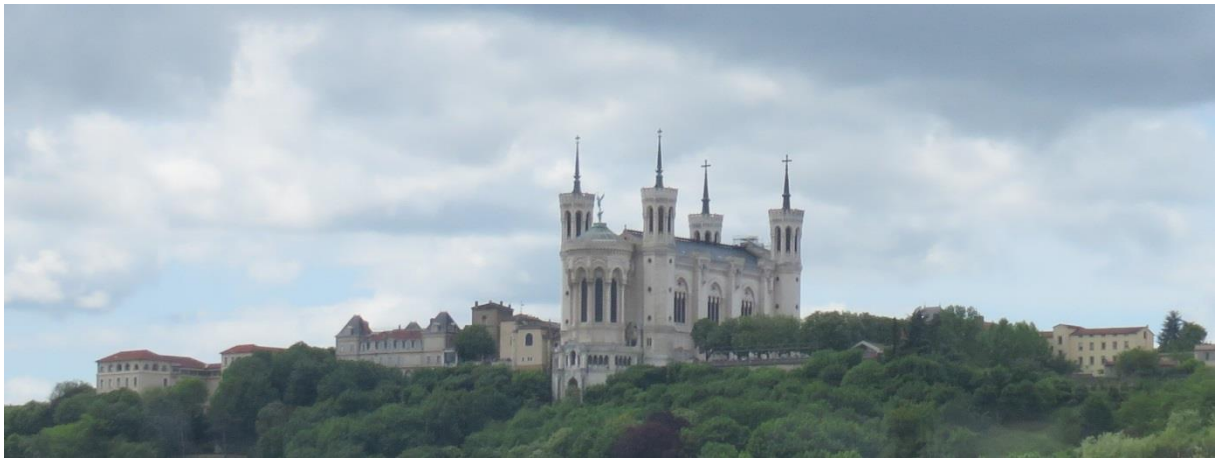
15th European Inter-Regional Conference on Ceramics

CIEC 15



5 – 7 September 2016, Lyon

Book of Abstracts

CIEC 15 Program

MONDAY 5TH Sept.

08h00		Participants Welcome
08h30		Conference Opening
09h00	Invited	Fabrice ROSSIGNOL
09h30		M. SCHMIDT
09h45		J. SARTHOU
10h00		J.J. ROA
10h15		J.M. TULLIANI
10h30	Posters	Coffee break
11h00		C. GAJDOWSKI
11h15		J. CROQUESEL
11h30		J.M. CHAIX
11h45		M. MARCINKOWSKA
12h00		R. MACAIGNE
12h15		G. KALFAYAN
12h30		P. NINZ
12h45		Lunch

MONDAY 5TH Sept.

14h00	Invited	Robert DANZER
14h30		I. TOUAIHER
14h45		R. BERMEJO
15h00		G. BEN GHORBAL
15h15		U. SCHMITT-RADLOFF
15h30	Highlight	Marc ANGLADA
16h00	Posters	Coffee break
16h30	Highlight	Martin BACHE
17h00		J.P. JONES
17h15		Z. QUINEY
17h30		C. MINGAZZINI
17h45		M. JIMENEZ
18h00		Posters session
19h00	Posters	Get together (buffet dinner)
20h00		

TUESDAY 6TH Sept.

08h30	Highlight	Maurice GONON
09h00		D. BOUVARD
09h15		T.P. VU
09h30		M. DUTTO
09h45		M. LASGORCEIX
10h00		E. JIMENEZ-PIQUE
10h15		E. ROITERO
10h30	Posters	Coffee break
11h00	Invited	Corrado PICONI
11h30		M. BOHNER
11h45		P. KRIEG
12h00		H. ELSAYED
12h15		S. RAMIREZ
12h30		R. GADOW
12h45		Lunch

TUESDAY 6TH Sept.

14h00	Highlight	Frank KERN
14h30		G. DE PORTU
14h45		D. MARCHAT
15h00	Highlight	Paola PALMERO
15h30		A. PINEL
15h45		D. ZIEGLER
16h00		
17h00		Confluences Museum visit
19h45		
20h00		*Conference Dinner*
23h00		

WEDNESDAY 7TH Sept.

