Liu<sup>\*1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

High entropy alloys (HEAs) are composed of 4-5 metallic elements with almost equiatomic proportions. HEAs have great potentials for applications in high temperature, high corrosion and high irradiation environment. Powder metallurgy becomes important in processing HEAs, because the process of casting, inhomogeneous and coarse microstructures usually are unavoidable. The presentation introduced our recently progress in processing HEAs by powder metallurgy. Both the gas atomization and mechanical alloying were used to synthesize HEA powders, and then the powders were spark plasma sintered, hot extruded or selective laser sintered. Powder metallurgical HEAs cannot be fully consolidated without primary particle boundaries and residual porosities due to the complex alloy systems. Hot extrusion leads to a fully recrystallized equiaxed microstructure, but it is difficult to obtain complex-shaped parts. Finally, using selective laser sintering, HEAs with fine microstructures and near net shape were successfully prepared. The PM HEAs have both higher tensile strength and ductility than cast alloys. The improvement of mechanical properties is caused by the solid solution strengthening, grain boundary strengthening and the homogeneous microstructure, which increases the work hardening ability. In general, powder metallurgy can be considered as a promising way for preparing large-sized and complex-shaped HEAs with high mechanical properties.

### 3:40 PM

#### (SINT-042-2017) Processing of Ti<sub>6</sub>Al<sub>4</sub>V compacts with graded porosity for biomedical applications

J. L. Cabezas-Villa<sup>3</sup>; D. Bouvard<sup>2</sup>; L. Olmos<sup>\*1</sup>; J. Lemus-Ruiz<sup>3</sup>

1. Universidad Michoacana de San Nicolás Michoacana, INICIT, Mexico
2. Université Grenoble Alpes, CNRS, Grenoble, SIMAP, France
3. Universidad Michoacana de San Nicolas de Hidalgo, IIMM, Mexico

Materials with graded porosity were processed by sintering two-layer Ti6Al4V alloy powder compacts and their microstructural and mechanical features are characterized. Several compacts with various compositions and configurations were fabricated by die pressing. The initial material for each layer may be fine (below 20 µm), coarse (76-106 µm) powder or fine powder mixed with coarse (100-400 µm) salt particles acting as pore formers. The volume fraction of salt particles may vary between 15 and 50%. Two geometrical configurations were tested, with either axial or radial distribution of the layers. After salt elimination, the two-layer compacts were debinded and next sintered in a vertical dilatometer at 1260 °C in Ar atmosphere. Their shrinkage kinetics are compared to the ones of homogeneous compacts. The microstructure of sintered materials was observed by both SEM and computed microtomography (CMT). The porosity of layers ranged between 5 and 75% depending on the initial material. No crack was detected at the interface between layers although adjacent layers had different densification kinetics. Quantitative analysis of 3D images from CMT provided microstructure features as size, shape and connectivity of pores. The mechanical properties of the materials were estimated from compression tests. CMT imaging of samples after interrupted compression tests provided information about failure mechanisms

### 4:00 PM

#### (SINT-043-2017) Sintering and processing of a novel titanium matrix composite

C. Zhou<sup>\*1</sup>; Y. Liu<sup>1</sup>; B. Liu<sup>1</sup>

1. Central South University, State Key Lab of Powder Metallurgy, China

Titanium matrix composite (TMC) is a type of materials combining attractive properties of both titanium matrix and reinforcing phase(s). The application for TMCs is limited due to a high material cost and difficulties in manufacturing. In this work, we introduced a novel Ti-HSS (high-speed steel) composite synthesized by using powder metallurgy (P/M) method. Unique microstructures such as particulate, layer, and fiber reinforcements can be designed and fabricated through processing methods including low-temperature sintering, heat treatment, and hot working. It is indicated that a composite with HSS particulate reinforcements was obtained after sintering. It was also found that during the sintering, reinforcing TiC particles were generated near the interface between HSS and Ti matrix, due to the diffusion of carbon from HSS phase to Ti matrix. The interface can be optimized by a heat treatment. Moreover, the as-sintered parts can be hot worked by using hot forging or rotary swaging to achieve higher densities. The hot working also created a fiber-strengthening Ti-HSS microstructure, which lead to improvement for the mechanical properties. This work provide a low-cost P/M method to fabricate Ti-HSS TMCs with potential for wide applications.

4:20 PM

### (SINT-182-2017) Fabrication of carbon fibre reinforced MAX-phase ( $\text{Ti}_2\text{AlC}$ ) composite

J. H. Nel<sup>1</sup>; D. C. Blaine<sup>\*1</sup>; L. Sigalas<sup>2</sup>

1. Stellenbosch University, Mechanical and Mechatronic Engineering, South Africa
2. University of the Witwatersrand, School of Chemical and Metallurgical Engineering, South Africa

A continuous carbon fibre reinforced MAX-phase composite, where the MAX-phase used is  $\text{Ti}_2\text{AlC}$ , (C- $\text{Ti}_2\text{AlC}$ ) was created as a prepreg material using a slurry infiltration process.  $\text{Ti}_2\text{AlC}$  powder particle size was reduced to 300 nm by attrition milling in a liquid medium. Milled powder was analysed using dynamic light scattering, energy-dispersive X-ray spectroscopy (EDS), powder X-ray diffraction (PXRD), and scanning electron microscopy (SEM). Infiltration processes were investigated and it was found that neither vacuum slurry infiltration nor electrophoretic deposition (EPD) of suspended powders could be used to infiltrate sufficient powder particles into interfibre spaces. Pressure slurry infiltration was used to create the C- $\text{Ti}_2\text{AlC}$  composite prepreg. Samples consisting of eight prepreg layers were sintered using spark plasma sintering (SPS) technique. Computed tomography (CT) scanning was used to establish similarity between sintered samples and to identify large voids and low density areas. The ball on three balls (B3B) test method was used to compare strength values of the sintered C- $\text{Ti}_2\text{AlC}$  discs to that of sintered  $\text{Ti}_2\text{AlC}$  discs. Fractured samples were investigated using optical microscopy and SEM. Finally, nano-CT scanning was used to show finer details within the sintered composite.

4:40 PM

### (SINT-181-2017) The sintering behavior of percolated metal-ceramic composites

A. S. Patnaik<sup>\*1</sup>; J. Antalek<sup>1</sup>; J. Rawson<sup>1</sup>

1. Morgan Advanced Materials, Technology, USA

The advanced materials industry routinely uses metal ceramic composites for many high stress or high demand environments ranging from components for sensors and drill supports for the oil and gas industry to automotive, aerospace and energy storage/generation applications. Often a cermet is used where the strength of the ceramics needs to be combined with the toughness of metals. In other cases the cermet is used for electrical heaters/ bushings/contacts in high temperature or aggressive environments to apply their thermal and electrical conduction properties. With the emerging technologies in the above mentioned fields especially those in highly corrosive or reactive environments or high temperature or both, the densification and microstructure of the cermet plays an important role. This presentation will first briefly introduce the percolation theory and then proceed to analyze how the sintering behavior of a cermet changes with the degree of percolation. The analysis will be based on the degree of densification, effect of sintering thermal profiles and sintering environment based on dilatometry, bulk microstructure, surface morphology and bulk electrical properties. The factors determining the transfer of the sintering process to a robust manufacturing process to yield high quality products will also be discussed.

## Sintering of Nanostructured Materials II

Room: Bayview III

Session Chairs: Gary Messing, Penn State University; Xiaoyan Song, Beijing University of Technology

2:00 PM

### (SINT-044-2017) Ordered pore structures at elevated temperatures (Invited)

R. Janssen<sup>\*1</sup>; K. Furlan<sup>1</sup>; R. Pasquarelli<sup>1</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany

Highly reflective coatings can be created using ordered pores in matrices with high refractive index, e.g. 'inverse opals' obtained by inversion of the colloidal crystals offering thereby pronounced photonic bandgaps. For reflecting radiation energy, structures with pores size around the  $\mu\text{m}$  range are interesting as they can be considered as candidates for thermal barrier coatings or selective emitter in thermophotovoltaics. At high temperature, however, these structures can exhibit significant undesired microstructural changes (due to phase changes, grain growth, sintering, etc.) that result in a loss of the desired photonic properties. At present, we focus on  $\text{Al}_2\text{O}_3$  and Mullite inverse opals prepared from templates by vertically self-assembled PS monospheres, ALD infiltration and subsequent calcination. In the presentation we will report the synthesis, microstructure as well as resultant properties. Special emphasis will be given to structural changes during heat treatments. These confirm that by respective tailoring coatings with ordered porosity can even serve up to 1400°C. In addition to  $\text{Al}_2\text{O}_3$  and Mullite, also  $\text{TiO}_2$  inverse opals have been investigated showing a yet unknown stability of the Anatas phase at high temperatures. In general, our results confirm that both mass transport by diffusion as well as phase changes are the key parameters for structural stability at elevated temperatures of these highly porous structures.

2:40 PM

### (SINT-045-2017) Thin films of surface water in nanocrystalline MgO: Influence on particle coarsening, coalescence and grain morphology evolution

O. Diwald<sup>\*1</sup>; D. Thomele<sup>1</sup>; A. Gheisi<sup>2</sup>; J. Bernardi<sup>3</sup>

1. University of Salzburg, Chemistry and Physics of Materials, Austria
2. Friedrich-Alexander-Universität Erlangen Nürnberg, Germany
3. Vienna University of Technology, Austria

An important question to conventional sintering is how and to what extent residual water inside metal oxide particle powders affects coarsening and coalescence during heat treatment. We employed powders of MgO nanocubes with size distributions in the range below 10 nm as a model system for transformation studies of nanocrystalline particle systems. Using X-ray Diffraction, Electron Microscopy and Sorption Analysis we characterized particle powders at different levels of dispersion and after heat treatment in different gas environments. In the course of these studies we were able to describe the impact of adsorbed water on the evolution of MgO grain size, morphology and the level of intergrowth. While in water free environments, which are guaranteed at base pressures of  $p < 10^{-5}$  and under continuous pumping up to annealing temperatures of  $T = 1173$  K, we obtained grains of almost cubic habit with predominant (100) planes, thin films of water were found to promote mass transport on such particle systems. This leads to substantial particle growth and intergrowth. At the same time, it stabilizes (110) faces and step edges on the grain surfaces and interfaces. The concomitant effects of kinetics and thermodynamics, which is based on the hierarchy of surface energies, to microstructure evolution inside MgO nanoparticle ensembles will be discussed.

**3:00 PM****(SINT-046-2017) Restrained nanosintering to produce designed nanostructured solids**B. N. Feigelson<sup>\*1</sup>; J. A. Wollmershauser<sup>1</sup>; K. Manandhar<sup>1</sup>

1. US Naval Research Laboratory, USA

With designed bulk nanostructured solids, one could potentially combine properties that are mutually exclusive in single bulk material, reveal new phenomena and obtain desired performance. However, a major research challenge and roadblock is how to produce 3D nanostructured materials consistently with the required phases arranged in designated spatial order that are at the same time fully dense without porosity and detrimental phases. We developed an enhanced high pressure sintering (EHPS) process for restrained nanosintering, to consolidate nanoparticles (NPs) into monolithic nanostructured solids while preserving nanoparticle initial size and structure. Fully dense nanocrystalline transparent ceramics with grain size down to 3.5 nm was produced. Such ceramics exhibit Hall-Petch type relation down to 15 nm with unusual behavior at the low end of the grain size. Applying restrained nanosintering process to core/shell NPs offer fundamentally new means for nanostructured solids design and tailoring their basic properties. To provide flexibility in core/shell NPs design, a particle atomic layer deposition (p-ALD) reactor was incorporated in the EHPS facility. The new setup allows to controlling environment during all stages of the nanoparticles processing, ALD and sintering. Core/shell nanocomposite ceramics was produced and characterized for the first time.

**3:40 PM****(SINT-047-2017) Ceramic/organic supercrystalline nanocomposites with exceptional isotropic mechanical properties**D. Giuntini<sup>\*1</sup>; B. Domenech<sup>1</sup>; B. Bor<sup>1</sup>; D. Benke<sup>1</sup>; G. Schneider<sup>1</sup>

1. Hamburg University of Technology (TUHH), Institute of Advanced Ceramics, Germany

A novel route to produce ceramic/organic nanocomposites presenting an exceptional combination of mechanical properties has been developed. Organically-coated ceramic nanoparticles are organized into supercrystals through a combined evaporation-sedimentation process, leading to the self-assembly of nanoparticles at much larger scales than the ones traditionally explored. A simple sequence of pressing and heating follows. The resulting nanocomposites present a unique combination of bending modulus, hardness and strength values. Differently from what usually proposed in the literature, these nanocomposites have isotropic characteristics, and the processing routine is applicable to a variety of material systems. Alternative organic ligands and nanoparticles' constituents and morphologies are being explored, while the manufacturing process is being perfected. Additionally, the nanocomposites are currently being employed as building blocks for the production of hierarchical materials. Spark plasma sintering (SPS) has also been applied to such materials, a process that has led to multiple interesting results. SPS allows the minimization of defects when shifting to macro-scale components, and the introduction of multifunctionality, by enhancing the magnetic properties of iron oxide in the bulk form.

**4:00 PM****(SINT-048-2017) Preparation of nanohydroxyapatite based scaffolds fabricated using magnesium as a porogen**G. Adamek<sup>\*1</sup>

1. Poznan University of Technology, Institute of Materials Science and Engineering, Poland

Hydroxyapatite based materials have the potential for orthopedic application due to their excellent biocompatibility and bioactivity. In this work a series of scaffolds based on nanohydroxyapatite (nHA) were prepared using magnesium as a porogen. The scaffolds were fabricated using different types of Mg particles (nano and micro scale powders) which is also biocompatible material. The nano

powders were prepared using high energy ball milling and the micro powders were commercially available. The phase, microstructure, surface characteristic, porosity and biocompatibility were studied. In their research authors used SEM, EDS, XRD and BET techniques to characterize the influence of type of initial powders on nHA based scaffold properties. Furthermore, biological evaluation based on osteoblasts and fibroblasts were carried out. The results show that the nHA based scaffolds with wide range of porosity and with small addition of Mg had high potential for clinical application in hard tissue implant performance.

**4:20 PM****(SINT-049-2017) Effect of SiC incorporation on microstructure and mechanical properties of hot-pressed Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub> ceramics with eutectic composition**A. Henniche<sup>\*1</sup>; J. Ouyang<sup>1</sup>; Y. Ma<sup>1</sup>; Z. Wang<sup>1</sup>; Y. Wang<sup>1</sup>; Z. Liu<sup>1</sup>

1. Harbin Institute of Technology, School of Materials Science and Engineering, China

In the present work, nanopowders of Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub> were prepared via a chemical co-precipitation method, then, β-SiC nanoparticles were incorporated into the oxide powders in different volume fractions of 10 vol. %, 15 vol. % and 20 vol. %, respectively. Fully densified Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub>-SiC ceramics were fabricated by hot pressing for 1h at sintering temperatures of 1773K, 1823K and 1873K, respectively. The as-sintered composite has a dense and uniform microstructure, The GdAlO<sub>3</sub> grains are entangled with α-Al<sub>2</sub>O<sub>3</sub> grains as indicated by SEM observations. SiC nanoparticles are found within the interior of α-Al<sub>2</sub>O<sub>3</sub> grains or at the interfaces of the entangled α-Al<sub>2</sub>O<sub>3</sub> and GdAlO<sub>3</sub>. Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub>-SiC composite with 10 vol. % SiC sintered at 1773K exhibits a flexural strength of 516 MPa at room temperature. With the increase of SiC contents from 10 vol. % to 20 vol. %, the Vicker hardness of Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub>-SiC composites increases from 17.5 GPa to 19.5 GPa, while the fracture toughness of Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub>-SiC composites increases from 8.49 MPa.m<sup>1/2</sup> to 9.28 MPa.m<sup>1/2</sup>, respectively, which is significantly improved with respect to that (7.5 MPa m<sup>1/2</sup>) of unmodified Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub> ceramic. The toughening mechanisms of Al<sub>2</sub>O<sub>3</sub>-GdAlO<sub>3</sub>-SiC composites are attributed to crack deflection and bridging due to the presence of intragranular SiC nanophases observed from the indentation.

**4:40 PM****(SINT-050-2017) Influence of sintering temperature on micromorphology and microstructure of electrospun mullite nanofibers with different contents of SiO<sub>2</sub>**J. Wang<sup>\*1</sup>; X. Song<sup>1</sup>; W. Liu<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

Mullite nanofibers with SiO<sub>2</sub> contents of 20wt. %, 24wt. %, 28wt. % and 32wt. % were successfully obtained using electrospinning method followed by sintering. The micromorphology and microstructure of the as-spun and sintered nanofibers were investigated. The results showed the as-spun nanofibers with different contents of SiO<sub>2</sub> were all continuous with smooth surface and had an average diameter of about 300nm. After sintering, the surface of nanofibers became coarser and the diameter decreased to about 200nm. The sintered nanofibers with higher content of SiO<sub>2</sub> had smoother surface and denser structure. When sintered at 873K, the nanofibers were all amorphous. With temperature increased to 1273K, mullite and γ-Al<sub>2</sub>O<sub>3</sub> phase were simultaneously observed in the nanofibers and with higher content of SiO<sub>2</sub> the content of γ-Al<sub>2</sub>O<sub>3</sub> phase decreased and mullite increased. After sintering at 1473K, they all changed to mullite and the grain sizes were bigger with higher content of SiO<sub>2</sub>. The nanofibers sintered at 1273K with 28wt. % SiO<sub>2</sub> content had optimal performance with smooth surface, dense microstructure and small grain size.



## Modeling and Simulation of Sintering at Multiple Scales I

Room: Mission I

Session Chair: Rajendra Bordia, Clemson University

### 2:00 PM

#### (SINT-051-2017) Influence of initial packing, pore formers and sintering on mechanical properties of porous ceramics: Discrete element method simulations (Invited)

D. Jauffres<sup>\*1</sup>; C. Martin<sup>1</sup>; R. Bordia<sup>2</sup>

1. University of Grenoble Alpes, SIMAP, France
2. Clemson University, Materials Science and Engineering, USA

Porous ceramics are used in an expanding field of functional applications in bioengineering (e.g. bone substitution), chemical engineering (e.g. catalyst support) or energy engineering (e.g. fuel cells, Li-ion batteries, insulation). Minimal mechanical properties requirement often guide the design of these materials by limiting the porosity amount and in turn their functionality. Partial sintering, with occasionally the addition of pore formers, is the main processing route used to obtain porous ceramics. Even up to relatively high density (~0.85) partially sintered ceramics still exhibit a granular character inherited from its powder processing route. This makes the Discrete Element Method (DEM) a tool of choice to study the mechanical properties of these materials. First, the use of a DEM sintering model to generate a realistic set of various microstructures will be described. Then, the influence of initial packing, pore former addition and sintering (in particular how the matter is redistributed) on the young's modulus and toughness will be studied via DEM simulation and discussed. In particular it will be shown that porosity is not the only parameter governing the mechanical properties and that, for a given amount of porosity, the microstructure could be optimized for better mechanical performance.

### 2:40 PM

#### (SINT-052-2017) In-situ de-agglomeration during powder consolidation

D. Giuntini<sup>\*1</sup>; A. Maximenko<sup>3</sup>; E. Olevsky<sup>2</sup>

1. Hamburg University of Technology (TUHH), Institute of Advanced Ceramics, Germany
2. San Diego State University, USA
3. NAS Ukraine, Ukraine

The issue of agglomeration, cause of the formation of hierarchical and inhomogeneous porous structures in powder compacts, is addressed with an analytical and numerical approach. Agglomeration issues affect in particular nanopowders, a growingly interesting material system for both conventional and field-assisted sintering, and pre-densification de-agglomeration treatments often do not provide the desired results. In-situ de-agglomeration is thus attempted by means of a temperature-based procedure. The correlations between temperature, porous material structure and tooling geometry are explored. Densification kinetics, sintering stress, bulk and shear moduli of agglomerated compacts are modeled as functions of the hierarchy characterizing the porosity and the nonlinear viscous rheology of the material. The material nonlinearity is expressed by the strain rate sensitivity parameter, which in turn is dependent on temperature. An empirically-based explicit formulation of strain rate sensitivity as a function of temperature is provided. A subsequent tuning of the strain rate sensitivity allows the optimization of the thermal regime in order to obtain in situ de-agglomeration of the SPS specimen or the production of tailored material structures.

### 3:00 PM

#### (SINT-053-2017) FE simulation of temperature distribution, densification and grain growth during field assisted sintering of 316L stainless steel

S. Sistla<sup>\*1</sup>; M. Hajeck<sup>1</sup>; A. Kaletsch<sup>1</sup>; C. Broeckmann<sup>1</sup>

1. RWTH Aachen University, IWM, Germany

Finite Element (FE) simulations of different sintering types (such as HIP, Field Assisted Sintering Technologies (FAST)) are accurate in predicting various sintering characteristics such as densification, grain growth, shape changes and temperature distribution in the samples. In FAST, due to the complex behavior that arises by combining electrical, thermal and structural effects into a single step, FE simulations are rarely investigated in literature. Normally, kinetics of densification and grain growth during FAST are analyzed under isothermal conditions. In this work, various FAST experiments at different sintering temperatures (800-1100°C) and different dwelling times (2-8 minutes) are conducted to determine the boundary conditions for the FE simulations. The accuracy of predicting the temperature distribution, density and the grain size are increased by determining the material properties dependent on temperature for Stainless Steel 316L and graphite. Furthermore, coupled electrical, thermal and structural simulations of FAST of 316L are carried out within this research work. The material behavior is defined by using user defined constitutive equations for conducting materials, which also include the grain growth kinetics. The study concludes with the comparison of different sintering characteristics (mentioned above) with the conducted experiments.

### 3:40 PM

#### (SINT-054-2017) Microscopic sintering forces acting among particles during sintering by grain boundary/surface diffusion (Invited)

F. Wakai<sup>\*1</sup>; S. Kanchika<sup>1</sup>; G. Okuma<sup>1</sup>; N. Nishiyama<sup>1</sup>

1. Tokyo Institute of Technology, Laboratory for Materials and Structures, Japan

Large scale simulation of sintering of a huge number of particles are successfully achieved by using discrete element method (DEM) recently. The mechanics underlying this method is a relationship between the relative velocity and the sintering force acting among neighbor particles. The macroscopic sintering force, which is the thermodynamic driving force for shrinkage, arises from microscopic sintering forces among many particles in the initial stage of sintering. Although the concept of sintering force has been defined to a circular grain boundary between two spherical particles, the contacts among multiply connected particles has complicated shapes generally. Here, we show that the concept of sintering force can be defined for any shape of contacts in sintering by grain boundary diffusion/surface diffusion. This result suggests that DEM simulation can be applied not only to the initial stage but also for the intermediate stage of sintering.

### 4:20 PM

#### (SINT-055-2017) Pattern formation during current sintering: Simulation models

L. Engelke<sup>\*1</sup>; M. Jongmanns<sup>1</sup>; C. Gorynski<sup>2</sup>; M. Winterer<sup>2</sup>; D. E. Wolf<sup>2</sup>

1. University of Duisburg-Essen, Physics, Germany
2. University of Duisburg-Essen, Nanoparticle Process Technology, Faculty of Engineering, Germany

A complex model is required to be able to check, in how far the following mechanisms are responsible for the observed pattern formation during current sintering in systems like aluminum doped zinc oxide: Temperature and stress dependent particle rearrangement, particle coarsening, mobility of dopants, phase transformation, heterogeneous Joule heating and Peltier effect. We combine transport calculations by means of a network model with discrete element simulations of particle motion and a kinetic model



for shrinkage and mass transfer between particles. This simulation tool will allow to simulate conventional sintering, hot pressing, current-activated pressure-assisted densification and other experimental protocols to convert a powder into a polycrystalline bulk solid. Crucial elements of the model have been successfully developed and applied to special cases: Our model of shrinkage and mass exchange between particles unifies and generalizes previous models for conventional sintering under the constraints of constant dihedral angle on the one hand, or of constant grain volume on the other. Our model of thermoelectric transport coupled to particle motion (Angst, Engelke, Winterer, Wolf, 2017) predicts segregation of a two-component mixture of thermoelectric materials or homogenization, depending on the model parameters.

#### 4:40 PM

##### (SINT-056-2017) Modeling of electric current assisted sintering for porous-graded materials design

A. M. Locci<sup>\*1</sup>; R. Orrù<sup>1</sup>; G. Cao<sup>1</sup>

1. Università degli Studi di Cagliari, Italy

Electric Current Assisted Sintering (ECAS) is a technology able to consolidate powders in shorter processing time with respect to conventional pressure-assisted methods. Its versatility makes ECAS also suitable for the production of porous-graded materials, which are attractive in several fields (biomedicine, solar energy absorbers, etc). The obtention of materials with tailored porosity gradients clearly calls for a thorough comprehension of all phenomena (heat transfer, current flow, etc) affecting sample shrinkage, which, in turn, depends upon material parameters, geometrical variables, surfaces properties, as well as operating conditions. Due to the large number of variables to be taken into account, mathematical modeling can be very useful for developing new experimental protocols aimed to produce the required spatial changes in material porosity. In this work, a suitable mathematical model able to quantitatively describe temperature, stress, velocity, density and electric current field inside the ECAS ensemble is developed. Numerical investigation of the effect of all parameters involved in the model will provide a useful design tool to develop porosity-graded-materials.

## Novel Sintering Processes I

Room: Mission II

Session Chairs: Olivier Guillon, Forschungszentrum Juelich; Stewart Silling, Sandia National Laboratories

#### 2:00 PM

##### (SINT-057-2017) Sintering by microwave heating for ceramics: New applications and recent developments (Invited)

F. Valdivieso<sup>\*1</sup>

1. Mines SE, France

The proposed communication will present both the research strategy used for general study of sintering by microwave heating and the main results obtained on ceramics materials in a consortium composed by three French research laboratories (CRISMAT, SIMAP and LGF). First of all, the instrumentation specifically dedicated to experimental study in a microwave furnace will be presented. Thanks to an accuracy control of the heating kinetics of the materials, it is possible to compare the processes involved and the difference with a conventional heating. Indeed, based on an MSC-type methodology, and kinetic studies, the determination of activation energies allows demonstrating the effects of the EM-field on sintering process. A complete study of different ceramics materials was carried out in order to compare microwave and conventional heating, both in terms of densification and microstructures evolution. Results concerning microwave sintering of various ceramics will be presented, like  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ,  $\text{ZnO}$ ,  $\text{CeO}_2$ , and composites materials  $\text{Al}_2\text{O}_3\text{-ZrO}_2$ ,  $\text{Al}_2\text{O}_3\text{-ZnO}$ . Moreover, thanks to the numerical simulation based on multiphysics coupling (electromagnetism, heat

diffusion) of the process it possible to determine the optimal experimental configuration in the optics of sintering varied-sized pieces in mono- or multi-mode cavity, but also to quantify the EM-field in a grained material.

#### 2:40 PM

##### (SINT-058-2017) Ultra-rapid microwave sintering of pure and $\text{Y}_2\text{O}_3$ -doped $\text{MgAl}_2\text{O}_4$

S. Egorov<sup>1</sup>; A. Ereemeev<sup>1</sup>; V. Kholoptsev<sup>1</sup>; K. Rybakov<sup>\*1</sup>; A. Sorokin<sup>1</sup>; Y. Bykov<sup>1</sup>

1. Institute of Applied Physics, Russian Academy of Sciences, Russian Federation

We report fast microwave sintering of pure and 1wt.%  $\text{Y}_2\text{O}_3$ -doped magnesium aluminate spinel ceramics to nearly full density with zero hold time. Nanosize high purity  $\text{MgAl}_2\text{O}_4$  powder with the specific surface  $S = 100 \text{ m}^2/\text{g}$  was pressed uniaxially into disks with a diameter of 20 mm and a thickness of 3 mm. Some samples were doped by dipping them into 0.5 mol/l water solution of  $\text{Y}(\text{NO}_3)_3$  for 10 minutes and subsequent annealing in air at 70 °C. The samples were heated in the applicator of a 24 GHz gyrotron system at a ramp-up rate of 15...200 °C/min to a preset temperature  $T_{\text{max}}$  in the range of 1250...1780 °C under a pressure of about 0.5 Pa. Upon reaching  $T_{\text{max}}$  the microwave power switched off automatically and the sample cooled down along with the thermal insulation. At all heating rates, when the absorbed microwave power density exceeded  $10 \text{ W}/\text{cm}^2$ , the density of the sintered undoped samples was above 98 %  $\rho_{\text{th}}$  at  $T_{\text{max}} \geq 1600 \text{ °C}$ . The density of the  $\text{Y}_2\text{O}_3$ -doped samples was above 99 %  $\rho_{\text{th}}$  at  $T_{\text{max}} \geq 1500 \text{ °C}$ . The XRD analysis revealed trace amounts of  $\text{Y}_4\text{Al}_2\text{O}_4$  formed in the doped samples microwave heated up to  $T_{\text{max}} = 1250 \text{ °C}$  and YAG in the samples heated up to 1600 °C. The SEM study has demonstrated a pronounced bimodal grain size distribution, abnormal grain growth, and fast segregation of Y atoms to the grain boundaries, indicating that the fast and full densification proceeds via liquid phase formation.

#### 3:00 PM

##### (SINT-059-2017) Mechanical properties of 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> composite ceramics prepared via conventional and microwave sintering

B. Liang<sup>\*1</sup>; C. Fang<sup>1</sup>; Y. Ai<sup>1</sup>; W. He<sup>2</sup>; W. Chen<sup>1</sup>; T. Chen<sup>2</sup>; C. Liu<sup>1</sup>

1. Nanchang Hangkong University, School of Materials Science and Engineering, China  
2. Nanchang Hangkong University, Instrumental Analysis Center, China

$\text{La}_2\text{O}_3/\text{Nb}_2\text{O}_5$  doped  $\text{Al}_2\text{O}_3$  ceramics (7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub>, volume percent) were prepared via conventional and microwave sintering processes. The effects of sintering temperature on densification, phase composition, microstructure and mechanical properties of 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> ceramics were compared between two sintering processes. The results show that both conventionally sintered and microwave sintered specimens consisted of  $\alpha\text{-Al}_2\text{O}_3$ ,  $\text{LaNbO}_4$  and  $\text{LaAl}_{11}\text{O}_{18}$ . The sintering temperature of  $\text{La}_2\text{O}_3/\text{Nb}_2\text{O}_5$  doped  $\text{Al}_2\text{O}_3$  ceramic was at least 100 °C lower than that of  $\text{Al}_2\text{O}_3$  ceramic. Microwave sintering processes remarkably accelerated the densification of 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> ceramics at lower temperature and within shorter time than conventional sintering. Some columnar grains, identified as  $\text{Al}_2\text{O}_3$ , were observed from the SEM images. The mechanical properties (Vickers hardness and fracture toughness) of microwave sintered specimens were better than that of conventionally sintered specimens, on account of high relative density, small grain size and the existence of columnar  $\text{Al}_2\text{O}_3$  grains. Microwave sintered 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> composite ceramics exhibited hardness of 12.4 GPa and fracture toughness of 6.2  $\text{MPa}\cdot\text{m}^{1/2}$  (1550°C, 15 min). The fracture toughness of 7.5La<sub>2</sub>O<sub>3</sub>/5Nb<sub>2</sub>O<sub>5</sub>-87.5Al<sub>2</sub>O<sub>3</sub> ceramics was above 100% higher than that of  $\text{Al}_2\text{O}_3$  ceramic.

### 3:40 PM

#### (SINT-060-2017) Microwave sintering of Al<sub>2</sub>O<sub>3</sub> base ceramics (Invited)

J. Chaix<sup>\*1</sup>; J. Croquesel<sup>1</sup>; D. Bouvard<sup>1</sup>; S. Saunier<sup>2</sup>; C. Carry<sup>1</sup>

1. University of Grenoble Alpes, SIMaP, France
2. Ecole des Mines de Saint-Etienne, France

Microwave sintering of  $\gamma$  and  $\alpha$  alumina, doped or not with MgO, and alumina-zirconia microcomposites was studied in both single mode and multimode microwave cavities. Specifically designed devices enabled to catch the densification kinetics of compacts during controlled heating rate experiments. FEM simulations allowed analyzing the heating process and estimating the electromagnetic field inside the Al<sub>2</sub>O<sub>3</sub> compacts, with and without susceptors. The results enlighten several aspects of the microwave sintering method. Pure alumina exhibits poor coupling ability with microwaves, so that susceptors are required to heat it in a multimode cavity. Accurate tuning of resonance in single mode cavity led to successful direct heating. A low amount of MgO makes direct heating possible in multimode cavities, and a very low amount of coupling second phase in microcomposite evidences an unexpectedly strong "internal susceptor" effect. Specific effects of microwaves on physical chemical mechanisms remain an open topic in the literature. The present work contributes on two aspects: the effect on  $\gamma$ - $\alpha$  phase transformation and the effect on sintering, including both densification and grain growth. Their identification requires careful experimental conditions and questioning about the effective electromagnetic field inside the compact during heating, which is affected by susceptors and by thermal insulating materials surrounding the compact.

### 4:20 PM

#### (SINT-061-2017) Effect of the absorbed microwave power density on the sintering of 3YSZ ceramics

I. Plotnikov<sup>1</sup>; S. Egorov<sup>1</sup>; A. Eremeev<sup>1</sup>; V. Kholoptsev<sup>1</sup>; K. Rybakov<sup>\*1</sup>; A. Sorokin<sup>1</sup>; Y. Bykov<sup>1</sup>

1. Institute of Applied Physics, Russian Academy of Sciences, Russian Federation

3 % yttria-stabilized zirconia (3YSZ) ceramic samples have been sintered to near-full density in ultra-rapid microwave heating processes with zero hold time. A crucial factor in the ultra-rapid ("flash") densification of ceramics is the onset of thermal instability. The development of the instability is determined by the balance between the microwave power absorbed volumetrically in the sample and the intensity of heat losses from the sample surface. To study the influence of the absorbed microwave power density on sintering, the intensity of heat losses was varied by using thermal insulation materials with different dielectric properties. The sintering experiments were carried out on a 24 GHz / 6 kW gyrotron system for microwave processing of materials. 3YSZ powder compacts were heated in air at a ramp-up rate of 15...150 °C/min to a preset temperature  $T_{max}$  in the range of 1100...1500 °C. Upon reaching  $T_{max}$  microwave power was switched off automatically and the sample cooled down along with the thermal insulation. It is demonstrated that the results of sintering depend substantially on the value of the absorbed microwave power density  $P$  (W/cm<sup>3</sup>). For example, the final density did not exceed 91 % of the theoretical value when the samples were heated to  $T_{max} = 1400$  °C with  $P < 5$  W/cm<sup>3</sup>, whereas at  $P = 80$  W/cm<sup>3</sup> a density of 95.8% was achieved at a temperature as low as 1100 °C.

## Randall German Honorary Symposium II

Room: Mission III

Session Chairs: Animesh Bose, Desktop Metal; Volker Piotter, Karlsruhe Institute of Technology

### 2:00 PM

#### (SINT-062-2017) Novel multi-use liquid phase for PM components (Invited)

J. Torralba<sup>\*1</sup>; M. Campos<sup>1</sup>; A. Galán-Salazar<sup>1</sup>

1. Universidad Carlos III de Madrid, Materials Science and Engineering and Chemical Engineering, Spain

Liquid phase sintering (LPS) is commonly used in powder metallurgy industry to enhance the performance and to tailor multi-phase microstructures. Nowadays, one of the main targets for LPS is to improve dimensional precision. This work tries to assess the liquid phase features that a multi-use liquid phase former should fulfill for sintering low-alloyed steels or for accomplishing sinter-brazing between PM steel components. The composition of the liquid phase promoter was designed according to thermodynamic and kinetic calculations in order to control solubility ratio (solid interface motion is in the  $\mu\text{m/s}$  range) and chemical activity. In this study, the spreading rate of the new composition was monitored by sessile drop experiments considering green (porous) and wrought (fully dense) substrates. After the test, in order to understand dissolution phenomena, new phases formation or penetration distance into the solid, the cross-section of the samples was examined. On the other hand, dimensional variation associated with the presence of a liquid phase in PM steels has been tackled during dilatometry trials. In the end, the ability to form a joint has been investigated as a possible filler material during sinter-brazing experiments with sintered steels.

