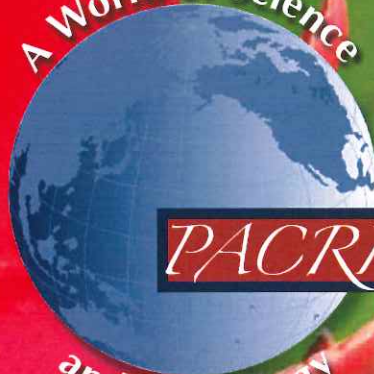


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PACRIM Symposium 06: Synthesis and Processing of Materials Using Electric Currents and Pressures

Electric Currents I

Room: King's I

Session Chairs: Takashi Goto, IMR Tohoku University; Javier Garay, University of California, San Diego

1:15 PM

(PACRIM-S6-001-2017) Thermal Runaway in Flash Spark Plasma and Microwave Sintering (Invited)

E. A. Olevsky^{*1}; C. Maniere¹

1. San Diego State University, USA

An ultra-rapid process of flash hot pressing (or ultra-rapid spark plasma sintering) is developed based on the conducted theoretical analysis of the role of thermal runaway phenomena for material processing by flash sintering. The present study experimentally addresses the challenge of uncontrollable thermal conditions by the stabilization of the flash sintering process through the application of the external pressure. The effectiveness of the developed flash spark plasma sintering technique is demonstrated by the few seconds-long consolidation of SiC powder in an industrial spark plasma sintering device. Similarly to flash spark plasma sintering, the experimentally known thermal instability of microwave sintering is theoretically explained. It is shown that the sample location has a great impact on the temperature distribution and decreasing the sample size promotes temperature homogenization thereby assisting the overall sintering stabilization.

1:45 PM

(PACRIM-S6-002-2017) Flash sintering of alumina: Evidences of oxide partial reduction

M. Biesuz^{*1}

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

Flash Sintering (FS) represents an innovative sintering technology for ceramics that allows a consistent reduction of consolidation time and temperature. Although the operative conditions for flash sintering and its applicability to several ceramics have been extensively studied, the mechanisms behind the process have not been clarified yet. In previous works a model for the thermal runaway, which provides a reasonably good description of the onset condition for FS has been proposed. Nevertheless, the reasons behind the enhanced electrical conduction and the rapid densification have not been completely explained. In the present work microstructural features and evolutions of flash sintered alumina have been studied in detail by using complementary experimental techniques. The results show anomalous grain growth phenomena and partial reduction of the oxide. This latter phenomenon could be responsible for the electronic conduction increase during the flash event and could accelerate the densification/grain coarsening phenomena as a result of a faster mass transport mechanism.

2:05 PM

(PACRIM-S6-003-2017) Direct Joule heated (Flash) Sintering of Ionic Conductive Ceramics in a Conventional Spark Plasma Sintering Furnace Using Standard Graphite Tooling

L. S. Walker^{*1}

1. Thermal Technology, USA

Direct joule heating of ionic conductive ceramics, also known as 'flash' sintering, has attracted great interest of recent due to the extremely high sintering rates achieved through the rapid heating process. The basic approach involves heating a powder compact of an ionic conductive ceramic such as zirconia in a furnace to a

temperature high enough for it to conduct electrical current and rapid joule heating to high temperature using a current controlled power supply to achieve rapid sintering. The apparatus for such an experiment is generally complex and control over the experiment is also generally limited to current control of the power supply. Presented here is a simplistic approach to achieve the 'flash' sintering effect in a conventional spark plasma sintering (SPS) furnace using either current control or temperature control. The method requires no pre-sintering or compaction of the powder and uses only conventional tooling typical to the SPS process without any sacrificial heaters. The setup and operation of the technique will be presented in addition to the scalability potential to larger samples.