09h00	Invited	Arturo DOMINGUEZ
09h30		M.S. RENOIRT
09h45		Ş. SEN
10h00		J. SEUBA
10h15		V. GUERINEAU
10h30	Posters	Coffee break
11h00	Highlight	Daniele MARI
11h30		V. GAUTHIER-BRUNET
11h45		M. TURON-VINAS
12h00		G. BAYRAK
12h15		N. PREUX
12h30		A. CELLARD
12h45		Lunch
14h00		Optional: visit of SETARAM

Since 1988, CIEC has been organised biannually by researchers of some European regions. Starting with 4 contributing regions, the number of participants has regularly increased and includes now Baden-Würtenberg, Catalunya, Canton de Vaud, Hainaut, Lombardia, Piemonte Valle D'Aosta, Rhône-Alpes and Wales.

The 15th European Inter-Regional Conference on Ceramics will be held from 5 to 7 September 2016 in Lyon (France). This conference will cover all the scientific aspects concerning ceramics as well as processing, structural characterization, properties and industrial applications of monolithic ceramics, ceramic composites, glass and ceramic coatings. The conference will consist of 4 invited keynotes, 6 highlights, as well as 43 oral and 36 poster contributions. A single session format as well as a lively poster session will allow detailed discussion of the relevant issues. This conference aims to promote particularly collaboration between young researchers and supports initiatives of European scientific networking.

On behalf of the scientific committee and of the local organizing committee, we thank you for your participation and for your venue in Lyon.

***Scientific Committee*:**

M.J. Anglada Gomila (Catalunya), U. Anselmi-Tamburini (Lombardia), M. Bache (Wales), J. Chevalier (Auvergne Rhône-Alpes), R. Gadow (Baden-Württemberg), M. Gonon (Hainaut), L. Montanaro (Piemonte), D. Mari (Canton de Vaud).

Local organizing committee:

J. Chevalier (MATEIS, INSA Lyon), J. M. Chaix (SIMAP, INP Grenoble), N. Douard (SainBiose, ENSMSE), G. Fantozzi (MATEIS, INSA Lyon), V. Garnier (MATEIS, INSA Lyon), D. Goeuriot (LGF, ENSMSE), L. Joly-Pottuz (MATEIS, INSA Lyon), S. Meille (MATEIS, INSA Lyon), C. Payen (MATEIS, INSA Lyon), L. Quillot (MATEIS, INSA Lyon), H. Reveron (MATEIS, INSA Lyon), P. Reynaud (MATEIS, INSA Lyon), M. R'Mili (MATEIS, INSA Lyon), V. Salles (LMI, UCB Lyon I), S. Tadier (MATEIS, INSA Lyon), F. Valdivieso (LGF, ENSMSE) and all the PhD students.

CIEC 15 PROGRAM

Monday 05/09/2016 morning

08h00 Participants Welcome

08h30 Conference Opening

09h00 Keynote lecture: Combining top-down and bottom-up approaches through additive manufacturing for biosensors fabrication, by Fabrice Rossignol

F. Rossignol[1], R. Trihan[1], C. Lefort[2], M. Lejeune[2]

[1]SPCTS, UMR CNRS 7315, 12 rue Atlantis, 87068 Limoges, France

[2]XLIM, UMR CNRS 7252, 123 avenue Albert Thomas, 87060 Limoges, France

09h30 **Addition of boron at molecular scale in Silicon Carbide Precursors : Effect on the pyrolysis behavior of precursors and on the final properties of silicon carbide**

M. Schmidt[1], G. Chollon[2], and S. Bernard[1]

[1]Institut Européen des Membranes, UMR 5635-CNRS-ENSCM-UM, Université de Montpellier, Place E. Bataillon, F-34095, Montpellier, France - [2]Laboratoire des Composites Thermostructuraux, CNRS-Herakles-CEA-Université de Bordeaux, 3 allée de La Boétie, F-33600, Pessac, France

09h45 **Synthesis and characterization of Yb:CaF_2 transparent ceramics using an innovative energy-saving wet-route fabrication process**