### 2:20 PM

#### (SINT-063-2017) Novel materials for micro powder injection molding (Invited)

V. Piotter<sup>\*1</sup>

1. Karlsruhe Institute of Technology, Institute for Materials Research, Germany

Powder injection molding represents an attractive technology for manufacturing micro components offering a remarkably wide range of materials. A survey will be given on current and future applications focused (but not limited to) on micro system technology products. Keywords, among others, are functional materials, magnetic metals, and ceramic matrix composites. Special topic will be the powder injection molding of high-entropy-alloys or compositionally complex alloys, respectively, which represent a quite new type of materials in metallurgical research. They are expected to offer unique material properties. The related interdependencies of alloy composition, atomization, injection molding parameters, sintering procedures, densities, and obtained microstructures will be evaluated. Recent results and examples will be presented.

### 2:40 PM

#### (SINT-064-2017) Influence of the green microstructure on sintering shrinkage of metallic powders (Invited)

A. Molinari<sup>\*1</sup>

1. University of Trento, Industrial Engineering, Italy

This lecture presents the results of an experimental work aimed at introducing the deformed microstructure of green parts in the shrinkage kinetic model. Due to plastic deformation particles are in contact over areas depending on particle size and green density. Such a geometrical condition affects the relationships between neck growth and linear shrinkage (geometrical activity). The material is strain hardened in the neck region, what implies an increased diffusivity due to the density of structural defects (structural activity). The geometrical characteristics of particles in the contact regions in

the green parts were investigated by metallography. Investigation of the structural characteristics needs for an experimental technique sensitive to the local dislocation density. FEM was used to estimate local plastic deformation. Moreover, shrinkage was investigated by dilatometry, and curves were fitted by a modified kinetics model accounting for geometrical activity. The effective diffusivity was determined and related to the density of structural defects through the dislocation pipe diffusion model. The effect of the green microstructure on sintering shrinkage and on its anisotropy is discussed. The limits of the experimental approach are discussed, highlighting strength and weakness, and the further work to improve shrinkage kinetics model accounting for the microstructure of the green parts.

### 3:00 PM

#### (SINT-065-2017) Densification pathways – does the journey matter? (Invited)

D. C. Blaine\*<sup>1</sup>

1. Stellenbosch University, Mechanical and Mechatronic Engineering, South Africa

The press-and-sinter process is typically designed so that the majority of densification occurs during compaction, and relatively little occurs during sintering. The degree of densification at each process step is dependent on the powder characteristics, as well as the compaction and sintering parameters. As such, there are a number of parameters that can be manipulated in order to produce the required PM material. This study reviews the densification pathways that were followed in a variety of press-and-sinter experiments. Different powder blends, of titanium and 60Al:40V master alloy powders, provided a range of particle size distributions that resulted in varying compressibility and, likewise, varying potential for densification during sintering. Powder blends that have different apparent density, compressibility and sintering densification responses, may still end up at the same final density through manipulation of the compaction and sintering parameters. However, even though the final density may be similar, different densification pathways result in different final microstructures, and, as can be expected, different mechanical properties. Property models for PM materials very often rely on density alone in order to predict the relationship of final to fully dense behaviour. This study reflects on the influence of densification pathway on the prediction of final material properties by evaluating the data.

### 3:40 PM

#### (SINT-066-2017) A history of liquid phase sintering in space (Invited)

J. L. Johnson\*<sup>1</sup>

1. Elmet Technologies LLC, USA

Liquid phase sintering under reduced gravity conditions is significantly different than on Earth. Initial experiments with tungsten heavy alloys using parabolic trajectory rockets in the 1980s showed extensive grain agglomeration after microgravity sintering for less than a minute. Further investigation of the gravitational role in liquid phase sintering required longer sintering times available only through space flights. Experiments with tungsten heavy alloys conducted as part of Spacelab-J, IML-2, and MSL-1 on board Space Shuttle missions in the 1990s provided significant experimental data for comparison to model predictions. Many of the original hypotheses were proven to be incorrect. Analysis led by Professor German resulted in new models for densification, distortion, solid-liquid segregation, pore coarsening, grain growth, and mechanical properties. The development of these theories is reviewed and areas for future research are outlined.

### 4:00 PM

#### (SINT-067-2017) Improving liquid-phase sintering of iron based Ti(C,N) cermets (Invited)

E. Gordo\*<sup>1</sup>; M. Dios<sup>1</sup>; P. Alvaredo<sup>1</sup>; Z. Gonzalez<sup>2</sup>; B. Ferrari<sup>2</sup>

1. University Carlos III of Madrid, Materiales Science and Engineering, Spain
2. Institute of Ceramics and Glass, CSIC, Spain

This work studies the microstructural evolution of a Ti(C,N)-Fe based cermet during liquid-phase sintering and the role of Ni and C additions to the metal phase. Iron is known to have a poor wettability on Ti(C,N), which compromises its potential as binder phase for cermets. The addition of C decreases the solidus temperature of iron, but also influences the stoichiometry of ceramic phase. Similarly, Ni has been studied as sintering aid for ultrahigh temperature ceramics and has a recognised role as binder in cermets. The study of combined effects of both additions would provide a better understanding and control of the microstructure during sintering. A systematic study is presented including: thermodynamic simulation to know about changes in solubility of Ti(C,N) on metal phase as well as critical temperatures; experimental studies to measure the contact angle of liquid metal on Ti(C,N) substrates combined with interfacial analysis; powder preparation by colloidal techniques to reach a high distribution of phases from the first steps of processing; sintering studies at different temperatures, atmospheres and time; and characterisation of sintered samples by means of porosity, XRD, SEM, Raman spectroscopy and quantitative microstructural analysis. The relationships between composition of metal phase, wettability, distribution of phases on the sintered samples and lattice parameters of ceramic phase are discussed.

### 4:20 PM

#### (SINT-068-2017) Enhancing properties through liquid phase sintering (Invited)

M. Campos\*<sup>1</sup>; J. Torralba<sup>1</sup>; A. Galán-Salazar<sup>1</sup>; E. Bernardo<sup>2</sup>;

R. De Oro Calderon<sup>3</sup>

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For enhancing the material's properties is possible to introduce a liquid phase former that accelerates densification and may contribute to minimize the porosity and modulate phase transformations. Liquid phase can be tailored to control mass transport phenomena, to adjust the dimensional change and to proceed the sintering in solid state. Different dissolutive capacity of the liquid phases could lead to different dimensional behavior: low dissolutive phenomena could favor a fast and homogenous liquid distribution but a higher risk of swelling is associated. Depending on the composition of liquid phase promoter and the starting green compact features the results might cover a wide range of possibilities. The proposed methodology for low alloyed steels, starts with the theoretical calculations, to identify the alloying systems that provide the liquid metal which fulfill the requirements. Special attention must be given to the solubility among the solid that coexists with the liquid during wetting step. Once the prototypes are defined, based on the sessile drop method and selecting a proper substrate is possible to study the wetting angle, the kinetics of the spreading, and the main parameters to validate the liquid behavior. This study is completed with dilatometries and microstructural analysis to optimize and predict the liquid metal evolution during sintering.



## Poster Session

Room: Regatta Pavilion

5:30 PM

### (SINT-P001-2017) Chemical reactions between natural chalcopyrite and copper in SPS sintering and the thermoelectric properties of the compacts

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In recent years the SPS sintering has been often applied for the preparation of bulk thermoelectric semiconductors. The authors have already reported a mixture of titanium-based oxide and non-oxide powder such as TiN reacted during SPS sintering to produce a reduced oxide body. Here, we examined chemical reactions between natural chalcopyrite and copper powders during the SPS sintering process for the purpose of widening the applicability of the reactive SPS sintering. The structure and texture as well as thermoelectric properties were measured as a function of the copper quantity added systematically. The natural chalcopyrite ore used here was found to include pyrite and quartz. As a result, with increase in quantity of copper addition, two chemical changes, (1) from pyrite to chalcopyrite and (2) chalcopyrite to bornite, were successively observed. Micro texture of exsolution into chalcopyrite and bornite was observed in the intermediate region. The product compacts showed an n type thermoelectricity in the compositional range we checked. The ZT value showed a maximum near a composition of 18 wt% copper added.

### (SINT-P002-2017) Microwave sintering of nuclear fuels: Specific development of a microwave device for UO<sub>2</sub> pellets

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Microwave sintering is a promising technique to bring innovative microstructures, while reducing temperature, processing time and energy consumption. This process could be used to improve nuclear ceramics sintering efficiency, reduce economic costs and to potentially improve fuels features. This paper describes the development of an instrumented and automated microwave setup qualified on fuel surrogates which allows reproducible and reliable comparison of microwave nuclear fuels experiments with those in conventional sintering. Finite elements modeling were used to design the setup and a hybrid heating technique with a specific sintering cell in a multimode furnace has been chosen to obtain uniform and controlled heating of the specimens under Ar/4%-H<sub>2</sub>. This original setup is used first to perform sintering tests on UO<sub>2</sub> pellets and then on MOX fuels. The sintering results (densification behavior and microstructural evolution) will be compared with conventional sintering experiments under the same thermal conditions (sintering rate and temperature) and for higher heating rates. The results will be used to evaluate the feasibility and the advantages of the microwave sintering of nuclear ceramics and also to identify possible microwave effects on densification and sintering temperature and to look at the future development of an industrial furnace.

### (SINT-P003-2017) Mechanical forces in the microscopic powder contact during field assisted sintering

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The electric Field Assisted Sintering Technique (FAST), also known as Spark Plasma Sintering (SPS) or Pulsed Electric Current Sintering (PECS), is an innovative method with growing employment in research and industrial production. It is typically used for hard to densify materials or the fast and efficient production of fine- or even nano-crystalline materials. The method is closely related to

hot pressing with the difference of the heating being accomplished by means of a pulsed electric current. If electrically conducting materials are densified, additional forces evolve besides those coming from the surface tension of the disperse material and the externally applied pressure. In this work, we evaluate the contribution of electrostatic and electrodynamic forces as well as the pinch effect in the electric current carrying microscopic contact between two powder particles. The results are discussed for varying particle sizes and current densities. For typical FAST conditions, these additional forces are negligible compared to the high externally applied mechanical force or the surface tension of fine particles.

### (SINT-P004-2017) Determination of the optimal temperature of spark-plasma sintering of ALON ceramics by using calculation of thermal fields in a graphite mold

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The research provides modeling of spark-plasma sintering process of aluminum oxynitride (ALON), specifically the distribution of temperature fields over the sample volume and mold at various stages of heating. Calculation was based on the experimental data on the measurement of temperature on the surface of the mold, inside the mold cavity and in its various internal parts including punches, in the absence of insulation felt. The experimental results allowed establishing the mechanism of change of the heat emission source, which moves from the contacts to the punches. The temperature dependencies of electric and contact resistance on the punch-matrix border were selected. The calculation model based on these dependencies shows a good match to the experiment model. Using the obtained data, it was possible to obtain a transparent ALON with high mechanical properties. Al<sub>2</sub>O<sub>3</sub> and AlN powder mixtures were used to synthesise ALON ceramics at temperature 1700 °C for 30 min at 40 MPa and a heating rate of 100 °C/min under nitrogen. The solid-phase synthesis of ALON with the spark-plasma sintering yielded positive results. The microstructure and mechanical properties of the produced ceramics were analysed. The highest hardness value was recorded to be 18.8 GPa, and the tensile strength was measured to be 187 MPa.

### (SINT-P005-2017) Forming micro components from ceramics materials by using FAST process

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Recent studies have been undertaken to investigate the possibility of forming micro-components by using the combination of micro-forming and Electrical-field activated sintering (Micro-FAST). The FAST process uses low voltage and high current, pressure-assisted sintering and synthesis technique, which has been used recently in materials processing and densification. This process shows the potential to produce solid parts from ceramic powder materials with and without any binders. This method is similar to hot pressing, but the mechanism of the heating and powder densification are different. Using several type of ceramic powders (Al<sub>2</sub>O<sub>3</sub>, MSZ and ZrO<sub>2</sub>), a number of processing parameters have been investigated, such as pressure, heating rate, heating temperature and holding time, which helped to gain optimum results. The Ceramics powders were loaded directly into the die, followed by electric-sintering under certain pressure. In this paper Ø4.00mm × 4.00mm and Ø2.00mm ×



2.00mm cylinder solid samples were produced. This experiment was conducted by use of a Gleeble 3800 thermal- mechanical simulator. Quite few properties of the solid samples, such as relative density, hardness and the microstructures, were examined, and these showed that good results have been found.

**(SINT-P006-2017) The effect of dislocations on flash behaviors in SrTiO<sub>3</sub> single crystals**

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2. National Institute for Materials Science (NIMS), Japan

Flash sintering is a very effective technique to enhance sintering rates for ceramics, which is characterized by the occurrence of a spike in specimen electric current. The sintering process immediately finishes with the current spike. The occurrence of the current spike is often discussed in terms of point/lattice defects caused by applied electric fields. In this study, the behaviors in specimen electric current during heating under DC electric fields were investigated for plastically deformed SrTiO<sub>3</sub> single crystals. Plastic deformation was conducted at room temperature up to about 1% strain. Current behaviors in non-deformed SrTiO<sub>3</sub> single crystals were found to depend on the crystallographic orientation against applied DC electric field. Current surging appears when electric field is applied in [110] direction of SrTiO<sub>3</sub> single crystal while it does not appear in [100]. On the other hand, flash temperature decreases in deformed SrTiO<sub>3</sub> single crystals. The difference in the current behaviors observed in non-deformed/deformed SrTiO<sub>3</sub> single crystals could be closely related to point defects and oxygen ionic conduction caused by applied electric fields.

**(SINT-P007-2017) Unmasking the PECS process in a typical Dr. Sinter™ apparatus**

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1. Clemson University, Physics and Astronomy, USA

Pulsed electric current sintering (PECS) offers rapid sintering of many materials compared with hot press sintering. Earlier studies and our recent studies demonstrated that neither sparks nor plasma occurred in the SPS or spark plasma sintering of a typical apparatus such as Dr. Sinter™. Hitchcock et al. showed that electromagnetic radio frequency (rf) emission occurred during the pulsing current. Our recent results show that this emission is an important augmentation to the hot press sintering in addition to the current flow in the specimen. By isolating the pulsed current flow in the specimen while maintaining, and enhancing, the pulsed current flow in the surrounding die we have demonstrated the importance of the rf radiation that continues to permeate the specimen and enables significant sintering.

**(SINT-P008-2017) Laser sintering of persistent luminescent (Sr,Ca)Al<sub>2</sub>O<sub>4</sub>:Eu<sup>2+</sup>RE<sup>3+</sup> ceramics**

N. R. Souza<sup>2</sup>; D. C. Silva<sup>2</sup>; D. V. Sampaio<sup>2</sup>; C. Kucera<sup>3</sup>; J. Ballato<sup>3</sup>; A. Trofimov<sup>4</sup>; L. G. Jacobsohn<sup>4</sup>; R. S. Silva<sup>\*1</sup>

1. Federal University of Sergipe / Clemson University, Brazil
2. Federal University of Sergipe, Brazil
3. Clemson University, Center for Optical Materials Science and Engineering Technologies, USA
4. Clemson University, USA

In this work, we present the use of a laser sintering technique to produce persistent luminescence MA<sub>2</sub>O<sub>4</sub>:Eu<sup>2+</sup>RE<sup>3+</sup> (M = Ca, Sr) ceramics. In this method, a CO<sub>2</sub> laser is used as the main heating source during the sintering, keeping the laser beam at the center of the sample throughout the process. For sintering, the laser power is raised at a linear rate up to maximum power, and kept at this value for few seconds. After the first face sample irradiation, this process is repeated on the other sample face. The main advantages of this method are rapid processing times, the potential for using high

heating and cooling rates (about 2000 °C/min) without crucibles thereby reducing the risk of contamination, and the potential for sintering materials with high melting points. The ceramics sintered at power density of 1.2 W/mm<sup>2</sup> (CaAl<sub>2</sub>O<sub>4</sub>) and 3.1 W/mm<sup>2</sup> (SrAl<sub>2</sub>O<sub>4</sub>) yielded homogeneous grain size distributions and transmittance up to 65% in the range of 500 nm to 800 nm. Upon sintering in air, the ceramics exhibited the characteristic emission from the Eu<sup>2+</sup> ion, corresponding to the 5d → 4f transition, and weak emission from Eu<sup>3+</sup> ions between 550 nm and 700 nm, corresponding to the <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>1</sub> transitions. The valence of europium ions was further studied by the X-ray absorption spectroscopy in the XANES region and those details are reported in the conference.

**(SINT-P009-2017) Electrical study of Li<sub>3-x</sub>La<sub>(2/3-x)</sub>TiO<sub>3</sub> ceramics sintered by laser and spark plasma techniques**

K. A. Barbosa<sup>2</sup>; L. M. Jesus<sup>2</sup>; Y. G. Santos<sup>2</sup>; J. S. Pereira<sup>3</sup>; F. Z. Guerrero<sup>3</sup>; Y. R. Barcelay<sup>3</sup>; J. A. Rivera<sup>3</sup>; A. Freitas<sup>3</sup>; J. C. Sales<sup>3</sup>; J. A. Moreira<sup>4</sup>; Y. L. Ruiz<sup>2</sup>; R. S. Silva<sup>\*1</sup>

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2. Federal University of Sergipe, Brazil
3. Federal University of Amazonas, Brazil
4. University of Porto, Portugal

Lithium Lanthanum Titanate (Li<sub>3-x</sub>La<sub>(2/3-x)</sub>TiO<sub>3</sub> - LLTO) is a fast lithium-ion conductor, as an effective coating material for cathode materials used in rechargeable lithium-ion batteries. In this work, spectroscopic impedance (SI) technique was used to perform the electric characterization of LLTO ceramics. The powders were synthesized by high-energy ball milling and sintered by both Spark Plasma and Laser sintering techniques. In the laser sintering method a CO<sub>2</sub> laser is used as the main heating source during the sintering, keeping the laser beam at the center of the sample throughout the process. The main advantages of this method are rapid processing times, the potential for using high heating and cooling rates (about 2000 °C/min) without crucibles thereby reducing the risk of contamination, and the potential for sintering materials with high melting points. The dielectric responses of the ceramics showed to be dependent on the processing method through the (micro)structural characteristics achieved during sintering. This complex dependence is discussed in terms of the effects expected from bulk versus grain boundaries in such materials. From SI results was found an intrinsic conductivity from 10<sup>-5</sup> to 10<sup>-4</sup> S/cm in the temperature range from 25 to 280 °C.

**(SINT-P010-2017) Ultrafine nanocrystalline scandia stabilized zirconia consolidated by deformable punch spark plasma sintering**

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Scandia stabilized zirconia (ScSZ) has a great technological interest as solid electrolyte in solid oxide fuel cells since it has the highest ionic conductivity among zirconia-based ceramics. However, ScSZ usually requires temperatures above 1600 °C to achieve relative densities higher than 95% by conventional sintering process. Besides this, an ordered rhombohedral phase formation at room temperature is a detrimental factor to application of the ScSZ solid solutions. In this work, fully dense nanocrystalline ScSZ specimens were obtained by recently proposed Deformable Punch Spark Plasma Sintering technique, DP-SPS. Samples of ZrO<sub>2</sub> containing 6, 8 and 10 mol% Sc<sub>2</sub>O<sub>3</sub> were synthesized by reverse strike coprecipitation method and calcined at 500 °C for 2 h. Grain sizes between 8 and 17 nm were obtained for fully dense ScSZ specimens sintered at low temperatures (700 to 800 °C) and high pressures (1.5 and 2 GPa). Raman spectroscopy analyses confirm cubic stabilization without rhombohedral phase in dense samples. It has been postulated that the DP-SPS method inhibits grain growth due to the low processing

temperatures and eliminates isolated residual pores due to high pressure application. The sintered ScSZ ceramics showed translucency in the visible spectrum, which is consistent with the full density of the samples.

### (SINT-P011-2017) Effect of CNT reinforcement on thermal diffusivity of Cu and Cu-CNT composites using the spark plasma sintering

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We have fabricated the high performance heat dissipation material using CNT functionalized copper (Cu) powder, interesting that the thermal diffusivity of Cu-CNT composite was significantly enhanced more than conventional copper alloy. The Cu-CNT composite was prepared by mechanical alloying of Cu-CNT composite powder and sintered by spark plasma sintering (SPS) method. The mechanical alloying was carried out planetary ball milling process to synthesize uniformly dispersed CNT with copper powder and SPS sintering was carried out of 600°C for 5 min under 70 MPa. We have investigated the structural properties of Cu-CNT by X-ray diffraction, SEM, TEM. Also, we have measured the thermal diffusivity of sintered Cu-CNT by laser flash analyser (50 to 200°C). Consequently, sintered sample have to investigate the mechanism of heat flux on interface between copper and CNT to enhance thermal diffusivity of sintered Cu-CNT.

### (SINT-P012-2017) Microstructure and mechanical properties of aligned electrospun mullite nanofiber yarns obtained at different temperatures

X. Song<sup>\*1</sup>; W. Liu<sup>1</sup>; Y. Ma<sup>1</sup>; J. Wang<sup>1</sup>; S. Xu<sup>1</sup>; Q. Cai<sup>1</sup>; S. Tang<sup>1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

Aligned mullite nanofiber yarns obtained by conjugate electrospinning approach combined with sol-gel method were prepared at different temperatures. The microstructure and mechanical properties were investigated. Thermal analyses of the as-spun nanofibers showed an exothermic peak at 914 °C ascribed to the formation of  $Al_4B_2O_9$  and a ceramic yield of 21 % at 1200 °C. X-ray diffraction (XRD) results revealed that mullite formed at 1100 °C, coupled with the transformation from  $Al_4B_2O_9$  to  $Al_{18}B_4O_{33}$ . Continuous and aligned structure of the nanofibers in mullite yarns retained during the whole calcination process, but their surface turned from smooth to rough when calcining temperature beyond 1100 °C due to the grain growth. The tensile strength increased when the calcined temperature increased from 800 °C to 1000 °C, while the strength decreased to 42 % after calcining at 1200 °C.

### (SINT-P013-2017) Compaction and sintering of mixed nano-micro YSZ powder combinations

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Nanosized ceramic powders suffer from a number of issues, e.g. low production hence high cost, handling problems caused by agglomeration, difficulty of remaining the nano-structure during sintering, etc. However, it is possible to improve both the processing and final properties of submicron grain sized ceramics by adding a little proportion of nanosized powder of the same composition. We used wet-route mixing method to disperse the YSZ nanopowders in submicron YSZ powder matrix. With homogeneous distribution in the matrix of submicron powder, the 10 wt% nanosized powders can increase the green density, and also help sintering process of conventional submicron ceramics. Furthermore, the final properties of ceramics are improved.

### (SINT-P014-2017) Investigation of sintering conditions of Mxene-ceramic composites

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Mxene, a ternary carbide or nitride with two-dimensional nanostructures, shows high strength and elastic modulus, and good resistance to oxidation, heat, and corrosion. It has been reported that Mxenes can be added into polymer matrices to enhance mechanical properties. Some researchers suggest that Mxene-ceramic composites could yield novel properties, but a limited number of people are working on how the Mxene performs when cooperating with ceramic materials. This study focuses on optimising the processing conditions, especially the sintering conditions, for Mxene-Ceramic composite fabrication. The results show that the Mxene phase acts as a binder since it increases the green density, and also helps sintering process. However, degradation of the Mxene phase has been observed during sintering.

### (SINT-P015-2017) Sintering and sintering resistance in nanostructure functional materials

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The particle size, surface area, crystallinity, morphology, etc. are important factors that determine the performance of nanostructure materials. Sintering significantly improves the crystallinity of the materials, while changes the morphology, particle size, and surface area of the nanoparticles, which leads to the change of the performance of the nanomaterials. Sintering and sintering resistance is therefore a trade-off for the performance of nanostructure materials. The morphology evolution and the performance of oxide nanowire arrays and metal/oxide composite nanostructures were investigated and their effects on the photoelectrocatalytic/catalytic performance were discussed.

### (SINT-P016-2017) A brief sintering analysis on sintering of pure and tin-doped magnesium aluminate spinel using differential scanning calorimeter

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In this work, the heat of sintering itself was used to quantify both surface and grain boundary energies in  $MgAl_2O_4$  spinel and some data on tin doped- $MgAl_2O_4$ . Surface and grain boundary energies are key parameters for understanding and controlling microstructural evolution. However, reliable thermodynamic data on interfaces of ceramics are relatively scarce, limiting the realization of their relevance in processes such as sintering and grain growth. Nanoparticles were compacted and heated inside a Differential Scanning Calorimeter (DSC) when densification and grain growth were observed. The evolved heat signal was quantitatively attributed to the respective microstructural evolution in terms of interfacial area change, allowing determination of average surface and grain boundary energies for  $MgAl_2O_4$  as 1.49 J.m<sup>-2</sup> and 0.57 J.m<sup>-2</sup>, respectively. These data was accompanied by a dilatometry study using the samples prepared with different size particles. Preliminary data concerning  $MgAl_2O_4$  doped with different amounts of Sn<sup>4+</sup>, where a significant mass loss indicates a temporary stabilization effect probably due an evaporation of tin segregated during densification.

**(SINT-P017-2017) High resolution load assisted dilatometry to measure radial and axial strain rate**

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5. Productos Químicos A.R., Mexico

A load dilatometer was designed to simultaneously measure radial and axial shrinkage during sintering. Three systems works at the same time: 1) Vertical tubular split Furnace Carbolite 1700; 2) Universal testing system INSTRON 5967; and two laser sensors BETA LaserMike. A table was designed to surround the oven and it is horizontally leveled. Over this table the laser transmitter and receiver are positioned in such a way that they can measure the cylindrical sample diameter and height during heating. The laser sensors are located over a NewPort linear translation stages. The height for the radial laser sensor and the horizontal tangential displacement for the height laser sensor are manipulated through a compact motorized actuator for both transmitters and receivers. This allows a better manipulation for the sample measurement. The better working parameters that have been reached for the laser sensors are: precision 3 -4 mm / 6.2 mm. This sinter-forging system offers the possibility to apply uniaxial stress on the sample. Two load cells are available for the universal testing system: 10 kN and 100 N. The maximum temperature that the full system can reach is 1400 °C. In summary, it is possible to study both free and constrained sintering. Uniaxial viscosity and viscous Poisson ratio are possible to measure as part of the solid mechanics description for sintering.

**(SINT-P018-2017) Synthesis and characterization of La-doped bismuth titanate by spark plasma sintering for enhancement of its ferroelectric properties**

J. R. Leyva Mendoza<sup>1</sup>; G. Hernández Cuevas<sup>1</sup>; S. D. Villalobos Mendoza<sup>1</sup>; Y. Espinosa-Almeyda<sup>1</sup>; H. Camacho Montes<sup>\*1</sup>; C. A. Rodríguez Gonzalez<sup>1</sup>; P. E. García Casillas<sup>3</sup>; O. Raymond Herrera<sup>2</sup>; S. Díaz de la Torre<sup>3</sup>

1. Universidad Autonoma de Ciudad Juarez, Fisica y Matematicas, Mexico
2. Universidad Nacional Autónoma de México, Mexico
3. Instituto Politécnico Nacional, Mexico

Ferroelectric properties for Lanthanum doped bismuth titanate ( $\text{Bi}_{4-x}\text{La}_x\text{Ti}_3\text{O}_{12}$  or La-BiT) with  $x=0.0, 0.5, 1.0, 1.5, 2.0$  are studied in the present work. Powder synthesis is developed by sol gel. Raman spectroscopy shows that the most affected vibrational modes are 270, 337, 538, 614 and 854  $\text{cm}^{-1}$  due the Lanthanum doping. Powder compact samples were obtained by Pressureless sintering (PLS) and Spark plasma sintering (SPS) with relative densities about 92% and 99% respectively. The crystallite sizes are 20.6 nm with particle sizes greater than 4000 nm with elongated morphology. The electric field has an intensity of 20 kv/cm for each sample. With the same compound, the maximum polarization was 0.5354  $\mu\text{C}/\text{cm}^2$ , coercive field 4.892 kV/cm and remnant polarization of 0.131  $\mu\text{C}/\text{cm}^2$  when exposed to an electric field of 20 kV/cm. The electrical permittivity also increases with the adding of dopant. Curie temperatures were set in a range between 650 and 675 °C in systems tested at 1MHz. For the SPS doped with  $x=2.0$  the better properties were obtained due to the high reached density and the incorporation in the structure of the dopant in both perovskita and fluorite-like. This causes high distortion in the geometry especially on the bonds where the bismuth is supposed to be located.

**(SINT-P019-2017) Synthesis and characterization of niobium doped bismuth titanate**

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3. Universidad Nacional Autónoma de México, Mexico

Pure and niobium doped bismuth titanate ceramics ( $\text{Bi}_4\text{Ti}_{3-x}\text{Nb}_x\text{O}_{12}$  (Nb-BiT)) with  $x$  ranging from 0% to 2% were prepared by sol-gel method. Pellets were sintered by pressureless sintering (PLS) and spark plasma sintering (SPS). X-ray diffraction showed no secondary phases and denotes texturing due SPS technique. The shape and size of the grains are strongly influenced by the niobium added to the system as reported by scanning electron microscopy (SEM). Raman spectroscopy indicates distortion in the structure due to a probably niobium substitution into the titanium sites. Ferroelectric hysteresis loops exhibit how the niobium and the texturing affect the ferroelectric behavior. As the niobium content increases, the grain size decreases for the sintering sample. Higher densities are reached for SPS samples. XRD diffraction patterns for sintering sample shows the development of a crystallographic texture. The SPS sintered showed preferential texturing on the plane  $c$  or (001) direction where polarization is not favored. Therefore, the highest values for polarization in the axial direction were obtained for the PLS sample with 2 % of Nb doping.

**(SINT-P020-2017) Solidification microstructure and mechanical properties of Au80Sn20 solder joints under different welding process**

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Au80Sn20 (wt. %) solder alloy is widely used in high powder chip packaging during to its perfect wettability with common substrates, high strength and excellent thermal conductivity. In this study, the solidification microstructure and mechanical properties of Au80Sn20/Cu joints under different reflow soldering and vacuum furnace welding process were investigated. The results showed that the solder matrix solidification structure was homogeneous eutectic structure under reflow due to its faster cooling rate, however the abnormal growth of eutectic phase was occurred under vacuum welding. With the increase of welding time, the average sizes and the thicknesses of  $(\text{Au,Cu})_3\text{Sn}$  intermetallic compounds (IMCs) layers on Cu substrate increased, the growth was controlled by diffusion and the growth index  $n=0.53376$ . In addition, the shear strength of the reflow solder joints was greater than vacuum furnace welding because of its uniform eutectic structure and IMC layers. The shear strength of the solder joints increased with the reflow time and then it decreased as the reflow time continue increased, the fracture locations of the solder joints transferred from  $(\text{Au,Cu})_3\text{Sn}$  layers to the Au-20Sn matrix.

**(SINT-P021-2017) Electrical study of multi-light sintered inkjet printed copper lines on flexible substrate**

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Use of Flexible substrates for the device is becoming important due to its lightweight and bending properties. However, due to low  $T_g$  (glass transition temperature), the flexible substrate can be easily damaged during its thermal treatment using the furnace. Consequently, the printed pattern can result in unexpected low electrical conductivity, due to its damaged substrate. To overcome this problem, we have used flash light sintering furnace where its irradiation energy density ( $\text{J}/\text{cm}^2$ ) can be controlled in milliseconds. The conventional inkjet printed materials can be Ag or Au. However,



these precious metals are rather expensive. Therefore, we have used Cu particles to improve its economical and electrical characteristics. Since Cu is prone to oxidation, we choose a dry coating method via self-assembled 1-Octanethiol. A single and multi-pulse sintering were compared for its microstructure with respect to off-time and light energy density. Finally, the resistivity values of the printed line after single and multi-pulse sintering methods resulted in  $532 \times 10^{-7} \Omega\text{m}$  and  $5.97 \times 10^{-7} \Omega\text{m}$  respectively. Moreover, bending test for multi-sintered sample was performed more than 4,000 times, showing a slight increase in resistivity value. Therefore, electrical property of flexible inkjet printed Cu pattern has improved through multi-pulse sintering.

### (SINT-P022-2017) FLASH sintered KNN – structural and microstructural studies

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Potassium Sodium Niobate, commonly known as KNN, is one of the most promising lead-free piezoelectric materials. It stabilizes in a perovskite structure that, due to small changes in its middle atom (Nb) positions, allows the material to have ferroelectric behavior. Despite its very promising properties, the processing of KNN is not trivial, especially because alkali metals tend to sublime during sintering, and monophasic dense parts are hard to obtain. In this work, a FLASH sintering (FS) technique was applied to consolidate the material and the effect of varying the holding time, during the current control regimen at constant electric field conditions ( $350 \text{ V/cm}$ ,  $20 \text{ mA/mm}^2$ ), was investigated. The FS samples were characterized by XRD, SEM and TEM. It was observed that the FS samples present differential densification areas and the relative extension of these areas are dependent on the holding time. The samples are monophasic with an orthorhombic structure and the TEM analysis of the densest regions did not reveal octahedral oxygen tilting, although distortion of the unit cells was found. The ferroelectric domain structure, analysed by bright field images, shows a mixture of stripe-like and fishbone-like nanodomains.

### (SINT-P023-2017) Selective laser sintering of WC powders sputter coated with stainless steel

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Tungsten carbide with micro-sized particle powders are commonly embedded in a tough binder metal for varied structural applications, from cutting tools to other mechanical parts with complex geometries and these last products represent an increasing market. Additive manufacturing processes as Selective Laser Sintering (SLS) might be the solution to overcome some of the manufacturing problems, if the difficulties resulting from difference of physical properties between tungsten carbide and the metallic binder, such as laser absorbance and thermal conductivity, are surpassed. An original approach of powder surface modification was here considered to prepare WC-metal composite powders and overcome these constraints, consisting on the sputter-coating of the WC particle with a nanocrystalline thin film of stainless steel binder. The coating improves the thermal behaviour and rheology of the WC particles and, at the same time, ensures a binder homogenous distribution. The feasibility of the SLS technology as manufacturing process for WC powder sputter-coated with 13 wt% stainless steel AISI 304L was explored with different laser power and scanning speed parameters. The consolidated layers were characterized regarding elemental

distribution, phase composition and morphology, and the results are discussed emphasizing the role of the coating on the successful SLS consolidation.

### (SINT-P024-2017) Sintering of WC - (Cu, Fe, Cr, Ni) nanocomposites

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The wettability of melted Cu on WC surface was shown to increase by alloying it with stainless steel (SS, AISI304L) and a systematic study on the sintering of WC bounded by (Cu, Fe, Cr, Ni) alloys, as alternatives to cobalt, was undertaken. A fixed total binder amount of 12 wt.% was considered and alloys with varied Cu:SS ratios were studied. The homogenization step was performed by high energy ball milling, to assure a good binder distribution, reduced particle size and mechanical alloying of binder elements. Dilatometric experiments under vacuum, up to  $1450 \text{ }^\circ\text{C}$ , showed that the shrinkage kinetics is very dependent on the binder composition and, despite the fact that the liquid phase appears at increasing temperatures with increasing the SS in the binder composition, higher final densification values could be obtained for the SS richest binders. For binders having Cu:SS ratio equal or lower than 1:2, high dense composites could be attained by vacuum sintering, followed by hot isostatic pressing. The microstructure of the densified composites depends on the binder composition and significant amounts of second phases could be formed during sintering of these nanometric powders; Densification was accompanied by grain coarsening, resulting on enlarged grain size distributions, with some abnormal grains in the micrometric range, but maintaining the average grain size in the nanometric range.