2:25 PM

(PACRIM-S6-004-2017) Energy Coupled to Matter for Electric Field-Enhanced Sintering (Invited)

R. E. Brennan^{*1}; B. McWilliams¹; V. L. Blair¹; J. Yu¹; M. Kornecki¹; F. Kellogg¹; S. V. Raju¹

1. US Army Research Laboratory, USA

Energy Coupled to Matter (ECM) research is focused on the study of field interactions with materials to produce outcomes that are unattainable through conventional means, expanding materials-by-design and processing/manufacturing science capabilities. Technological advancements in ECM can have a significant impact on Army efforts for developing novel materials with tailored microstructures to produce unprecedented properties, and enhancing processing and manufacturing capabilities for rapid production of unique components. For electric field-enhanced sintering, a number of methods are currently being explored, including electric field-assisted sintering, flash sintering, and single-mode microwave sintering. All of these methods use electric fields for field-enhanced heat treatment of materials, significantly reducing temperature and time requirements for enabling full densification. The ability to rapidly densify materials under less extreme processing conditions exemplifies a major advantages of ECM, as the smaller final grain size can be uniquely preserved, resulting in enhancement of physical and mechanical properties that are relevant to Army applications (i.e. strength, hardness, fracture toughness, etc.). Breakthrough results for fabricating ceramics, metals, and hybrid materials using the aforementioned techniques will be presented to demonstrate the implications of these exciting capabilities.

2:55 PM

(PACRIM-S6-005-2017) Spark plasma sintering: From Finite Element Modeling of the process up to the elaboration of complex shapes (Invited)

C. Maniere¹; L. Durand²; E. Brisson³; H. Desplats³; P. Carre³; P. Rogeon³; C. Estournes^{*1}

1. CIRIMAT, LCMIE, France

2. CEMES, France

3. LIMATB, France

Pulsed Electric Current Sintering (PECS) techniques have known a huge development over the last two decades. In particular, Spark Plasma Sintering (SPS) is an extremely powerful technique to sinter all classes of powders (metallic, ceramic) as well as composites. Recently, the modeling of Spark Plasma Sintering by finite element method has known drastic development. Coupling three main physics, Electric Thermal and Mechanic (ETM), it allows now to predict the evolutions of temperature, grain size and porosity during the process. The electrical and thermal parts of the ETM model are used to calibrate the contact (thermal and electrical) resistances and to calculate the temperature at any point of the SPS tool and column. Ex-situ measurements of contact resistances were also performed in different conditions to compare the results of the calibrations. Creep parameters are identified on dense and porous materials, and sintering models (Olevsky and Abouaf) are used to predict the densification of the powders to be sintered. Last, a grain growth

law coupled with the densification model may also be considered. Finally this type of modeling enables the definition of the optimized SPS parameters and tool geometry in order to minimize the porosity and microstructure gradients in a complex shape part.

3:40 PM

(PACRIM-S6-006-2017) Flash sintering of TCP bioceramics

M. Frasnelli^{*1}; V. M. Sglavo¹

1. University of Trento, Industrial Engineering, Italy

In this work, sintering behavior of tricalcium phosphate (TCP) ceramics under the effect of an external electrical field (in flash sintering configuration) was analyzed to obtain dense bio-resorbable components. The aim was to understand the physical condition leading to the flash phenomenon and to study the effect of consequent reduction in sintering time and temperature on the undesired $\beta \rightarrow \alpha$ -TCP phase transition occurring at high temperature. TCP powders were synthesized by conventional solid state reaction and then shaped into cylindrical green body by uniaxial cold-pressing. Their sintering behavior was studied by dilatometry under different E-field at constant rate heating. The presence of α -TCP and the microstructure were investigated by XRD and SEM techniques. It is shown that a flash phenomenon takes place at furnace temperature below 1000°C. In addition, although $\beta \rightarrow \alpha$ transition occurs at ~1150°C for pure TCP, the detection of both polymorphs within the sintered bodies indicates that higher temperature is reached in the material, which is very likely associated to Joule effect induced by the current flow along the sample.