J. Sarthou[1], P. Aballéa[1], G. Patriarche[2], H. Serier-Brault[3], A. Suganuma[1], P. Gredin[1,4], M. Mortier[1]

[1]PSL Research University, Chimie ParisTech, CNRS, Institut de Recherche de Chimie Paris, 75005 Paris, France - [2]Laboratoire de Photonique et de Nanostructure (LPN), CNRS, Université Paris-Saclay, Route de Nozay, F-91460 Marcoussis, France - [3]Institut des Matériaux Jean Rouxel, Université de Nantes, CNRS, 2 rue de la Houssinière, BP 32229, 44322 Nantes cedex, France - [4]Université Pierre et Marie Curie, 4 Place Jussieu, 75005 Paris, France

10h00 **Structural and Mechanical Characterization of 3Y-ZrO_2 produced by Rapid Prototyping**

J.J. Roa[1,2], G. Fargas[1,2] and M. Anglada[1,2]

[1]CIEFMA, Department of Materials Science and Metallurgical Engineering, ETSEIB, Universitat Politècnica de Catalunya-Barcelona Tech, Avda. Diagonal 647, 08028, Barcelona, Spain - [2]CRnE, Campus Diagonal Sud, Edificio C', Universitat Politècnica de Catalunya-Barcelona Tech, C/ Pascual i Vila 15, 08028, Barcelona, Spain

10h15 **Elaboration and characterization of humidity sensors based on micro-carbonized bamboo particles**

A.S. Afify[1], S. Ahmad[2], R.A. Khushnood[3], P. Jagdale[1] and J.-M. Tulliani[1]

[1]Department of Applied Science and Technology, Politecnico di Torino, INSTM Research Unit PoliTO, LINCE Laboratory, Corso Duca degli Abruzzi 24, 10129 Torino, Italy - [2]Mirpur University of Science and Technology (MUST), 10250 Mirpur, Azad Kashmir, Pakistan - [3]Institute of Civil Engineering (NICE), National University of Sciences and Technology (NUST), 44000 Islamabad, Pakistan

10h30 Coffee break

11h00 **Transparent polycrystalline ceramics for ballistic application**

C. Gajdowski[1,2], J. Böhmler[1], E. Barraud[1], S. Lemonnier[1], Y. Lorgouilloux[2], S. d'Astorg[2], A. Leriche[2]

[1]Institut franco-allemand de recherches de Saint-Louis, 5 rue du Général Cassagnou, 68301 Saint-Louis, France - [2]Laboratoire des Matériaux Céramiques et des Procédés Associés, Boulevard du Général de Gaulle, 59600 Maubeuge, France

11h15 **Microwave sintering of nuclear ceramics**

J. Croquesel[1], S. Pillon[1], F. Valdivieso[2] and S. Saunier[2]

[1]CEA Marcoule, DEN/DTEC/SECA/LFC, BP 17171, 30207 Bagnols sur Cèze cedex, France - [2]Ecole des Mines de Saint-Etienne, Laboratoire Georges Friedel CNRS UMR 5307, 158 cours Fauriel, 42023 Saint Etienne, France

11h30 **Flash sintering of zirconia and zirconia-alumina composites**

E. Bichaud[1,2], M.C. Steil[1], J.-M. Chaix[2] and C.P. Carry[2]

[1]LEPMI, Univ. Grenoble Alpes, CNRS, F-38000 Grenoble, France - [2]SIMAP, Univ. Grenoble Alpes, CNRS, F-38000 Grenoble, France

11h45 **Field assisted sintering processing of bioinspired ceramic/metal composites**

M. Marcinkowska[1,2], E. Maire[1], S. Meille[1], J. Chevalier[1] and S. Deville[2]

[1]Université de Lyon, INSA-Lyon, MATEIS CNRS UMR5510, Villeurbanne 84306, France - [2]Laboratoire de Synthèse et Fonctionnalisation des Céramiques, UMR3080 CNRS/Saint-Gobain, Cavaillon F-69621, France

12h00 **Study of the Influence of Different Dopants on the Densification of Spinel by Microwave Heating**

R. Macaigne[1,2], S. Marinel[1], E. Savary[3], G. Riquet[1], C. Meunier[2], S. Saunier[2], D. Goeuriot[2]

[1]CRISMAT Laboratory, UMR 6508 CNRS-ENSICAEN-UCN, 6 Boulevard Maréchal Juin, 14050 Caen cedex, France - [2]Ecole Nationale Supérieure des Mines de Saint-Etienne, 158 Cours Fauriel, 42023 Saint-Étienne, France - [3]CNRS-Délégation Normandie, 6 Boulevard Maréchal Juin, 14050 Caen cedex, France