### (SINT-P025-2017) Effect of additives and sintering condition on grain growth of $\text{UO}_2$ pellet

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The development of PCI resistant  $\text{UO}_2$  pellet for the LWRs is mainly focused on the large grain and soft pellet as they can reduce corrosive fission gas release and pellet-clad-interaction. So, many nuclear fuel companies developed PCI resistant  $\text{UO}_2$  pellet as a ADOPT, Cr doped pellet and alumina-silicate fuel. In this paper, to develop the PCI resistant  $\text{UO}_2$  pellet, we fabricated dopant  $\text{UO}_2$  pellets using various oxide additives and sintering condition. The measurement of sintered density, grain size and compressive creep for fabricated  $\text{UO}_2$  pellet was performed. And then the results of test was evaluated in comparison with a commercial  $\text{UO}_2$  pellet. Particularly, the results of test indicated that a titanium oxide binary system among the varied additives can effectively promote the grain growth of  $\text{UO}_2$  pellet by conventional sintering. This paper describes the effect of a small amount additive on grain growth and compressive creep behaviour of PCI resistant  $\text{UO}_2$  pellets.

### (SINT-P026-2017) Self-sizing ceramic powders

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The particle sizes of raw materials play a key role in determining the physical attributes of ceramics. Hence, the powders used in the manufacture of engineered ceramics are chosen with tight specifications. Commodity raw materials often do not meet all the requirements, making expensive milling and classification necessary. In this paper we describe a novel method for making ceramic powders, which does not use traditional comminution and classification techniques to achieve the required narrow particle size distribution (PSD). The process control parameters for achieving these are the sintering temperature and time. We found that this process lends itself very well to the production of ceramic powders which have a mismatch in their lattice thermal expansion



coefficients. The resultant powder particles can either be single grained or a conglomerate of multiple grains. We will present data to show the affects of the various process parameters on the resultant PSD, and also a way of moving from single grained particles to multi-grained ones.

#### (SINT-P027-2017) $\beta$ -SiAlON-BN ceramic composites obtained by spark-plasma sintering

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Composites  $\beta$ -SIALON – BN are highly promising for metallurgical applications such as tubes for metal pouring, pipe heaters, nozzles, dozer units, annular breakers, buckets, crucibles, lining plates, thermocouple casing, etc. Combustion synthesis (CS) is a convenient technique for production of SIALON powders with desired composition, particle size, and morphology. Spark plasma sintering (SPS) is a newly developed process that allows pulsed direct current to pass through the die and sample to heat them. Compared with conventional hot pressing, SPS allows higher heating rates and a very short holding time. The spark plasma sintering of  $\beta$ -SiAlON/0–30 wt % BN ceramic composites was investigated. It was studied the microstructure and the strength of the samples ( $\beta$ -SIALON – BN) consolidated by SPS. The raw materials ( $\beta$ -Si<sub>5</sub>AlON<sub>7</sub> and BN powders) were prepared by infiltration-mediated combustion synthesis. High-density (>95%)  $\beta$ -SiAlON–BN ceramic composites with improved strength properties (flexural strength of 250–300 MPa) were prepared by spark plasma sintering of combustion synthesis-produced  $\beta$ -Si<sub>5</sub>AlON<sub>7</sub> and BN powders. Thus obtained machinable ceramics seem promising for fabrication of machine parts and items operating in severe conditions of strong thermal shock and corrosion-active media. This work was financially supported by the Russian Science Foundation (grant no. 16-19-10213).

#### (SINT-P028-2017) Sintering behavior and mechanical properties of Ti<sub>6</sub>Al<sub>4</sub>V compacts over a broad range of porosity

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This study evaluates the effect of pore fraction and characteristics on the sintering behavior and mechanical properties of Ti64 alloy powder compacts. Low-porosity compacts were fabricated by pressing a Ti6Al4V powder with small (below 20  $\mu\text{m}$ ) intermediate (20–45  $\mu\text{m}$ ) or large (45–75  $\mu\text{m}$ ) size particles. High-porosity samples were fabricated with fine powder mixed with 3 to 50 vol.% of 100–400  $\mu\text{m}$  salt particles. Sintering of samples was performed in a vertical dilatometer from 900 to 1260°C, during 1h in Ar atmosphere. The densification kinetics and final density of the various materials were analyzed as function of the starting powder. Interestingly, samples with the same value of porosity and different pore size distributions were obtained. The microstructures of the samples were examined using both SEM and computed microtomography (CMT). In particular pore characteristics, as interconnectivity, size and shape, were estimated from CMT 3D images. Interparticle pore size was found to depend on the particle size and the sintering temperature, whilst large pore size was fixed by the salt particle size. Compression tests were performed at a strain rate of  $10^{-3} \text{ s}^{-1}$ . It was found that Young's modulus mostly depends on the relative density, while the yield stress appeared to be more affected by the sintering conditions since interparticle bonding plays a significant role

#### (SINT-P029-2017) Correlation between anisothermal sintering shrinkage and structural activity of cold compacted iron parts

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Microstructural analysis on metallic green parts obtained by uniaxial cold compaction highlights that the powder particles result deformed, flattened in the compaction plane. The length of the contacts in the plane and along the compaction direction is different, being the contacts that contribute to the shrinkage in the longitudinal direction longer than those contribute to the transversal shrinkage. The geometrical and structural condition of the powder particles affects the dimensional variations on sintering. A shrinkage kinetic equation accounting for the geometrical activity allows the evaluation of the structural activity introduced by prior cold compaction along the different directions, and of its anisotropy. Dilatometric tests have been carried out on longitudinal and transversal specimens. The anisothermal shrinkage values measured during the heating ramp have been considered in order to calculate the corresponding effective diffusion coefficients which are directly proportional to the dislocation densities in the longitudinal and transversal direction.

#### (SINT-P030-2017) Multistep method application within ceramics sintering consolidation

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Time scale calculus (TSC), a relatively new physical-mathematical method is widely utilized for description of various types of kinetics: classical mechanics, fluid mechanics, physical chemistry kinetics, biology kinetics, and represents major challenge for contemporary scientists. Generally speaking, processes in those systems are described with discrete time scale. Reasonably it could be assumed that such treatment of time scale could be applied to other process parameters. Until nowadays the ceramics synthesis process is not described by means of TSC. Let us assume that ceramics with temperature over discrete scale is basic process of sintering and microstructure consolidation parameter. In this work, BaTiO<sub>3</sub>-ceramics discrete temperature thermodynamics properties with temperature step h and basic temperature point a in the process of sintering and microstructure consolidation is investigated. Multi-step differential transformation method (MSDTM) could be used for solving corresponding discrete model.

#### (SINT-P031-2017) Post-sintering of a HfO<sub>2</sub> ceramic

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Hafnia is a high-temperature ceramics material used for some special applications, e.g., thermocouple shielding, optical coatings, due to its high melting point, dielectric, optical and electrical properties. A commercial HfO<sub>2</sub> thermocouple ceramic part was post-sintered in a new high-temperature dilatometer up to 1680 °C in air atmosphere. The sample showed a sintering step starting around 1435°C. The influence of different heating rates was investigated and thermokinetic analysis data will be presented.

#### (SINT-P032-2017) Effect of sintering conditions on the transparent AlON during two-stage reaction sintering

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AlON is a new advanced ceramic based on cubic spinel crystal structure aluminium oxynitride composition. It is hard, durable and transparent in the broad wavelength range of light. For sintering of AlON, MgO and Y<sub>2</sub>O<sub>3</sub> were used as a sintering aid to form an

Al<sub>2</sub>O<sub>3</sub>-AlN mixed powder and then sintered by two-stage reaction sintering. Primary sintering was performed at 1600 to 1650°C, and secondary sintering was performed at 1800 to 1920°C in a nitrogen atmosphere using gas pressure sintering (GPS) under 0.5MPa pressure. After secondary sintering at 1920°C, the sample was sintered to almost the theoretical density, and it was possible to obtain a high transmittance. The effect of sintering conditions such as temperature, retention time on AlON was analyzed and discussed.

### (SINT-P033-2017) Modeling of compaction mechanisms during hot isostatic pressing of a 316L steel

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In order to understand the various metallurgical and mechanical phenomena involved during HIP compaction, experimental and numerical tests were carried out on a 316L steel. The powder used was produced by gas atomization and was in the form of spherical particles. First, compaction tests were carried out by SPS to determine the sintering parameters. The HIP tests were then carried out on a cylindrical 304L container, under a pressure of 1200 bar and a temperature of 1200°C during 4 hours. Measurements of the density at different points of the compacted part were carried out using the method of the Archimedes thrust. The microstructure of the sintered material was characterized by optical and scanning electron microscopes in order to establish the link with the final mechanical properties of the part. In parallel, numerical simulations are developed on ABAQUS, based on an implementation of the Abouaf model and compared to others using the "Creep" subroutine. The objective is, first to be able to predict the final density of the part at any point, as well as the deformations and the final shape of the part and, second to be able to access the temperature and stress fields that influence the microstructural changes during compaction. The first simulation results are in good agreement with the experimental observations.

### (SINT-P034-2017) Anisotropic shrinkage of a single pore in simulation of viscous sintering by finite element method

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A spherical pore shrinks isotropically and disappears ultimately in the final stage of sintering. However, it is often observed that non-spherical residual pores are fracture origin of ceramics and glasses. The origin and evolution of non-spherical residual pores are important for fundamental understanding of reliability of sintered products. Here, we performed a finite element simulation of pore shrinkage in the final stage of viscous sintering. In the micromechanical point of view, a closed pore shrinks by viscous flow driven by surface tension. The driving force for the shrinkage, i.e., sintering stress, is expressed by surface energy tensor, and makes the pore more spherical. The purpose of present study is to find a quantitative relationship between the strain rate tensor for pore shrinkage and the sintering stress tensor. While a spherical pore shrinks to a point, the simulation result shows that a prolate pore shrinks to a needle, and an oblate pore becomes a disk. It is revealed that the evolution of anisotropic pore depends on external pressure on sintered body, gas pressure inside the pore, and the initial pore shape, which is not a sphere in the real sintering situations.

### (SINT-P035-2017) Sintering analysis of LSGM by construction of the master sintering curve

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Oxygen-ion conductors based on strontium- and magnesium-doped lanthanum gallate have been proposed for use as solid electrolyte in solid oxide fuel cells operating at intermediate temperatures

(500-700 °C), due to their high ionic conductivity and stability over a wide range of oxygen partial pressures. Sintering of this solid electrolyte is usually carried out at high temperatures, yielding loss of gallium and consequent formation of impurity phases. In this work, the sintering process of the composition La<sub>0.9</sub>Sr<sub>0.1</sub>Ga<sub>0.8</sub>Mg<sub>0.2</sub>O<sub>3-δ</sub> prepared by solid-state reaction was investigated by dilatometry and by construction of the master sintering curve (MSC). The aim of this work was to investigate the sintering evolution of powder compacts, and to construct time-temperature-density plot for predicting reliable density data for arbitrary thermal treatments. Non-isothermal dilatometry experiments were carried out and the activation energy for densification was determined after the MSC analysis. The microstructure evolution was investigated by electron microscopy and X-ray diffraction analysis, further correlated to dilatometry results.

### (SINT-P036-2017) Grain growth analysis of sintered alpha alumina

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The purpose of this research is to observe the grain growth in low pressure compacted alpha alumina samples upon sintering. The bulk and surface grain growth will be analyzed during the first phase of this research to see the variance of the growth and how effectively the grains fill the porous regions in the samples. In order to do this, a Field Emission Scanning Electron Microscope will be used to view the grain growth of the different samples. The grain growth of the sintered samples will additionally be compared to that of the samples which were not sintered to see their grain growth differences. After the grain growth is analyzed, the final phase of this research will involve observing dielectric property variance of these different samples through the use of a dielectric spectrometer at an Army Research Lab.

### (SINT-P037-2017) Microstructure and phase evolution of direct laser sintering Ti/Al lightweight gradient material

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Ti/Al lightweight gradient materials is combined the performance of titanium alloy and aluminum alloy, which can greatly meet the demand of lightweight and performance of structural parts. Accordingly, the Ti/Al lightweight gradient material will widely used in the industries and fields of aerospace, automotive, machinery manufacturing, etc. In the present work, a novel Ti/Al lightweight gradient material was fabricated successfully by direct laser sintering process, with the continuous composition changing from 100vol.% Ti6Al4V to 100vol.% AlSi10Mg. The microstructure of the FGM was characterized. Elemental concentration and the hardness distribution were also evaluated. The phase transformation and microstructural evolution along the compositional gradient in direct laser sintering processes were analyzed. The results show that, a series of phase evolutions along the compositional gradient occurred:  $\alpha$ -Ti  $\rightarrow$   $\alpha$ -Ti + AlTi<sub>3</sub>  $\rightarrow$  AlTi<sub>3</sub>  $\rightarrow$  AlTi<sub>3</sub> + AlTi<sub>2</sub>  $\rightarrow$  AlTi<sub>2</sub> + AlTi  $\rightarrow$  AlTi + Al<sub>3</sub>TiV<sub>0.2</sub>  $\rightarrow$  Al<sub>3</sub>Ti + Al + Ti<sub>17</sub>Al<sub>5</sub>Si<sub>12</sub>  $\rightarrow$  Al + Si. The hardness change quasi-continuously with composition changes which decrease from approximately 500 HV<sub>0.3</sub> at the Ti substrate side to about 110 HV<sub>0.3</sub> at the top layer as the AlSi10Mg content increases.

### (SINT-P038-2017) Effects of magnetic field on the interfacial microstructure and shear strength of Sn-3.5 wt%Ag-(0-0.1 wt%Fe) during sintering process

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The magnetic field was used to help refine the microstructure during arc-melting and other techniques. In our study, the magnetic field not only enhance the reaction between SnAg solder and copper substrate, but it also changed the growth of intermetallic compounds

during following sintering process. Various amount of iron was added so as to strengthen the effect of magnetic field. The way of field-applying did influence the particle of iron to flow in the melt when the solder reached its melting point. The chaos solidification brought by the flow of iron particles refine the grains of solder joint during cooling process.

#### (SINT-P039-2017) Grain evolution during free-sintering of nanoparticles

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Sintering is generally carried out using green bodies, or pre-formed, compacted particle agglomerates. During conventional sintering, microstructural evolution is dominated by grain growth and diffusion-driven densification. In loose nano-powders, however, grain evolution appears to follow significantly different rules due to the availability free surfaces and increased rotational degrees of freedom. These rules were first explored within the crystal growth community, where "oriented attachment" was found to lead to the formation of single crystal, many-particle agglomerates. Results from recent computational and experimental efforts will be presented in this poster which elucidate the dominant processes by which nanoparticles align themselves under dry, free-sintering conditions. These results are motivating future work on the development of sintering methodologies to tailor microstructure in metallic and semiconductor thin films.

#### (SINT-P040-2017) Spark plasma sintering of pure titanium powders

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Titanium and its alloys are well-known throughout engineering applications for their combined desirable properties of high specific strength, low density properties, biocompatibility and relatively good corrosion resistance. The sintering of titanium powders is of interest because of the possibility to produce of near-net shape components at very low cost. One method of interest recently has been spark plasma sintering (SPS) wherein direct heating of powder is done to fully densify the material by simultaneous application of pulsed current and pressure is utilized for rapid consolidation. Recent studies have shown that SPS technique can produce dense titanium with relatively improved properties. In this study, an investigation on spark plasma sintering of pure titanium powders was done. The sintering was performed at a constant dwell time of 3min at varying temperature and pressure in the range of 550-900 C and 25-75MPa in vacuum atmosphere respectively. A full densification with high Vickers hardness values was obtained at 800 Cat 25MPa and 75MPa for Ø8mm and Ø20mm sintered pellets. Different microstructure evolution with respect to increasing temperature was observed in the sintered material. The results were discussed in terms correlating relative density, Vickers hardness and microstructure and they indicated that good properties can be obtained at very low temperature at short period of sintering.

#### (SINT-P041-2017) Spark plasma sintering of ZnO nanoparticles for thermoelectric applications

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Zinc oxide ZnO, an n-type semiconductor, is a promising material for high temperature thermoelectric generator because of his good chemical stability, high electrical conductivity and high Seebeck

coefficient [1-5]. The use of Spark Plasma sintering allows to obtain high densified ceramics, with limited grain growth, in a very rapid way, and leads to reduced oxide, because of reducing atmosphere during sintering. Further thermal treatment after sintering permit to control the oxygen stoichiometry in the material, and hence control the thermoelectric properties. Pure commercial Zinc Oxide (ZnO) powder has been prepared by coprecipitation synthesis followed by a calcination treatment. For comparison, commercial powder has been studied as well. These nanometric powders were then densified using SPS. The sintering parameters have been optimized in order to achieve high densification. Preliminary results have indicated that the as-sintered pellets were conductive with resistivity of about 2.5 Ω.cm and Seebeck Coefficient of -250 μV/K at room temperature. These properties are attributed to the lack of oxygen in the material. Annealing the sample drastically changed the resistivity and Seebeck coefficient to 0.3 MΩ.cm and -600 μV/K, respectively. The results show that SPS sintered ZnO is a promising thermoelectric material by carefully tuning the annealing process.

#### (SINT-P042-2017) Recycling of thermoplastic waste in the industry

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In this article; the subject, accomplished in two steps; the party elaboration was undertaken at the company industrial of electromanager (ENIEM), then the party characterization was accomplished in university Mouloud Mammeri (UMMTO) and in other national laboratories such as Algerian petroleum institute (IAP). Mainly the work is concentrated on study of influence of Polystyrene (PS) crushed shock rate's, on the mechanical characteristics of the Polystyrene (PS) virgin shock, to determine the optimal concentrations eligible to not affect the physical, morphological, thermal and mechanical products; set up a comparative study with various percentages of the Polystyrene (PS) shock and the Polystyrene (PS) crushed on a part obtained by plastic injection.

#### (SINT-P043-2017) Freeze-casting to create micro-channels in $\text{La}_{0.66}\text{Ca}_{0.33-x}\text{Sr}_x\text{Mn}_{1.05}\text{O}_3$ ceramics

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A novel freeze-casting setup with enhanced control of freezing rate by in-situ determination of the freezing front position and the application of a Peltier element for cooling is presented. Freeze-casting is a processing technique where a ceramic aqueous suspension is directionally frozen, resulting in the growth of anisotropic ice crystals. Water is sublimated from the frozen body by freeze-drying, leaving voids where the ice crystals were and thereby creating macro-pores or channels in the green body. Subsequent sintering results in a structure with parallel, well-defined micro-channels with widths of ~10 to 100 μm and solid ceramic walls. The channel size and homogeneity of the channels through the sample are primarily dependent on the freezing rate. The more constant the rate is, the more homogenous the channels become. Thus, enhancing control of the freezing rate enhances control of channel size and results in homogenous micro-channels throughout the sample. The perovskite  $\text{La}_{0.66}\text{Ca}_{0.33-x}\text{Sr}_x\text{Mn}_{1.05}\text{O}_3$  ceramic is well known for its desirable magnetocaloric properties. The Curie temperature can be controlled by varying x, making this ceramic ideal for application as regenerator-material in magnetic refrigeration. In this presentation, results on the use of freeze-casting to make engineered porous structures of this material will be presented and further use in magnetic refrigeration will be evaluated.



### (SINT-P044-2017) Synergistic reinforcement of tungsten carbide and carbon nanotubes for improving the thermal shock resistance of ultra-high temperature ceramic

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TiC (TiC-SD), TiC-3.5 wt.% WC (TW-SD) and TiC-3.5 wt.% WC-2 wt.% CNT (TWC-SD) pellets were consolidated using spark plasma sintering technique at 1600°C 1600°C and 50 MPa pressure. Spray drying technique was used for achieving uniform distribution of CNTs in TiC-WC matrix. The HR-TEM and Raman analysis has confirmed the survival of CNTs in TWC-SD with increased defect concentration. Incorporation of WC increased the density of TiC from 89% to 94%, while the addition of CNTs to TiC-WC matrix has further increased the density value to 99%. The average grain size of TW-SD and TWC-SD pellets were reduced by ~17% and ~31% respectively compared to TiC-SD pellet. In addition to that, the interfacial analysis performed at the TiC-CNT interface shows that a shear stress greater than 0.28-0.33 GPa is required for removal of CNT from TiC interface. Addition of both WC and CNT has improved the thermal shock resistance of TiC by ~40%, which is because of hindrance provided by CNTs in transporting oxygen atoms during thermal shock.

### (SINT-P045-2017) Thermal and mechanical properties of SKD61 base tool steel alloys manufactured by selective laser melting process

J. Yu<sup>\*1</sup>; J. Yun<sup>1</sup>; J. Choe<sup>1</sup>; S. Yang<sup>1</sup>; D. Yang<sup>1</sup>; Y. Kim<sup>1</sup>; C. Lee<sup>2</sup>

1. Korea Institute of Materials Science, Powder Technology Research, Republic of Korea  
2. Korea Institute of Mechanics and Materials, Republic of Korea

Hot-work tool steel is used in precision molds for hot-press forming, die casting and plastic injection mold because of advantages such as high resistance to thermal fatigue, thermal shock, abrasion resistance, heat resistant, and dimensional stability. In this study, we designed SKD61 tool steel alloys for additive manufacturing to improve the thermal and mechanical properties. The alloy powders were fabricated by gas atomization process. We used to powder bed fusion process to fabricate of samples and evaluated thermal and various mechanical properties. In the result, hardness of ingots was measured by Rockwell hardness and was showed about 41 HRC. Also, density of ingots was measured by Archimedes principle and was showed about 7.9 g/cm<sup>3</sup>. The best sample, which has thermal conductivity among ingots was confirmed through measurement of thermal conductivity by LFA 467 Hyperflash. Thermal conductivity of sample was about 36.7 W/mK. We are attempting establishment of optimum conditions through control of processing parameters such as laser power, layer thickness, overlap, and scan speeds to fabricate specimens of evaluation of mechanical properties.

### (SINT-P046-2017) On the stoichiometry and sintering behavior of boron suboxide

W. Shoulders<sup>\*1</sup>; H. Payne<sup>2</sup>; K. D. Behler<sup>1</sup>; J. LaSalvia<sup>1</sup>; L. Vargas<sup>1</sup>

1. US Army Research Laboratory, USA  
2. Pennsylvania State University, USA

Boron suboxide (B<sub>2</sub>O<sub>3</sub>) is an attractive high-hardness, low-density armor ceramic with additional applications in thermoelectrics and cutting tools. The theoretical maximum hardness is predicted to occur at full oxygen occupancy and full density, which is difficult to achieve with the current state of the art in powder processing and reasonable industrial scale densification conditions. This work reviews the progress of ARL in the synthesis of B<sub>2</sub>O<sub>3</sub> powders by the oxidation/reduction reaction of amorphous boron in either dry air or in the presence of powdered B<sub>2</sub>O<sub>3</sub> and subsequent hot-pressing. More recent efforts in improving B<sub>2</sub>O<sub>3</sub> processing at ARL include reactive hot-pressing and controlled atmosphere processing, which

are also presented. The role of these processes in densification and stoichiometry control are explored through methods including the examination of reaction thermodynamics and stoichiometry determination through Rietveld refinement of x-ray diffraction patterns. Experimental results are presented and discussed.

## Tuesday, November 14, 2017

### Plenary Session II

Room: Bayview I

Session Chair: Didier Bouvard, Univ. Grenoble Alpes

#### 8:30 AM

### (SINT-PL-002-2017) Contact formation and densification during early stages of spark plasma sintering of metal powders (Invited)

B. Kieback<sup>\*1</sup>; J. Trapp<sup>1</sup>; T. Wallmersperger<sup>2</sup>; A. Semenov<sup>2</sup>; O. Eberhardt<sup>2</sup>; M. Nöthe<sup>1</sup>

1. Technische Universität Dresden, Institute of Materials Science, Germany  
2. Technische Universität Dresden, Chair Mechanics of Multifunctional Structures, Germany

The role of the electric field and electric current during Spark Plasma Sintering of metallic powders were discussed in numerous publications with the aim to explain the fast densification in this technique. The aim of the current paper is a systematic analysis of the conditions and processes during pulsed current sintering on a microscopic scale. Based on typical electric parameters and applied pressures during SPS the role of electric currents, temperature fields and stress distributions were analysed by simulation and model experiments using spherical metal powders. The results show, that only at extreme conditions contact melting happens at the particle contacts. The role of temperature gradients, electric field and current distribution on contact formation will be discussed in detail. Using the two particle model for the early stages of sintering, the growth of contacts and shrinkage will be discussed for various mechanisms: curvature driven diffusion, pressure assisted sintering, creep deformation of particle contact areas. The results show, that some of the processes proposed for SPS do not contribute in a significant way. Although the experimental results were obtained only for copper and steel powders, the discussion should help give a realistic view also for other systems and process conditions.

### Spark-plasma and Flash Sintering III

Room: Bayview I

Session Chairs: Javier Garay, University of California, San Diego; François Valdivieso, Mines SE

#### 10:00 AM

### (SINT-069-2017) FAST/Flash: A combination of FAST/SPS and flash sintering (Invited)

J. Raethel<sup>\*1</sup>

1. Fraunhofer IKTS, Sintering & Characterization, Germany

FAST/SPS and flash sintering are interesting and fast consolidation techniques. A combination of both at voltage <10V and electrical field of 10V/cm maximum have been already shown for zirconia. For investigations of mechanisms present at higher electric fields a hybrid-heated FAST/SPS, equipped with an additional voltage support providing up to 180V was used. The flash effect was investigated under these conditions for zirconia and silicon carbide. Clear correlations between applied voltage, current and pressure could be observed. Main focus was on the onset of flash sintering for better understanding of differences comparing FAST/Flash with flash sintering. Results are accompanied by measurement of electrical resistivity of several ceramic powder materials under applied load.



Besides using this data for understanding of FAST/Flash these values are also discussed in context of standard FAST/SPS processes.

#### 10:40 AM

##### (SINT-070-2017) Spark plasma sintering of titanium based alloys: From finite element modeling to the fabrication of near net shape (NNS) specimens (Invited)

D. Martins<sup>1</sup>; U. Kus<sup>1</sup>; C. Maniere<sup>1</sup>; G. Chevallier<sup>1</sup>; P. Sallot<sup>2</sup>; K. Mocellin<sup>3</sup>; M. Bellet<sup>3</sup>; D. Delagnes<sup>4</sup>; J. Huez<sup>5</sup>; C. Estournes<sup>\*1</sup>

1. CIRIMAT, UPS/CHIMIE, France
2. Safran Tech, France
3. CEMEF, France
4. ICA Mines Albi, France
5. CIRIMAT, ENSIACET, France

SPS is a process belonging to the powder metallurgy. A Load and a high intensity DC-pulsed current are simultaneously applied on tools containing powder to be sinter. To use SPS at industrial scale, modelling is needed to predict densification and temperature profile both dependent on sintering conditions/geometry. The aim of the present work was to develop near net shape complex parts based on titanium alloys and to evaluate the reliability of this technique. The presentation will be divided in two parts. In the first one a fully coupled numerical computation model on TiAl 48-2-2 commercial powder densification will be presented. The experimental relative density map will be presented and the correlation with the numerical model will be analyzed, both on simple and complex shape specimens. The evolution of the microstructure during creep test but also after densification of a complex part will be presented and correlation with numerically assessed temperature field will be discussed. The second part will be devoted to Ti-6Al-4V alloy. On simple shape, sintering cycles below or above  $T_p$  allow to be obtained, in repeatable manner, fully dense samples with controlled and homogeneous microstructures. Fully dense dog-bone specimens have been obtained and their mechanical behavior have shown high values and very homogeneous of ultimate tensile strength.

#### 11:20 AM

##### (SINT-071-2017) Intrinsic electric current effects on densification behavior of fine zirconium nitride powders

G. Lee<sup>\*1</sup>; C. Maniere<sup>1</sup>; J. McKittrick<sup>2</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Mechanical Engineering, USA
2. University of California, San Diego, Mechanical and Aerospace Engineering, USA

Previous works indicate that the densification kinetics of conductive powders can be significantly improved by spark plasma sintering (SPS) process. However, the effect of electric current during SPS was not clarified mainly due to the difficulty of the deconvolution of the Joule heating and possible non-thermal field phenomena. In the present work, three SPS heating modes are utilized to overcome this obstacle. One SPS setup restricts the electric current flow through the powder, and the other two setups allow small or large amounts of the electric current passing through the powder specimen, respectively. All the three setups are employed under the same sample temperature regime. The electric current effect is quantified by finding the fraction of the electric current flowing through ZrN powder during SPS using finite element modeling and taking into consideration the inter-particle neck area evolution during sintering. The constitutive equation for sintering with and without the influence of electric current is applied for the analysis of the porosity evolution in ZrN powder in order to find the densification mechanism of ZrN for the three considered SPS modes.

#### 11:40 AM

##### (SINT-072-2017) Large scale direct current based spark plasma/field assisted sintering: Uniformity and densification kinetics

L. S. Walker<sup>\*1</sup>

1. Thermal Technology, USA

Direct Current Sintering (DCS) also known as Field Assisted and Spark Plasma Sintering (FAST, SPS) has achieved great success in academia and industrial based research with its ability to rapidly produce high quality material systems. However, there is a significant lack of information on processing large scale parts (>10cm) and the kinetics of densification as part sizes increase significantly in both thickness and diameter. Due to limited availability of large direct current sintering equipment the general research community does not have an opportunity to routinely experiment on this scale. Here we present the production of large scale parts and investigate the impact of scale-up on the densification of material systems processed in an industrial scale DCS furnace that is publicly accessible for research and industrial usage. Material systems based on insulators and conductors will be presented across a range of sizes to identify the role of scale-up on the densification process from the lab scale to >10cm diameter.

#### 12:00 PM

##### (SINT-073-2017) Traveling flash-sintering

J. Lebrun<sup>\*1</sup>; E. Sortino<sup>3</sup>; A. Sansone<sup>2</sup>; R. Raj<sup>3</sup>

1. Saint-Gobain, Innovative Materials, USA
2. Lucideon, United Kingdom
3. University of Colorado, USA

Flash sintering is a process where green ceramic bodies are sintered in a few seconds at low furnace temperatures, under modest applied electric fields. Until now, the method has been applied to dog-bones or small specimens in the shape of cylinders. In this presentation, we address the possibility of Traveling Flash-Sintering, where large ceramic sheets move progressively under the electrodes thus allowing a continuous process of sintering ceramics. In the flash process the electric field (and temperature) controls the initiation of the flash, while the current flowing through the specimen determines the extent of densification. Therefore, single pass flash sintering of specimens with a large surface area, for example a tile sheet, would require power supplies with huge current capabilities, and risk localization of current flow. We present a simple method of continuous sintering, where line-shaped electrodes are pressed against a flat sheet moving continuously through the electrodes, at velocities of up to 10 cm min<sup>-1</sup>. We present a model that describes the fundamental limit to the relative velocity between the workpiece and the electrodes. Safe and fail maps are developed to prescribe the regime of electrical parameters and relative velocity in order to avoid the localization of current flow (which produces inhomogeneous microstructure). Experiments were conducted on a whiteware green body. Supported by Lucideon Inc., U.K.

#### Microstructural Evolution in Sintering Processes II

Room: Bayview II

Session Chair: TBA

#### 10:00 AM

##### (SINT-074-2017) Electric field induced equilibrium grain boundary configurations in perovskite ceramics (Invited)

K. van Benthem<sup>\*1</sup>

1. University of California, Davis, Materials Science and Engineering, USA

Electric fields can enable the accelerated consolidation of materials during field assisted sintering techniques, including spark plasma sintering and flash sintering. Although such techniques are already employed for the synthesis of a wide variety of microstructures with unique macroscopic properties, a fundamental understanding

of the atomic-scale mechanisms for grain boundary formation and subsequent migration in the presence of electrostatic potentials is still debated. To study the effects of electrostatic potentials on grain boundary formation we have designed new diffusion bonding experiments for the formation of SrTiO<sub>3</sub> bicrystals. Under otherwise identical processing conditions subsequent high resolution TEM characterization reveals that the atomic and electronic grain boundary structures are remarkably different depending whether or not an electrostatic potential was applied across the grain boundary plane. It was found that the electric field interacts mostly with the oxygen sublattice, which leads to different interface widths, grain boundary core structures, and local chemical compositions.

**10:40 AM**

### (SINT-075-2017) Solute-drag versus solute-acceleration of grain boundary mobility

W. D. Kaplan\*<sup>1</sup>

1. Technion - Israel Institute of Technology, Dept. of Materials Science and Engineering, Israel

The role of dopants in processing ceramics has been an important issue for many years, especially given the contradicting reports of retarded or accelerated grain growth by key dopants and impurities. New analysis of grain boundary (GB) mobility of alumina as a function of dopant concentration has shown that some segregating dopants increase the GB mobility, i.e. the opposite of solute-drag. The segregating dopants are associated with 2-D structural and compositional transitions at the GBs, and possible changes in the mechanism of GB migration. This presentation will review recent GB mobility measurements and the concept of 2-D GB transitions and their potential role on the mechanism of GB motion. A comparison will be made with recent measurements of the remarkable increase of GB mobility due to the application of external electrical fields.

**11:00 AM**

### (SINT-076-2017) Boundary Structural Transition and Grain Growth Behavior during Sintering

S. L. Kang\*<sup>1</sup>

1. Korea Institute of Ceramic Engineering and Technology (KICET), Republic of Korea

Grain boundaries can be categorized into two types in terms of their morphology: rough (atomically disordered) and faceted (atomically ordered). For a given system, the structural transition of boundaries between rough and faceted can be induced by changing the temperature and atmosphere and by adding dopants. The migration of a faceted boundary was found to be governed by the attachment of atoms at the low energy singular plane of the facet. Its migration kinetics was nonlinear with respect to the driving force, showing a critical driving force,  $\Delta g_c$ , for appreciable migration of the boundary. The migration appears to be governed by the interface reaction and the diffusion of atoms below and above the critical driving force, respectively. Because of the nonlinear migration of faceted boundaries, various types of grain growth can appear in the same system with  $\Delta g_c$  relative to the maximum driving force,  $\Delta g_{max}$ , for the growth of the largest grain (the principle of microstructural evolution). The microstructural evolution principle has been supported by many experimental observations in both ceramics and metals. The technical development of the solid-state conversion of single crystals is explained as an application example of the principle

**11:20 AM**

### (SINT-077-2017) Effect of nanopowder addition on grain growth behavior of ultracoarse cemented carbides

X. Liu\*<sup>1</sup>; X. Song<sup>1</sup>; H. Wang<sup>1</sup>; C. Hou<sup>1</sup>; Z. Nie<sup>1</sup>

1. Beijing University of Technology, College of Materials Science and Technology, China

The ultracoarse cemented carbides, generally with WC grain sizes larger than 6mm, are used widely in mining and construction parts, due to their good combination of resistance against wear, thermal fatigue and shock. In the present study, the nanoscale composite powder was used as additive to prepare ultracoarse WC-Co cemented carbide bulk material by sintering. The effect of nanoscale powder on the grain growth behavior in the sintering process of WC-Co powder mixture was investigated, and the mechanisms of grain coarsening during liquid state sintering was proposed. The features of microstructure and grain size distribution of the sintered cemented carbide bulk materials were characterized for different sintering stages. The results show that the addition of nanoscale composite powder to the raw WC based powder plays a significant role in causing growth of WC grains in the whole sintering process. Due to the addition of nanoscale powder, at early sintering stage with lower temperatures, the WC grains grow rapidly as a result of higher driving force from the surface energy of mixed powders. Moreover, the eutectic temperature of WC and Co is decreased by addition of nanoscale powder, which facilitates continuous growth of WC grains.