4:00 PM

(PACRIM-S6-007-2017) Magnetic Field Processing and Sintering of Rare-Earth Doped Aluminum Oxide

V. L. Blair^{*3}; N. Ku¹; C. A. Moorehead²; J. Elward¹; B. C. Rinderspacher³; R. E. Brennan³

1. ORISE, USA

2. Drexel University, USA

3. US Army Research Laboratory, Weapons and Materials Research Directorate, USA

Energy Coupled to Matter (ECM) is an emerging technology area that goes beyond the traditional limits of materials research by exploring the use of applied physics-based fields and their influence over materials structure, properties, and response. The objective of the current research effort is to characterize and exploit effects of various energetic fields (magnetic, electric, microwave, etc.) on microstructural development of materials in order to formulate a physical explanation of the fundamental mechanisms of material interactions during processing. This presentation will focus on unexpected results observed during high magnetic field processing of diamagnetic ceramic materials. Over the past three years, ARL has successfully doped rare earth cations into nanosized alumina powders and aligned the doped powders under a magnetic field in a ceramic/polymer composite. The result was the unexpected alignment of the alumina particles to the (006) plane, which can lead to future crystallographic texture. Additionally, ARL has shown that crucial phase transformation temperatures in aluminum oxide synthesis can be affected by an applied magnetic field during the calcination process, which has implications in future densification studies. Current and future work will also be described; detailing new synthesis procedures under exploration and densification of ceramics under applied fields.

4:20 PM

(PACRIM-S6-008-2017) Industrial Applications of Direct Current Based Spark Plasma/Field Assisted Sintering; Large Components and Uniformity

L. S. Walker^{*1}

1. Thermal Technology, USA

Direct current sintering also known as field assisted and spark plasma sintering has achieved great success in academia and industrial based research with its ability to rapidly produce high quality material systems. Industrial acceptance, however, has been slow to evolve due to limited access to large industrial scale systems for proof of concept work and experienced operators. Here we present the production of large parts (>10cm) for industrial applications while achieving high part uniformity and part-to-part consistency. In-situ temperature measurements within the tooling are used to measure the process uniformity at multiple sites. Microstructure and material properties will be investigated to show the results of sintering larger samples using different material systems, and the process uniformity achieved.

4:40 PM

(PACRIM-S6-009-2017) A Finite Element Based Model to Validate Temperature Distribution Measurements in Electrical Insulator and Electrical Conductor Ceramics Using SPS

E. L. Corral^{*1}

1. The University of Arizona, Materials Science and Engineering Department, USA

Spark plasma sintering (SPS) is capable of precisely controlling material microstructures and achieving non-equilibrium phases due to rapid heating and cooling rates through the simultaneous application of pressure and direct current. Due to these characteristics, SPS is an ideal for processing high temperature ceramics at temperatures greater than 1500°C. However, desired microstructure must be maintained throughout the geometry of the part thus, we developed a finite element analysis model to ensure processing conditions result in desired microstructures. Our model is a coupled thermal-electrical finite element analysis model to investigate the effect of tooling, contact resistance, and current density on the resultant processing conditions for an electrical conductor, zirconium diboride, and an electrical insulator, silicon nitride. The model is experimentally verified and material properties are optimized in order to calibrate the model.

PACRIM Symposium 13: Advanced Structural Ceramics for Extreme Environments

Structural Stability in Extreme Environments

Room: Kohala 4

Session Chairs: Raj Singh, Oklahoma State University; Dechang Jia, Harbin Institute of Technology

1:15 PM

(PACRIM-S13-012-2017) Cyclic fatigue durability of EBC coated 3D SiC/SiC composites under thermal gradient conditions at 2700°F in air