12h15 **Microwave joining process of an alumino-silicate ceramic material for radioactive waste containers**

G. Kalfayan[1,2], D. Goeuriot[1], S. Saunier[1], C. Meunier[1] and N. Texier-Mandoki[2]

[1]Centre Sciences des Matériaux et des Structures, CNRS-UMR 5307, ENS des Mines de St-Etienne, 158 cours Fauriel, 42023 St-Etienne, France - [2]Andra, 1-7 rue Jean Monnet, 92298 Chatenay-Malabry Cedex

12h30 **Ceramic substrates for laser induced selective metallization**

P. Ninz[1], F. Kern[1], E. Ermantraut[2], H. Müller[3], W. Eberhardt[3], A. Zimmermann[2,3] and R. Gadow[1]

[1]Institut für Fertigungstechnologie keramischer Bauteile (IFKB), University of Stuttgart, Allmandring 7b, D-70569, Stuttgart, Germany - [2]Institute for Microintegration, University of Stuttgart, Allmandring 9b, D-70569, Stuttgart, Germany - [3]Hahn-Schickard-Institute for Microassembly Technology, Allmandring 9b, D-70569, Stuttgart, Germany

12h45 Lunch

Combining top-down and bottom-up approaches through additive manufacturing for biosensors fabrication

Fabrice Rossignol[1], Romain Trihan[1], Claire Lefort[2] and Martine Lejeune[1]

[1]SPCTS, UMR CNRS 7315, 12 rue Atlantis, 87068, Limoges, France

[2]XLIM, UMR CNRS 7252, 123 avenue Albert Thomas, 87060 Limoges, France

Abstract

We will give an overview of what is currently being done in the field of additive manufacturing (AM) dedicated to the fabrication of biosensors at the laboratory of Science of Ceramic Processes and of Surface Treatments (SPCTS) in Limoges, France. More specifically, we will focus our talk on examples of recent works dealing with cancer diagnostic and therapy based on the development of endoscopic devices suitable for the early detection and treatment of tumors. The devices are fabricated by combining Inkjet Printing (IJP), with Evaporation Induced Self-Assembly (EISA), to produce mesoporous silica microdots arrays that can be afterwards specifically functionalized with biomarkers by click chemistry. We will show how such a general approach can be used to treat both the diagnostic issues through Fluorescence Resonance Energy Transfer (FRET) and the therapy issues through PhotoDynamic Therapy (PDT).

Keywords : Additive manufacturing, Inkjet printing, Biosensors

Addition of boron at molecular scale in Silicon Carbide Precursors : Effect on the pyrolysis behavior of precursors and on the final properties of silicon carbide

Marion Schmidt[1], Georges Chollon[2], and Samuel Bernard[1]

[1]*Institut Européen des Membranes, UMR 5635-CNRS-ENSCM-UM, Université de Montpellier, Place E. Bataillon, F-34095, Montpellier, France*
[2]*Laboratoire des Composites Thermostructuraux, CNRS-Herakles-CEA-Université de Bordeaux, 3 allée de La Boétie, F-33600, Pessac, France*

Abstract

Silicon carbide (SiC) has attracted interest for environmental and energy applications due to its good properties at high temperature.
The synthesis of SiC was initiated by Acheson in 1892 and is still today applied to produce commercial *SiC* powders. Traditional manufacturing techniques such as sintering are energy-intensive, inappropriate for complex shapes and the ability to control purity and crystallinity is also restricted. Future industrial challenges for SiC components require materials with compositions, shapes and microstructures that are tuned on demand. This can be achieved by combining both molecular and materials chemistries. The *Polymer-Derived Ceramics* (PDCs) route is a good example of this strategy. The chemistry, the processing properties and the reactivity of polymers can be tailored to supply, after shaping and pyrolysis, ceramics with the desired phase composition and distribution. This method was used to prepare boron-modified SiC. The polymers were synthesized by the reaction of allylhydridopolycarbosilane (AHPCS) with borane dimethylsulfide. The boron content has an effect on the chemistry and processability of precursors, as well as on the properties of final materials. Infrared, NMR spectroscopies and elemental analyses give us information about the chemical properties of the boron-modified SiC precursors. Their pyrolysis behavior is investigated by solid-state NMR coupled with thermogravimetric analyses. The final materials are characterized by X-ray diffraction, elementary analysis and Raman spectroscopy. Applications of the PDC route to the processing of ceramic composites will be discussed.

