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14. ABSTRACT
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a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU	UU		Rishi Raj
					19b. TELEPHONE NUMBER 303-492-1029

# RPPR Final Report

## as of 08-Jan-2019

Agency Code:

Proposal Number: 68148MSCF

Agreement Number: W911NF-16-1-0079

### INVESTIGATOR(S):

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DUNS Number: 803033104

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**Report Date:** 03-Dec-2016

Date Received: 07-Jun-2018

**Final Report** for Period Beginning 04-Mar-2016 and Ending 03-Sep-2016

**Title:** Electric Field Assisted Sintering and Related Phenomena Far From Equilibrium Conference

**Begin Performance Period:** 04-Mar-2016

**End Performance Period:** 03-Sep-2016

**Report Term:** 0-Other

Submitted By: Rishi Raj

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**Distribution Statement:** 1-Approved for public release; distribution is unlimited.

### STEM Degrees:

### STEM Participants:

**Major Goals:** This overall objective of this conference, the first of its kind, is to discuss how electrical and electromagnetic fields can influence the generation and the movement of defects in ceramics. Mass transport in ionic ceramics is controlled by the diffusion of defects. Electrical conductivity depends on the transport of charged defects as well as holes and electrons. Recent research has shown that mass transport and electrical conductivity can change, together, and abruptly, under a critical combination of applied field and temperature. The most remarkable evidence of this non-linearity is so-called "flash sintering" where ceramics sinter from a powder compact to a dense body in just a few seconds, at furnace temperatures that are hundreds of degrees below conventional sintering. That nearly half the mass can be transported by solid-state diffusion in mere seconds is striking. The phenomenon has been shown to occur in several oxide ceramics, and more recently, in non-oxides as well. The rise in electrical conductivity, which coincides with flash sintering, leads to Joule heating, adding considerable complexity to the fundamental understanding of this phenomenon.

Spark Plasma Sintering (SPS) is another process which accelerates sintering.

Whereas flash sintering is a low power process which employs fields of about 100 Vcm<sup>-1</sup> and currents that are much less than one Ampere, SPS is a high power, low voltage process. In SPS high currents are used to rapidly heat a graphite die. They produce very high heating rates, which combined with applied pressure achieve fast sintering. SPS is often used to sinter materials which are otherwise very difficult to fabricate.

A third process for field assisted processing of ceramics is microwave sintering, which has been shown to induce high rates of sintering while preserving small grain size. It has been shown to apply to several ceramics.

The simplicity of the flash sintering experiments which can be carried out in conventional furnaces, heated to relatively low temperature, with the electrical field being applied directly to the specimens with a pair of electrodes, opens up the possibility of fundamental elucidation of the mechanisms by which electrical fields act to accelerate sintering. The electrical parameters can now be controlled at the millisecond time scale to resolve the underlying atomistic mechanisms. The design of these experiments makes them amenable for real time experiments in the laboratory and at X-ray synchrotrons for in-situ characterization of structural events at the atomic scale. Such experiments hold the potential of revealing a unified understanding of field assisted sintering.

Recent in-situ experiments at APS and Pohang Light Source II are beginning to provide exciting new results related to the flash effect. Diffusional and martensitic transformations that switch on and off with applied fields have been measured. The rates of chemical reactions are unusually accelerated. Sintering has been shown to be accompanied by optical emission which has been identified as electroluminescence with distinct spectral lines

## RPPR Final Report as of 08-Jan-2019

suggesting dynamic electronic energy states that develop and extinguish with the applied field. Silicate glasses have been shown to become extremely fluid and luminesce intensely under electrical fields. Together these observations suggest new science. This scientific journey is in its infancy. The proposed conference will take place at the cusp of the emergence of this new field.

The influence of electrical fields extends beyond sintering into a topic that has come to be known as electro-chemo-mechanics, where electrical and mechanical driving forces act in tandem to influence the outcome of a materials phenomenon. This coupling has come to the fore in the field of Li<sup>+</sup> batteries where the diffusion of Li, driven by electrical fields produces volume expansion in electrode materials. The stresses generated by this expansion and contraction produce failure. The overlap with SPS is evident, where large pressures are applied, deliberately, to promote the movement of atoms. The compressive stress in SPS produces densification. In batteries the stress is generated electrochemically. It can be tensile as well as compressive. The relaxation of these stresses by atom movements can determine whether or not the battery will fail. We seek to include a discussion of this phenomenon at the meeting as well.