**11:40 AM**

### (SINT-078-2017) Liquid-like grain boundary complexation and sub-eutectic activated sintering in CuO-doped TiO<sub>2</sub>

J. Nie\*<sup>1</sup>; J. M. Chan<sup>1</sup>; M. Qin<sup>1</sup>; N. Zhou<sup>1</sup>; J. Luo<sup>1</sup>

1. University of California, San Diego, USA

The eutectic temperature and composition of the TiO<sub>2</sub>-CuO system were carefully measured to be 1010 ± 10°C and 83CuO:17TiO<sub>2</sub>, respectively. Subsequently, a TiO<sub>2</sub>-CuO phase diagram was computed, representing a correction and major improvement from the phase diagram available in literature. Dilatometry measurements and isothermal sintering experiments unequivocally demonstrated the activated (enhanced) sintering of TiO<sub>2</sub> with the addition of CuO, occurring at as low as >300°C below the eutectic temperature. High resolution transmission electron microscopy (HRTEM) characterization of water-quenched specimens revealed the formation of nanometer-thick, liquid-like, intergranular films (IGFs), a type of grain boundary (GB) complexation (a.k.a. 2-D interfacial phase), concurrently with accelerated densification and well below the bulk eutectic temperature. Consequently, activated sintering is explained from the enhanced mass transport in this premelting-like complexation. An interfacial thermodynamic model was used to quantitatively explain and justify the stabilization of liquid-like IGFs below the eutectic temperature and the temperature-dependent IGF thicknesses measured by HRTEM. A GB λ diagram was computed, for the first time for a ceramic system, to represent the thermodynamic tendency for general GBs in CuO-doped TiO<sub>2</sub> to disorder.

**12:00 PM**

### (SINT-079-2017) Prediction of microstructure evolution of solid oxide fuel cell electrodes during sintering through Kinetic Monte Carlo (KMC) simulations

Z. Yan\*<sup>1</sup>; S. Hara<sup>2</sup>; Y. Kim<sup>1</sup>; N. Shikazono<sup>1</sup>

1. University of Tokyo, Industrial Institute of Science, Japan  
2. Chiba Institute of Technology, Department of Mechanical Engineering, Faculty of Engineering, Japan

The Potts Kinetic Monte Carlo (KMC) model is used to predict the microstructure evolutions of electrodes in solid oxide fuel cells (SOFCs) during sintering. In KMC models, the microstructures are controlled by sintering temperatures ( $K_b T$ ) and frequencies ( $f$ ),

i.e.,  $K_B T_{gg}$ ,  $f_{gg}$  for grain growth,  $K_B T_{pm}$ ,  $f_{pm}$  for pore migration, and  $K_B T_{vf}$ ,  $f_{vf}$  for vacancy formation and annihilation. These parameters are calibrated using artificial neural networks against three dimensionally reconstructed microstructures of sintered samples by Focus Ion Beam Scanning Electron Microscopy (FIB-SEM). The real particles' 3D geometries are obtained by FIB-SEM reconstruction of raw powder. Irregularly shaped particles are represented with clumped multispheres (multi-sphere model) and volume-equivalent single sphere (single-sphere model), respectively, based on their 3D geometries. The discrete element method (DEM) is used to reproduce 'virtual' powder with multi-sphere and single-sphere models. The DEM-generated microstructures and the real microstructure are submitted to KMC simulations. It shows that the multi-sphere model predicts more realistic microstructure than the single-sphere model. We will also present an adapted KMC model to mimic the anisotropy behavior during constrained sintering of a thin layer on a rigid substrate.

### Sintering of Nanostructured Materials III

Room: Bayview III

Session Chairs: Ricardo Castro, University of California, Davis; Caroline Lee, Hanyang University

#### 10:00 AM

##### (SINT-080-2017) Sintering property of bimodal metal nanopowder mixtures with various size ratios (Invited)

J. Lee\*<sup>1</sup>; J. Song<sup>1</sup>; E. Hong<sup>1</sup>

1. Hanyang University, Materials Engineering, Republic of Korea

Powder Metallurgy (PM) processing of nanoscale metal powders has been of great interest in that it can realize fabrication of high performance PM parts with saving process-energy and using cost effective materials. The present presentation introduces our researches on finding a breakthrough how to process a high strength iron PM parts using Fe nanopowder. The most important issue is a question; "Is it possible to achieve a high strength pure iron PM parts only by grain refinement effect without alloying treatment including carbon?" To solve this problem, our researches have been focused on understanding the sintering of bimodal nanopowders with various size ratios (three Fe bimodal powder systems of large micro-nano, fine micro-nano, nan-nano powder mixtures). Sintering experiments revealed that the bimodal nanopowders in three powder systems underwent a full densification and slow grain growth during sintering. As a result, mechanical properties were remarkably improved. The key idea of these studies is based upon the optimization of structure design and full density sintering of micro-nano or nano-nano type bimodal powder mixtures into fine-grained Fe PM parts. Especially understanding the roles of hierarchical interfaces in nanopowder agglomerates on densification and grain growth during sintering is stressed.

#### 10:40 AM

##### (SINT-081-2017) Microstructural evolution in a four group element ODS ferritic steel (Y-Al-Ti-Zr) consolidated by SPS

E. Macia<sup>1</sup>; J. Cornide<sup>1</sup>; A. García-Junceda<sup>2</sup>; M. Campos\*<sup>1</sup>; J. Torralba<sup>1</sup>

1. Universidad Carlos III de Madrid, Department of Materials, Spain  
2. IMDEA Materials, Spain

Oxide Dispersion Strengthened (ODS) ferritic Steels are extraordinary candidates for nuclear applications due to their good behavior at high temperature and under irradiation conditions. In this work, ODS ferritic steel was produced by mechanical alloying (MA) and SPS to obtain a complex nanostructure. A four group elements (Y-Ti-Al-Zr) was selected to improve the characteristics of the precipitates. The addition of Zr would refine and increase the density of oxides particles forming complex oxides as Y-Zr-O instead of Y-Al-O. These stable nanoparticles improve the thermal stability of the fine structure. Besides, the heterogeneous distribution

of stored energy due to the high-energy attrition of MA powder will produce an inhomogeneous recrystallization during the consolidation step. After SPS consolidation, an heterogeneous grain size distribution was attained, as recrystallization depends on the plastic deformation degree, on the composition of each particle and on the oxide dispersion promoted. Therefore, two sintering cycle was performed at 1273, 1373 K following fast heating rates (from 100 to 600 °C/min) to minimize porosity. The final microstructures were characterized by XRD and electron microscopy (SEM and TEM). In addition, Vickers microhardness and tensile tests was performed to analyze the mechanical response at R.T.

#### 11:00 AM

##### (SINT-082-2017) Microstructural changes on Co-9Al-9W alloy produced by SPS and Ti and Ta additions

M. Carton Cordero<sup>1</sup>; M. Campos<sup>2</sup>; J. Torralba\*<sup>2</sup>

1. IMDEA Materials, Spain  
2. Carlos III University, Materials Science and Engineering, Spain

The Co-based superalloys are strong candidates for high temperature and extreme conditions applications, due to their good strength, excellent and better wear resistance and high oxidation resistance. In this work, a pre-alloyed atomised powder -Co-9Al-9W (at%)- was used as precursor to produce alloys for high temperature applications. In order to enhance the formation of the  $\gamma'$  phase, 2% Ti and 2% Ta (at%) were added by mechanical alloying to the Co-9Al-9W atomised powder. Afterwards, with the aim of obtaining a fully dense material with an improved mechanical properties, processed alloys were consolidated by spark plasma sintering (SPS) to hold the ultrafine grain size microstructure (UGS). To precipitate  $\gamma'$ , it was study the microstructural changes promoted after heat treatments (solubilisation + ageing) in relation to the added alloying elements. The effect of quinary elements on  $\gamma'$  solvus temperature of the CoAlW base alloy was investigated through thermal analysis.

#### 11:20 AM

##### (SINT-083-2017) Effect of nano-powder addition on sintering of water atomized iron powder

S. Manchili\*<sup>1</sup>; J. Wendel<sup>1</sup>; A. Zehri<sup>2</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>; J. Liu<sup>2</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden  
2. Chalmers University of Technology, Department of Microtechnology and Nanoscience, Sweden

Press and sinter powder metallurgy (PM) steel offers cost-effective solutions for structural applications. There is a constant drive for improvement of the density of these PM steels, which will expand their usage in applications demanding higher performance than what they deliver today. One of the promising ways to improve densification is through the utilization of bimodal mixes. Blending nano powder can contribute to a higher sinter-density of micron sized powder typically used for manufacturing PM steel components. The higher surface to volume ratio in nano powder is hypothesized to suppress the melting point and can further contribute to densification (liquid phase). Even when the nano powder does not melt at the desired sintering temperature, it is expected to increase in the inter-particle contact area. This is supposed to enhance the material transport path and improve the densification. In the present investigation, pure iron and low carbon steel nano powder were chosen and mixed with water atomized iron powder (-45  $\mu\text{m}$ ) in the ratio of 5% and 95% respectively. The powder mixes were compacted at different pressures: 400, 600 & 800 MPa followed by sintering at 1350 °C in pure H<sub>2</sub> atmosphere. Both loose powder and compacted mixes were investigated to elucidate the effect of nano particle on densification. The sintering phenomena were studied and the activation energy for sintering was estimated.



11:40 AM

## (SINT-084-2017) Reaction spark plasma sintering for ceramic matrix nanocomposites (Invited)

A. V. Ragulya\*<sup>1</sup>; V. V. Skorokhod<sup>2</sup>

1. Frantsevich Institute for Problems in Materials Science, Dpt. Nanostructured Ceramics and Nanocomposites, Ukraine
2. Frantsevich Institute for Problems in Materials Science, Director Office, Ukraine

Chemical reaction activated by electric pulses during SPS represents a unique instrument for the manufacturing of a broad variety of ceramic materials and ceramic matrix nanocomposites. The passage of the direct pulsed electric current through the particulate body and application of external pressure creates special conditions for the accelerated heat, mass transfer, chemical reactions, and phase transformations which allow control the grain size, phase composition, density and combination of properties in ceramic materials. Unlike traditional hot-pressing method, the spark plasma sintering occurs several times faster, but results in higher relative density, usually above 99%. Examples of SPS are given for nanostructured multiphase materials. The grain size control under reaction SPS occurs through the acceleration of the nucleation and inhibition of nuclei growth and strongly depends on number of phases. Several details of reaction SPS are shown for the composites TiN/TiB<sub>2</sub>, ZrN/ZrB<sub>2</sub> and TiN(ZrN)/TiB<sub>2</sub>(ZrB<sub>2</sub>). The ultrafast flash reaction spark plasma sintering has been carried out within a few tens of seconds. In this case, the grain growth was completely impeded, but full density was achieved. The discussions of the most probable mechanisms of the reaction SPS have been presented.

## Fundamental Aspects of Sintering II

Room: Mission I

Session Chairs: Alberto Molinari, University of Trento;  
Charles Maniere, San Diego State University

10:00 AM

## (SINT-085-2017) The role of thermodynamics in controlling sintering and grain growth of ceramics (Invited)

R. H. Castro\*<sup>1</sup>

1. University of California, Davis, Materials Science & Engineering, USA

Sintering and grain growth are thermodynamically favorable processes, meaning that in both cases excess energy coming from interfaces is released as an exothermic phenomenon to decrease the system energy. However, it has been proposed that since the rate of interface elimination is linked to its energies, one can use the later to better understand and control processing. Here we show how dopants can indeed modify interface energies and consequently affect densification and grain growth. For instance, Mn doped Ytria Stabilized Zirconia (YSZ) presents stronger densification as compared to YSZ alone. This can be linked to a change in the interfacial energies directly measured by calorimetry accompanying by (or interconnected to) the modification of the mass transport mechanism to plastic flow. Mn is observed to segregate to interfaces and cause both thermodynamic and kinetic effects, suggesting that a full description of sintering cannot neglect an interface energetic description, which in turn can be the key to interpret changes in mass transport mechanisms themselves. In another example, Gd is demonstrated to virtually stop grain growth in fully dense nanocrystalline YSZ prepared by Spark Plasma Sintering. The phenomenon was related to the existence of a zero-energy grain boundary condition, as demonstrated by calorimetry, evidencing grain growth can be controlled by targeting driving forces.

10:40 AM

## (SINT-086-2017) Full field study of sagging during sintering: A phase field and Navier-Stokes based approach

E. Borukhovich\*<sup>1</sup>; R. Babu<sup>1</sup>; D. Chasoglou<sup>2</sup>; J. Odqvist<sup>1</sup>

1. KTH Royal Institute of Technology, Enheten Struktur, Sweden
2. Höganäs AB | Global Development, Sweden

Shape loss during the sintering process, called sagging, caused by lack of stability of the metal powder is a severe issue in Metal Injection Molding (MIM) and Additive Manufacturing (AM) and has not been yet modelled in a satisfactory manner. The current contribution faces this issue with a full field description using a framework consisting of the Navier-Stokes equation coupled with a phase field model. The chosen approach does not only allow to resolve pressure and forces in the full space but it is also able to consider arbitrary powder morphology. In order to benefit from the latter advantage of the used calculation method, three-dimensional representation of real powder particles obtained using the Focused Ion Beam (FIB) sectioning in a dual beam microscope is used as input data. Using the experimentally obtained data of individual particles and particle size distributions of actual powders, a non-trivial realistic microstructure is generated. The data generated by the computational method specified above is interpreted statistically allowing characterization of the general morphological stability for different powder morphologies. Finally, the two cases of powders obtained by water atomization and by gas atomization are compared.

11:00 AM

## (SINT-087-2017) Variation with strain rate of the contribution of pore surface diffusion to bulk sintering viscosity accounting for evolving coordination

F. Delannay\*<sup>1</sup>; L. Brassart<sup>2</sup>

1. Université catholique de Louvain, Institute of Mechanics, Materials and Civil Engineering, Belgium
2. Monash University, Department of Materials Science and Engineering, Australia

The quasi-static approximation, which neglects the contribution of surface diffusion to sintering viscosity, is most often implicit in physically-based constitutive laws commonly used in sintering simulations by FEM. The validity of this approximation becomes questionable when the ratio of grain boundary to surface diffusivity is larger than  $10^{-2}$  and when strain rate is large, which is the case with the various field assisted sintering processes that are increasingly popular nowadays. We have developed a model for the prediction of the influence of wide ranges of straining rate, diffusivity ratio, dihedral angle, and pore volume fraction on the relative magnitude of the contributions of grain boundary diffusion and surface diffusion to bulk viscosity  $K$ . The evolution of average grain coordination is taken to be linked to the evolution of pore volume fraction. The essential physics is captured in 2D under plane strain. The solution of the partial differential equation of the problem is decomposed into steady state and transient parts. The steady state solution reveals a transition from Newtonian viscosity at low strain rate to non-Newtonian viscosity at high strain rate. The contrasts between convex and concave pore curvature as well as between negative or positive straining rate (i.e. densification or extension) are enlightened.



11:20 AM

**(SINT-088-2017) Quasi-static model for the dependence of shear sintering viscosity on dihedral angle, grain boundary friction, relative density and grain coordination**F. Delannay<sup>\*1</sup>; L. Brassart<sup>2</sup>

1. Université catholique de Louvain, Institute of Mechanics, Materials, and Civil Engineering, Belgium
2. Monash University, Department of Materials Science and Engineering, Australia

Microstructural models for the prediction of the dependence of shear sintering viscosity,  $G$ , on relative density during sintering dominated by grain boundary diffusion have commonly been based on regular 3D arrangements of grains with integer coordination number. The viscosity tensor can then be calculated using an isostrain hypothesis, and the compliance tensor is derived by inversion of the viscosity tensor. Consideration of regular 3D arrangements of grains does not allow accounting for the monotonic evolution of grain coordination with relative density. This contribution presents an approach for the modelling of shear viscosity in a system in which average grain coordination evolves with relative density. The method is developed for a random array of grains in plane strain. Voigt and Reuss bounds for  $G$  are calculated in the framework of the quasi-static hypothesis (i.e. the contribution of surface diffusion is neglected) using successively strain-controlled and stress-controlled boundary conditions. The roles of dihedral angle and grain boundary friction are thoroughly analysed. The computational results for full density can be compared to literature references on the prediction of shear viscosity in creep dominated by grain boundary diffusion.

11:40 AM

**(SINT-089-2017) Fractal correction of Coble's sintering model (Invited)**V. Mitic<sup>\*1</sup>; L. Kocic<sup>2</sup>; V. Paunovic<sup>2</sup>; Z. Nikolic<sup>2</sup>

1. Serbian Academy of Sciences, Institute of Technical Sciences, Serbia
2. Faculty of Electronic Engineering, Serbia

Studying of the complex electrodynamic properties of sintered materials needs understanding ceramic grain contacts morphology. The microstructures of sintered BaTiO<sub>3</sub>-ceramics, observed with the SEM method, reveals how complex this morphology is. It grossly differs from classic Euclidean geometry. Several variations of Coble's two-sphere model are reviewed in this paper by setting the stress on fractal nature of these contacts. The area of grains' surface is calculated using fractal correction and fractal dimension. This gives a more precise numerical representation of the parameters and related properties of electronic ceramics. A more realistic Coble's model is considered regarding the neck profile. The cross-section with the rotation symmetry plane reveals the circular external neck border rather than a linear one. The graphical interpretation of the cross-section of the common lens-like zone is given together with the relative position of the "neck". In particular, the role of the dielectric constant, being correlated with the fractal nature of intergranular morphology, causes corrections to the Heywang model and Curie-Weiss law. Considering the obtained results, new frontiers are established for deeper and higher level of microelectronic integration of electronic circuits, which practically results in a new framework for fractal electronics.

**Novel Sintering Processes II**

Room: Mission II

Session Chairs: Kirill Rybakov, Institute of Applied Physics, Russian Academy of Sciences; Jean-Michel Missiaen, Univ. Grenoble Alpes

10:00 AM

**(SINT-090-2017) Reactive laser sintering of electrophoretically deposited nanoparticulate films**V. Mackert<sup>\*1</sup>; C. Notthoff<sup>1</sup>; J. Gebauer<sup>1</sup>; M. Winterer<sup>1</sup>

1. University of Duisburg-Essen, Nanoparticle Process Technology, Germany

Laser sintering is an alternative to conventional sintering. Especially, resonant excitation above the band gap ( $E_g$ ) allows the use of low laser power for local heating and direct coupling of laser energy compared to frequently used infrared laser sintering. In this work, we present how nanoscaled microstructures of colloidal processed films respond to various laser parameters. Therefore, chemical vapor synthesized nanoparticles (NPs) of zinc oxide (ZnO) and tin dioxide (SnO<sub>2</sub>) are processed into stable dispersions to form granular films via Electrophoretic Deposition (EPD). Pure and composite films of SnO<sub>2</sub> ( $E_g=3.6$  eV) and ZnO ( $E_g=3.4$  eV) NPs are deposited and treated using UV laser ( $\lambda=325$  nm,  $E_g=3.8$  eV). The influence of UV laser sintering is studied as a function of writing speed and laser power for pure and mixed EPD films. Microstructure, morphology and composition of nanoparticulate films before and after laser sintering is investigated by grazing incidence XRD, HRSEM and EDS. XRD patterns after UV laser sintering of composite ZnO-SnO<sub>2</sub> films show the presence of a complete new phase, which is identified as inverse cubic spinel zinc stannate (Zn<sub>2</sub>SnO<sub>4</sub>) structure. Results confirm that the UV laser sintering is not only suited to preserve and manipulate nanoscaled microstructures but also to synthesize complex metal oxides like Zn<sub>2</sub>SnO<sub>4</sub> by a new preparation method.

10:20 AM

**(SINT-091-2017) Reactive spark plasma sintering of ZrB<sub>2</sub> and TiB<sub>2</sub> from elemental mixtures**S. R. Bakshi<sup>\*1</sup>; K. NS<sup>1</sup>; M. BS<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

In this study, reactive spark plasma sintering (RSPS) of ball milled Zr/B, Ti/B, Zr/B/SiC and Ti/B/SiC mixtures was carried out resulting in formation of dense fine grained ZrB<sub>2</sub> and TiB<sub>2</sub> and SiC reinforced composites. Systematic investigations were carried out to understand the mechanisms of Reactive sintering. Two densification mechanisms were found to be operating during RSPS. The first stage of densification was due to self-propagating high temperature synthesis (SHS) reaction leading to formation of ZrB<sub>2</sub> and TiB<sub>2</sub> compacts with relative density of ~48% and ~65%, respectively. The second stage of densification occurred at temperatures more than 1100 °C and resulted in final relative density of more than 98%. Interrupted RSPS samples as well as dense samples were analysed using electron backscatter diffraction and electron microscopy. Studies showed deformed grains and presence of slip steps while grain orientation spread map and pole figure analysis confirmed plastic flow. Plastic flow aided pore closure is shown as major mechanism during reactive sintering. Reactive spark plasma sintering is shown to be an attractive method for fabrication of ultra high temperature diborides and their composites.

10:40 AM

## (SINT-092-2017) Electric-field assisted bonding of layered YSZ/ alumina for improvement in thermal barrier coat adhesion

C. Grimley<sup>\*1</sup>; J. Schwartz<sup>2</sup>; A. Prette<sup>2</sup>

1. North Carolina State University, Materials Science and Engineering, USA
2. Lucideon, United Kingdom

Thermal barrier coatings (TBCs) are thermally insulating ceramic layers used to protect turbine engine components from their high operating temperatures. The most common TBC material is 7wt% yttria-stabilized zirconia (YSZ), which is bonded to the metallic pieces by an intermediate thermally grown oxide (TGO) composed of alumina. This layered structure provides interfaces along which the coating often fails mechanically due both to the weakened nature of the interface as well as the continued growth of the TGO layer during operation. Improved adhesion, reduced interfacial impurity, and reduced oxygen content at the TBC/TGO interface have been identified as pathways which may improve the TBC performance and lifetime. Electric-field assisted sintering- including variants flash and spark plasma sintering- is a family of techniques which have been shown to not only reduce the times and temperatures required for ceramics processing but to also produce benefits at grain boundaries such as impurity relocation. Here, we present a study investigating the improvement of adhesion between 7YSZ and alumina via electric-field assisted sintering. The resulting interface was examined for improvements such as decreased impurity content, decreased interfacial porosity, and enhanced adhesion using SEM, EDS, and mechanical testing.

11:00 AM

## (SINT-093-2017) The effect of a small cation off-stoichiometry on flash sintering for SrTiO<sub>3</sub> and BaTiO<sub>3</sub>

T. Yamamoto<sup>\*1</sup>; T. Yoshino<sup>1</sup>; H. Yoshida<sup>2</sup>; T. Tokunaga<sup>1</sup>

1. Nagoya University, Department of Materials Design Innovation Engineering, Japan
2. National Institute for Materials Science (NIMS), Japan

The effect of a small cation off-stoichiometry on sintering behaviors during flash sintering was investigated for SrTiO<sub>3</sub> and BaTiO<sub>3</sub>. It was found that the temperature at flash event and the shrinkage behaviors are very sensitive to a small cation off-stoichiometry, i.e., Sr/Ti or Ba/Ti ratios change. The flash temperature and the final density decrease with increasing in excess Ti, while it increases with increasing in excess Sr or Ba. In addition, the electric current behaviors also depends on the small cation off-stoichiometry. The specimen electric current exhibits surging behaviors in Ti-excess compacts while it does not in Sr or Ba-excess compounds. HRTEM and EEL analysis have revealed that grain boundaries are reduced in Ti-excess flash sintered compacts. Thus, the difference in the flash sintering behaviors could be considered to be closely related to point defects variation due to a small cation off-stoichiometry.

11:20 AM

## (SINT-094-2017) A mesoscopic treatment of sintering in ceramics: Application to direct write additive manufacturing

F. Abdeljawad<sup>\*1</sup>; D. Bolintineanu<sup>1</sup>; A. W. Cook<sup>1</sup>; H. J. Brown-Shaklee<sup>1</sup>; D. Kammler<sup>1</sup>

1. Sandia National Laboratories, USA

Direct write additive manufacturing (AM) has emerged as a powerful technology for building objects by adding material layer-upon-layer. Materials processed via AM exhibit microstructures that are greatly influenced by AM process parameters and several phenomena at different scales. Herein, we present a diffuse interface model of solid-state sintering that is capable of accounting for bulk thermodynamics, free energies of grain boundaries (GBs) and free surfaces, and capturing various mass transport mechanisms. In addition to diffusional fluxes, the framework incorporates an advective flux to allow for rigid body motion of particles due to

the annihilation of defects at GBs. We start our exploration of the model by examining simple particle geometries, where simulation results recover dynamical scaling laws of pore shrinkage kinetics. Then, quantitative analysis of the roles of particle size/distribution, equilibrium dihedral angles, and spatial arrangement of filaments on the evolution of grain microstructures is presented. With the aid of several statistical measures, the spatio-temporal evolution of the pore space and densification rates are quantified in order to identify regions in parameter phase space with optimal microstructures. In broader terms, the proposed modeling framework provides an avenue to explore sintering kinetics of materials processed via direct write AM.

11:40 AM

## (SINT-095-2017) Spark plasma sintering of structure-tailored ultra-high temperature components: First step to being a complex shape

X. Wei<sup>\*1</sup>; O. Izhvanov<sup>2</sup>; C. A. Back<sup>2</sup>; C. D. Haines<sup>3</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, USA
2. General Atomics, USA
3. US Army ARDEC, USA

The net-shaping capability in spark plasma sintering (SPS) of ultra-high temperature ceramics has been explored in the present study. The annular shape or the ring-like shape is considered here as the first step to being a more complex geometry compared to a cylinder or a disk. ZrC powders have been SPS-processed in specially designed graphite tooling to achieve the annular shape geometry. Experimental runs have been carried out to determine the optimal processing parameters for producing highly dense annular shape ZrC specimens. Finite element modeling framework has been constructed to determine the internal stress evolution as well as the densification during the SPS of annular shape ZrC. The formulated processing schemes for annular shape ZrC have been adapted to process SiC/ZrB<sub>2</sub> composite powder with the purpose of making tooling components for SPS applications. The applicability of the obtained composite SPS tooling has been evaluated at high temperature and high pressure SPS regimes.

12:00 PM

## (SINT-096-2017) Producing functional gradient material by high-voltage consolidation of powders

E. G. Grigoryev<sup>1</sup>; A. V. Yudin<sup>1</sup>; E. Olevsky<sup>\*2</sup>

1. NRNU MEPhI, Key Laboratory for Electromagnetic Field Assisted Materials Processing of Novel Materials, Russian Federation
2. San Diego State University, USA

The densification of electrically conductive powders by high voltage consolidation is studied. High voltage consolidation method (HVC) includes the simultaneous exposure of a powder sample to mechanical pressure and to a short (< 300 μs) high voltage (> 1 kV) electric discharge with the pulse current amplitude of a few hundred kA/cm<sup>2</sup>. The densification kinetics of industrial iron powder is analyzed by ultra-rapid video-recording. The integral temperature of the sample has a maximum value at the beginning of the densification process. The compaction process lasts less than 16 ms for all the values of the parameters studied. The shortness of the densification process in comparison with the cooling of the sample provides a constant temperature throughout the entire compaction process. Based on the analysis of the obtained experimental results a mathematical model of high rate wave mode compaction of a powder material under conditions of HVC is formulated. The constitutive equation of the mechanical behavior of the consolidated material accounts for the plastic flow of powder particles and for the collapse of the inter-particle pores. The numerical simulation results reveal optimal values of the dimensionless parameters controlling the HVC process. The computer simulation results are in good agreement with the experimental values of porosity distribution in the consolidated carbonyl iron samples.

**Randall German Honorary Symposium III**

Room: Mission III

Session Chairs: Kathy Lu, Virginia Tech; Hideshi Miura, Kyushu University

**10:00 AM****(SINT-097-2017) Developing grain aligned Alnico type-8 permanent magnets in near-final shape by novel compression molding and sintering treatments (Invited)**I. E. Anderson\*<sup>1</sup><sup>1</sup>. Ames Laboratory (USDOE), Division of Materials Sciences and Engineering, USA

Economic uncertainty in the rare earth (RE) permanent magnet marketplace and an expanding electric drive vehicle market that favors permanent magnet AC synchronous drive motors motivated renewed research in RE-free permanent magnets like “alnico,” based on Al-Ni-Co-Fe. Thus, high-pressure gas atomized isotropic type-8H pre-alloyed alnico powder processing was developed for compression molding to near-final shape and sintering to >99% of theoretical density (7.3g/cc). To produce microstructurally aligned alnico magnets for improved energy product and magnetic remanence, uni-axial stress was added during final sintering to bias grain growth texture, avoiding directional solidification that provides alignment in alnico 9. This solid-state approach produced alnico with large-grained, textured microstructure approaching the 15-degree beneficial threshold of Durand-Charre for enhancing magnetic energy density. Further development should enable aligned anisotropic alnico magnets of highest energy density to be mass-produced. Supported by DOE-EERE-VTO-EDT Program at the Ames Laboratory, operated for the U.S. DOE under contract no. DE-AC02-07CH11358.

**10:20 AM****(SINT-098-2017) Engineering challenges in additive manufacturing of metal components fabricated by selective laser melting (SLM) (Invited)**A. Z. Bunsch<sup>1</sup>; A. P. Ventura<sup>2</sup>; R. Cunningham<sup>3</sup>; W. Z. Misiolek\*<sup>2</sup>

1. AGH University of Science and Technology, Poland
2. Lehigh University, Materials Science & Engineering, USA
3. Carnegie Mellon University, Materials Science & Engineering, USA

There is a need for better understanding of powder characteristics of the material used in the SLM process. This summary presents a broad investigations including powder morphology, optimized powder processing conditions as well as the measurements of the residual stresses present in SLM Cu-4.3%Sn components. The source Cu-4.3%Sn powder was analyzed for basic characteristics, then the residual stress state (by the x-ray diffraction  $\omega\sin^2\psi$  method) was determined for samples with different geometries and heat treatments. Nonhomogeneous residual stresses are present within Cu-4.3%Sn SLM components. Biaxial tensile stress was revealed in the interior of as-printed cube samples and was reduced by annealing, whereas thin strip samples contained biaxial compressive stresses and after annealing the stress becomes more equi-biaxial but is not reduced to zero near the sample surface. This investigation provides insight on the residual stresses in copper alloys fabricated via SLM, which has seen limited study. The methodology of utilizing the  $\omega\sin^2\psi$  can be widely applied in standard x-ray diffraction facilities to study additively manufactured alloys.

**10:40 AM****(SINT-099-2017) Review of some non-conventional rapid hot consolidation techniques**A. Bose\*<sup>1</sup>; R. Sadangi<sup>2</sup>

1. Desktop Metal, USA
2. Armament R&D Engineering Center, USA

Hot consolidation of high performance materials is increasingly being used to improve the structure and properties of materials. Conventional hot processing techniques require longer processing times, resulting in significant grain growth. There are several rapid, non-conventional hot consolidation approaches that have been developed to limit the grain growth and yield rapid consolidation. This paper reviews few of these non-conventional hot consolidation processes.

**11:00 AM****(SINT-100-2017) Development of super-high performance injection molded Ti alloy compacts for aerospace application**H. Miura\*<sup>1</sup>

1. Kyushu University, Research Center for Steel, Japan

Ti-6Al-4V is the most common Ti alloy and has been used in various industrial fields including aerospace and power plant because of their high tensile strength, high fatigue strength and light weight. However, their low thermal conductivity, high hardness and low Young's modulus make them difficult to produce the complicated shapes via machining. On the other hand, metal injection molding process offers net shape production, high design flexibility, and high cost efficiency, which is useful for forming the materials with poor workability such as Ti alloys. In our previous studies, the injection molded Ti-6Al-4V alloy compacts were improved the properties by addition of third elements such as Cr and Mo, and they showed high tensile strength and high elongation comparable to wrought materials. However, the fatigue strength was significantly lower than that of wrought materials. In order to improve the fatigue strength of injection molded Ti-6Al-4V alloy compacts, refining of microstructure was treated by addition third elements including TiB<sub>2</sub> powders. Eventually, we obtained the same fatigue strength as that of wrought materials. In addition, the relationship of grain size and fatigue strength showed good agreement with Hall-Petch relations.

**11:20 AM****(SINT-101-2017) Ni-based superalloy sintered engineered porous layer for direct surface cooling**E. M. White\*<sup>1</sup>; A. J. Heidloff<sup>1</sup>; D. Byrd<sup>1</sup>; R. Anderson<sup>1</sup>; I. E. Anderson<sup>1</sup>

1. Ames Laboratory of USDOE, Materials Sciences & Engineering, USA
2. Praxair, Inc., USA

To increase gas turbine system efficiencies to reach aerospace and land-based power targets, combustor operating temperatures must be increased. However, the temperatures of present combustor turbine engine sections are at or exceeding the disk and blade material limits for high temperature oxidation/corrosion, creep resistance, and strength. Advanced airfoil designs with thermal barrier coatings provide cooling at or near the metal substrate deformation temperatures. To increase operating temperatures while maintaining airfoil temperatures below melting or rapid creep deformation limits, even greater heat removal through new cooling strategies and novel blade and vane designs is required. Porous engineered coatings to permit direct surface cooling of airfoils have been modeled and produced from experimental gas atomized Ni-based superalloy powders that were sieved to an ideal size range for the partial sintering experiments. The resulting layers have been characterized using microscopy, density and mechanical properties. Funding provided by NETL ORD FWP-2012.03.02 and DOE-OS-DMSE through Ames Lab (DE-AC02-07CH11358).



Wednesday, November 15, 2017

## Plenary Session III

Room: Bayview I

Session Chair: Suk-Joong Kang, KAIST

**8:30 AM**

### (SINT-PL-003-2017) Investigating the sintering of multilayer components with advanced experimental and modelling tools (Invited)

D. Bouvard\*<sup>1</sup>

1. University of Grenoble Alpes, SIMAP, France

Powder processing is particularly appropriate to the fabrication of multilayer components, possibly including both ceramic and metal layers. However, due to the different sintering behaviors of the stacked materials, stresses develop during heating, sintering or cooling, which affect dimensional changes and may damage the materials. Thus obtaining near net-shape and defectless components is often challenging. Coupling adapted experimental work and model development has proven to be valuable for better understanding and optimizing the sintering of such components. Three examples of such efforts are presented. They concern, respectively, SOFC stacks comprising porous and dense layers, which often exhibits cambering during sintering, two-layer ceramic-metal parts that crack during heating and multilayer ceramic capacitors that develop local defects in the course of sintering. Appropriate experimental tools include optical dilatometry and computed microtomography whereas modelling tools comprise finite element analysis and discrete element method.

## Spark-plasma and Flash Sintering IV

Room: Bayview I

Session Chairs: Claude Estournes, CIRIMAT

**10:00 AM**

### (SINT-102-2017) Grain growth in oxides in the presence of an electric field (Invited)

W. Rheinheimer<sup>1</sup>; J. H. Preusker<sup>1</sup>; S. Karras<sup>3</sup>; J. P. Parras<sup>2</sup>; R. A. De Souza<sup>2</sup>; R. Garcia<sup>3</sup>; M. J. Hoffmann\*<sup>1</sup>

1. Karlsruhe Institute of Technology, Institute for Applied Materials, Germany
2. RWTH Aachen University, Institute of Physical Chemistry, Germany
3. Purdue University, School of Materials Engineering, USA

In the last years the impact of electric fields on microstructure evolution was investigated in vast detail, e.g. in the field of SPS and flash sintering. Very strong effects were reported, e.g. sintering of zirconia at temperatures below 400°C or sintering within seconds. However, the active mechanisms for these processes are still under debate. Unfortunately the highly needed basic research in this field is very challenging, since the experiments suffer from a poor controllability. For example if currents flow through the sample, true temperatures are hard to estimate. The present study focuses on the impact of electric fields on grain growth in oxide ceramics in order to connect defect concentrations to field assisted microstructure evolution. Well-defined model experiments were used (i.e. no joule heating, since insulating electrodes block electric currents). It is shown that for perovskites the effects in electric field can be well-understood by combining the knowledge on field-free microstructural evolution, defect chemistry and space charge. The findings are used to understand field assisted grain growth in other oxide materials.