C. Smith^{*2}; B. J. Harder²; D. Zhu²; R. Bhatt¹; S. Kalluri¹

1. Ohio Aerospace Institute, USA

2. NASA Glenn Research Center, USA

Ceramic matrix composites (CMCs) such as SiC/SiC are currently being designed and implemented in high temperature sections of aerospace turbine engines. Such components will be subject to through-thickness thermal gradients, which may affect the durability. In this study, SiC/SiC CMCs with a hybrid chemical vapor

*Denotes Presenter

Applications of PDCs II

Room: King's 3

Session Chair: Samuel Bernard, CNRS

10:15 AM**(PACRIM-S4-030-2017) Highly porous silicate bioceramics from preceramic polymers and reactive fillers (Invited)**E. Bernardo^{*1}; H. Elsayed¹; L. Fiocco¹

1. University of Padova, Dept. of Industrial Engineering, Italy

Highly porous foams, based on akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$) and wollastonite (CaSiO_3)-diopside ($\text{CaMgSi}_2\text{O}_6$) ceramics, may be produced with commercial silicones mixed with CaCO_3 and $\text{Mg}(\text{OH})_2$ micro-sized particles. An extensive foaming is achieved simply by water release, at 300–350 °C, from decomposition of sodium borate or phosphate hydrated salts, used as secondary fillers and forming liquid phase (favouring ionic interdiffusion) upon firing at 1100°C. Operating with anhydrous salts, silicone-based pastes can be applied also for direct ink writing, leading to 3D reticulated structures, later ceramized as above. Optimized formulations lead to materials with remarkable compressive strength (well exceeding 5 MPa, with a total porosity in the order of 70%) and bioactivity, confirmed by in vivo cell tests. The overall approach may be extended even to Ca-Zn bioactive silicates. In this case, foaming and phase evolution are associated with the use of calcium borate, leading to novel B-doped hardystonite ceramics ($\text{Ca}_2\text{Zn}_{1-x}\text{B}_x\text{Si}_2\text{O}_7$).

10:45 AM**(PACRIM-S4-031-2017) Polymer-Derived Ceramic Membranes and Sensors (Invited)**A. Gurlo^{*1}

1. Technische Universität Berlin, Chair of Advanced Ceramic Materials, Germany

Tunable synthesis of nanocomposites possessing desired functionalities has received considerable attention in the last decades. In the field of the hydrogen economy such materials are applied as catalysts for hydrogen production, hydrogen storage materials, hydrogen separation membranes, and hydrogen sensors. In the present work we report a precursor-based approach towards composite materials composed of polymeric matrix with incorporated nanoscaled metallic, oxide and nitride particles. After thermal treatment under desired conditions (temperature, ambient gas) such polymeric nanocomposites transform into ceramic nanocomposites. Their porosity is tuned by an appropriate choice of thermolysis and annealing conditions. Case studies include the synthesis and characterization of (i) microporous ceramic membranes and (ii) catalytically active nanocomposites.

11:15 AM**(PACRIM-S4-032-2017) Boron-modified Silicon Oxycarbide Composite Electrode for Electrochemical Energy Storage (Invited)**M. Abass¹; G. Singh^{*1}

1. Kansas State University, Mechanical and Nuclear, USA

Heteroatom modification of polymer-derived ceramics is one of the sustainable means of improving their electrochemical energy storage properties as electrode materials. In electrochemical energy storage application, boron is believed to improve the electronic conductivity and chemical stability of SiOC by modifying its nano-domain structure. Herein we report synthesis of freestanding films of boron nitride nanotube (BNNT)-modified silicon oxycarbides (SiOC) ceramic. The composite films were synthesized via vacuum filtration technique with reduced graphene oxide (rGO) as conducting agent. The layer-by-layer configuration ensured uniform dispersion of the ceramic on rGO sheets. Thin films containing varying loading of BNNTs were tested as sodium-ion battery (Na-IB) and supercapacitor electrodes. SiOC containing 0.5 wt % BNNTs (SiOCB-0.5wt.%)

displayed optimum electrochemical properties. As observed in this study, superior capacity of SiOCB-0.5 wt % as an electrode material for Na-IB and supercapacitor suggests the existence of an optimum doping level of boron required to enhance desirable properties of SiOC composites.