**Accomplishments:** Goals were met at the conference through oral presentations, poster presentations and extensive discussion. More than 80 people attended the meeting including leaders in the field.

**Training Opportunities:** Nothing to Report

**Results Dissemination:** Nothing to Report

**Honors and Awards:** Nothing to Report

**Protocol Activity Status:**

**Technology Transfer:** Nothing to Report

### **PARTICIPANTS:**

**Participant Type:** PD/PI

**Participant:** Rishie Raj

**Person Months Worked:** 1.00

Project Contribution:

International Collaboration:

International Travel:

National Academy Member: N

Other Collaborators:

**Funding Support:**

*Program*

**Electric Field Assisted Sintering and Related  
Phenomena Far from Equilibrium**

**March 6 – 11, 2016**

**Hotel dos Templários  
Tomar, Portugal**

**Conference Chair**

**Rishi Raj**

University of Colorado at Boulder, USA

**Conference Co-Chair**

**Thomas Tsakalacos**

Rutgers University, USA



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**Sunday, March 6, 2016**

16:00 – 18:00	Conference Check-in
18:00 – 19:30	Welcome Reception (Lobby Bar)
19:30 – 22:00	Dinner followed by Reception

***NOTES***

- Audiotaping, videotaping and photography of presentations are strictly prohibited.
- Speakers – Please leave at least 5 minutes for questions and discussion.
- Please do not smoke at any conference functions.
- Turn your cellular telephones to vibrate or off during technical sessions.
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**Monday, March 7, 2016**

07:30 – 08:30 Breakfast

08:30 – 08:35 Welcome – Conference Chairs

Introductory Remarks:  
ECI Liaison – Lawrence Kabacoff

**Session 1: Flash, Microwave, SPS and Grain Growth – I**

**Session Chair:** Rishi Raj, University of Colorado at Boulder, USA

08:35 – 09:05 **Electric field–induced electronic conduction in bulk oxide ceramics**  
Anthony R. West, University of Sheffield, United Kingdom

09:05 – 09:35 **Flash microwave sintering of oxide ceramics**  
Kirill I. Rybakov, Institute of Applied Physics, Russian Academy of Sciences;  
Lobachevsky State University of Nizhny Novgorod, Russia

09:35 – 10:05 **Flash sintering of covalent non–oxide ceramics at low temperatures with low DC electric fields: An *in situ* EDXRD study by synchrotron probe**  
Thomas Tsakalakos, Rutgers University, USA

10:05 – 10:25 Coffee Break

10:25 – 10:55 **Field–assisted and flash sintering of nanocrystalline yttria: Densification and microstructural evolution**  
Hidehiro Yoshida, National Institute for Materials Science, Japan

10:55 – 11:25 **Electroluminescence and Heating Rates during Flash**  
Rishi Raj, University of Colorado Boulder, USA

11:25 – 11:55 **Highly transparent spinel windows by microwave sintering**  
Jasbinder Sanghera, Naval Research Laboratory, USA

11:55 – 12:25 Coffee Break

12:25 – 12:55 **The influence of fields and dopants on grain boundary mobility**  
Wayne D. Kaplan, Technion – Israel Institute of Technology, Israel

12:55 – 13:25 **Spark plasma sintering of a functionally graded material consisting of a high–alloyed CrMnNi–steel and varying Mg–PSZ content**  
Sabine Decker, Technical University Bergakademie Freiberg, Germany