**10:40 AM**

### (SINT-103-2017) Integration of nanoscale phases: Leveraging kinetics to densify functional nano-composites (Invited)

Y. Kodera<sup>1</sup>; A. Volodchenkov<sup>1</sup>; K. Chan<sup>1</sup>; J. E. Garay\*<sup>1</sup>

1. University of California, San Diego, Dept. of Mechanical and Aerospace Engrg., USA

Composites are often designed with two distinct materials with each component having a particular property—a highly conductive phase and an insulating phase or a ferromagnetic phase and antiferromagnetic phase for example. Highly successful cases of sintered composites with micrometer scale constituents are numerous whereas composites with nanoscale components are significantly rarer. The inherent difficulty is in densifying the powder while retaining grain size and without unwanted reaction between the constituent phases. We will present results on producing nanoscale composites composed of similar (oxide/oxide) and dissimilar material (metal/oxide) classes using current activated pressure assisted densification (CAPAD). The materials have a crystallite sizes below 100 nm and minimal unwanted reaction between phases. We discuss the results in light of differences in densification and reaction kinetics. We will also present property measurements—emphasizing magnetic properties—to highlight the success of pairing dissimilar materials at the nanoscale.

**11:20 AM**

### (SINT-104-2017) Densification of FeO and FeS with pulsed electric current sintering

M. Nanko\*<sup>1</sup>; K. Ueda<sup>1</sup>; N. Yamaguchi<sup>1</sup>

1. Nagaoaka University of Technology, Department of Mechanical Engineering, Japan

FeO and FeS are typical compounds on high-temperature corrosion of ferritic materials. However, their fundamental properties are still not evaluated. In order to evaluate their mechanical or physical properties, dense bulks are required. In the present study, densification of FeO and FeS with pulsed electric current sintering was discussed. Direct contact of their iron compounds to graphite die causes their reduction at high temperatures. Powder compacts of their iron compounds mounted in BN powder was sintered with pulsed electric current sintering. Dense FeO and FeS bulks were successfully obtained.

**11:40 AM**

### (SINT-105-2017) Effect of austenite fraction on the microstructure and properties of duplex stainless steels consolidated by field-assisted sintering

A. García-Junceda<sup>1</sup>; C. D. Rivera<sup>2</sup>; V. G. Torralba<sup>3</sup>; M. Campos<sup>2</sup>; J. Torralba\*<sup>2</sup>

1. IMDEA Materials Institute, Spain
2. Universidad Carlos III, Spain
3. Universidad Rey Juan Carlos, Spain

This study assesses the possibility of designing new Duplex Stainless Steels (DSS) with austenite contents varying between 70-90 wt.%, using a PM route including the consolidation by Field-Assisted Sintering Technology (FAST). In particular, a novel technique named as Field-Assisted Hot Pressing (FAHP), based on the simultaneous application of an alternating current and a uniaxial pressure, is applied to obtain almost full-density samples. Apart from ferritic and austenitic constituents, another phase with higher local misorientation, elastic modulus and nanohardness is found in the ferrite/austenite interface. The increase in the ferrite fraction may involve a major fraction of Cr available for diffusion, leading to a thicker interdiffusion phase. Although the ferrite is the main responsible of the strength, this investigation suggests some additional contribution of this interdiffusion phase to the final tensile strength and hardness of DSS with austenite contents above 70 wt.%. This work confirms



FAHP as a promising technique to process DSS with homogeneous microstructures and outstanding mechanical properties in terms of elastic modulus, hardness, ultimate tensile strength (UTS), yield strength and ductility.

#### 12:00 PM

##### (SINT-106-2017) Sintering kinetics of titanium silicon carbide MAX phase densified by field assisted sintering

J. Erauw<sup>\*1</sup>; V. Dupont<sup>1</sup>; M. Demuyne<sup>1</sup>; P. Aubry<sup>1</sup>; V. Lardot<sup>1</sup>; F. J. Cambier<sup>1</sup>

1. Belgian Ceramic Research Centre, Belgium

In the present study, the sintering kinetics of a commercial  $Ti_3SiC_2$  powder densified by field assisted sintering has been investigated. Sintering experiments have been performed on a FCT HPD10 system in the temperature range 1100°C-1400°C. The heating rate was varied between 50 °C/min and 250 °C/min, the applied pressure between 30 MPa to 60 MPa and the holding time at maximum temperature between 0 and 15 minutes. Full density samples were achieved above 1300°C irrespective of the heating rate and applied pressure, for holding times in excess of 5 minutes. Besides the detailed analysis of the influence of the sintering cycle on the overall densification behaviour, its influence on the microstructure and evolution of the secondary phases present in small amount in the starting powder (namely titanium carbide and titanium di-silicide) has also been assessed. Finally, the effect of the applied pressure on the anisotropy of selected mechanical properties such as hardness and toughness has been investigated and will be reported. Acknowledgement : the European Regional Development Fund (ERDF) and Wallonia for financial support (Project N° 1610282 - CeraMAX)

#### Microstructural Evolution in Sintering Processes III

Room: Bayview II

Session Chairs: Wayne Kaplan, Technion - Israel Institute of Technology; Torsten Staab, University Wuerzburg

#### 10:00 AM

##### (SINT-107-2017) Two stage sintering – does it work for alumina? (Invited)

K. Drdlikova<sup>1</sup>; D. Drdlik<sup>1</sup>; R. Klement<sup>2</sup>; K. Maca<sup>1</sup>; D. Galusek<sup>\*2</sup>

1. Central European Institute of Technology, Czech Republic
2. Alexander Dubcek University of Trencin, Centre for Functional and Surface Functionalized Glasses, Slovakia

Two-stage sintering provoked intensive discussion in sintering community. The questionable concept of the kinetic window, limited applicability only to ceramics with cubic crystal structure, and the fact that in many reports the authors failed to achieve relative density > 96-98 %, ignoring the fact that the majority of grain growth occurs in the final stage of sintering, when the last 2-3 % of porosity is eliminated, added to the confusion. We summarize our results obtained during several years of study of two-stage sintering in polycrystalline alumina with, and without dopants that affect the grain boundary energy, especially, Mg, Zr, and Y, but also rare earth elements, e.g. Eu and Er. The results can be summarized as follows: Two-stage sintering of undoped sub-micrometer polycrystalline alumina resulted in measurable microstructure refinement, when compared to conventionally sintered material. However, entire suppression of the grain growth could not be achieved. Almost entire suppression of grain growth in the final stage of sintering was achieved in aluminas doped with up to 500 ppm of Mg, Y and Zr. The mechanism is not quite clear, but some evidence indicates that long isothermal heating at lower temperature results in more homogeneous distribution of dopants at grain boundaries. This effect was with success applied in preparation of Eu- and Er-doped transparent polycrystalline alumina with luminescent properties.

#### 10:40 AM

##### (SINT-108-2017) Sintering of pure, doped, and graded magnesium diboride for optimised superconducting properties

C. E. Dancer<sup>\*1</sup>

1. University of Warwick, Warwick Manufacturing Group, United Kingdom

The production of high quality magnesium diboride is necessary to optimise superconducting performance in proposed bulk applications, such as for superconducting magnetic bearings. A number of processing procedures have been carried out to establish key parameters for sintering pure magnesium diboride. Results indicate that pressure-assisted routes such as spark plasma sintering or hot pressing under in a reducing atmosphere are necessary to achieve dense magnesium diboride without the formation of large amounts of deleterious impurity phases. Porosity, impurity content, hardness, grain size, and critical current density ( $J_c$ ) were measured and magnetic flux trapping performance was assessed. Maximum trapped magnetic field for pure magnesium diboride occurred for sample density over 90% of the theoretical maximum, but this was limited by a significant variation in local  $J_c$  across the radius of the sample. Further computational and experimental studies examined the use of dopant additions to enhance microstructural connectivity and so increase  $J_c$  locally to attain a uniform  $J_c$  in the sample. Dopants were distributed in the cylindrical sample both homogeneously and such that the composition varied radially. Results indicate that for optimised superconducting properties a strategy of maximising density and use of selected dopant additions graded through the sample should be employed.

#### 11:00 AM

##### (SINT-109-2017) The effects of particle grading on the sintering behavior, microstructures and mechanical properties of solid-state-sintered SiC ceramics

H. Wu<sup>\*1</sup>; Z. Huang<sup>1</sup>

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, State Key Laboratory of High Performance Ceramics and Superfine Microstructure, China

Effects of grain grading on the sintering behavior, microstructures and mechanical properties of S-SiC ceramics were comprehensively investigated, using the mixtures of 0.8  $\mu\text{m}$  and 8.6  $\mu\text{m}$  powder as the starting materials for the first time. When the proportion of introduced coarse powder was increased to 50 wt%, the sintering shrinkage was reduced low to 12.2 %, and the densification degree exceeded 96 % as well regardless of the existence of slim decrease. Based on the results of sintering rate and microstructure evolution in the densifying process, sintering mechanism of graded powders can be deduced as the bonding of coarse SiC particles through solid-state sintering of fine powder. The pinning effect of introduced coarse particles successfully suppressed abnormal grain growth, resulting in an equiaxial-grain microstructure. Dramatically improved fracture toughness reached up to 3.77  $\text{MPa}\cdot\text{m}^{1/2}$  at 50 wt% of added coarse powder, accompanied with excellent flexural strength (349-431 MPa) and high elasticity modulus (308-342 GPa).

#### 11:20 AM

##### (SINT-110-2017) Densification and grain growth of transparent polycrystalline alumina by two-step pulsed electric current sintering

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1. Nagaoka University of Technology, Japan

Transparent polycrystalline alumina (TPA) is a potential material to replace single crystal sapphire in many optical applications. However, its transparency is still poor, especially at large sizes such as  $\Phi 30$  mm. In order to improve its transparency, densification and grain growth during sintering process need understanding. In this study, those of  $\Phi 30$ -mm polycrystalline alumina were investigated during two-step pulsed electric current sintering. The sintering

program included two temperature-holding steps, 1<sup>st</sup> step at 930°C for 1 h and 2<sup>nd</sup> step at 1130°C for 20 min, with the heating rate of 10 or 100 °C/min. The sintering program was stopped at various positions within the entire program. The temperature of the samples was measured by an R-type thermal couple inserted inside the graphite punch. The temperature at the center of the samples differed 20-40°C from the outer surface of die. This temperature distribution is supposed to be one of the reasons caused the grain size distribution, which is generally larger at the center than near the edge of Al<sub>2</sub>O<sub>3</sub> samples. In conclusions, although the densification was mainly uniform through the entire sintering process, the heterogeneity of grain size was generated mostly in the 2<sup>nd</sup> step. The lower heating rate (10 °C/min) not only restricted the grain growth but also reduced the heterogeneity of grain size.

### 11:40 AM

#### (SINT-111-2017) Pattern formation during current sintering: Experiments

C. Gorynski<sup>\*2</sup>; L. Engelke<sup>1</sup>; M. Jongmanns<sup>1</sup>; D. E. Wolf<sup>1</sup>; M. Winterer<sup>2</sup>

1. University of Duisburg-Essen, Computational and Statistical Physics, Germany
2. University of Duisburg-Essen, Nanoparticle Process Technology, Germany

We observe pattern formation during current sintering of aluminum doped zinc oxide. The formation of bands perpendicular to the current direction occurs on the micrometer scale, much larger than the microstructure in the green body consisting of nanocrystalline particles. Our hypothesis is that the pattern formation is due to a feedback between Joule heating and segregation of Al-Zn-spinel as an insulating phase in doped zinc oxide as conducting matrix. We want to understand the underlying mechanism and control the pattern formation to either eliminate or minimize segregation or deliberately generate tailored patterns. Nanocrystalline aluminum doped zinc oxide is synthesized using Chemical Vapor Synthesis (CVS). Green bodies are formed by uniaxial compaction. They are current sintered with a systematic variation of electrical current and flow geometries. The resulting microstructures are investigated using HRSEM, EDX and XRD. Aluminum is homogeneously distributed in the nanocrystalline zinc oxide particles after CVS although, thermodynamically, it is insoluble. Therefore, segregation and second phase formation is observed after sintering. Pattern formation occurs only during current and not during thermal sintering. We propose a feedback between the electrical current used for the Joule heating during sintering and the generation of an insulating phase as possible mechanism for the self-organized pattern formation.

### 12:00 PM

#### (SINT-112-2017) Al/P/Yb-doped active optical fibers produced using a modified powder sintering method

V. Velmiskin<sup>\*1</sup>; O. Egorova<sup>1</sup>; L. Iskhakova<sup>1</sup>; S. Semjonov<sup>1</sup>

1. FORC RAS, Russian Federation

Today powder sintering for silica optical fibers production is of great interest and is being actively developed due to its unique potentiality. Unfortunately, there is a number of problems, one of which is difficulty in introduction of volatile elements like phosphorus to silica glass. At the same time, Al/P-co-doping of silica allows not to increase its refractive index. This is promising for production of active fibers with large mode area. In this paper, we produced Al<sup>3+</sup>/P<sup>5+</sup>/Yb<sup>3+</sup> doped silica fibers through a new type of the «powder-in-tube» technology. Used as precursors highly purified powders of SiO<sub>2</sub>, Yb<sub>2</sub>O<sub>3</sub> and AlPO<sub>4</sub> were dry mixed in the correct proportion and sintered in an extra pure silica tube. Optical properties of the resulting glass were improved using a procedure of concentration and optical inhomogeneity reducing specially developed by us. A set of optical fibers in a polymer coating was fabricated using sintered samples. The fibers had both homogeneous doping and low and flat

refractive index profile of the active core. The Al<sup>3+</sup>/P<sup>5+</sup>/Yb<sup>3+</sup> contents were 1.85/ 1.8/ 0,18 at%, respectively. Background optical loss level was acceptable (400 dB/km at 1200 nm). 80%-efficiency lasing was obtained for 2 m long single-mode optical fiber using a continuous cladding pumping at 915 nm. Thus, the using of AlPO<sub>4</sub> helps to solve the problem of phosphorus doping in powder sintering method.

## Emerging Topics in Sintering and Micro Development I

Room: Bayview III

Session Chairs: Jianhua Tong, Clemson University; Deborah Blaine, Stellenbosch University

### 10:00 AM

#### (SINT-113-2017) Relationship between grain growth and thermal conductivity in liquid-phase sintered silicon carbide ceramics (Invited)

Y. Kim<sup>\*1</sup>; T. Cho<sup>1</sup>; Y. Kim<sup>1</sup>

1. University of Seoul, Dept. of Materials Science & Engineering, Republic of Korea

Post-annealing after sintering influences the thermal conductivity of liquid-phase sintered SiC (LPS-SiC) ceramics. Two contradictory results were reported on the influence of grain growth on thermal conductivity of LPS-SiC ceramics: post-annealing of SiC ceramics after sintering with La<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> increased its thermal conductivity, whereas post-annealing of SiC ceramics after sintering with Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> decreased its thermal conductivity significantly. In this research, the effect of grain growth on thermal conductivity of SiC ceramics sintered with Gd<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> was investigated and compared with literature data. During prolonged sintering at 2000°C in argon or nitrogen atmosphere, the β→α phase transformation, grain growth, and reduction in lattice oxygen content took place in the ceramics. Effects of these parameters on thermal conductivity of LPS-SiC ceramics were investigated. The results suggest that (1) grain growth achieved by prolonged sintering at 2000°C accompanies the decrease of lattice oxygen content and the occurrence of the β→α phase transformation; (2) the reduction of lattice oxygen content plays a dominant role in enhancing thermal conductivity; and (3) the thermal conductivity of the LPS-SiC ceramic was insensitive to the occurrence of the β→α phase transformation.

### 10:40 AM

#### (SINT-114-2017) Sintering of glass frit

A. L. Fry<sup>\*1</sup>; H. Lee<sup>1</sup>; W. Carty<sup>1</sup>

1. Alfred University, Ceramic Engineering and Materials Science, USA

The hypothesis of this work is that glass frits (i.e., glass particles) sinter at a specific viscosity. Therefore, the sintering temperature is dictated by the viscosity-temperature relationship for any glass composition. As a corollary, it is proposed that glass frit sintering is independent of particle size and the corresponding surface area, since viscous flow sintering mechanisms are not dependent on atomic diffusion (as would be necessary for solid-state sintering). To evaluate this hypothesis, several glass frits were consolidated and sintered using a gradient furnace and a hot-stage microscope (HSM). The HSM uses area variance as a function of temperature to determine both sintering and glass transition temperatures (T<sub>G</sub>). HSM estimated sintering temperatures were typically significantly greater than densification temperatures determined using the gradient furnace and microscopy. Furthermore, the densification temperature for all of the tested frits was slightly above their respective T<sub>G</sub>. The viscosity-temperature relationships were determined via modeling using the Mauro-Yu-Ellison-Gupta-Allan model (MYEGA) and beam-bending viscosity measurements.

11:00 AM

**(SINT-115-2017) Sintering of a zirconia green body investigated by dilatometry and laser flash analysis**E. Post\*<sup>1</sup>

1. NETZSCH Geraetebau GmbH, Germany

The zirconia ceramics are usually produced by sintering of a green body containing some amounts of organic binder. The binder burn-out and sintering parameters are influencing the quality of the ceramics. Sintering and density change of ceramics are traditionally studied by dilatometry. With the laser flash method the temperature diffusivity and the thermal conductivity of the sintered and the raw material can be determined. This contribution will show the sintering curves and kinetic analysis results of a zirconia green body measured by dilatometry. Additionally, the thermal diffusivity (TD) during the sintering process was measured by Laser Flash analysis (LFA). The TD values were corrected for temperature-dependent length change measured by dilatometry. Usually dilatometer measurements are performed at a dynamic heating rate. For LFA measurements isothermal temperature steps are needed where the laser shots were taken. This means the temperature treatment of the LFA and dilatometer samples are different and the shrinkage during the isothermal steps is usually not taken into account for the length correction. In this paper the influence on the TD results were discussed using length-change data from dynamic dilatometer measurements and data of dilatometer measurements with identical temperature profile like the LFA measurements.

11:20 AM

**(SINT-116-2017) Development of SiC nanopowder coated with a very thin additives and its utilization to low temperature SPS**K. Shimoda\*<sup>1</sup>; H. Murakami<sup>1</sup>

1. National Institute for Materials Science (NIMS), Research Center for Structural Materials, Japan

SiC is one of the promising structural materials for high-temperature applications. SiC is difficult to densify without additives because of covalent nature of Si-C bonding and low self-diffusivity. While homogeneous dispersion of additives in SiC matrix is essential for high temperature applications, such ideal mixing is quite troublesome so far. On the other hand, the development of a new nanopowder, on which the desirable kind of additives with a controlled thickness are coated, could lead to the densification of SiC at lower temperatures. In this study, a new nanopowder system, produced by a laser pyrolysis method was applied to fabricate bulk SiC, using SPS at 1600-1800 °C. Surface structure of the nanopowder was analyzed in detail by TEM, HRTEM and XPS. The average particle size was almost 50 nm with the very thin coated layer of Al-containing oxide with thickness below 5nm. The effect of sintering temperature on mechanical and thermal properties of SiC was investigated. Sintered densities above 97% were obtained at a temperature as low as 1700 °C. Toughened microstructures, which consisted of platelet SiC grains and relatively small equiaxed grains, have been obtained when sintered at 1800 °C. Typical flexural strength, fracture toughness, and thermal conductivity of the sintered SiC were 592 MPa, 5.4 MPam<sup>1/2</sup>, and 80 W/mK at room temperature, respectively.

11:40 AM

**(SINT-117-2017) Inverse Hall-Petch behavior of nanocrystalline oxide ceramics fabricated by high-pressure SPS**M. Sokol\*<sup>1</sup>; B. Ratzker<sup>1</sup>; S. Kalabukhov<sup>1</sup>; N. Frage<sup>1</sup>

1. Ben-Gurion University of the Negev, Materials Engineering, Israel

High pressure (up to 1 GPa) spark plasma sintering (HPSPS) allows to fabricate polycrystalline ceramics at relatively low temperatures with short sintering time. Polycrystalline transparent magnesium aluminate spinel (PMAS) obtained by HPSPS technique displays nano-structure (20 -30 nm) and a unique combination of optical and mechanical properties, comparable or even better than the best

results reported in literature for a two-stage fabrication process (pressureless sintering followed by hot isostatic pressing or vacuum sintering). Our recent experimental results on densification of PMAS and yttria stabilized zirconia (YSZ) have raised some fundamental questions related to microstructure evolution, densification and grain growth kinetics. Furthermore, for the first time, a minimal grain size (>40 nm) where a Hall-Petch relation is valid was established and an inverse Hall-Petch correlation was clearly observed for ceramics with grain size less than 30 nm. In the present study, a model explaining the effect of the grain size of nanostructured ceramics on the mechanical properties was developed and will be discussed.

12:00 PM

**(SINT-118-2017) Graphene nanoplatelet and carbon nanotube reinforced titanium diboride composites by reactive spark plasma sintering**S. R. Bakshi\*<sup>1</sup>; K. NS<sup>1</sup>; M. BS<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

In this study, carbon nanotube (CNT) and graphene nanoplatelet (GnP) reinforced TiB<sub>2</sub> based composites have been prepared by reactive spark plasma sintering of ball milled Ti/B/CNT and Ti/B/GnP mixtures. Self propagating high temperature synthesis reaction results in formation of TiB<sub>2</sub> along with small quantity of TiC. High adiabatic temperature of the reaction results in simultaneous synthesis and densification. Highly dense compacts (>99%) can be produced at low temperatures of 1400 °C. Nanograins are formed in case of CNT addition. Addition of 2 vol.% and more of CNT prevents SHS reaction while GnP does not affect the process. Graphene nanoplatelet acts as template resulting in formation of large plate-like grains imparting basal texture in the sample. Texture formation in GnP reinforced TiB<sub>2</sub> is confirmed using XRD and EBSD studies. Significant increase in indentation fracture toughness is observed with addition of 4 vol.% of nano-carbon reinforcements. Textured samples are shown to have higher indentation fracture toughness. Elastic modulus and hardness are found to be very high and reduce marginally with nano-carbon addition.

**Fundamental Aspects of Sintering III**

Room: Mission I

Session Chairs: Jian Luo, UCSD; Aravind Mohanram, Mott Corporation

10:00 AM

**(SINT-119-2017) Sintering behavior of compacted water-atomized iron powder: Effect of initial green state**J. Wendel\*<sup>1</sup>; S. Manchili<sup>1</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden

Press and sinter is the traditional powder metallurgy approach in which water-atomized powder is mixed with a lubricant and compacted at high pressures, giving a compact of desired shape. The compacts are then delubricated and finally sintered in order to strengthen the material. The compaction gives different initial densities as well as different levels of particle deformation. Depending on the particle size, the deformations can pass through the entire particle or be confined to its rim. This will influence the material transport mechanisms responsible for sintering neck formation and densification. In this study, the sintering behavior of two types of water-atomized iron powder, sieved to -45 and -75 μm, was investigated. The powder was mixed with a lubricant and compacted at three different pressure levels of 400, 600, and 800 MPa, as well as with and without graphite as carbon source. Electron back-scatter diffraction (EBSD) was employed to analyze the initial state of the green compacts in terms of the deformed particles. The sintering



behavior of the compacts was investigated by dilatometry using a hydrogen-nitrogen based atmosphere to reveal the effects of compaction. Information from the sintering, including total shrinkage, sintering rates, and calculated activation energies, was then correlated to the initial compact density and respective deformation on the particle level.

### 10:20 AM

#### (SINT-120-2017) Effect of surface chemistry and processing conditions on densification of carbonyl iron powder

J. Wendel<sup>\*1</sup>; R. Shvab<sup>1</sup>; E. Hryha<sup>1</sup>; L. Nyborg<sup>1</sup>

1. Chalmers University of Technology, Department of Industrial and Materials Science, Sweden

Metal injection molding (MIM) is an attractive manufacturing method enabling high-volume production of metallic components with complex shapes. The typical MIM powder has near-spherical morphology, fine particle size, and a narrow size distribution which offers excellent sinterability based on its physical attributes. However, different surface and bulk chemistries also influence the sintering of the powder. In this study, the sintering behavior of two types of carbonyl iron powder, a standard grade and a hydrogen annealed grade, was investigated. Characterization with scanning electron microscopy (SEM) was performed on the powder to ascertain the physical attributes whereas the surface chemistry was investigated more thoroughly with energy dispersive X-ray analysis (EDX) and X-ray photoelectron spectroscopy (XPS). The thermal stability of the powder in terms of the reduction and mass loss behavior was determined with thermogravimetric analysis (TGA). Injection molded and fully debinded brown parts were sintered in a dilatometer using a hydrogen-rich atmosphere. Sintering cycles with different heating rates were also employed to study the effect of the ferrite-austenite phase transformation on shrinkage and densification.

### 10:40 AM

#### (SINT-121-2017) Gravitational role on dimensional control in liquid phase sintering

R. M. German<sup>\*1</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Engineering, USA

By the time of this sintering conference, the research team will have launched 49 test samples for microgravity liquid phase sintering on the International Space Station. The ground-based research is showing how gravity plays a role in densification, pore stratification, microstructure evolution, and most importantly dimensional variation. Many study of sintering practice demonstrated part-part variations, with isolation of factors such as powder lot, equipment changes, and processing conditions. The motivation in this research is to isolate the very specific role of gravity on component dimensional change, especially the role of component mass and substrate drag on nonuniform sintering shrinkage. This research uses an array of liquid phase sintered tungsten alloys processed for various hold times (0 to 60 min). The compositions are a variety of tungsten heavy alloys (W-Cu-Ni-Mn) with theoretical densities ranging up to 18 g/cc. Composition changes give different solid:liquid ratios and dihedral angles. The sintering trajectory in terms of dimensional uniformity depends on gravity. The findings are relevant to current industrial practice and anticipated space-based repair and fabrication concepts. This paper will combine recent precise measurements on sintered dimensions with concurrent simulation models to map the combinations of factors most typically inducing nonuniform sintered dimensions.

### 11:00 AM

#### (SINT-122-2017) Powder chemistry effects on grain boundaries and sintering of Bayer alumina

T. Frueh<sup>\*1</sup>; E. R. Kupp<sup>1</sup>; C. Compson<sup>2</sup>; J. Atria<sup>2</sup>; G. L. Messing<sup>1</sup>

1. Pennsylvania State University, USA
2. Almatix, Inc., USA

Commercial high purity Bayer alumina powders are typically 99.8 - 99.9% pure and contain Na<sub>2</sub>O, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CaO as main impurities and MgO as a sintering aid. During heating these impurities and aids can combine to form a nanometer thick liquid grain boundary phase. High-resolution TEM and EDS studies reveal how physical and chemical characteristics of the grain boundaries change as a function of powder chemistry. We show that the viscosity and solubility of Al<sub>2</sub>O<sub>3</sub> in the liquid phase are strongly affected by impurity chemistry and systematically investigate how the sintering kinetics and fundamental sintering mechanisms of Bayer alumina change as a function of the total amount and relative ratios of the impurities and dopants. By comparing the sintering kinetics, grain boundary chemistries and structures of MgO-free and MgO-doped Bayer aluminas, we identify how MgO affects the sintering process. We then develop a model to predict the evolution of thickness and properties of grain boundaries during densification.

### 11:20 AM

#### (SINT-123-2017) Experimental study of electromagnetic field impact on surface diffusion in zinc oxide ceramic

A. Ducoulombier<sup>\*1</sup>; F. Valdivieso<sup>1</sup>; J. Bruchon<sup>1</sup>; P. Ganster<sup>1</sup>; S. Saunier<sup>1</sup>; C. Meunier<sup>1</sup>

1. Mines Saint Etienne, SMS/MPE, France

Sintering is a key step to obtain a dense material from powders. Compared to conventional treatment, microwave heating is a promising way to obtain a faster densification, and at lower temperature. Controlling this process includes a well understanding of how the electromagnetic wave coupling with matter would affect diffusion mechanisms and consequently sintering kinetics. The aim of this work is to study sintering mechanism comparing the kinetic of surface diffusion by two heating ways. So that, an experimental work is dedicated here to compare the mechanism of diffusion which occurs during the sintering in conventional and microwave heating (5.8 GHz single-mode microwave furnace) using ZnO samples. In this goal, an adapted method has been set up to produce samples with high densification, large grain size, and very low rough surfaces. First, the surfaces were polished, and in inner grains scratches were realized using FIB technics. Then, the leveling of these scratches and their widths are followed for various temperature stages with different dwell time. Finally, the leveling evolution was measured using AFM. Based on V.W. Mullins works, surface diffusion coefficients and activation energy associated were computed from these experimental works for conventional and microwave heating at low temperature (below 700°C).

### 11:40 AM

#### (SINT-124-2017) Evolution of uniaxial viscosity and microstructure during field-assisted sintering of rare earth doped ceria

M. Mutter<sup>\*1</sup>; C. Cao<sup>1</sup>; R. Mücke<sup>1</sup>; O. Guillon<sup>1</sup>

1. Forschungszentrum Jülich GmbH, IEK-1, Germany

Rare earth doped ceria is a promising material for the use as electrolyte in solid oxide fuel cells and as gas separation membrane. The field-assisted sintering offers the possibility to densify the material in short time scale. However, the effects of the applied field on the sintering parameters and microstructure evolution still need to be investigated in detail. For this purpose, green bodies with a density of about 60 % were manufactured using nano-scaled powder and isothermally sintered under the application of mechanical and electrical field in a sinter-forging device equipped with laser scanner



and programmable power supply. Particular attention is paid on the influence of the electric field on the densification behavior. The uniaxial viscosity is determined by discontinuous sinter-forging as a function of density and enables prediction of the sintering behavior of the material on a macroscopic scale. Temperature as well as current flowing through the sample is controlled thoroughly. The microstructure evolution is evaluated experimentally on the sintered samples at various sintering stages. In parallel, a kinetic Monte Carlo (kMC) simulation is developed that incorporates anisotropic effects of grain growth and pore migration induced by applied directed fields for a deeper insight on the fundamental processes connected with the microstructural evolution.

## Sintering of Multi-material and Multi-layer Systems II

Room: Mission II

Session Chairs: Chris Haines, US Army ARDEC; Zuoren Nie

### 10:00 AM

#### (SINT-125-2017) Synthesis of multiphase Nb/SiCN ceramic composites by reaction of niobium with polysilazanes (Invited)

G. Motz\*<sup>1</sup>; M. Seifert<sup>1</sup>

1. University of Bayreuth, Ceramic Materials Engineering, Germany

Due to their extraordinary temperature stability refractory metal based composites containing carbide, nitride and silicide phases are very interesting for ultrahigh temperature applications. Based on the high melting points of refractory metals and their carbides, nitrides and silicides processes like self-combustion high temperature synthesis (SHS), spark plasma sintering (SPS) or reactive hot pressing (RHP) are required to manufacture bulk composites. In comparison, polymer derived ceramics (PDC) technology allows the manufacturing of complex shaped ceramics at lower temperatures. By using the increased reactivity of an organopolysilazane (OPSZ) during pyrolysis the reaction with niobium powder as active filler with content variations from 40 to 70 vol% various, specially tailored multiphase Nb(C,N)/Nb<sub>5</sub>Si<sub>3</sub> ceramic composites were designed after heat treatment up to 1600 °C. X-ray analysis proved the formation of Nb<sub>5</sub>Si<sub>3</sub>, metastable Nb<sub>5</sub>Si<sub>4</sub>C and different Nb(C,N) phases depending on the starting OPSZ/Nb ratio and the pyrolysis conditions. After milling, the resulting multiphase powders were densified by spark plasma sintering (SPS). The SPS approach led to very dense samples at 1600 °C and an applied uniaxial pressure of 100 MPa. XRD measurements and EBSD analysis confirmed that SPS retains the original phase composition and grain size of the multiphase powders during sintering.

### 10:40 AM

#### (SINT-126-2017) Electric field flash sintering of multiphase ceramics (Invited)

M. Mecartney\*<sup>1</sup>; D. Kok<sup>1</sup>; Y. Yang<sup>1</sup>; R. Raj<sup>2</sup>

1. University of California, Irvine, Chem. Engr. & Materials Science, USA

2. University of Colorado, Boulder, USA

This talk will focus on the use of an applied electric field in air without the use of significant pressure to flash sinter multiphase composite ceramics. Multiphase materials with different compositions are evaluated for suitability for flash sintering including alumina, yttria-stabilized zirconia (YSZ), spinel, magnesia, and monazite. High power densities can sinter three phase alumina-spinel-YSZ ceramics in seconds with furnace temperatures as low as 1150C. While all multicomponent compositions are stable during conventional sintering, if a range of stoichiometry is possible, such as for MgAl<sub>2</sub>O<sub>4</sub>, adjoining phases such as Al<sub>2</sub>O<sub>3</sub> can be consumed to form high-alumina spinel with flash sintering with extremely fast kinetics. The converse, high-MgO spinels, is considerable more difficult as predicted by the shape of the solid solution region on the phase diagram. It is well known that YSZ flash sinters relatively easily, and the minimum amount of YSZ required to achieve

flash sintering depends not only on the temperature but also on the applied field. Lastly, from these experiments, conclusions about the relative ease of flash sintering related to the thermal conductivity and ionic/electronic conductivity of the individual components will be discussed.

### 11:20 AM

#### (SINT-127-2017) Utilizing cold sintering process for the fabrication of ceramics and ceramic-based composites

J. Guo\*<sup>1</sup>; H. Guo<sup>1</sup>; A. Baker<sup>1</sup>; S. Funahashi<sup>2</sup>; M. Lanagan<sup>1</sup>; C. Randall<sup>1</sup>

1. Pennsylvania State University, Department of Materials Science & Engineering, USA

2. Murata Mfg. Co., Ltd., Japan

The sintering process is the most basic part of processing materials, which involves the compaction and forming of a solid material through the application of thermal energy and/or pressure. Owing to the high melting points, most of the ceramics are sintered at high temperatures, using the conventional thermal sintering process. Cold sintering process (CSP) is a new sintering technique that we are exploring to sinter ceramics and ceramic-based composites with the assist of a transient liquid phase, such as water and acids. With CSP, the sintering temperatures of ceramics can be decreased from several hundreds of degrees or even tens of hundreds of degrees to lower than 300 °C. The low sintering temperature makes it possible to co-sinter thermoplastic polymers and ceramic materials in a one-step sintering process. A bulk ceramic, a multilayer ceramic, a bulk ceramic/polymer composite, and a ceramic/carbon-nanofiber thick film with different applications, are selected to show the feasibility of CSP. Cold sintering is more general and a diverse range of inorganic materials and composites can be sintered at temperatures in the range of room temperature and 300 °C. We anticipate that CSP can provide a simple, effective and energy-saving route for the fabrication of ceramics and ceramic-based composites.