PACRIM Symposium 06: Synthesis and Processing of Materials Using Electric Currents and Pressures**Electric Currents II**

Room: King's 1

Session Chairs: Yasuhiro Kodera, University of California, San Diego; Manshi Ohyanagi, Ryukoku University; Claude Estournes, CIRIMAT

8:30 AM**(PACRIM-S6-010-2017) Progress in Spark Plasma Sintering (SPS) Method and Cost-Effective Technology to Produce Functionally Graded Materials (FGMs) on the Large Scale (Invited)**M. Tokita^{*1}

1. NJS Co., Ltd., SPS R&D Center, Japan

Functionally Graded Materials (FGMs) is being considered in many high-tech and high-performance applications in the field of mold & die, cutting tools, electronics, automotive, clean energy and aerospace industries. The FGMs will always cost more than metallic or ceramic, monolithic or composite alternative materials because of the inherent cost of manufacturing method. The potentials for an industrial use of FGMs are hampered by the lack of an infrastructure for high volume manufacturing facilities. In order to overcome those existing issues, the low cost production systems and processes are desired. The SPS has a high potential to be a major FGMs manufacturing method in the various industries provided the developments necessary, therefore, is now challenging both high-value added small scale and large scale area using by the 5th generation SPS systems with new process. For example, industrially commercialized application on ZrO₂/Ti alloy FGMs for Ultra-sonic Homogenizer system developed in Japan will be introduced in this talk. A weldable WC/Ni system FGM having Hv1400-1500 surface hardness was already applied to a screw component of extruding machine and achieved more than 3 times longer life time at economical running cost. The factors influencing the cost for industrialization of SPS method on advanced FGMs are discussed.

9:00 AM**(PACRIM-S6-011-2017) Spark plasma sintering of Diamond/Si₃N₄ composite (Invited)**M. Ohyanagi^{*1}; H. Inoue¹; K. Shirai¹; Z. Munir²

1. Ryukoku University, Japan

2. University of California, Davis, Department of Chemical Engineering and Materials Science, USA

The feature of SPS method includes very short processing time and very high-heating rate compared to the conventional hot-pressing process and hot isostatic pressing, which makes it possible to densify metastable materials by suppressing the kinetic change of the metastable phase. The unique SPS process was developed so that the loading pressure can go up to 1 GPa by using a set of specially-modified high pressure die (consisting of inner and outer dies). We describe, herein, the fabrication of 50vol% diamond/Si₃N₄ composite under 1 GPa at 1550°C which the condition for the consolidation is thermodynamically not stable for diamond. The key issue is to suppress the transformation of diamond to graphite under relatively high pressure, for short time and just above at the threshold temperature for the graphitization. When the 50vol% diamond/

^{*}Denotes Presenter

Abstracts

Si₃N₄ composite was consolidated with 8wt% and 12 wt% of Al₂O₃-Y₂O₃ additives at 1550°C under 1.0GPa for 5min., the relative density reached 93.5% and 96.2%. The Raman spectrum of the composite showed that most of diamond structure was maintained, but the low intensity of DLC signal was also detected in the composites. The abrasion test for the dense composite of 50vol%diamond/Si₃N₄ with 96.2% of the relative density was performed. The abrasion amount in the denser composite with 12wt% of the additive was much less than that of the composite with 8.0wt% of additive.