13:25 – 14:30 Lunch

**Monday, March 7, 2016 (continued)**

**Session 2: Flash, Microwave, SPS and Grain Growth – II**

**Session Chair:** Thomas Tsakalakos, Rutgers University, USA

- 14:30 – 15:00      **Electrical conduction mechanism at high voltages and dielectric breakdown strength in bulk ceramic insulators**  
Gerold A. Schneider, Hamburg University of Technology, Germany
- 15:00 – 15:30      **Oxygen vacancy formation due to DC electric fields during flash sintering in BaTiO<sub>3</sub>**  
Takahisa Yamamoto, Nagoya University, Japan
- 15:30 – 16:00      **Effect of electric field/current on liquid phase sintering**  
Jesus Gonzalez-Julian, Forschungszentrum Jülich GmbH, Germany
- 16:00 – 16:30      Coffee Break
- 16:30 – 17:00      **Influence of an electric field on grain growth and sintering in strontium titanate**  
Fabian Lemke, Karlsruhe Institute of Technology (KIT), Germany
- 17:00 – 17:30      **First stage in flash sintering of zirconia based ceramics**  
Paul Carry, SIMaP Laboratory, France
- 17:30 – 18:00      **Effect of (external) electric fields on heterogeneous solid state reactions – Special role of grain boundary diffusion**  
Carsten Korte, Forschungszentrum Jülich GmbH, Germany
- 18:00 – 18:30      Coffee Break
- 18:30 – 19:00      **Spark and plasma aided densification mechanisms during spark plasma sintering of ceramic powders**  
Rachman Chaim, Technion–Israel Institute of Technology, Israel
- 19:00 – 19:30      **The modification of the temperature gradient in a large sample sintered by SPS**  
Mirva Eriksson, Stockholm University, Sweden
- 19:30 – 21:00      Dinner
- 21:00 – 22:00      Social Hour

**Tuesday, March 8, 2016**

07:30 – 08:30 Breakfast

***Session 3: Flash – Various (Experimental)***

**Session Chair:** Antti Makinen, Office of Naval Research, USA

08:30 – 09:00 **Field and thermal factors in field-assisted consolidation of powder materials**  
Eugene Olevsky, San Diego State University, USA

09:00 – 09:30 **Direct microwave sintering of gamma-alumina powder: Effect of alpha seeding and magnesia doping**  
Paul Carry, SIMaP Laboratory, France

09:30 – 10:00 **Phase transformations in real time during flash**  
Jean-Marie Lebrun, University of Colorado, USA

10:00 – 10:30 Coffee Break

10:30 – 11:00 **Role of defects in electric-field-assisted sintering**  
Steve Hellberg, Naval Research Laboratory, USA

11:00 – 11:30 **Flash sintering of complex oxides**  
Luis Perez-Maqueda, Materials Institute, Spain

11:30 – 12:00 Coffee Break

12:00 – 12:30 **Electric field-assisted flash sintering of fine-grained and high-permittivity  $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$  electroceramics**  
Lilian M. Jesus, University of São Paulo, Brazil

12:30 – 13:00 **Flash sintering of ZnO, TiO<sub>2</sub> and other oxides: The origin of onset flash and effects of atmosphere, doping and particle size**  
Jian Luo, University of California San Diego, USA

13:00 – 13:30 **Estimating ionic conductivity during flash sintering of 8ysz**  
Daniel Marinha, Saint-Gobain CREE, France

13:30 – 14:30 Lunch

14:30 – 19:00 Afternoon guided excursion to the Templar Castle (Convento de Cristo), a UNESCO World Heritage Site

19:30 – 21:00 Dinner on your own

21:00 – 22:00 Social Hour

**Wednesday, March 9, 2016**

07:30 – 08:30 Breakfast

**Session 4: Flash and SPS – Various**

**Session Chair:** Lawrence Kabacoff, USA

08:30 – 09:00 **Flash sintering of glass containing alumina bodies**

Mattia Biesuz, University of Trento, Italy

09:00 – 09:30 **The influence of doping on flash sintering condition in  $\text{SrTi}_{1-x}\text{Fe}_x\text{O}_{3-\delta}$**

Neta Shomrat, Technion, Israel

09:30 – 10:00 **Electric field effects on grain boundary formation and grain growth**

Klaus van Benthem, University of California Davis, USA

10:00 – 10:30 Coffee Break

10:30 – 11:00 **Flash obviates constrained sintering**

Rishi Raj, University of Colorado Boulder, USA

11:00 – 11:30 **Electrical field effects in spark plasma sintering of hyperstoichiometric  $\text{UO}_2$**

Marco Cologna, European Commission, Joint Research Centre (JRC), Institute for Transuranium Elements (ITU), Germany

11:30 – 12:00 **Low-temperature spark plasma sintering of transparent ceramics by using SiC molding set**

Byung-Nam Kim, National Institute for Materials Science, Japan

12:00 – 12:30 **Electric current as a driving force for interphase growth in spark plasma sintered dielectric composites**

Catherine Elissalde, ICMCB-CNRS, France

12:30 – 13:00 **Discoloration of spark-plasma-sintered transparent  $\text{MgAl}_2\text{O}_4$  spinel**

Koji Morita, National Institute for Materials Science, Japan

13:00 – 14:30 Lunch

**Session 5: SPS – Various**

**Session Chair:** Claude Estournès, Université de Toulouse; UPS, INP; Institut Carnot Cirimat, France

14:30 – 15:00 **Engineering of lightweight ceramic composites by spark plasma sintering**