### 11:40 AM

#### (SINT-128-2017) Sintering of titanium carbonitride reinforced alumina tool ceramics

M. Szutkowska\*<sup>1</sup>; B. Smuk<sup>1</sup>; P. Putyra<sup>1</sup>; M. Podsiadlo<sup>1</sup>

1. The Institute of Advanced Manufacturing Technology, Poland

Recent advances in tool materials have focused on reinforcing alumina with different carbides, oxides and nitrides in order to improve hardness, fracture toughness and wear resistance. The present study reports results obtained by reinforcing alumina ceramics with Ti(C,N) in amount 30 wt.% prepared on the basis micro and nanoscale trade powders. The pressureless sintering in a vacuum and FAST method of sintering were used. Vicker's hardness, apparent density, wear resistance and fracture toughness ( $K_{IC}$ ) at room and elevated temperatures up to 800°C characteristic for tool work were evaluated. Properties of the ceramics based on the powders in microscale were compared with ceramics comprising nanoscale phase in a range from 2 to 36 wt.%. Tested ceramics with nanoscale phase reveal lower  $K_{IC}$  (approx. 10-30%) at ambient temperature in comparison to ceramics based on powders in microscale. However, in the elevated temperatures their fracture toughness increases from 10% to 30%. The observation of the microstructure of specimens was carried out using scanning electron microscopy. Preliminary industrial tests confirm the high cutting performance of tested ceramic cutting inserts.

12:00 PM

## (SINT-129-2017) Heterogeneity effects during sintering for tile clay traditional materials

H. Camacho Montes<sup>\*1</sup>; C. I. Villa Garcia<sup>2</sup>; A. Garcia Reyes<sup>2</sup>; R. Bordia<sup>3</sup>

1. Universidad Autonoma de Ciudad Juarez, Fisica y Matematicas, Mexico
2. InterCeramic Technological Center, Mexico
3. Clemson University, USA

Ceramic industry is one of the most important activities in the north part of Mexico. Tile and wall covers are popular products in the market. These ceramics pieces can be described as a three laminated composite made of different materials. By far, this is only one cause of heterogeneity during firing. Temperature and density distributions are also a source for heterogeneity. These three features induced a constrained sintering behavior that is very common for traditional ceramic processing. In the present work, we try to describe the firing process based on continuum mechanics. Three problems are stated. As the first step, the thermal problem to calculate the temperature profiles evolution is developed. We consider that thermal expansion does not depend on density. However, due to thermal gradients and the involved three different materials are supposed to induce thermal expansion stresses. These stresses are calculated in the thermo-elastic problem. Finally, the temperature and the thermo-elastic stresses distributions are imported into a viscous problem focused on describing densification and deformation during sintering. All the mentioned problems are solved applying the Finite Element Method. This way, a continuous analysis can be used to better characterize traditional ceramic firing. It is also worthily to mention that set of needed properties is also a useful tool for characterization.

## Chemical Interactions during Sintering

Room: Mission III

Session Chair: Zhigang Fang, University of Utah

10:00 AM

## (SINT-130-2017) Reactions during sintering of metallic compacts that involve interstitial elements (Invited)

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1. Technische Universität Wien, Institut für Chemische Technologien und Analytik, Austria

Traditionally, studies on sintering of metallic systems have been focused on processes in the metallic phase, such as neck formation and growth, pore elimination and homogenization. On the other hand, for PM bodies one prominent feature is the very large specific surface that makes them highly reactive, much more than cast and wrought metals. The main reaction partners are interstitial elements that may emerge from the starting powders, such as C and O, or from the surrounding atmosphere, which includes C, O, N and H. In the present work, the reactions of interstitials occurring during sintering are described and discussed, and their respective impacts on sintering behaviour and resulting properties are assessed.

10:40 AM

## (SINT-131-2017) Quantitative analysis of the effect of refractory particles on the sintering of Cu: From preventing swelling to hindering shrinkage

A. Papillon<sup>2</sup>; J. Missiaen<sup>\*1</sup>; J. Chaix<sup>3</sup>; S. Roure<sup>3</sup>; H. Schellekens<sup>4</sup>

1. University of Grenoble Alpes, Grenoble INP, France
2. Schneider Electric, Site Technopole, France
3. Schneider Electric, 38EQI, France
4. Schneider Electric, Usine 38V, France
5. University of Grenoble Alpes, CNRS, France

Addition of refractory particles to a copper powder compact usually hinders shrinkage, as inert particles inhibit the displacement of sintering particles. Atypical behaviour was observed during sintering

of Cu-Cr powder compacts: a maximum uniaxial shrinkage was observed during sintering for 10-20 vol% addition of refractory particles. The beneficial effect at low Cr content is related to the getter effect of Cr particles, which prevents gas trapping in the closed porosity and therefore avoids swelling of the powder compact. A critical amount of refractory particles is necessary for trapping the vapour phase impurities formed during heating. On the opposite, sintering is hindered at high Cr content due to the resistance to shrinkage of the inert particle skeleton. By quantifying both effects and taking into account shrinkage anisotropy, the range of refractory volume fraction for optimizing the final sintered density was estimated and is found in agreement with experimental observations.

11:00 AM

## (SINT-132-2017) Effect of the sintering parameters and process atmosphere on the thermodynamics and kinetics of the sintering of prealloyed water-atomised PM steels

E. Hryha<sup>\*1</sup>

1. Chalmers University of Technology, Materials and Manufacturing Technology, Sweden

Particles of the typical water atomised steel powder are covered by heterogeneous surface oxide, formed by thin (~6 to 8 nm) iron oxide layer, covering about 95% of the powder surface, and particulate features formed by thermodynamically stable oxides. Full removal of the iron oxide layer is required in order to develop strong inter-particle necks. Inability to remove iron oxide layer at low temperatures bring significant risk of the oxide transformation and particulate oxide growth in the areas with poor thermodynamic conditions – e.g. growing inter-particle necks. Despite the low thermodynamic stability of the iron oxide layer, its removal in the powder compact is very complex process, determined by a number of parameters such as temperature profile, sintering atmosphere (composition and purity), compact properties (geometry, density, etc.), powder properties (alloy composition, powder size, surface chemistry, etc.), additives, etc. Paper summarised effect of the mentioned above sintering process parameters and powder properties on the final component properties based on the kinetics and thermodynamics of the surface oxide transformations, studied by thermogravimetry analysis, dedicated microscopy and surface analysis of the initial powder and fracture surface of the sintered components as well as thermodynamic simulations.

11:20 AM

## (SINT-133-2017) Effect of C/Ti ratio on densification, microstructure and properties of TiC<sub>x</sub> prepared by reactive spark plasma sintering

S. R. Bakshi<sup>\*1</sup>; V. Kombamuthu<sup>1</sup>

1. Indian Institute of Technology Madras, Metallurgical and Materials Engineering, India

TiC is known to be a hard ceramic having potential application in coatings and as a reinforcement in metal matrix composites. TiC is highly non-stoichiometric with C/Ti ratio extending from 0.4 to 1. In the present study, TiC<sub>x</sub> (x = 0.34 to 0.78) has been prepared using reactive spark plasma sintering of ball milled Ti and carbon black powders. Wet milling in toluene is shown to contribute excess carbon due to decomposition of toluene. Two stage sintering process consisting of gradual formation and densification of TiC<sub>x</sub> at the beginning followed by plastic flow assisted densification was observed. The lattice parameter of TiC<sub>x</sub> was correlated with the C/Ti ratio and compared with previously reported results. The intensity ratio of (111) peak to (200) peak was analysed to relate it with the C/Ti ratio and the texture in the sample. The onset of second stage involving plastic flow is found to be significantly dependent on the C/Ti ratio. Dense (>99%) compacts could be prepared by reactive sintering at 1400 °C. Grain size was found to reduce significantly with increase in C/Ti ratio. It is observed that the hardness and

elastic modulus respectively increased from 16 to 27 GPa and 201 to 377 GPa with increase in C/Ti ratio from 0.34 to 0.78.

#### 11:40 AM

##### (SINT-134-2017) Effect of boriding on mechanical, thermal and oxidation properties of TZM alloy prepared by spark plasma sintering process

B. Yavas<sup>\*1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgical and Materials Engineering, Turkey

TZM alloy is one of the most important molybdenum (Mo) based alloy which has a nominal composition containing 0.5–0.8 wt% titanium (Ti), 0.08–0.1 wt% zirconium (Zr) and 0.016–0.02 wt% carbon (C). It is a possible candidate for high temperature applications in a variety of industries. However, the rapid oxidation of TZM alloys at high temperature in air and low wear resistance are considered to be the main drawbacks. In this study, the effect of boriding on mechanical, thermal and oxidation properties of TZM alloy was investigated. The boriding of TZM alloy carried out by spark plasma sintering (SPS) process by using boron carbide (B<sub>4</sub>C) powder as a boron source. Both sintering and boriding process were performed in a single step at constant temperature of 1420 °C in various pressure (40–60 MPa) and holding time (5–15 min) under vacuum. Molybdenum boride layers having a thickness in the range of 137–213 μ were formed on the surface of TZM alloy after sintering process. The formation of boride layers composed of MoB and Mo<sub>2</sub>B contributed to improvement of the hardness, thermal resistance and oxidation properties of TZM alloy

#### Spark-plasma and Flash Sintering V

Room: Bayview I

Session Chairs: Rachman Chaim, Technion - Israel Institute of Technology; Raymond Brennan, US Army Research Laboratory

#### 2:00 PM

##### (SINT-135-2017) The effect of lithium doping on the sintering and grain growth of non-stoichiometric magnesium aluminate spinel (Invited)

S. Hayun<sup>\*1</sup>

1. Ben-Gurion University of the Negev, Materials Engineering, Israel

In the present talk, the effects of lithium doping on the sintering and grain growth of non-stoichiometric nano-sized magnesium aluminate spinel will be presented. Li-doped nano-MgO·nAl<sub>2</sub>O<sub>3</sub> spinel (n = 1.06 and 1.21) powders containing 0, 0.20, 0.50 or 1.00 at. % Li were synthesized by the solution combustion method and dense specimens were processed spark plasma sintering (SPS) apparatus at 1200 °C and under an applied pressure of 150 MPa. The SPS-processed samples showed mutual dependency on the lithium concentration and the alumina-to-magnesia ratio. For example, the density and hardness values of near-stoichiometry samples (n = 1.06) showed an incline up to 0.51 at. % Li, while in the alumina rich samples (n = 1.21), these values remained constant up to 0.53 at. % Li. Studying grain growth revealed that in the Li-MgO·nAl<sub>2</sub>O<sub>3</sub> system, grain growth is limited by Zener pinning. Moreover, the effect of the applied electric field on cations distribution will be discussed.

#### 2:40 PM

##### (SINT-136-2017) Effect of electric current on high temperature flow behavior of 8Y-ZrO<sub>2</sub>

K. Morita<sup>\*1</sup>; H. Yoshida<sup>1</sup>; B. Kim<sup>1</sup>; K. Hiraga<sup>1</sup>; Y. Sakka<sup>1</sup>

1. National Institute for Materials Science (NIMS), Japan

Flash sintering phenomena can succeed to lower the sintering temperature of several ceramics. SPS technique is also regarded as one of the external current applied sintering methods under pressure. For the SPS method, since the sintering occurs through

deformation processes, especially in the initial and intermediate stages, the understanding of the current effect on the deformation is important. Conrad et al. examined the effect of electric bias on the tensile properties of 3Y-TZP and confirmed that the bias can lower the flow stresses. Although the enhanced deformation was explained by suppressed grain growth, the effect of electric field on the deformation is still unclear. In order to clarify the current effect, therefore, this study was examined the tensile behavior under current conditions using fine grained 8Y-ZrO<sub>2</sub>. By applying the current higher than a critical value E<sub>c</sub>, flash event similar to that of powder sintering occurs even in the bulk 8Y-ZrO<sub>2</sub>. For lower than E<sub>c</sub>, the applied field increases sample temperature depending on the applied value, but does not enhance the rate of deformation. For higher than E<sub>c</sub>, the current can enhance the rate of the deformation to >10 times as compared with that of no current conditions. The enhanced deformation cannot be explained only by the increment of sample temperature and is likely to occur by the flash event similar to the sintering.

#### 3:00 PM

##### (SINT-137-2017) Rapid rate sintering of highly transparent AlON ceramics

Y. Shan<sup>1</sup>; X. Wei<sup>\*2</sup>; X. Sun<sup>1</sup>; E. Olevsky<sup>2</sup>; J. Xu<sup>1</sup>; Q. Qin<sup>3</sup>

1. Dalian Maritime University, Department of Materials Science and Engineering, China
2. San Diego State University, College of Engineering, USA
3. Australian National University, Research School of Engineering, Australia

Using bimodal AlON powder synthesized by carbothermal reduction and nitridation method, spark plasma sintering is adopted to rapidly sinter transparent aluminum oxynitride (AlON) ceramics, and the influence of heating rate on phase assemblages, densification and microstructure is investigated. After holding 1min at 1600°C under 60MPa, high density specimens are obtained at heating rate of 50–250°C/min, and all of the fabricated specimens show higher transmittance, i.e., the maximum transmittance for 1.8mm thick specimens is up to 70.29%–77.53%. Moreover, it is observed that the faster the heating rate is, the higher transmittance is measured. It indicates that highly transparent AlON ceramics can be fabricated by rapid rate sintering of the bimodal AlON powder. The phase assemblage analysis reveals that the phases of sintered specimens also depend on the heating rate due to the phase transformations between AlON, α-Al<sub>2</sub>O<sub>3</sub> and AlN. Besides AlON, α-Al<sub>2</sub>O<sub>3</sub> and a trace of AlN phases were detected also in the specimens heated at 50°C/min. However, only AlON can be detected in the specimens fabricated at 100–250°C/min, which indicates that the transformation of the AlON phase can be affected by fast heating. Also, the processed specimens exhibit high mechanical properties, i.e. Vickers hardness of ~16.5GPa and fracture toughness of ~2.5MPa<sup>1/2</sup>. This should be attributed to the fine grain structure formed during rapid sintering.

#### 3:40 PM

##### (SINT-138-2017) Processing and properties of transparent anisotropic ceramics using applied currents and pressures

Y. Kodera<sup>\*1</sup>; E. Penilla<sup>1</sup>; A. Wieg<sup>1</sup>; J. E. Garay<sup>1</sup>

1. University of California, San Diego, Dept. of Mechanical and Aerospace Engrg., USA

Light scattering due to birefringence has prevented the use of polycrystalline ceramics with anisotropic optical properties in applications such as laser gain media. However, continued development of processing technology has allowed for very low porosity and fine grains, significantly improving transparency and is paving the way for polycrystalline ceramics to be used in demanding optical applications. We will discuss the important microstructural features that contribute to the optical properties and some processing techniques that allow for microstructural control. Specifically, we will discuss



the role of point defects as purposely added dopants and unwanted contaminants as well as secondary phases in oxides and nitrides. We will emphasize the important role of the powder (purity, morphology and particle size). We will also show property measurement oriented results from ongoing efforts to develop ceramics for light emission (solid state lasers and lighting). In the photoluminescence case, we discuss the role of dopant concentrations and dopant agglomeration on the optical properties.

**4:00 PM**

### **(SINT-139-2017) Flash sintering of a three-phase alumina, spinel, and yttria-stabilized zirconia composite**

D. Kok<sup>\*1</sup>; S. K. Jha<sup>2</sup>; D. Yadav<sup>2</sup>; E. Sortino<sup>2</sup>; R. Raj<sup>2</sup>; M. Mecartney<sup>1</sup>

1. University of California, Irvine, Chemical Engineering and Material Science, USA
2. University of Colorado at Boulder, Mechanical Engineering, USA

Three-phase ceramic composites constituted from equal volume fractions of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, MgAl<sub>2</sub>O<sub>4</sub> spinel and cubic 8 mol% Y<sub>2</sub>O<sub>3</sub>-stabilized ZrO<sub>2</sub> (8YSZ) were flash-sintered under the influence of DC electric fields. The temperature for the onset of rapid densification (flash sintering) was measured using a constant heating rate at fields of 50-500 V/cm. The experiments were carried out by heating the furnace at a constant rate. Flash sintering occurred at a furnace temperature of 1350°C at a field of 100 V/cm, which dropped to 1150°C at a field of 500 V/cm. The sintered densities ranged from 90-96%. Higher electric fields inhibited grain growth due to the lowering of the flash temperature and an accelerated sintering rate. During flash sintering, alumina reacted with the spinel phase to form a high-alumina spinel solid solution, identified by electron dispersive spectroscopy and from a decrease in the spinel lattice parameter as measured by X-ray diffraction. Synchrotron in-situ X-ray diffraction experiments have shown that this phase transformation happens in 3 seconds or less. It is proposed that the solid solution reaction was promoted by a combination of electrical field and Joule heating.

**4:20 PM**

### **(SINT-140-2017) Spark plasma sintering of SiC fiber-reinforced SiC composite**

M. Ohyanagi<sup>\*1</sup>; K. Shirai<sup>1</sup>; R. Ozaki<sup>1</sup>

1. Ryukoku University, Japan

Much attention has been paid on SiC with a high melting point for the application to turbine blades of aircraft engines. Since SiC exhibits extremely low fracture toughness, its ceramic fiber-reinforced ceramics composite (CFCC) has been developed, and the improvement of toughness is devised. In the case of SiC-CFCC, a preform made of a bundle of SiC fibers (SiC<sub>f</sub>) coated with h-BN is fabricated and the voids in the bundles are filled with SiC by repeating the process for several times in which a filling of the polyorganosilane into the voids of bundles made of SiC<sub>f</sub> and subsequent pyrolysis to form SiC. A reactive sintering with filling SiC, Si, C particles in the voids of bundles may also be processed. Conventionally, a gas phase method such as CVD has been used to coat h-BN on the SiC<sub>f</sub> surface. We, herein, have investigated a method of coating h-BN on the surface of SiC<sub>f</sub> in the fabric woven by liquid phase method. The BN-coated SiC<sub>f</sub> was consolidated with stacking-sequence disordered SiC under the temperature which the shape of fiber was maintained through inactive BN without the direct sintering between the fibers and the SiC matrix. The sintered SiC-CFCC was also evaluated for mechanical properties.

## **Modeling and Simulation of Sintering at Multiple Scales II**

Room: Bayview II

Session Chairs: Bernd Kieback, Technische Universität Dresden / Fraunhofer IFAM; Fumihiko Wakai, Tokyo Institute of Technology

**2:00 PM**

### **(SINT-141-2017) Modeling a material from packing, through sintering and to the final microstructural properties (Invited)**

R. Bjørk<sup>\*1</sup>; K. Nielsen<sup>1</sup>

1. Technical University of Denmark, Department of Energy Conversion and Storage, Denmark

We present a combination of numerical models that can together simulate the initial packing of particles, followed by sintering and finally the resulting microstructural properties. For the latter we here focus on the magnetism of a sintered sample, and the associated coupling between heat and magnetism known as the magnetocaloric effect. We present a 3-dimensional time-dependent numerical model that spatially resolves samples down to the grain size, and includes the demagnetizing field, chemical inhomogeneity realized as a spatial variation of Curie temperature across the sample, local hysteresis and heat transfer. We can thus model how particle size, packing, sintering and chemical inhomogeneity affect the observed properties of magnetocaloric samples. For example, we show that even a modest distribution in Curie temperature ( $T_C$ ) across the sample results in a significant broadening and lowering of the total entropy change of the sample around  $T_C$ . We discuss how clustering of grains with similar values of  $T_C$  across the sample influences the results.

**2:40 PM**

### **(SINT-142-2017) Modelling sintering by cellular Automata-Monte Carlo approach**

X. Wang<sup>\*1</sup>; A. Atkinson<sup>1</sup>

1. Imperial College London, Dept of Materials, United Kingdom

Sintering is a complicated process involving multiple mass transport processes and complex microstructure changes, typically leading to both densification and coarsening. Theoretical modelling plays an important role in improving understanding of the sintering process. We have developed a Cellular Automata-Monte Carlo (CA-MC) approach for modelling the microstructure evolution and sintering of materials. This modelling approach, based on a new concept of 'structural imbalance' of interfaces and surfaces, is capable of incorporating multiple mass transport mechanisms acting in parallel and dealing with real 3D microstructures without unnecessary simplification or assumptions regarding particle and pore shapes. We will show that this approach can generate not only accurate quantitative kinetic information of sintering (both coarsening and densification), but also the evolution of 3D microstructure details. Our model also takes into account the effect of shrinkage mismatch among different neck-connected particle pairs on the rigid body motion of particles associated with grain boundary collapse. The simulation approach is illustrated by applying it to investigate the influence of particle packing and surface transport, relative to grain boundary transport, on densification kinetics. The predictions of the simulations are compared with experimental observations of the sintering of nickel powder compacts.



**3:00 PM****(SINT-143-2017) A thermodynamically consistent phase-field framework for solid-state sintering**Q. Yang<sup>\*1</sup>; C. Liu<sup>2</sup>; J. Shen<sup>3</sup>; L. Chen<sup>1</sup>

1. Pennsylvania State University, Materials Science and Engineering, USA
2. Pennsylvania State University, Mathematics, USA
3. Purdue University, Mathematics, USA

A novel phase-field-based approach to modeling sintering process is proposed. It incorporates the convective mass transport due to particle rearrangement in addition to different mass diffusion mechanisms and the convective velocity field through the Navier-Stokes equation. All the governing equations are consistently derived from a thermodynamic model for inhomogeneous systems. The proposed model is applied to studying microstructure evolution in solid-state sintering. It is shown that the convective mass transport is critical to correctly model the solid state sintering process. The presented model can also be employed to simulate other types of sintering processes, for example, liquid phase sintering where convection effect is expected to be even more significant.

**3:40 PM****(SINT-144-2017) Electromagnetic thermal mechanical modeling of microwave sintering: From simple to complex shapes (Invited)**C. Maniere<sup>\*1</sup>; T. Zahrah<sup>2</sup>; E. Olevsky<sup>1</sup>

1. San Diego State University, Engineering, USA
2. Matsys Inc., USA

Microwave sintering presents several benefits compared to the conventional sintering such as high heating rates, volumic sample heating, acceleration of the sintering kinetics and energy savings. However, this process remains unstable and difficult to control. These instabilities are caused by the materials properties runaway, the resonance phenomena, and the unequal wave distribution in the cavity. In order to predict and adjust these parameters for realistic shapes, a fully coupled electromagnetic thermal mechanical (EMTM) model has been developed. This model is able to predict the microwave distribution in the cavity, the temperature field, the densification and distortions of the sample with advanced simulation tools such as the surface to surface radiation and the proportional integral derivative (PID) regulation. The EMTM model has been tested for different sample dimensions, direct/hybrid heating configurations, and for simple and complex specimen shapes.

**4:20 PM****(SINT-145-2017) Peridynamic modeling of sintering (Invited)**S. A. Silling<sup>\*1</sup>; V. Tikare<sup>1</sup>; B. G. Van Bloemen Waanders<sup>2</sup>; D. Z. Turner<sup>1</sup>

1. Sandia National Laboratories, Multiscale Science Department, 1444, USA
2. Sandia National Laboratories, USA

The peridynamic theory is a generalization of the standard theory of continuum mechanics that treats all internal forces as nonlocal. This means that in the peridynamic theory, material points interact directly through forces across finite distances, as determined by the deformation and material model. This is in contrast to the standard theory, which assumes that deformations are continuous and that material points interact only when they are in contact with each other. In sintering, densification by diffusion of mass occurs to minimize interfacial energies. The Potts model simulates densification and mass transport by minimizing energy of the system by changing the local configuration of grains and pores. The peridynamic approach to sintering translates these variations in interfacial energy into long-range forces. These forces are applied in the peridynamic momentum balance equation to simulate the interactions between grains undergoing sintering to simulate microstructural evolution. The peridynamic approach to the modeling of sintering appears to show encouraging results in computational simulations of spherical, deformable, interacting grains with random initial distributions of

radius and position. In this talk, we will describe the peridynamic approach to the modeling of sintering and show computational examples.

**5:00 PM****(SINT-146-2017) Discrete Element Sinter Modeling of Two-Phase Structures**R. Janssen<sup>\*1</sup>; R. Besler<sup>1</sup>; M. Marcel Rossetti da Silva<sup>1</sup>; K. Furlan<sup>1</sup>; C. Ohmstede<sup>2</sup>; S. Heinrich<sup>2</sup>; M. Dosta<sup>2</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. TU Hamburg-Harburg, Inst. of Solids Process Eng. & Particle Technology, Germany

Ceramic structures are often particulate composites, where one phase forms the matrix and the other is heterogeneously or homogeneously distributed in this matrix. Modeling the sintering behavior using discrete elements offers the potential to mimicry these complex structures with a high degree of flexibility and tailorability. In the present paper we concentrate on two different structures (I) an inverse opal structure with large fractions of homogeneously ordered, interconnected pores embedded in a matrix of fine grained alumina with random nano-sized pores, and (II) alumina nickel composites with metal to ceramic ratios from 0 to 100% thereby addressing metal as well as ceramic matrix composites. In both cases, the discrete element method takes the particulate structure into account and allows the differentiation of the developed contacts. It will be shown that the modeling can predict in the first case the local disintegration of the strut/nod networks, and in the later case the contact radius evolution of each phase as well as the sintering retardation. Additional emphasis will given to theoretical constraints of DEM in sintering simulation and the option to transfer the DEM results to a bond model for further stress analysis.

**Novel Sintering Processes III**

Room: Bayview III

Session Chairs: Manshi Ohyanagi, Ryukoku University;  
Johannes Trapp, Technische Universität Dresden**2:00 PM****(SINT-147-2017) From field-assisted sintering to water-assisted sintering: FAST/SPS as a tool to understand and enable ultra-low temperature sintering (Invited)**O. Guillon<sup>\*1</sup>

1. Forschungszentrum Juelich, Germany

In this presentation I will give an overview of the sintering behavior of one model material, namely zinc oxide, under different conditions. Field Assisted Sintering Technique / Spark Plasma Sintering offers the possibility of applying uniaxial mechanical pressure as well as high heating rates. Under low voltage conditions, the electric field effect is however negligible. Higher electric fields are required, with or without direct current flow through the specimen, to affect both densification rate and microstructure. Thorough experiments carried out in a sinter-forging device enable to investigate this behavior, including "flash" sintering which induces a massive heating of the specimen itself. With the presence of water added to the powder, FAST/SPS unveils new, attractive possibilities for the sintering of ZnO (and other hydrophilic oxide materials) at extremely low temperatures (for example 250°C). The defect stoichiometry and structure of sintered zinc oxide was analyzed by several spectroscopic methods, Kelvin probe force microscopy enables the mapping of local changes in defect concentrations and properties of grain boundaries in contrast to grains. The mechanisms for water-assisted densification characterized by a lower activation energy is attributed to hydroxide-ion diffusion mechanism.

2:40 PM

### (SINT-148-2017) Room temperature sintering of alkali halide salts

G. L. Messing<sup>\*1</sup>; E. R. Kupp<sup>1</sup>; J. Anderson<sup>1</sup>; N. Pulati<sup>1</sup>

1. Pennsylvania State University, USA

Since the early studies of Kingery and Berg, metal halides like NaCl have been of interest as model systems for sintering. This paper first reviews how various process and powder characteristics affect coarsening and densification of NaCl. This literature is contrasted to recent studies on water-based, room temperature pressureless sintering of NaCl at room temperature. NaCl and KCl powders are shown to undergo densification and grain growth when exposed to constant relative humidities of <85%; conditions at which very small concentrations of water are adsorbed by salts. Densification is interpreted with a liquid phase sintering model and grain growth by a liquid phase enhanced process. The underlying liquid phase enhanced processes are further extrapolated to interpret pressure-induced 'cold sintering' of metal oxides.

3:00 PM

### (SINT-149-2017) Advances in flame assisted flash sintering

A. Hunt<sup>\*1</sup>

1. nGimat LLC, USA

This presentation addresses recent developments toward maturity of the flame assisted flash sintering (FAFS) technology. The FAFS process invented at nGimat LLC for fast sintering of ceramic films onto electrically conductive substrates follows the same fundamental principles as flash sintering but instead of using a rigid top-electrode relies on a flame medium for transferring the electrical current into a ceramic layer. The superior ceramic-to-metal adhesion resulting from the directional electron flow across the ceramic/metal interface is the key advantage of FAFS. Recent attempts for the application development demonstrate the FAFS capability for ceramic coating of heat exchangers, bioimplants and barrier coatings, just to name a few. The technology is applicable to a variety of materials from single-phase to composite materials. Most recent additions to the FAFS technology have significantly improved reproducibility, facilitated scalability and broadened the potential applications to the ceramic coatings onto metal substrates with complex geometries.

3:40 PM

### (SINT-150-2017) Non-conventional sintering routes of a sodium-ion conducting NASICON material for battery application

J. Pereira da Silva<sup>\*1</sup>; A. Laptev<sup>1</sup>; J. Gonzalez-Julian<sup>1</sup>; Q. Ma<sup>1</sup>; F. Tietz<sup>1</sup>; M. Bram<sup>1</sup>; O. Guillon<sup>1</sup>

1. Forschungszentrum Juelich, Germany

Scandium-substituted NASICON  $\text{Na}_{3.4}\text{Sc}_{0.4}\text{Zr}_{1.6}\text{Si}_2\text{PO}_{12}$  is a promising electrolyte material for sodium-ion solid state batteries, with highest ionic conductivity reported to date for a NASICON material. Low-temperature densification and control of microstructure are important factors to enable the low-cost manufacturing of such new battery type. Non-conventional sintering techniques such as Field Assisted Sintering Technology / Spark Plasma Sintering (SPS) and Cold Sintering are compared to conventional free sintering. FAST/SPS enables to get rapidly dense samples (99% TD) at lower temperatures than the ones required by conventional sintering routes and with similar electrical properties. Cold sintering experiments, involving the addition of aqueous solutions as sintering aids and high mechanical pressure enable a moderate densification, but at temperatures as low as 250°C. Densification behavior, microstructure evolution as well as material properties such as ionic conductivity are investigated and compared in detail.

4:00 PM

### (SINT-151-2017) Fast sintering of advanced oxide ceramics by conventional heating

K. Maca<sup>\*1</sup>; D. Salamon<sup>1</sup>; V. Pouchly<sup>1</sup>; V. Prajzler<sup>1</sup>

1. Brno University of Technology, Czech Republic

Fast sintering (heating rate up to hundreds of °C/min) of tetragonal zirconia and  $\alpha$ -alumina (both with two different particle size) in a specially designed conventional resistance furnace was studied. Isostatically pressed samples reached after fast sintering relative density up to 97.8 % t.d. for zirconia and 99.9 % t.d. for alumina, without forming any cracks in samples. The role of radiation heat transfer and the conditions for forming and elimination of core-shell sample structure were discussed. The sintering activation energy during fast sintering was compared for pressure-less conventional sintering and pressure-assisted Spark Plasma Sintering.

4:20 PM

### (SINT-152-2017) Aerosol deposition: Mechanical sintering of ceramics, one particle at a time

P. Fuierer<sup>\*1</sup>

1. New Mexico Tech, Materials Engineering, USA

Aerosol deposition (AD), or vacuum kinetic spray (VKS), refers to a novel process for the fabrication of ceramic films based on the high velocity impact of fine particles upon a substrate. The process offers exciting possibilities for both rapid prototyping and large scale manufacturing of thick film functional ceramics because of several attributes: a wide range of film thickness (<1 to >100 microns), high deposition rate and room temperature processing. Fundamentally, no ceramic material is precluded from AD, but powder and process parameters must be determined for each experimentally, as the mechanisms for deposition are not fully understood. Because the kinetic energy of individual particles rather than supplied thermal energy provides the energy for consolidation, AD results retains nano-crystallinity while achieving theoretical density. This talk will describe the unique microstructure, texture, and stress state of AD films revealed by SEM, AFM and scanning white light profilometry. Example materials, performance, and potential applications will be described.

4:40 PM

### (SINT-153-2017) Doping effect on flash-sintering behavior in nanocrystalline yttria

H. Yoshida<sup>\*1</sup>; K. Morita<sup>1</sup>; B. Kim<sup>1</sup>; T. Yamamoto<sup>2</sup>

1. National Institute for Materials Science (NIMS), Japan

2. Nagoya University, Japan

Electric field-assisted sintering (FAST) is gaining interest in recent years due to the accelerated consolidation compared to conventional, pressureless sintering. In particular, flash-sintering, where densification occurs almost immediately (typically <5 seconds) under high DC electric field, has attracted extensive attention as an innovative sintering technique.  $\text{Y}_2\text{O}_3$  has special chemical and physical properties such as high resistance to halogen-plasma corrosion and thermal stability, and is therefore known as a promising environment-resistant or optical material. However,  $\text{Y}_2\text{O}_3$  is difficult to sinter. Dense, polycrystalline  $\text{Y}_2\text{O}_3$  ceramics have been developed by pressureless sintering in vacuum or hydrogen atmosphere at high temperature (typically >1600°C), by hot press sintering, and by hot isostatic pressing process. We recently demonstrated that high-purity, undoped  $\text{Y}_2\text{O}_3$  can be fully densified by flash-sintering. For instance, full densification is achieved at 1133°C under a field of 500 V/cm. More recently, it has been shown that 1mol%  $\text{Ni}^{2+}$ -doping can reduce the onset temperature for the flash event in  $\text{Y}_2\text{O}_3$  by about 200°C. In the present paper, the effect of cation-doping on the field-assisted sintering/flash-sintering behavior in  $\text{Y}_2\text{O}_3$  will be systematically investigated.

## In situ Measurements and Analysis of Sintering

Room: Mission I

Session Chairs: Jean-Marc Chaix, Univ. Grenoble Alpes, CNRS, Grenoble INP; Dusan Galusek, Alexander Dubcek University of Trencin

### 2:00 PM

#### (SINT-154-2017) Temperature mapping in SPS using binder evaporation

E. M. Heian<sup>1</sup>; I. Pikus<sup>2</sup>

1. Gentherm Inc., USA
2. Tungsten Parts Wyoming, USA

Difficulties in measuring sample temperature during SPS are well-known. In the present work, we describe a method of inferring the local temperature in a sample during sintering by observation of the vacuum level. During die filling, a small amount of polymer binder is included with the sample powder. The binder can be placed in a specific location within the sample. When the binder decomposes and evaporates during sintering under high vacuum, the pressure in the SPS chamber increases, producing a measurable signal. Results are presented using polyvinylpyrrolidone (PVP: evaporation temperature 500-600°C) and polypropylene carbonate (evaporation temperature 240°C).

### 2:20 PM

#### (SINT-155-2017) Flash sintering of B<sub>4</sub>C: A time resolved energy dispersive x-ray diffraction study with an ultrahigh energy synchrotron probe

I. Savkliyildiz<sup>1</sup>; K. Akdogan<sup>2</sup>; H. Bicer<sup>3</sup>; T. Ozdemir<sup>2</sup>; C. Haines<sup>4</sup>; Z. Zhong<sup>5</sup>; T. Tsakalakos<sup>2</sup>

1. Selcuk University, Metallurgical and Materials Engineering, Turkey
2. Rutgers University, Materials Science and Engineering Department, USA
3. Dumlupinar University, Materials Science and Engineering Department, Turkey
4. United States Army ARDEC, ARDEC, USA
5. Brookhaven National Laboratory, USA

In a highly covalent ceramic such as B<sub>4</sub>C, we found a colossal  $16 \times 10^{-2} \text{ \AA}^3/\text{Ampere}$  hydrostatic expansion in the rhombohedral unit cell when a specimen was furnace heated at 30 °C/min while a 45 V/cm electric field was superimposed on it if Joule heating was prevented. The diffusivity of B<sub>4</sub>C is predicted to increase by ~800%, accounting for sintering to 95% density in as little as <30 seconds in an nonisothermal fashion (688-711 °C) by what we call burst mode densification (BMD) and define as a transient state phenomenon. Our results show that ultrahigh atomic mobility can be imparted to covalent ceramics with low energy density input (<400 W/cm<sup>3</sup>), causing a reduction in sintering time by 1-2 orders of magnitude, and keeping the temperature to <0.3 of the melting temperature. The finding of this study has strong technological implications on the sintering technology of ultrahigh melting temperature and conductive nonoxide ceramics such as carbides, borides, and nitrides.