9:30 AM

(PACRIM-S6-012-2017) Development of Electric current activated/assisted sintering (ECAS/SPS) (Invited)

Y. Sakka^{*1}

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

Essentially, SPS exploits the same punch/die system concept as the more familiar hot pressing (HP) process. The well-established advantages of SPS are: (a) low power consumption, (b) the absence of sintering aids, (c) control of the thermal gradient (for functional graded materials (FGMs)), (d) selective control of the density in specified regions, (e) accurate control of the porosity, (f) single step sintering-bonding, (g) particle surface cleaning, (h) high heating rate and (i) near-net-shape capability. In the SPS process, however, the measured temperature is not directly related to the sintering temperature. The combined experimental and FEM simulation analysis permitted to obtain the optimum process and mold design which permitted to have direct control of the final microstructure. The electric conductivity of the material plays a fundamental role on the current and temperature distribution inside the sample. The SPS method was successfully applied to electric conductive ceramics such as pure WC, WC-diamond, (Zr,Hf)B₂-systems, MAX phase ceramics, semiconductors such as B₄C, B₄C compounds, and low electrical conductive ceramics such as transparent alumina, CNT-alumina nanocomposites. Finally, we will summarize future direction and problems of the SPS technology.

10:15 AM

(PACRIM-S6-013-2017) Modification of pulsed electric current sintering conditions for the reduction of processing time

M. Mikami^{*1}; K. Kubo²; N. Uchiyama²; H. Miyazaki³; Y. Nishino³

1. National Institute of Advanced Industrial Science and Technology, Japan
2. Atsumitec Co., Ltd., Japan
3. Nagoya Institute of Technology, Japan

Pulsed electric current sintering is an effective method of controlling microstructure in sintered material because of its high heating and cooling rate. In various kinds of functional material, transport properties can be controlled by microstructure refinement in order to optimize its performance. Especially for a thermoelectric material, the reduction of thermal conductivity and the improvement of mechanical strength can enhance utility for thermoelectric energy conversion devices. However, typical whole processing time of the pulsed electric current sintering is the order of tens of minutes or longer and it is too long for the mass fabrication at low cost. In this study, in order to reduce the processing time, heating conditions such as amount of current, current flow region, sintering mold were modified according to the finite element simulation. Using the modified sintering process, sintered compact was obtained by current feed within several seconds. The microstructure and transport properties of the sintered compact will be presented.

10:35 AM

(PACRIM-S6-014-2017) Deformable Punch Spark Plasma Sintering for Processing of Fully Dense Nanocrystalline Oxides (Invited)

R. Castro^{*1}

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

Fully dense nanocrystalline oxides are extremely difficult to be obtained due to the high stability of isolated pores formed during the final stage of sintering. While techniques such as Spark Plasma Sintering can improve densification by plastic yielding, this densification mechanism is hindered by the high stability of the isolated pores. This leaves pores to be eliminated only with some grain growth. From a thermodynamic perspective, grain growth decreases the stability of the pores by changing equilibrium contact angles attained at the interface between pores and grain boundaries. In this work, we introduce an alternative manner to de-stabilize pores by utilizing a deformable punch in the die. This is designed to complaint at the final stage of sintering, such that grains are forced to slide along and naturally cause a change in the equilibrium angle. Using cemented WC punch, MgAl₂O₄ nanoparticles with sizes below 5nm were sintered using this technique and the results were fully dense, transparent ceramics, with grain sizes below 10nm. This unprecedented achievement allowed assess of nano-related properties, such as the extension of the Hall-Petch relationship down to those small grain sizes -. We further used the deformable punch to sintering BaTiO₃, obtaining a highly transparent sample with grain sizes below 20nm, and has the potential for application in many other systems.

11:05 AM

(PACRIM-S6-015-2017) Synthesis and processing of magnetic nanocomposites through CAPAD

Y. Kodera^{*1}; K. Chan¹; A. Volodchenkov²; J. E. Garay¹

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

A core mission for material scientists is the developing new materials via innovative processing approaches. One example is overcoming thermodynamic limits using kinetics to obtain materials with far from equilibrium (FFE) state. Bulk material with nano-sized grains that possess non-conventional properties is great example. Obtaining FFE bulk materials through powder consolidation approaches, requires the optimizations of the powder synthesis method specifically for the consolidation method and the consolidation method to maintain FFE state as bulk form. This is because an appropriate energy balance (heat) is required; if FFE materials are over heated, they will convert to equilibrium phase/state and exhibit conventional properties. Here we will present results on the chemical synthesis of metastable material/phases and the integration into a consolidated nanocomposite via Current Activated Pressure Assisted Densification (CAPAD). We will show processing of iron oxide/silica hard bulk magnets with coercive fields comparable to typical rare-earth magnets. Also an increase in energy product of ferrite by forming nano-composite with 3d transition metal will be presented.