Oleg Vasylykiv, National Institute for Materials Science, Japan

**Wednesday, March 9, 2016 (continued)**

- 15:00 – 15:30      **Development of electric current activated/assisted sintering (ECAS/SPS)**  
Salvatore Grasso for Yoshio Sakka, National Institute for Materials Science,  
Japan
- 15:30 – 16:00      **Benefits of SPS sintering on microstructure and piezoelectric properties of  
KNN-based ceramics**  
Florian Jean, GREMAN, France
- 16:00 – 16:30      Coffee Break
- 16:30 – 17:00      **Generating MgAl<sub>2</sub>O<sub>4</sub> whiskers using carbothermic reactions and SPS/FAST**  
Anton Salomon, TU Bergakademie Freiberg, Germany
- 17:00 – 17:30      **The mechanisms of field assisted sintering in metallic systems**  
Johannes Trapp, TU Dresden IfWW, Germany
- 17:30 – 18:00      **Microscopic densification mechanisms of metallic systems by spark  
plasma sintering**  
Jean-Philippe Monchoux, Centre d'élaboration de matériaux et d'études  
structurales, France
- 18:00 – 18:30      Coffee Break
- 18:30 – 19:00      **Exploring length scale–property relationships in dense nanocrystalline  
materials**  
Javier Garay, University of California, San Diego, USA
- 19:00 – 19:30      **SPS for chemical preparations**  
Ihor Veremchuk, Max Planck Institute for Chemical Physics of Solids, Germany
- 19:30 – 20:00      **Ceramics of potassium sodium niobate by field–assisted sintering  
techniques: Spark plasma sintering versus spark plasma texturing**  
Paula Maria Vilarinho, University of Aveiro, Portugal
- 20:00 – 21:00      Dinner
- 21:00 – 22:30      Poster Session / Social Hour

**Thursday, March 10, 2016**

07:30 – 08:30 Breakfast

**Session 6: Techniques, Methods and Simulations–I**

**Session Chair:** Michael Bakas, Army Research Office, USA

08:30 – 09:00 **Microstructure evolution in spark plasma sintered hafnium–tantalum carbides**

Ben Boesl, Florida International University, USA

09:00 – 09:30 **Consolidation behavior of bulk amorphous glasses and foils: The effect of current on devitrification kinetics**

Olivia A. Graeve, University of California, San Diego, USA

09:30 – 10:00 **Processing high strength low oxide and metal impurity ZrB<sub>2</sub> ceramics using boron carbide and spark plasma sintering**

Erica L. Corral, University of Arizona, USA

10:00 – 10:30 Coffee Break

10:30 – 11:00 **Hybrid heated FAST/SPS with additional voltage support**

Jan Raethel, Fraunhofer IKTS, Dresden, Germany

11:00 – 11:30 **A dynamic bifurcation criterion for thermal runaway during the flash sintering of ceramics**

Joao Gustavo Pereira da Silva, TUHH, Germany

11:30 – 12:00 Coffee Break

12:00 – 12:30 **Hybrid heated FAST/SPS with additional voltage support**

Jan Raethel, Fraunhofer IKTS, Dresden, Germany

12:30 – 14:00 Lunch

**Session 7: Techniques, Methods and Simulations–II**

**Session Chair:** Olivier Guillon, Forschungszentrum Jülich GmbH, Germany

14:00 – 14:30 **Field effects during consolidation of metallic powders**

Brandon McWilliams, US Army Research Laboratory, USA

14:30 – 15:00 **Microwave and flash processing of functional materials: Are there (m)any similarities?**

Bala Vaidhyanathan, Loughborough University, United Kingdom

15:00 – 15:30 **Flash sintering: New opportunities**

Salvatore Grasso, Queen Mary University of London (QMUL), United Kingdom

**Thursday, March 10, 2016 (continued)**

- 15:30 – 16:00      **Ceramics sintering and shaping using the electrical field assisted sintering method**  
Amiya Mukherjee, University of California, USA
- 16:00 – 16:30      Coffee Break
- 16:30 – 17:00      **Spark plasma sintering: Comparison between a fully coupled process numerical simulation and experimental data**  
David Martins, CEMEF / CIRIMAT / SAFRAN, France
- 17:00 – 17:30      **Growing larger: Scaling up during spark plasma sintering of high-temperature ceramics**  
Oleg Vasykiv, National Institute for Materials Science, Japan
- 17:30 – 18:00      **Energy coupled to matter for field-assisted processing**  
Raymond Brennan, U.S. Army Research Laboratory, USA
- 18:00 – 18:30      Coffee Break
- 18:30 – 19:00      **Energy coupled to matter for field-assisted processing**  
Raymond Brennan, U.S. Army Research Laboratory, USA
- 19:00 – 19:30      **Field assisted sintering: Challenges in scale-up from buttons to body armor**  
Christopher D. Haines, US Army ARDEC, USA
- 19:30 – 21:30      Banquet followed by Social Hour