### 2:40 PM

#### (SINT-156-2017) Abnormal unit cell expansion during flash sintering: A time-resolved in-situ EDXRD study with an ultrahigh energy synchrotron probe

I. Savkliyildiz<sup>1</sup>; K. Akdogan<sup>2</sup>; H. Bicer<sup>3</sup>; T. Ozdemir<sup>2</sup>; Z. Zhong<sup>4</sup>; T. Tsakalakos<sup>2</sup>

1. Selcuk University, Metallurgical and Materials Engineering, Turkey
2. Rutgers University, Materials Science and Engineering, USA
3. Dumlupinar University, Materials Science and Engineering Department, Turkey
4. Brookhaven National Laboratory, USA

Effects of superimposed thermal and electric field on the sintering of 8% yttria doped zirconia (8YSZ) ceramics (200 nm particle size) were studied as a function of applied electric field magnitude and at

20 °C/min heating rate using time-resolved in-situ high temperature energy dispersive x-ray diffractometry with synchrotron probe. Nonisothermal densification was observed in the range 790–930 °C when the applied field magnitude was varied from 320 V/cm to 143 V/cm. Typical sintered densities were found vary from 95-98 % of the theoretical value. No local melting at particle-particle contacts and grain growth were observed in scanning electron micrographs. Typical final grain size was 250 nm. The temperature at which burst-like densification was observed decreased with increasing applied field magnitude. The sudden increase in current at the onset temperature is accompanied by an anomalous elastic volume expansion of unit cell by 1% to 3% that is proportional of the current. a time dependent change in the (211) and (112) peak widths was observed for 790 °C-930 °C. We attribute the observed changes in peak width to temporary oxygen vacancy declustering phenomenon. We propose defect pile-up at particle-particle contacts and subsequent tunneling as a mechanism creating the “burst-mode” discontinuous densification at the singularities.

### 3:00 PM

#### (SINT-157-2017) Understanding the mechanism of flash sintering with in situ EDXRD experiments

S. K. Jha<sup>1</sup>; H. Charalambous<sup>1</sup>; T. Tsakalakos<sup>1</sup>

1. Rutgers University, MSE, USA

Flash sintering has three variables: electric field, current and hold time. The relation of electric field and temperature is inverse; meaning higher is the temperature of furnace, lower is the electrical field needed to flash and vice versa. Once the material goes into the state of flash under electrical field, the current controls the behavior of material (the diffusion kinetics and hence sintering, grain growth and chemical reaction) and electric field finds an equilibrium based on the chance in conductivity. Hold time is the time for which the material was kept under flash. The temperature, estimated by thermal lattice expansion does not match to the temperature needed to explain such a high rate of sintering. With the in-situ Energy Dispersive X ray diffraction technique (EDXRD) we analyze the relation of this excited state and enhanced diffusion with lattice expansion and try to separate the contribution of electric field generated defects and electrical joule heating because of resistance of sample through cyclic flash experiments on Yttria stabilized Zirconia.

### 3:40 PM

#### (SINT-158-2017) Using thermo-optical-measurement technique (TOM) to characterize sintering and melting behavior of ceramic and glass parts under atmospheric control

A. Diegeler<sup>\*1</sup>

1. Fraunhofer ISC, Germany

Thermo-optical measurement devices have been developed at the Fraunhofer ISC during the last 12 years - recently also with atmospheric control (TOM-AC). Thus it is possible to follow shrinkage or warpage of oxide and non-oxide ceramic parts during the whole temperature cycle inside the furnace. However, this method allows to follow changes in the shapes of samples (up to 50mm in diameter) with an accuracy of about 0.3 micron by contact-less measurements. Due to the purely optical detection samples of any shape can be monitored inside the furnace. A sophisticated algorithm detects the contour of samples - and thus dimensional changes. Possible applications include simply measuring the shrinkage or shape distortions of ceramic parts in a contact-less manner as well as measuring the wetting angle of melts, especially on glass. By a computer controlled program we realized full atmospheric control, i.e. changing the gas atmosphere inside the furnace (inert gas at different pressures, forming gas or vacuum) during the sintering process. We present several examples of experiments with ceramic and glass parts of unusual shapes where e.g., nevertheless, shrinkage curves can be

\*Denotes Presenter



obtained and compared to those taken for the same material with a standard dilatometer.

**4:00 PM**

**(SINT-159-2017) The temperature and electrical conductivity related to flash-sintering cubic zirconia**

J. Liu<sup>\*1</sup>; Y. Wang<sup>2</sup>; D. Liu<sup>1</sup>; L. An<sup>3</sup>

1. Southwest Jiaotong University, School of Mechanics and Engineering, China
2. Northwestern Polytechnical University, School of Materials Science and Engineering, China
3. University of Central Florida, Department of Materials Science and Engineering, USA

In this paper, we studied the effect of applied electric-field strength, current density and furnace temperature on the flash-sintered dense 8YSZ ceramics. The results reveal that the flash-sintered samples exhibit clearly different microstructures and properties compared to the conventionally-sintered sample. The nonuniform temperature distribution have been detected on the surface of the specimens during flash sintering. The obvious nonlinear increase in electrical conductivity have been recorded at incubation stage. The results are discussed by considering the nature of the flash sintering and the point defects.

**4:20 PM**

**(SINT-160-2017) Experimental characterization of sintering mechanism using optical dilatometer and push rod dilatometer: A comparative study**

K. K. Telkicherla<sup>\*1</sup>; V. N. Nurni<sup>2</sup>; H. Ahmed<sup>1</sup>; C. Andersson<sup>1</sup>

1. Lulea University of Technology, Civil, Environmental and Natural Resources, Sweden
2. Indian Institute of Technology Bombay, Metallurgical Engineering and Material Science, India

In Iron and Steel industries, sintering process is used in producing agglomerates either as iron ore sinter or as heat hardened iron ore pellets. It is also used extensively used in producing ceramic or metallic powder compacts as well as in additive manufacturing processes. The thermal excursion of the green powder compact determines the extent of sintering and its subsequent evolution of desired properties. Therefore, it is necessary to understand the sintering phenomena for a single green compact. Sintering phenomena of iron ore pellets – magnetite and hematite are investigated in this study using two types of dilatometers. Optical Dilatometer has been used to study in-situ intrinsic sintering kinetics of pellets experimentally at different temperatures, and compared with that of widely used Push Rod Dilatometer. Unlike optical dilatometer, push rod dilatometer uses a load to keep the rod in contact with the compact. Interestingly, it has been found that the intrinsic sintering characteristics changes with the load imposed. These findings can also be extended to other materials manufactured by sintering phenomenon. Understanding on sintering mechanisms of material compacts with and without load will help in evaluating kinetic parameters considering the conditions compact encounter in further applications, and design accordingly.

## Sintering of Electronic Materials

Room: Mission II

Session Chairs: Herbert Danninger, Technische Universität Wien; Monica Campos, Carlos III University of Madrid

**2:00 PM**

**(SINT-161-2017) Flash light sintered pattern on flexible substrate using 1-Octanethiol-coated copper nano-ink via inkjet printing technology (Invited)**

C. S. Lee<sup>\*1</sup>; Y. Son<sup>1</sup>; M. Kang<sup>1</sup>

1. Hanyang University, Republic of Korea

Flexible substrate is widely used for various electronic device such as smart card, disposable electronics. Inkjet printing is suitable to print metal lines on flexible substrate. It uses noble metals such as silver. Nowadays, copper has been studied to replace the noble metal due to its low cost with high electrical conductivity. However, copper is prone to oxidation. In this study, copper nano particles were coated with 1-Octanethiol via dry coating for anti-oxidation to be inkjet printed. Conventionally, inkjet printed pattern has been sintered in the furnace. However, this heat treatment can damage the flexible substrate. Recently, flash light is used to sinter with high-power photons varying duration of pulses at short time. Flash light sintering uses a power in MW scale but short period time in msec per pulse whereas conventional sintering involves certain degree of temperature for several hours. We have observed that light sintering for only short period time can increase necking dramatically within green body with enough diffusion among nano particles due to its photo-thermal effect. Therefore, this method can sinter printed lines more densely, without damaging the substrate. We have varied energy density of the flash light by controlling power, number of pulses and time, to optimize its sintering condition for metal lines on flexible substrate.

**2:40 PM**

**(SINT-162-2017) Enhancement of lead-free NBBT piezoelectric ceramics via employing nanopowders and texturing technique (Invited)**

D. Zhang<sup>\*1</sup>

1. Central South University, State Key Laboratory of Powder Metallurgy, China

Piezoelectric materials are widely used in actuators, sensors and transducers, and mostly utilize lead based compositions which are becoming serious environmental concerns.  $(1-x)\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ - $x\text{BaTiO}_3$  (NBBT) ceramics with a morphotropic phase boundary (MPB) have attracted great interest because of their potential to replace lead based piezoelectric materials. In this talk, grain refinement and texturing techniques will be presented in details to demonstrate their contribution to the improvement of such lead-free system. NBBT nanoparticles were synthesized by hydrothermal method. The piezoelectric coefficient ( $d_{33}$ ) and electromechanical coupling factor ( $k_p$ ) of NBBT ceramics reached 171 pC/N and 0.31, respectively. The grain oriented NBBT ceramics were textured with 10 wt % of NBBT templates and sintered at 1125 °C, 1150 °C, 1175 °C and 1200 °C for 5-50 hrs, respectively. The microstructural evolution, strain behavior and temperature-dependent permittivity of NBBT textured ceramics were systematically investigated. The NBBT ceramic with 89% degrees of grain orientation which was sintered at 1175 °C for 50 h exhibited up to 0.7 % free strain at 7 kV/mm at room temperature where the value of field-induced  $S_{\text{max}}/E_{\text{max}}$  was calculated to reach as high as 1000 pm/V.



3:20 PM

**(SINT-163-2017) Silver sintering: An emerging technology to substitute soldered connections in the electronic industry**A. Steindamm\*<sup>1</sup>

1. SMT Maschinen- und Vertriebs GmbH &amp; Co. KG, Project Management, Germany

In the electronic industry soldering is still the main connection technology of surface-mounted devices (SMD), mostly based on tin-silver-copper based solders. The increase of current densities in high power applications as well as the increasing preconditions for security relevant devices led to the development of nearly void free connections i.e. by using vacuum soldering. But still the rather low melting temperatures of the solders in the range of 200-300°C are a limiting factor in the long term stability of those connections for an increasing number of applications. Especially high power devices for electro mobility and remote renewable energy utilities like offshore wind parks demand for improved durability. An advanced approach for forming very stable connections with much higher melting points without the need to expose the electronic parts to those temperatures during bonding was found in a sintering process using silver particles. Additionally the silver layers exhibit an improved electrical and thermal conductivity. In this talk the principles of silver sintering in the electronic industry as well as its benefits compared to soldering are presented. Also challenges of silver sintering in a high throughput production line and solutions for implementing this technique are highlighted.

4:00 PM

**(SINT-164-2017) The impact of intergranular regions on defect formation and electronic reducibility in nanocrystalline TiO<sub>2</sub> and In<sub>2</sub>O<sub>3</sub>**O. Diwald\*<sup>1</sup>; M. Elser<sup>2</sup>; N. Siedl<sup>2</sup>; D. Thomele<sup>1</sup>; J. Bernardi<sup>3</sup>

1. University of Salzburg, Chemistry and Physics of Materials, Austria  
 2. Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany  
 3. Vienna University of Technology, Austria

Intergranular regions inside nanocrystalline networks of reducible metal oxide nanoparticle based materials play a critical role for oxygen vacancy formation, stoichiometry changes and associated self-doping effects. With a combination of analytical techniques (electron paramagnetic resonance (EPR), FT-IR, UV-Vis-NIR absorption, XRD, and TEM) we addressed ion vacancy formation on TiO<sub>2</sub> and In<sub>2</sub>O<sub>3</sub> particle systems. In addition to optical property changes we identified a strong impact of particle contacts and grain boundaries on annealing induced stoichiometry changes and, thus, on the doping level of the processed nanostructures. Dielectric loss effects observed for nonstoichiometric In<sub>2</sub>O<sub>3-x</sub> nanoparticles inside the cavity of an EPR spectrometer system were used to determine trends in oxygen deficiency and n-type doping level for differently consolidated nanoparticle powders. The observation of a clear correlation between reducibility achieved by vacuum annealing and the amount of intergranular interface area underlines the multiple role of intergranular interfaces. Inside ensembles of semiconducting oxide nanoparticles, they not only provide diffusion paths for charge carriers, they also offer a handle to adjust the n-type doping level via heat treatment in reducing environments.

4:20 PM

**(SINT-165-2017) Additive-engineered reactive sintering for high-performance protonic ceramics with well-designed microstructures**S. Mu<sup>1</sup>; Y. Hong<sup>1</sup>; Z. Zhao<sup>1</sup>; F. Peng<sup>1</sup>; J. Tong\*<sup>1</sup>

1. Clemson University, Materials Science and Engineering, USA

A novel reactive sintering technique based on a small amount of proper additives was discovered for processing protonic ceramics with well-controlled microstructures. It was demonstrated that the fully densified and large-grained protonic ceramic electrolytes and the nanostructured porous protonic ceramic electrodes (e.g. doped

barium zirconates and doped barium cerates) could be successfully fabricated from cost-effective raw materials such as binary metal oxides and carbonates. This additive-engineered reactive sintering (RS) technique has been successfully used to fabricate protonic ceramic fuel cells in a single firing step at a moderate temperature (e.g. 1400°C). The protonic ceramic electrolytes showed greatly improved proton conductivity, which further demonstrated a significant improvement in the power density and stability of protonic ceramic fuel cells. Furthermore, the mechanistic understanding of this additive-engineered RS process was systematically studied, which was used to guide the selection of the additives for desired microstructures of protonic ceramics. This novel technique has the potential to be expanded to process other ionic oxide ceramics with well-designed microstructures.

**Sintering Phenomena in Additive Manufacturing**

Room: Mission III

Session Chairs: Sundar Atre, University of Louisville; Tatsuki Ohji, National Institute of Advanced Industrial Science and Technology (AIST)

2:00 PM

**(SINT-166-2017) Microstructures, properties and applications of laser sintered metal powders (Invited)**S. V. Atre\*<sup>1</sup>

1. University of Louisville, Mechanical Engineering, USA

Laser sintering and melting of metal powders have gained much interest in the additive manufacturing field. However, the differences in processing outcomes relative to conventional sintering technologies have not received as much attention. This paper uses laser sintering studies on metal powders with a wide range of melting temperatures from aluminum alloys to tungsten to compare microstructures and properties obtained from pressureless sintering. The materials addressed in this paper include an Al 6061 alloy, a 90Cu10Sn bronze, 17-4PH and 420 stainless steels, C-103 Nb alloy, and W from our recent studies to map out a comparative analysis of microstructures, properties and application spaces. The purpose of the work is to assess opportunities and gaps in laser v/s pressureless sintering of metals.

2:40 PM

**(SINT-167-2017) Hybrid structures processed by joining dense EBM-prepared lattices and partially sintered powder**O. Liashenko<sup>1</sup>; D. Bouvard\*<sup>1</sup>; R. Dendiev<sup>1</sup>

1. University of Grenoble Alpes, SIMAP, France

New methods are sought for fabricating filters and heat exchangers with mechanical strength and controlled permeability. One of them can be a coupling of different consolidation techniques. It was shown that preserving residual powders around dense EBM printed parts and additional sintering can be used for the production of composite structures with reasonable mechanical properties. Nevertheless, such technique may have some imperfections when printed structures have complex geometries. For example, flaws are found below inclined dense printed element due to the lack of packing of the non-melted powder during printing or sticking of powder at the melt front. During the following sintering treatment these gaps may increase in size, leading to detrimental internal defects. In this work we propose an alternative procedure consisting in manual filling of porous EBM-printed structures with loose powder, followed by sintering. Such approach allows us to achieve a compact packing of powder around the dense printed parts, as it will be demonstrated by optical and SEM microscopy and X-ray tomography and modelling of microstructural evolution using dp3D discrete element code. It is found that due to more effective particle packing in manually filled structures compared to EBM in-process filled structures, sintering results in a defect-poor structure.

**3:00 PM**

**(SINT-168-2017) Distortion and cracking during the densification of 3D printed green bodies**

Z. C. Cordero<sup>1</sup>; R. Carazzone<sup>\*1</sup>; M. Bonar<sup>1</sup>

1. Rice University, Materials Science and NanoEngineering, USA

Several techniques have been developed for 3D printing green bodies with intricate shapes otherwise impossible to achieve through traditional processing routes. It remains a challenge, however, to densify these parts without affecting their form. For example when a 3D printed part is sintered, it can creep under its own weight, or crack as a result of shrinkage stresses generated by external constraints. Here, I will describe our recent efforts to study these distortion and damage processes by imaging 3D printed cantilevered beams and notched plates as they are being sintered. Strategies for minimizing distortion and mitigating sinter-cracking will be discussed in light of our results.

**3:40 PM**

**(SINT-169-2017) Selective laser sintering techniques for improved porous media performance (Invited)**

V. Palumbo<sup>1</sup>; J. Steele<sup>2</sup>; S. Lagocki<sup>1</sup>; A. Mohanram<sup>\*1</sup>

1. Mott Corporation, R&D, USA

Conventional powder Metallurgy techniques utilizing compaction and sintering has been in use for decades to fabricate porous media for filtration and flow control devices. Mott Corporation has developed novel methods of manufacturing utilizing a patent pending technique to fabricate porous metal media utilizing Additive Manufacturing (AM). This new AM technique utilizes selective laser sintering to produce porous media covering a wide range of pore sizes for filtration and flow control devices. These new devices provide a narrower pore size distribution along with equivalent flow at significantly lower pressure drops compared to conventionally processed porous media. Additionally, porous media fabricated via the AM technique does not exhibit the density gradients observed in conventionally pressed parts. Furthermore, the AM technique avoids aspect ratio limitations commonly encountered with certain pressed geometries. We present examples showing improved performance of AM parts compared to conventionally processed media along with several examples of complex shapes that can be fabricated using additive manufacturing that cannot be manufactured using traditional press and sinter methods.

**4:20 PM**

**(SINT-170-2017) Additive manufacturing of advanced ceramics and components (Invited)**

T. Ohji<sup>\*1</sup>

1. National Institute of Advanced Industrial Science and Technology (AIST), Japan

After giving brief overviews of R&D trends on additive manufacturing of metals and ceramics in Japan, this paper is focused on the R&D project "High-Value Added Ceramic Products Manufacturing Technologies", which has been conducted since 2014 under the sponsorship of the Japanese government, with target of "removing restrictions of conventional manufacturing". This project deals with two principal technologies; additive manufacturing of ceramics aimed for complex shaping and reducing lead-time, and hybrid ceramic coating on 3D polymers/metals for enhancing their reliability and functionality. The paper will introduce the latest research achievements in addition to the objectives, research contents, research schemes. Particular focus is placed on the R&Ds on the additive manufacturing, which contains ceramic powder layer manufacturing (powder bed fusion, or indirect selective laser sintering), slurry layer manufacturing (stereolithography) and ceramic laser sintering (direct selective laser sintering) which realizes concurrent forming and sintering. The recent R&D trends

worldwide on additive manufacturing of ceramics also will be introduced. This work was conducted as a part of "High-value added ceramic products manufacturing technologies project" supported by CSTI, SIP, "Innovative design/manufacturing technologies (managed by NEDO)".

**Thursday, November 16, 2017**

**Microstructural Evolution in Sintering Processes IV**

Room: Bayview I

Session Chairs: Jai Sung Lee, Hanyang University; Shmulik Hayun, Ben-Gurion University of the Negev

**8:30 AM**

**(SINT-171-2017) Superior reliability via two-step sintering: Barium titanate based ceramics (Invited)**

X. Wang<sup>\*1</sup>

1. Tsinghua University, Materials Science and Engineering, China

Reliable ceramics with a high electrical or mechanical strength and a narrow strength distribution are commonly associated with fine-grain ceramics of a narrow grain size distribution. Two-step sintering (TSS) in a reducing atmosphere has been employed to obtain fine-grain BaTiO<sub>3</sub> ceramics with a core shell microstructure, a more uniform grain-size distribution, and superior reliability for multilayer ceramic capacitor applications. Compared to ceramics of the same composition conventionally sintered for about the same time, TSS ceramics feature a thinner shell thickness thus a stronger dopant localization, which leads to a lower oxygen vacancy concentration, higher internal resistance and more dopant-oxygen vacancy association. Improved reliability is manifest in a 50% higher breakdown strength at ambient temperature and a 400% longer endurance time to withstand DC stress at 185°C, in addition to a less field- and temperature-dependent capacitance. A scaling analysis of the oxygen vacancy redistribution and endurance dynamics identifies oxygen vacancy transmission across the shell-grain-boundary region as the critical element beneficially impacted by core-shell structure and two-step sintering.

**9:10 AM**

**(SINT-172-2017) Mechanics and evolution of microstructural topology in viscous sintering observed by X-ray microtomography**

G. Okuma<sup>\*1</sup>; S. Tanaka<sup>2</sup>; F. Wakai<sup>1</sup>

1. Tokyo Institute of Technology, School of Material and Chemical Technology, Japan

2. Nagaoka University of Technology, Material Design Engineering Group, Japan

Recent advances in X-ray microtomography revealed that the 3D microstructural evolution during sintering is far more complicated than the simplified model. This limits the applicability of classical models in real situations. The direct measurement of a 3D structure, which is now available from X-ray microtomography, is the first step to understand the realistic microstructure relationship during sintering. In the continuum model of sintering, shrinkage rate is a mechanical response of porous body to the thermodynamic driving force, i.e., the sintering stress, and is inversely proportional to the bulk viscosity. The macroscopic quantities such as sintering stress and bulk viscosity can be determined rigorously for some idealized microstructures in equilibrium. However, for real porous structures which are nonequilibrium, non-periodic, and nonuniform, it is still a challenge to estimate macroscopic quantities from the statistical analysis of microstructural characteristics, such as relative density, specific surface area, surface curvature, and particle size. Here, we

show that both sintering stress and bulk viscosity can be derived as functions of relative density directly from microtomographic images. We also discuss about the quantitative knowledges on topological properties, which provide insights for distinguishing stages of microstructural evolution in sintering.

#### 9:30 AM

##### (SINT-173-2017) Predicting functionally graded microstructure of ZnO sintered in a large induced thermal gradient

C. L. Cramer<sup>\*1</sup>; T. B. Holland<sup>1</sup>

1. Colorado State University, Mechanical Engineering, USA

Advanced sintering techniques have led to advanced analysis of sintering. Specifically, transient spark plasma sintering (SPS) of a specimen under a large induced thermal gradient has caused some constrained effects on the sintering body when trying to process a sample with a graded microstructure. By making an isothermal predictive map in the form of a density-grain size-time (DGT) plot, the predictions are an over estimate of the microstructure compared to the sample sintered under a large thermal gradient. The constraint comes from differential sintering where the hotter side of the specimen experiences higher strain and shields the underlying material from the same stress state. The strains anywhere axial are never equal, and the hot side will experience lower strain than the cold side at a point in time. Advanced sintering analysis has put a value on the constraint using the constituent equations for the visco-elastic assumption of sintering. Using an average value of the strain differences calculated from the DGT during sintering in the equation has led to a constraint at about 16 MPa for the ZnO system analyzed. Applying that much extra mechanical load to a thermal gradient sample allows for the thermal gradient to align with the DGT isothermal predictions for a graded microstructure within 2-4%.

#### 10:10 AM

##### (SINT-174-2017) Evaluation of nanograin growth of TiO<sub>2</sub> fibers fabricated by centrifugal jet spinning

H. Aminirastabi<sup>\*2</sup>; Z. Xiong<sup>2</sup>; G. Ji<sup>1</sup>; Z. Xiong<sup>2</sup>; H. Xue<sup>2</sup>

1. Xiamen University, Aerospace College, China

2. Xiamen University, Materials College, China

Manmade fibers with high surface area and controllable Nanostructures and porosity are one of the most widely investigated morphologies among the nanomaterials. Properties of fibers with Nanostructure made them useful materials that can be used in many possible technological and commercial applications. In this paper, TiO<sub>2</sub> fibers were successfully obtained via sol-gel route with Centrifugal jet spinning, using tetrabutyltitanate as the main raw reagent. One batch of the prepared samples was sintered at different temperatures, from 300°C to 800°C, for 2 hours, and the rests were treated at 700°C for different soaking times (1 to 600 minutes). The phase compositions of the fibers were identified by X-ray diffraction (XRD). The microstructure of the samples were observed by a scanning electron microscope (SEM) and transmission electron microscope (TEM). Experimental results indicated that a well-developed crystallinity and an ideal morphology of TiO<sub>2</sub>, from Nano to micro-scales, could be synthesized above 500°C for 2 hours. The crystallinity and morphology of grains of fibers are sensitive to the reaction temperature and soaking time. However, image analysis indicates that sintering temperature had a great impact on the crystallinity and grain size of the samples above 700°C. Kinetic exponents and fractal dimension of grain growth were also obtained by the aid of an image processing software.

#### 10:30 AM

##### (SINT-175-2017) Manipulating carbon content and sintering schedule to increase the density of pressureless sintered B<sub>4</sub>C

M. J. Dawes<sup>\*1</sup>

1. University of Birmingham, Chemical Engineering, United Kingdom

Boron carbide (B<sub>4</sub>C) is one of the hardest materials but it has proved notoriously difficult to densify through pressureless sintering. This has limited its uptake in certain applications, as currently only hot pressing can achieve one-hundred percent density. Addition of carbon to B<sub>4</sub>C powder compacts in the form of a binder is known to greatly improve densification in pressureless sintering through removal of the oxide layer present on the surface and pinning of the grain boundaries. Currently there are questions over the applicability of this knowledge to a wider range of B<sub>4</sub>C powders, as surface oxidation, free carbon and agglomeration have been shown to have a marked effect on the final properties of the material. Three B<sub>4</sub>C powders with nominally the same surface area and particle size but different sinterability have been selected to demonstrate the influence that sintering schedule can have on pressureless sintered B<sub>4</sub>C. B<sub>4</sub>C powders are doped with different amounts of carbon, compacted isostatically and sintered. The sintered articles are analysed to establish the role of carbon in reducing porosity and investigate manipulating powder formulation to acquire residual carbon in the microstructure. The findings are used to increase the final density of pressureless sintered B<sub>4</sub>C to levels only seen in hot pressed and spark plasma sintered B<sub>4</sub>C.

#### 10:50 AM

##### (SINT-176-2017) Sintering map of barium cerate based protonic conductors

H. E. Araujo<sup>\*2</sup>; K. Ramos<sup>1</sup>; D. Souza<sup>1</sup>

1. Federal University of Sao Carlos, Materials Engineering Department, Brazil

2. Federal Institute of Sao Paulo, Brazil

The development of solid electrolytes for Solid Oxide Fuel Cells is a challenge to enabling this technology as sustainable energy resource. The improvement of properties by optimized processing lies on control of sintering and processing features. Ceramics based on Yttrium doped-Barium cerate are potential candidates for this application due to electrical properties at intermediate temperatures as an alternative to YSZ (Yttria Stabilized Zirconia). Although the studies on proton conductors to solid electrolytes have been carried out since the 1990s, the correlation between ceramic processing and the understanding of sintering mechanism remains unclear. Ceramics based on Yttrium Doped Barium Cerate (BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3-d</sub>) were prepared and analyzed aiming build a sintering profile related to geometrical features of microstructure and ceramic processing. The pellets were processing by several processing ways including different powder routes, milling time, powder and milling balls ratio, heating and cooling rates, dwell time and temperature. The Scanning Electron Microscopy results shown by fractured surface micrographs that the fracture occurs along and trough the grain boundaries depending on processing parameters. High homogeneity of ceramics powders leads to fractures along grain boundaries. Additionally, were observed sintering by liquid and solid state depending on processing route.

#### 11:10 AM

##### (SINT-177-2017) Densification and microstructural development of a new varistor ceramic

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1. Federal University of Rio de Janeiro, Metallurgy and Materials Science, Brazil

Varistors are nonlinear voltage dependent ceramic resistors used to suppress and limit transient voltage surges. The work reported in this paper involves the relationship between densification behavior during sintering and microstructural development which affects



the varistor performance of ZnO-based ceramics doped with rare-earth oxides. Appropriate molar reagent grades were used to prepare a varistor formulation ZM (mol %) 99.00.ZnO + 0.50.CoO + 0.50.MexOy, where MexOy = 25.00.Pr<sub>6</sub>O<sub>11</sub> + 15.00.La<sub>2</sub>O<sub>3</sub> + 15.00.Nd<sub>2</sub>O<sub>3</sub> + 15.00.Sm<sub>2</sub>O<sub>3</sub> + 15.00.Eu<sub>2</sub>O<sub>3</sub> + 15.00.Dy<sub>2</sub>O<sub>3</sub>. The investigations on the densification and sintering processes that occur in this varistor ceramic were made based on the phenomenological approach developed by Su and Johnson [1] and Hansen [2], also used by Choron et al. [3] in a recent work on a similar ceramic system, which makes it possible to group the set of kinetic (time) and thermal (energy) parameters in the global thermal-time function (GTT), which describes the densification behavior during the sintering, allowing identify their stages.

### Modeling and Simulation of Sintering at Multiple Scales III

Room: Mission II

Session Chair: Rasmus Bjørk, Technical University of Denmark

8:30 AM

#### (SINT-178-2017) Sintering mechanisms of functionally graded WC-Co materials (Invited)

I. Konyashin\*<sup>2</sup>; B. Ries<sup>1</sup>; A. Zaitsev<sup>2</sup>; E. Levashov<sup>2</sup>

1. Element Six GmbH, Germany
2. National University of Science and Technology MISiS, Russian Federation

The fabrication of cemented carbides with functionally graded composition, microstructure and properties has been an issue of great importance for a long time. Articles of functionally graded cemented carbides having gradients of hardness and cobalt content are fabricated due to creating Co drifts during liquid-phase sintering by controlling the carbon content and/or WC mean grain size in the surface layer and core region. Up to now mechanisms explaining binder migration phenomena during fabrication of the functionally graded cemented carbides were not understood. Novel mechanisms explaining the binder migration phenomena will be presented. The mechanisms are based on considering capillary forces in parts of carbide articles with various carbon contents and/or different WC mean grain sizes. The variance in capillary forces is found to play a decisive role when sintering carbide samples having different WC mean grain sizes. The wettability of tungsten carbide by liquid Co-based binders with various carbon contents is found to be complete at low carbon contents and becomes incomplete when increasing the carbon content, so that carbide regions with low carbon contents attract the liquid binder from those with high carbon contents. The novel mechanisms explain all the phenomena of Co migration during liquid-phase sintering of all the known functionally graded cemented carbides.

9:10 AM

#### (SINT-179-2017) Advanced complex shapes and productivity improvements in spark plasma sintering

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1. San Diego State University, Engineering, USA
2. CIRIMAT, France
3. CEMES, France

The spark plasma sintering (SPS) technique is presented as one of the most advanced processes for the densification of a wide variety of materials (from metals to ultra high temperature ceramics and composites) with microstructure control. Despite its advantages in terms of advanced material properties, the production of parts by SPS at industrial level is still a significant challenge. The reasons for this challenge are the difficulties to produce homogeneous densified complex shapes and the relatively low productivity of this technique.

To improve these aspects of SPS, an electro-thermal-mechanical-microstructural (ETMM) model has been employed. Different multiple sample SPS approaches able to consolidate 37 and 111 samples simultaneously and addressing the SPS productivity limitations are introduced. In these approaches, the ETMM model is used to predict unusual electric currents path control solutions to homogenize the temperature, densification and grain size. To address the SPS net shape limitations, a new sacrificial material approach able to process complex shapes and resulting in homogeneous densification and microstructure is introduced.

9:30 AM

#### (SINT-180-2017) Sintering processes for dual-phase solid oxide fuel cell electrodes

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1. University of Science and Technology of China, Materials Science and Engineering, China

The solid state sintering is essentially indispensable in preparing porous dual-phase electrodes for solid-state electrochemistry devices, such as solid oxide fuel cells, electrolyzers, and gas separation membranes. The sintering process is not for densification but to tailor the sintering conditions to enlarge the three-phase boundary (TPB) length for electrode reaction, enable feasible pore for gas transportation, achieve high electrocatalyst surface area for adsorption and disassociation, and to establish electron and oxygen-ion conduction pathways. Here we present models simulate the isothermal and pressureless sintering processes and formulates volumetric TPB length and composite porosity as a function of sintering time, surface/interface energies, grain boundary diffusivities, particle sizes, and dual-phase composition. The models describe the sintering processes of various dual-phase electrodes including conventional composites formed through random mixing of the precursor particles and nanostructured electrodes formed through infiltration process. Lanthanum strontium manganite - yttria-stabilized zirconia composite is used as an example to depict the effect of sintering temperature and time on microstructure characteristics such as TPB length, surface area and pore size. Parametric study is performed for the model parameters, illustrating novel insights into the sintering kinetics.

### Sintering of Multi-material and Multi-layer Systems III

Room: Mission II

Session Chair: Rolf Janssen, TU Hamburg-Harburg

10:10 AM

#### (SINT-183-2017) Predicting flexural deformation in multi-layered ceramic systems utilised in investment casting moulds

E. Douse\*<sup>1</sup>; S. Blackburn<sup>1</sup>

1. University of Birmingham, United Kingdom

The flexural deformation of multi-layered ceramics has been studied; it is proposed that the deformation (bending) results from differing thermal expansion and contraction behaviour occurring within each layer upon firing. Evidence for this mechanism is provided by measuring the degree of shrinkage experienced by each layer individually under the firing cycle by linear dilatometer method. Additionally, 3 point bend dilatometer method was undertaken on multi-layered ceramics to assess the extent of the flexural deformation in real time throughout the firing cycle. This was achieved by orientating the multi-layered material in both bottom-side up and bottom-side down orientations within the 3 point bend method and measuring the difference in push rod displacement between the 2 directions. It was then possible to predict this real time flexural deformation observed in the 3 point bend dilatometer method by calculating the expected bending as a result of the difference in



thermal expansion between the layers. The 2 methods of assessing flexural deformation match well, validating that the cause for the deformation is due to differing thermal expansion/contraction of each layer experienced in the firing cycle. It is proposed that this method can be extended for use in other multi-layered ceramic systems in order to prevent flexural deformations.

### 10:30 AM

#### (SINT-184-2017) Sintering of laminated oxide-oxide FRCMC with tailored internal and surface structure

R. Janssen<sup>\*1</sup>; D. Blaese<sup>1</sup>; P. Guglielmi<sup>2</sup>

1. TU Hamburg-Harburg, Inst. of Advanced Ceramics, Germany
2. HzG, Germany

For advanced ceramics composites, precise microstructural tailoring is the most essential prerequisite for successful commercial use. In the past, we developed at TUHH a new processing route for FRCMC based on the lamination of thermoplastic prepregs which allows not only affordable manufacturing but also precise tailoring of the internal structure (both intra-bundle and inter-textile) as well as the surface topography of complex shaped products. In the presentation, the focus is on  $\text{Al}_2\text{O}_3/\text{ZrO}_2$  matrix reinforced by NEXTEL 610 textile, denier 1500, using the porous matrix approach to achieve the desired interface crack deflection. Thereby, composites based on 8 layers infiltrated and impregnated textile exhibit a bending strength of 440 MPa with graceful failure behavior, e.g. a stepwise stress reduction after peak nominal stress. The fracture of these FRCMC is controlled by a series of interfacial delamination events, which enhance energy dissipation during failure. The results are reasoned considering theoretical predictions from the well-known He and Hutchinson model as well as the sintering activities of the constituents of the composites. Lastly, emphasis will be given to the synthesis of dense coatings enabling composite service in harsh abrasive and corrosive environments.