11:25 AM

(PACRIM-S6-016-2017) Nanostructured zinc oxide by FAST/SPS and cold sintering

J. Gonzalez-Julian^{*1}; K. Neuhaus²; M. Bernemann²; M. Bram¹; O. Guillon¹

1. Forschungszentrum Juelich, Germany
2. University of Muenster, Germany

Retention of nanocrystallinity in dense ceramic materials is one of main challenge in material science. To achieve this objective, the sintering temperature has to be strongly reduced in order to avoid unwanted grain growth mechanisms. Different approaches

have been largely explored, such as sintering methods assisted by mechanical pressure (Hot pressing), electric field/current (flash sintering) and combination of both (Field Assisted Sintering/Spark Plasma Sintering, FAST/SPS). Recently, one of the most promising approaches is to incorporate a transient liquid phase, typically water, to promote the full densification at low temperature. This method has been observed in FAST/SPS, and also demonstrated in several ceramic materials under high uniaxial pressures, which has been called "cold sintering". In this work, sintering behavior of ZnO with different contents of water will be analyzed using FAST/SPS and cold sintering. Highly dense nanocrystalline ZnO was obtained for maximal sintering temperatures of 400 °C and 250 °C, using graphite and non-graphite tools (to increase the mechanical pressure), respectively. Regarding the cold sintering, ZnO was highly densified keeping the nanostructure at only 250 °C. Sintering behavior, microstructure of the consolidated nanostructures, and the characterization by different techniques such AFM and photoluminescence will be evaluated to reveal the involved mechanisms.

PACRIM Symposium 13: Advanced Structural Ceramics for Extreme Environments

New Materials and Properties

Room: Kohala 4

Session Chairs: Stephan Schmidt-Wimmer, Airbus Defence and Space; Hailong Wang, Zhengzhou University

8:30 AM

(PACRIM-S13-029-2017) Oxidation behavior of porous Si₃N₄ ceramics

H. Liang^{*1}; Y. Zeng¹

1. Shanghai Institute of Ceramics, Chinese Academy of Sciences, China

Being an almighty ceramic, Si₃N₄ possess the high performance of high strength, high hardness, good resistance to thermal shock and oxidation, low dielectric constant and dielectric loss. Especially, compared with other engineering ceramics, porous Si₃N₄ ceramics have great advantage over other porous ceramics due to the extensive existence of rod-like grains in the matrix. As a consequence, porous Si₃N₄ ceramics have great potential to be used in various industry sectors. Generally speaking, high temperature and oxidation atmosphere are often encountered when porous Si₃N₄ ceramics are under service. So oxidation of porous Si₃N₄ ceramics should be taken into consideration when they are adopted. However, in spite that the oxidation behavior of dense Si₃N₄ ceramics have been widely studied in the past several decades, those of porous Si₃N₄ ceramics have been rarely concerned. In this investigation, the oxidation behavior of porous Si₃N₄ ceramics were studied in detail including the weight change, phase transformation, residual flexural strength and morphology evolution etc. The oxidation mechanism of porous Si₃N₄ ceramics were also analyzed.