**Friday, March 11, 2016**

07:30 – 08:30 Breakfast

**Session 8: Innovative Materials**

**Session Chair:** Rolf Janssen, TUHH, Germany

08:30 – 09:00 **Modelling and FEM simulation of electric field assisted sintering of tungsten carbide (WC)**

Sree Koundinya Sistla, RWTH–Aachen University, Germany

09:00 – 09:30 **Spark plasma sintering: From the thermal gradient to advanced ceramic composites**

Dmytro Demirskyi, Nanyang Technological University, Singapore

09:30 – 10:00 **Densification of dense nano crystalline zinc oxide under electric field**

Olivier Guillon, Forschungszentrum Jülich GmbH, Germany

10:00 – 10:30 Coffee Break

10:30 – 11:00 **Features of grain growth and grain boundary formation under microwave and spark plasma sintering conditions**

Andrey Ragulya, Institute for Problems of Materials Science (IPMS), Ukraine

11:00 – 11:30 **Fabrication of a vitreous porcelain by conventional sintering and spark plasma sintering**

Wirat Lerdprom, Imperial College London, United Kingdom

11:30 – 12:00 **Application of microwave energy to consolidate titanium powder**

Christopher Haines, US Army ARDEC, USA

12:00 – 12:30 **Modification of the interdiffusion process in the Fe–Al system by SPS and field assisted sintering**

Hanka Becker, Technical University Bergakademie Freiberg, Germany

12:30 – 13:00 **Application of steel as an alternative tool material for field assisted sintering in SPA**

Alexander Laptev, Forschungszentrum Julich GmbH, Germany

13:00 – 13:30 **Abnormal grain growth in pressure assisted sintering of BaLa<sub>4</sub>Ti<sub>4</sub>O<sub>15</sub>**

Ana Senos, University of Aveiro/ CICECO, Portugal

13:30 Lunch and Departure

## **Poster Session**

**Session Chair:** Rolf Janssen, TUHH, Germany

1. **Flash sintering of SrTiO<sub>3</sub>**  
Fabian Lemke, KIT, Germany
2. **Flash sintering of TCP bioceramics: Effect of particle size and influence on  $\beta \rightarrow \alpha$  transition**  
Matteo Frasnelli, DII - University of Trento, Italy
3. **FAST sintering of alumina, spinel and yttria-stabilized zirconia three-phase composites**  
David Kok, University of California, Irvine, USA
- 4.
5. **Fast one-step synthesis and sintering of materials promoted by electric fields**  
Lilian M. Jesus, University of São Paulo, Brazil
- 6.
- 7.
8. **Observations on Flash Sintering of Uranium Dioxide**  
Kenneth J. McClellan, Los Alamos National Laboratory, USA
9. **Microstructure and mechanical properties of Spark Plasma Sintered tungsten-copper-zinc composites**  
Thabiso Langa, Tshwane University of Technology, South Africa
10. **Self-organized structure in current-activated pressure-assisted densification (CAPAD)**  
Sebastian Angst, University of Duisburg-Essen, German
- 11.
- 12.
- 13.
- 14.
15. **Growth behavior of faceted Na<sub>1/2</sub>Bi<sub>1/2</sub>TiO<sub>3</sub>-BaTiO<sub>3</sub> grains in single and two-step sintering in support for the microstructural evolution principle**  
Seok-Young Ko, Korea Advanced Institute of Science and Technology (KAIST), South Korea

## Engineering Conferences International

### Participants List

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Tomar, Portugal

3/6/2016 through 3/11/2016

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## Engineering Conferences International

### Participants List

#### Electric Field Assisted Sintering

Tomar, Portugal

3/6/2016 through 3/11/2016

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### Participants List

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Tomar, Portugal

3/6/2016 through 3/11/2016

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3/6/2016 through 3/11/2016

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