### Novel Sintering Processes IV

Room: Palm Ballroom

Session Chair: Andrii Maximenko, Institute for problems in materials science NAS Ukraine; Thomas Tsakalakos, Rutgers University

### 8:30 AM

#### (SINT-185-2017) Effect of pressure on electric field-assisted pressureless sintering yttria-stabilized zirconia

S. G. Carvalho<sup>\*1</sup>; E. N. Muccillo<sup>1</sup>; R. Muccillo<sup>1</sup>

1. Energy and Nuclear Research Institute, Center of Science and Technology of Materials, Brazil

Electric field-assisted pressureless sintering has been extensively reported in tetragonal and cubic yttria-stabilized zirconia. We report here electric field-assisted sintering in  $\text{ZrO}_2:3 \text{ mol\% Y}_2\text{O}_3$  (3YSZ) green pellets under pressure, either upon heating from room temperature (dynamic) or at a fixed temperature (isothermal). The experiments were carried out positioning uniaxially and isostatically pressed cylindrical specimens in an alumina sample holder with a pressure load up to 1 MPa, inserted in a programmable furnace. Platinum electrodes on both parallel surfaces of the specimen were connected with platinum wires to a power supply. Under the same applied electric field, dynamic experiments in 3YSZ under pressure showed the trigger of the electric current pulse at a temperature 30°C lower than in the pressureless experiment. Impedance spectroscopy measurements on electric field-assisted sintered specimens show that the bulk conductivity remains unchanged while the grain boundary conductivity decreases in the specimens under pressure. Scanning electron microscopy images show that the application of pressure promotes increase the grain size. An explanation based on the pressure induced creation of preferential paths for the electric current pulse through the stressed specimen is proposed.

### 8:50 AM

#### (SINT-186-2017) Flash sintering of samaria-doped ceria/carbon nanotube composites

R. Muccillo<sup>\*1</sup>; A. Ferlauto<sup>2</sup>; E. N. Muccillo<sup>1</sup>

1. IPEN, Brazil
2. Federal University of Minas Gerais, Brazil

Studies of the effect of mixing carbon nanotubes (CNTs) to  $\text{CeO}_2: 20 \text{ mol\% Sm}_2\text{O}_3$  (20SDC) on electric field-assisted (flash) pressureless sintering were performed. Uniaxial and cold-pressed  $5 \text{ mm} \times 5 \text{ mm}$  20SDC - 10 vol.% CNT pellets were submitted during 2 min to AC electric fields of  $200 \text{ V cm}^{-1}$ , 1 kHz, in the temperature range 750-950 °C. The experiments were carried out in ambient atmosphere with the composite pellets positioned inside a vertical dilatometer furnace with Pt electrodes connected either to a power supply for applying the electric field or to an impedance analyzer for collecting  $[-Z''(\omega) \times Z'(\omega)]$  data. The flash sintering results show that 20SDC achieved > 95% relative density with elimination of the CNTs by Joule heating produced by the electric current pulse through the specimen. In another experiment, the thermal evolution of the CNTs was monitored with a mass spectrometer connected to the gas exit of a thermogravimetric/differential thermal analysis equipment. The oxidation of the CNTs may promote an improvement of the microstructure of 20SDC, enhancing the percolation of the charge carriers at the grain boundaries of the polycrystalline samples during flash sintering.

### 9:10 AM

#### (SINT-187-2017) Innovative control systems for Flash-Sintering

A. Prette<sup>\*1</sup>

1. Lucideon, Advanced Materials and Processing, United Kingdom

We present how to achieve microstructural control of various ceramics through development of innovative control systems for the application of electric fields for flash sintering. How to manipulate electric field input using physical hardware and electrical control software to achieve a homogeneous microstructural evolution is explored. The results cover a wide range of industrially-relevant ceramics - from multiphase materials used for tileware and abrasives, to high purity structural and technical ceramics used for medical devices and electronics - where flash sintering leads to some surprising outcomes in comparison to conventional processes in terms of resulting microstructure and mechanical properties. As a case study, we present how flash sintering can increase fracture toughness of alumina and zirconia by ~30%. We also compare how the optimal field input requirements are materials dependent, and how we can use the knowledge generated to date to accelerate future developments. We discuss Lucideon's progression of the technology in terms of understanding how best to control the field input, including through contact and potentially contactless field application. Building an understanding of ceramic behaviour under different flash sintering conditions, alongside solving the engineering challenges presented by scaling up to larger, commercial-scale production, will set the standard for a new paradigm in ceramic manufacture.

### 9:30 AM

#### (SINT-188-2017) Densification and structural transformation during spark plasma sintering of WC-Co-YSZ-cBN systems

P. A. Olubambi<sup>1</sup>; T. G. Langa<sup>\*2</sup>; T. Shabalala<sup>3</sup>; M. R. Mphahlele<sup>1</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa
3. Element Six Pty, South Africa

WC-Co cermets are hard metals that possess a combination of excellent mechanical properties and high fracture toughness. However, WC-Co has specific limits in application as a structural components where high temperature strength, oxidation and corrosion resistance are required. Recent studies have been reported on the

use of zirconia, having good binding strength and adhesion with the matrix, as a novel binding phase for WC. In this study, efforts were made focused on the partial replacement of cobalt content of WC with milled  $ZrO_2$  stabilized with 7.5wt%  $Y_2O_3$  (YSZ) and further reinforcement with cubic boron nitride (cBN). The synthesis focuses on full densification of 4 different compositions (WC-Co (control sample), WC-Co-YSZ, WC-Co-cBN and WC-Co-YSZ-cBN) using spark plasma sintering all at 1300°C and 50MPa with the ultimate goal of developing a WC-YSZ-cBN composite with no or little sintering aid. The effect of isothermal holding time on the densification, mechanical properties and microstructural & phase evolution were studied and will be presented. The results show that full densification, retention of tetragonal-YSZ phase, no cBN to hBN phase transformation and improved hardness & fracture toughness was obtained on the newly developed composite.

**10:10 AM**

**(SINT-189-2017) Microstructural evaluation of spark plasma sintered (SPS) and atmospheric plasma sprayed (APS) CYSZ / bond coat thermal barrier coatings**

F. Kirbiyik<sup>\*1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgy and Material Engineering Dept., Turkey

Thermal barrier coatings (TBCs) have been extensively used on hot-section metallic components of the gas turbine to protect them from harsh effect of the high temperature and oxidation. TBC systems generally have been produced with Atmospheric Plasma Spraying (APS). In this study, CYSZ / NiCo bond coat as a TBC systems were prepared with two different production methods which are Spark Plasma Sintering and Atmospheric Plasma Spraying. Microstructural and surface hardness properties of the TBCs were compared each other in order to evaluate the effect of production method

**10:30 AM**

**(SINT-190-2017) Structural evolution in TiAl-CNT nanocomposites during in-situ alloying and consolidation by spark plasma sintering technique**

P. A. Olubambi<sup>\*1</sup>; E. Nsih-Baafi<sup>2</sup>; A. O. Adegbenjo<sup>2</sup>; M. B. Shongwe<sup>2</sup>; M. M. Ramakokovhu<sup>2</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa

The demand for high performance, lightweight and high strength materials suitable for the harsh and unstable service conditions in aerospace applications remains unsatisfied till date. Hence, as a contribution to bridging this gap, the microstructural and phase evolutions in spark plasma sintered multi-walled carbon nanotube (MWCNT) reinforced TiAl nanocomposites was investigated in this study. 0.5, 1 and 2 wt % MWCNT were dispersed in blended TiAl powders by gentle ball milling and the composite powders were sintered at optimized SPS conditions of 1300 °C sintering temperature, 16 MPa applied pressure and 8 min isothermal holding time respectively. Microstructural and phase evolution studies during in-situ alloying and consolidation of the composite samples were performed by optical, scanning electron microscopy and X-ray diffraction techniques. Phase analysis revealed the formation of carbide phases which were observed to increase with increasing MWCNT weight fraction while the  $\alpha_2$ -Ti<sub>3</sub>Al phase decreased with MWCNT increase in the TiAl matrix. The lamellae phase ( $\gamma$ -TiAl +  $\alpha_2$ -Ti<sub>3</sub>Al) in the duplex microstructure of the consolidated composites also faded away with increase in MWCNT addition. This study revealed that the phase and microstructure of MWCNT/TiAl composites is strongly dependent on MWCNT content.

**10:50 AM**

**(SINT-191-2017) Effect of graphene addition on mechanical and thermal properties of ZrC-TiC composites prepared by spark plasma sintering**

B. C. Ocak<sup>\*1</sup>; O. Yucel<sup>1</sup>; F. Sahin<sup>1</sup>; G. Goller<sup>1</sup>

1. Istanbul Technical University, Metallurgical and Materials Engineering, Turkey

ZrC ceramic is characterized by an excellent combination of properties comprising high melting point, hardness, wear resistance, thermal conductivity, good creep resistance, and excellent chemical stability. However, consolidation difficulties, poor fracture toughness generally restrict the extensive applications of monolithic ZrC. Moreover, recent studies have demonstrated that graphene nanoplatelets (GNP) addition can significantly enhance the mechanical properties of ceramic matrices. In this study, GNPs were incorporated into (20-x)TiC-80ZrC (vol.%) composite powder with x = 0-9 vol.% contents. The resulting powder mixtures were sintered by spark plasma sintering at temperatures of 1700°C for 300 s under a pressure of 40 MPa. Fully dense ZrC-TiC-GNP composites with a relative density of more than 99 % were obtained. The microstructure and mechanical properties of the ceramics have been characterised. It was observed that TiC additions effectively promoted the densification process by forming (Zr,Ti)C solid solution. The sintered composites were then characterized with respect to their densification, microstructure, and mechanical properties change with the combination of TiC and graphene nanoplatelets. Improvement in mechanical and thermal properties with incorporation of GNPs will be discussed in this presentation.

**11:10 AM**

**(SINT-192-2017) Microstructural characterization of spark plasma sintered binderless Ti-based cermets**

P. A. Olubambi<sup>1</sup>; M. B. Shongwe<sup>2</sup>; M. S. Ramatja<sup>2</sup>; M. R. Mphahlele<sup>\*1</sup>

1. University of Johannesburg, South Africa
2. Tshwane University of Technology, South Africa

Sintering of titanium-based cermets has lately received attention and several researchers have noted the challenge in reaching high densities. Thus the purpose of this work was the optimization of the spark plasma sintering conditions to achieve high densification for pure titanium based cermets, namely - pure titanium carbide (TiC), titanium carbonitride (TiCN) and titanium diboride (TiB<sub>2</sub>) with particle sizes of 2-6, 1.0-2.0 and 2.5-3.5  $\mu$ m respectively. Characterization of the received powders was done using SEM and XRD to study the powder morphology. X-ray Diffraction (XRD) was done using a Cu K $\alpha$  radiation. Sintering was done using spark plasma sintering at a temperature between 1850-1900 °C, heating rate of 100 °C/ min at a constant pressure of 50MPa and holding time of 10 minutes. The relative densities of sintered solids were determined by Archimedes method using distilled water as wetting liquid and relative densities obtained for the composites ranged from 97-99%.

## Emerging Topics in Sintering and Micro Development II

Room: Mission I

Session Chairs: Guenter Motz, University of Bayreuth; Dou Zhang, Central South University

**8:30 AM**

### (SINT-193-2017) Preparation of calcium carbonate porous ceramics

S. Umemoto<sup>\*1</sup>; M. Tajika<sup>1</sup>; H. Unuma<sup>3</sup>; S. Shimai<sup>2</sup>

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2. Shanghai Institute of Ceramics, Chinese Academy of Sciences, R&D Center For Structural Ceramics and Composite, China
3. Yamagata University, Graduate School of Science and Engineering, Japan

Calcium carbonate, CaCO<sub>3</sub>, is known as a widely used material for industrial products and also to have high biocompatibility and restorability in vivo. So CaCO<sub>3</sub> porous ceramics is expected to be used in many applications such as for biomaterials, ceramic filters, and catalyses. However, it has generally been recognized that synthetic calcium carbonate powder is difficult to sinter because it decomposes to calcium oxide and carbon dioxide on heating above 900 K in ambient atmosphere. Therefore, a high-pressure of CO<sub>2</sub> or a hydrothermal condition has been used to suppress the decomposition of calcite in sintering. In this study, we have established a fabrication method of CaCO<sub>3</sub> porous ceramics using foaming and gel-casting method followed by pressureless sintering in air. Calcite powder consisted of monodispersed particles with 150 nm in diameter. Methyl cellulose was used as gelation agent. To obtain porous ceramics, calcite powder and gelation agent were dispersed in water by using a polyacrylate-based dispersant to prepare a slurry containing 40 vol% of calcite powder. The slurry was whisked, dried at 353 K for 16 h, and then sintered in air at 783 K for 3h. The obtained CaCO<sub>3</sub> porous ceramics consisted of densely sintered frameworks and aligned macroscopic pores containing microspores in their internal walls and had open cell structure. The apparent porosity was controlled by changing the starting slurry concentration.

**8:50 AM**

### (SINT-194-2017) Effects of alumina incorporation by particle atomic layer deposition on the sintering of Y-TZP and YSZ for biomedical and electrochemical applications

C. Bartel<sup>2</sup>; R. J. O'Toole<sup>\*2</sup>; M. Kodas<sup>2</sup>; A. Drake<sup>2</sup>; A. Horrell<sup>2</sup>; C. Gump<sup>1</sup>; R. Hall<sup>1</sup>; C. Musgrave<sup>2</sup>; A. W. Weimer<sup>2</sup>

1. ALD NanoSolutions, Inc., USA
2. University of Colorado, Chemical and Biological Engineering, USA

The crystal structure of zirconium oxide (ZrO<sub>2</sub>) can be tailored by the incorporation of aliovalent dopants, most commonly, yttrium oxide (Y<sub>2</sub>O<sub>3</sub>). The concentration of dopant dictates the thermodynamically stable crystal phase where 3 mol% Y<sub>2</sub>O<sub>3</sub> (Y-TZP) stabilizes the mechanically strong tetragonal phase and 8 mol% Y<sub>2</sub>O<sub>3</sub> (YSZ) stabilizes the oxygen-conducting cubic phase. Small additions of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) have been shown to decrease the high sintering temperature required to reach near theoretical density while not limiting (and, in some cases, improving) dense part properties which are critical to application (e.g., flexural strength for Y-TZP and ionic conductivity for YSZ). Sintering aid incorporation traditionally involves the mechanical mixing of additive (i.e., Al<sub>2</sub>O<sub>3</sub>) particles with primary ceramic particles (i.e., ZrO<sub>2</sub>). In this work, we report on the conformal deposition of Al<sub>2</sub>O<sub>3</sub> on individual Y-TZP and YSZ particles using the gas-phase deposition technique, particle atomic layer deposition (ALD). The effects of atomically deposited Al<sub>2</sub>O<sub>3</sub> at varying concentrations (i.e., film thicknesses) on the densification of Y-TZP and YSZ are assessed using a dilatometer for constant rate of heating experiments and electron microscopy for characterizing the as-deposited thin films and the microstructure at varying densities.

\*Denotes Presenter

**9:10 AM**

### (SINT-195-2017) Assessment of sintering using pyrometric products

T. A. McInerney<sup>\*1</sup>

1. The Edward Orton Jr. Ceramic Foundation, USA

Measurement of uniformity and accuracy of temperature in producing sintered parts can be done by the use of pyrometric products placed throughout the sintering furnace. Through the use of such products measurement of heatwork, uniformity, repeatability and accuracy of the furnace can be monitored and improved. This study examines the effectiveness of Pyrometric cones and calibrated shrinkage devices when used to monitor a sintering furnace.

**9:30 AM**

### (SINT-196-2017) From MAX phase to MXenes: 2D anodic materials for sodium-ion batteries

S. Marchionna<sup>\*1</sup>; F. Cernuschi<sup>1</sup>; M. Balordi<sup>1</sup>; R. Riccardo<sup>2</sup>; M. Fiore<sup>2</sup>; M. Merlini<sup>3</sup>

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2. Milan-Bicocca University, Materials Science department, Italy
3. University of Milan, Earth science dept., Italy

The development of suitable storage devices based on low-cost technologies and abundant materials is fundamental for a better exploitation of the growing energy production due to the non-programmable renewable sources. In the field of electrochemical storage, the current advantages shown by Lithium-ion batteries (LIB) collide with the limited natural abundance of certain elements involved in this technology (e.g.: Li and Co). The development of Na-Ions batteries (NIB) can overcome this limitation, and offering a potential solution for a low cost storage systems with high performance and sustainable mass-production. Recently, MXenes, a new class of 2D materials obtained by the chemical exfoliation of lamellar ternary carbides/nitrides known as MAX phases, have been proposed as suitable anodic materials for NIB devices. In our work, we have studied all the main steps to obtain optimal Ti<sub>3</sub>C and Ti<sub>3</sub>C<sub>2</sub> MXenes powders suitable for high-performance NIB anodes. Spark Plasma sintering has been used to produce pure MAX phases. MAX phases grinding and chemical exfoliation have been also optimized to identify the critical experimental parameters to maximize the electrochemical performances (e.g. specific capacitance). We will describe the main results achieved for each of the steps investigated, showing the correlation between experimental parameters and electrochemical analysis in prototypal NIB half-cell.

**10:10 AM**

### (SINT-197-2017) Numerical simulations towards microstructural design of solid oxide fuel cell anodes

S. Hara<sup>\*1</sup>; Z. Yan<sup>2</sup>; Z. Jiao<sup>2</sup>; N. Shikazono<sup>2</sup>

1. Chiba Institute of Technology, Mechanical Engineering, Japan
2. The University of Tokyo, Japan

Solid oxide fuel cells (SOFCs) is one of the most promising energy conversion technologies because of its high power generation efficiency. It is well known that the electrochemical performance of SOFCs closely correlates with an anode microstructure fabricated by a sintering process of composite ceramic powders. In this study, a new computational technique that allows us to design efficient and reliable electrode microstructures of SOFCs is presented. By utilizing a discrete element, kinetic Monte Carlo and phase field method, we show that the proposed approach is capable of predicting the microstructural evolutions of composite materials during sintering and reduction which are the major fabrication processes of solid oxide fuel cell anodes. The simulations also reveal the effect of an initial powder shape on an electrochemical performance of anodes.



10:30 AM

### (SINT-198-2017) The effect of heat treatment on remanufactured H13 Steel / Stellite<sup>®</sup> 21 moulds and dies

G. L. Payne<sup>\*1</sup>; C. Cullen<sup>2</sup>; S. Fitzpatrick<sup>2</sup>; T. Konkova<sup>2</sup>; P. Xirouchakis<sup>1</sup>; W. Ion<sup>2</sup>

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2. Advanced Forming Research Centre, Design, Manufacture & Engineering Management, United Kingdom

The use of Additive Manufacturing (AM) to Remanufacture dies, moulds and other high value components is subject of global industrial interest, and as a result it is being met with pragmatic academic research activity. Understanding the role and influence of process parameters in Laser Metal Deposition with Powder (LMD-p) on microstructure morphology, material properties, as well as the distribution of residual stresses in formed parts will expand existing knowledge and will be used to optimise the manufacturing process in order to achieve desired characteristics in a final product. Heat treatment is a well documented process for augmenting the material properties of metals for their intended application, its role within AM is still under consideration. In the presented work, 'mock' H13 tool steel forging dies have been machined to remove a simulated surface defect followed by filling with Stellite<sup>®</sup> 21 using LMD-p and lastly, machining back to its 'original' surface - completing the remanufacturing process. A series of pre and post heat treatments have been performed on four specimens. Following this, residual stress (Contour Method) measurement, microstructural analysis, microhardness testing and mercury intrusion porosimetry (MIP) were sequentially conducted to identify optimum process conditions for remanufacturing the die using LMD-p.

10:50 AM

### (SINT-199-2017) Effects of sintering parameters on spark plasma sintered Ti-Zr alloys

B. L. Bayode<sup>\*1</sup>; L. Lethabane<sup>2</sup>; P. A. Olubambi<sup>1</sup>; L. Sigalas<sup>3</sup>; S. Mxolisi<sup>2</sup>; M. Ramakokovhu<sup>2</sup>

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There is increasing need for improved fabrication routes for novel Ti-alloys. Ti-Zr alloys were consolidated by spark plasma sintering on a hybrid spark plasma sintering furnace. Effects of sintering temperature and formulation ratios were investigated. The temperatures were varied from 1000°C to 1100°C, while the formulation ratios varied between Ti-25Zr to Ti-35Zr (at%) at 1100°C. Microstructural and crystallographic analyses were conducted using scanning electron microscopy, energy dispersive spectrometry, and X-ray diffraction, to study the evolution of microstructures and phases in the bulk alloys. It was noted that increase in sintering temperature improved chemical homogeneity of the sintered products with some noticeable amounts of martensite features at 1100°C. Conversely, decrease in the amount of Zr yielded microstructural and homogeneity improvement. Furthermore, relative density measurements and mechanical properties of the sintered products were tested. The relative density increased monotonically with sintering temperature. But micro-hardness and indentation fracture toughness were different in trend. A 99,2% rel. density was achieved at 1100°C for Ti-30Zr, and 99,4% for Ti-28Zr. Highest micro-hardness value of 717 Hv was obtained in Ti-35Zr. Fracture toughness of 7.871 MPa.m<sup>1/2</sup> was recorded in Ti28Zr. Measured properties of the alloys suggest they may be suitable for biomedical applications.

11:10 AM

### (SINT-200-2017) BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3-d</sub>: A correlation between electrical properties, processing and microstructure

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Solid oxide fuel cells are electrochemical devices that convert chemical energy in electrical energy. The state-of-art of electrolyte used is YSZ, which requires high operating temperatures. Thereby, efforts have focused on decreasing the operating temperature using proton conductors based on Barium Cerate ceramics. Although the studies on proton conductors to solid electrolytes have been carried out since the 1990s, the correlation between ceramic processing and the understanding of sintering mechanism remains unclear. The synthesis of BaCe<sub>0.9</sub>Y<sub>0.1</sub>O<sub>3</sub> (BCY10) requires high sintering temperature compromising the material stoichiometry. Thus, the understanding of sintering step of these material is a challenge. BCY10 was synthesized by solid state reaction and sintered through several conditions. The samples were analyzed by density measurements by Archimedes Principle, X-ray diffraction, scanning electron microscopic and impedance spectroscopy aiming a correlation between geometrical characterization, performed with ImageJ software, of microstructure evolution and electrical properties. The ceramic processing of milling influences the densification and microstructure profile of the material. Samples sintered at 1400°C for 10 hours showed highest electrical conductivity and this results is more related to microstructural aspects than stoichiometry or crystalline features.

### Stress-assisted Sintering II

Room: Mission III

Session Chair: Eduard Hryha, Chalmers University of Technology

8:30 AM

### (SINT-201-2017) Pressure-assisted sintering of Ca<sub>3</sub>Co<sub>4</sub>O<sub>9</sub> multilayers for thermoelectric applications

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The development of competitive thermoelectric generators using oxide ceramics and multilayer technology requires the investigation of suitable sintering procedures. The sintering of Ca<sub>3</sub>Co<sub>4</sub>O<sub>9</sub>, which is the most promising p-type oxide regarding its figure of merit, is a special challenge, as the material starts to decompose at 926 °C. Pressure-assisted sintering (PAS), as known from large-scale production of low temperature co-fired ceramics, was used to sinter multilayers of Ca<sub>3</sub>Co<sub>4</sub>O<sub>9</sub> green tape at 900 °C with different pressures and dwell times. In-situ shrinkage measurements, microstructural investigations and electric measurements were performed. Relative density increases from 57.4 % after free sintering for 24 h to 93.7 % after 2 h of PAS with 10 MPa axial load. The combination of tape casting and PAS induces a pronounced alignment of the anisotropic grains. In comparison to freely sintered multilayers, the electrical conductivity in the casting direction is thereby strongly increased by a factor of 5. With respect to dry-pressed bars, the improvement amounts to a factor of 15. This study shows that PAS is a proper technique to produce dense Ca<sub>3</sub>Co<sub>4</sub>O<sub>9</sub> panels with good thermoelectric properties similar to hot-pressed tablets, even in large-scale production.



**8:50 AM****(SINT-202-2017) Comparison of hot pressing and spark plasma sintering in the densification behavior of indium tin oxide – borosilicate glass composites**T. Rudzik<sup>\*1</sup>; R. A. Gerhardt<sup>1</sup>

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The goal of this study was to fabricate borosilicate glass matrix composites with high optical transmittance and high conductivity by forming percolated segregated networks of ITO in the microstructure. ITO nanoparticles and borosilicate glass microspheres were mechanically mixed with ITO concentrations varying from 0-2.99 vol%. The mixes were then consolidated using either hot pressing (HP) or spark plasma sintering (SPS). The effects of changing sintering methods, along with varying the other processing parameters, were investigated to optimize the percolation threshold and optical properties. Ac impedance spectroscopy (IS), SEM, and EDS indicated the successful formation of a grain-like microstructure of the sintered glass, with the ITO particles segregated to the boundary regions, in all samples. IS results indicated percolation threshold values between 0.154 and 0.307 vol% ITO in the HP samples and between 0.307 and 0.764 vol% ITO in the SPS samples, with resistivities on the order of  $10^1 \Omega \cdot \text{cm}$  at 2.99 vol% ITO. Contrary to conventional belief, it was found that samples made using SPS required far higher temperatures to fully densify, with all other processing conditions being the same, compared with HP. The cause of this behavior is still under investigation.

**9:10 AM****(SINT-203-2017) Influence of the elaboration process on the microstructure and durability of hard coatings obtained from metal powders**J. Chateau-Cornu<sup>\*1</sup>; M. Ardigo-Besnard<sup>1</sup>; Y. Kchaou<sup>1</sup>; L. Minier<sup>1</sup>; F. Bernard<sup>1</sup>

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The shutters globe valves used in the primary coolant circuits of nuclear power plants are submitted to important closing pressures. For this reason, the valve seating surfaces need to be coated with a material having high corrosion, mechanical and galling wear properties. The material chosen and tested in this study is an austenite-ferritic hypereutectoid stainless steel, the Norem 02 (24 % Cr, 4% Ni, 4 % Mn, 3 % Si, 2 % Mo, 1,3 % C). This material intends to replace Stellite cobalt-based alloys, which in the primary coolant circuit produce highly radioactive <sup>60</sup>Co by radiative capture of neutrons. The Norem 02 is usually deposited by PTAW (Plasma Transferred Arc Welding). In our study, we compare its microstructure to the ones obtained by powder compaction by SPS (Spark Plasma Sintering) and HIP (Hot Isostatic Pressing). Diffusion phenomena at the coating/substrate interface are also investigated. The microstructure of the as-deposited PTAW Norem 02 is complex, exhibiting large austenitic dendrites, ranging from about 6 to 20  $\mu\text{m}$ . The interdendritic eutectic phase is composed of  $\text{Cr}_7\text{C}_3$ -type carbides and Mo-rich zones. On the other hand, the microstructures obtained by SPS and HIP present fine grains of austenite and ferrite with homogeneously distributed carbides, leading to better mechanical and durability properties.

**9:30 AM****(SINT-204-2017) Evolution of anisotropic microstructures in hierarchical porous ceramics during sinter-forging**R. Bordia<sup>\*1</sup>; H. Shang<sup>2</sup>; A. Mohanram<sup>3</sup>; E. Olevsky<sup>4</sup>

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2. University of Washington, USA

3. Mott Corp, USA

4. San Diego State University, Dept of Mechanical Engrg, USA

Several technologically important ceramics are porous. One approach to make high porosity ceramics is to use fugitive pore formers followed by partial densification leading to hierarchical porosity: extrinsic large pores from the burnout of the pore former; and intrinsic small inter-particle pores. We present experimental results on the development of pore shape anisotropy during sintering forging of hierarchical porous ceramics. The effect of applied stress on the microstructure was investigated. As expected during sinter forging, the pore shape becomes anisotropic and the pores orient preferentially. However, the intrinsic pores preferentially align parallel while the extrinsic pores align perpendicular to the applied stress. For both intrinsic and extrinsic pores, the degree of anisotropy increases with applied stress, reaches a maximum and then decreases with further increase in stress. Applied stress leads to finer grain microstructures at a particular density. Possible explanations are proposed to explain these observations.

**10:10 AM****(SINT-205-2017) Modeling pore shape evolution during sinter-forging of LSM-YSZ composites**E. Olevsky<sup>\*1</sup>; A. Maximenko<sup>1</sup>; D. Giuntini<sup>1</sup>; R. Bordia<sup>2</sup>

1. San Diego State University, Dept of Mechanical Engrg, USA

2. Clemson University, Materials Science and Engineering, USA

A theoretical framework for understanding the differences between the small and large pore evolution based on the specifics of viscous and diffusional mass transport during the pressure-assisted sintering of hierarchical LSM-YSZ structures using pore formers is developed. A pore size at which the transition from intrinsic to extrinsic topology of mass transport is experimentally obtained and a hypothesis for this behavior, based on the competition between densification and creep deformation, is discussed. Multi-scale finite-element simulations are then implemented in order to monitor the evolution of large pores' shape and size while monitoring the overall densification process. The obtained results are important for understanding and optimization of high temperature processing of the components of high performance electrochemical systems (e.g. electrodes for solid oxide fuel cells – such as based on strontium-doped lanthanum manganite - yttrium-doped zirconia powder composites, gas separation membranes and batteries) which include graded, hierarchical and/or anisotropic porous microstructures.



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## MEETINGS & EXPOSITIONS

# 2018

### JANUARY 17 – 19

2018 CONFERENCE ON ELECTRONIC AND ADVANCED MATERIALS, ORGANIZED BY THE ACERS ELECTRONICS AND BASIC SCIENCE DIVISIONS (FORMERLY ELECTRONIC MATERIALS AND APPLICATIONS)

DoubleTree by Hilton Orlando at Sea World Conference Hotel | Orlando, Fla. USA

### JANUARY 21 – 26

42<sup>ND</sup> INTERNATIONAL CONFERENCE AND EXPO ON ADVANCED CERAMICS AND COMPOSITES (ICACC'18)

Hilton Daytona Beach Resort and Ocean Center | Daytona Beach, Fla. USA

### MARCH 21 – 22

54<sup>TH</sup> ANNUAL ST. LOUIS SECTION/REFRACTORY CERAMICS DIVISION SYMPOSIUM ON REFRACTORIES

Hilton St. Louis Airport Hotel | St. Louis, MO USA

### APRIL 30

CERAMIC BUSINESS LEADERSHIP SUMMIT  
Airport Marriott | Cleveland, Ohio USA

### MAY 1 – 3

4<sup>TH</sup> CERAMICS EXPO  
I-X Center | Cleveland, Ohio USA

### MAY 20 – 24

GLASS AND OPTICAL MATERIALS DIVISION MEETING (GOMD 2018)  
Hilton Palacio del Rio | San Antonio, Texas USA

### JUNE 5 – 8

ACERS STRUCTURAL CLAY PRODUCTS DIVISION & SOUTHWEST SECTION MEETING IN CONJUNCTION WITH THE NATIONAL BRICK RESEARCH CENTER MEETING

Columbia, S.C. USA

### JUNE 11 – 12

9<sup>TH</sup> ADVANCES IN CEMENT-BASED MATERIALS (CEMENTS 2018)

Pennsylvania State University | University Park, Pa. USA

### JUNE 17 – 21

7<sup>TH</sup> INTERNATIONAL CONGRESS ON CERAMICS (ICC7)  
Hotel Recanto Cataratas | Foz do Iguaçu – PR, Brazil

### JULY 22 – 27

12<sup>TH</sup> INTERNATIONAL CONFERENCE ON CERAMIC MATERIALS AND COMPONENTS FOR ENERGY AND ENVIRONMENTAL APPLICATIONS (CMCEE2018)

Singapore

### AUGUST 20 – 23

MATERIALS CHALLENGES IN ALTERNATIVE & RENEWABLE ENERGY (MCARE 2018)

Sheraton Vancouver Wall Centre Hotel | Vancouver, BC Canada

### OCTOBER 14 – 18

MATERIALS SCIENCE & TECHNOLOGY 2018, COMBINED WITH ACERS 120<sup>TH</sup> ANNUAL MEETING (MS&T18)

Greater Columbus Convention Center | Columbus, Ohio USA

### NOVEMBER 5 – 8

79<sup>TH</sup> CONFERENCE ON GLASS PROBLEMS (79<sup>TH</sup> GPC)

Greater Columbus Convention Center | Columbus, Ohio USA



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11 Na 22.98976928 Sodium	12 Mg 24.305 Magnesium	13 Al 26.9815386 Aluminum	14 Si 28.0855 Silicon	15 P 30.973762 Phosphorus	16 S 32.065 Sulfur	17 Cl 35.453 Chlorine	18 Ar 39.948 Argon										
19 K 39.0983 Potassium	20 Ca 40.078 Calcium	21 Sc 44.955912 Scandium	22 Ti 47.867 Titanium	23 V 50.9415 Vanadium	24 Cr 51.9961 Chromium	25 Mn 54.938045 Manganese	26 Fe 55.845 Iron	27 Co 58.933195 Cobalt	28 Ni 58.6934 Nickel	29 Cu 63.546 Copper	30 Zn 65.38 Zinc	31 Ga 69.723 Gallium	32 Ge 72.64 Germanium	33 As 74.9216 Arsenic	34 Se 78.96 Selenium	35 Br 79.904 Bromine	36 Kr 83.798 Krypton
37 Rb 85.4678 Rubidium	38 Sr 87.62 Strontium	39 Y 88.90585 Yttrium	40 Zr 91.224 Zirconium	41 Nb 92.90638 Niobium	42 Mo 95.96 Molybdenum	43 Tc (98.906) Technetium	44 Ru 101.07 Ruthenium	45 Rh 102.9055 Rhodium	46 Pd 106.42 Palladium	47 Ag 107.8682 Silver	48 Cd 112.411 Cadmium	49 In 114.818 Indium	50 Sn 118.71 Tin	51 Sb 121.76 Antimony	52 Te 127.6 Tellurium	53 I 126.90447 Iodine	54 Xe 131.293 Xenon
55 Cs 132.9054 Cesium	56 Ba 137.327 Barium	57 La 138.90547 Lanthanum	58 Ce 140.12 Cerium	59 Pr 140.90765 Praseodymium	60 Nd 144.242 Neodymium	61 Pm (144.9128) Promethium	62 Sm 150.36 Samarium	63 Eu 151.964 Europium	64 Gd 157.25 Gadolinium	65 Tb 158.92535 Terbium	66 Dy 162.5 Dysprosium	67 Ho 164.93032 Holmium	68 Er 167.259 Erbium	69 Tm 168.93421 Thulium	70 Yb 173.054 Ytterbium	71 Lu 174.9668 Lutetium	
87 Fr (223) Francium	88 Ra (226) Radium	89 Ac (227) Actinium	90 Th 232.03806 Thorium	91 Pa 231.03688 Protactinium	92 U 238.02891 Uranium	93 Np (237) Neptunium	94 Pu (244) Plutonium	95 Am (243) Americium	96 Cm (247) Curium	97 Bk (247) Berkelium	98 Cf (251) Californium	99 Es (252) Einsteinium	100 Fm (257) Fermium	101 Md (288) Mendelevium	102 No (259) Nobelium	103 Lr (262) Lawrencium	

58 Ce 140.116 Cerium	59 Pr 140.90765 Praseodymium	60 Nd 144.242 Neodymium	61 Pm (144.9128) Promethium	62 Sm 150.36 Samarium	63 Eu 151.964 Europium	64 Gd 157.25 Gadolinium	65 Tb 158.92535 Terbium	66 Dy 162.5 Dysprosium	67 Ho 164.93032 Holmium	68 Er 167.259 Erbium	69 Tm 168.93421 Thulium	70 Yb 173.054 Ytterbium	71 Lu 174.9668 Lutetium
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