8:45 AM

(PACRIM-S13-030-2017) New Ceramic Material AlB₁₂-AlN: A Combination of High Boron Content, Hardness and Thermal Conductivity

O. Vasiliev^{*1}; V. Kartuzov¹; V. Muratov¹; P. Mazur¹; V. Garbuz¹; Y. Kartuzov¹

1. Institut for Problems of Materials Sciences NAS of Ukraine, Ukraine

The purpose of the work was to combine in a ceramic material, on the basis of theoretical studies with computer modeling elements, the properties of AlB₁₂ (high boron content, hardness, chemical and wear resistance, etc.) and AlN, as softer constituent with high thermal conductivity. AlB₁₂ and AlN powders were produced by vacuum-thermal synthesis with CVD elements from the inexpensive raw materials, without elemental boron, and compacted using hot

pressing. To vary the ratio of the components in the ceramics, AlB₁₂ was separated from AlN by wet chemistry. The result of the synthesis was a mixture of nanopowders ($d_n < 100$ nm) AlB₁₂ and AlN. Because of small particle sizes, the temperature of hot pressing was only 1800 °C. Ceramics with 25% AlB₁₂ and 75% AlN has hardness of 24 GPa, and retains its values above 16 GPa at loads up to 500 N (Vickers test). There is no interaction between components in the AlB₁₂-AlN system, allowing for the material properties variation according to the mixture rule. This way we estimated the thermal conductivity of the ceramics 100 W/(m·K). In conclusion, a new ceramic composite AlB₁₂-AlN was obtained, which possesses high boron content (neutron absorption), high mechanical properties and thermal conductivity level; the method of its preparation allows for the variation of operational properties by control of the ratio between the components.

9:00 AM

(PACRIM-S13-031-2017) Processing and Properties of ZrB₂-SiCw Composites Sintered by Spark Plasma Sintering

H. Wang^{*1}; G. Shao¹; B. Fan¹; H. Lu¹; R. Zhang²

1. Zhengzhou University, Materials Science and Engineering, China

2. Zhengzhou Institute of Aeronautical Industry Management, China

The ZrB₂-SiC_w composite powder was synthesized by sol-gel method using zirconium diboride, tetraethoxysilane (TEOS) and activated carbon as starting materials, then densified by SPS at 1700 °C under a pressure of 40 MPa for 5 min. The processing parameters including carbon source, the ratio of silicon and carbon, temperature and holding time for the synthesis of ZrB₂-SiCw powders were optimized, and the synthetic mechanism were investigated. Microstructure observations revealed that a large number of crooked SiC whiskers appear on the surface of ZrB₂ particles. Moreover, full dense ZrB₂-SiCw ceramics were achieved by SPS. Both fracture toughness and flexural strength of ZrB₂-SiCw composites were greatly improved with increasing amount of SiC whiskers. ZrB₂-30vol% SiC_w composite showed the highest fracture toughness of 6.1 MPa·m^{1/2} and flexural strength of 350 MPa, which were attributed to the formation of SiC whisker.

PACRIM Symposium 18: Microwave Dielectric Materials and Their Applications

Microwave Dielectric Materials and Their Applications III

Room: Kohala 2

Session Chair: Chonglin Chen, University of Texas San Antonio

8:30 AM

(PACRIM-S18-015-2017) Defect Engineered Complex Oxide Thin Films with Tunable Multiferroic Properties (Invited)

C. Chen^{*1}

1. University of Texas San Antonio, Physics, USA

Complex oxides have demonstrated various important physical properties such as various dielectric and unusual magnetic properties. These extraordinary phenomena are highly dependent upon the degrees of the freedom of the charge distribution, spin and orbital status, and the lattice structures. Complex cobalt oxide can exhibit different cobalt and oxygen coordination from tetrahedral, pyramidal to octahedral dependent on the oxygen content in it, leading to various crystal structures with a great flexibility of the oxygen frameworks. Thus, oxygen nonstoichiometry is a very crucial parameter for tuning their physical properties determined by their crystal structure. For instance, LnBaCo₂O_{5+d} (LnBCO, Ln= rare transition metal elements) systems exhibit various unique physical properties because of not only the presence of A-site disordered and A-site ordered structures, due to the close ionic sizes of Ln and

^{*}Denotes Presenter