Keywords : Polymer-Derived-Ceramics, Silicon carbide, Composites

Synthesis and characterization of Yb:CaF_2 transparent ceramics using an innovative energy-saving wet-route fabrication process

Julia Sarthou[1], Pierre Aballéa[1], Gilles Patriarche[2], Hélène Serier-Brault[3], Akiko Suganuma[1], Patrick Gredin[1,4], Michel Mortier[1]

[1]*PSL Research University, Chimie ParisTech, CNRS, Institut de Recherche de Chimie Paris, 75005 Paris, France*

[2]*Laboratoire de Photonique et de Nanostructure (LPN), CNRS, Université Paris-Saclay, Route de Nozay, F-91460 Marcoussis, France*

[3]*Institut des Matériaux Jean Rouxel, Université de Nantes, CNRS, 2 rue de la Houssinière, BP 32229, 44322 Nantes cedex, France*

[4]*Université Pierre et Marie Curie, 4 Place Jussieu, 75005 Paris, France*

Abstract

Since the first Nd:YAG transparent ceramic for laser applications demonstrated the advantages of ceramics with respect to single crystals, rare-earth doped polycrystalline ceramics as solid-state laser host material have experienced an increasing interest. In the past few years, our team has been focusing on a new class of transparent ceramics for near-IR solid-state lasers, Yb:CaF_2. This compound is very promising due to the combined properties of its fluoride matrix and ytterbium ions. We report here a new fabrication process for Yb:CaF_2 transparent ceramics using a complete wet-route for the green body shaping and a strainless sintering of the ceramic. No post-treatment is used. The fabrication process is thus energy-saving. Microstructural characterizations conducted on the obtained transparent ceramics show that the grain size is about 200nm, and the grain boundaries appear to be thin, with no amorphous oxygen compounds. The optical transmission of the obtained ceramics reaches more than 93% in the near-IR, which is better than the transmission of fluoride ceramics obtained with traditional dry-route fabrication processes. The laser properties also prove to be improved since the maximum power extracted from a 2.71mm thick 4at.% Yb:CaF_2 transparent ceramic reaches 1.6W (with a 6% coupler), which is the highest result recorded for powder-based Yb:CaF_2 ceramics.

Keywords : Transparent ceramic; laser application; calcium fluoride; ytterbium; wet-route

Structural and Mechanical Characterization of 3Y-ZrO2 produced by Rapid Prototyping

J.J.Roa[1,2], G.Fargas[1,2] and M.Anglada[1,2]

[1]CIEFMA, Department of Materials Science and Metallurgical Engineering, ETSEIB, Universitat Politècnica de Catalunya-Barcelona Tech, Avda. Diagonal 647, 08028, Barcelona, Spain

[2]CRnE, Campus Diagonal Sud, Edificio C', Universitat Politècnica de Catalunya-Barcelona Tech, C/ Pascual i Vila 15, 08028, Barcelona, Spain

Abstract

Engineering ceramics present a unique combination of mechanical, thermal and chemical properties becoming increasingly important in the nowadays-industrial landscape. Unlike other engineering ceramic materials, zirconia displays high strength and fracture toughness and biocompatibility, which makes it a candidate material for a wide range of applications in the biomedical field. However, manufacturing ceramic components has still a major limitation in production of highly complex 3D shapes, micro features or structures with tailored porosity. Present new technologies enable adding up complex three-dimensional structures layer by layer. Powder based rapid prototyping technique is one of such versatile methods with unique flexibility in material and geometry. In this work, 3Y-ZrO_2 based ceramic materials were designed and produced by Rapid Prototyping with the main goal to study the influence of printing geometries on the microstructure and mechanical properties from macro-to microscopic length scale. The results of density, hardness, sliding tests, stress-strain curves obtained through micro-compression tests and indentation fracture toughness, were discussed and compared with 3Y-ZrO_2 bulk ceramic materials produced under conventional production routes. A detailed characterization of the microstructure was performed by X-ray diffraction, Field Emission Scanning Electron Microscopy and Focus Ion Beam. A direct correlation is found between both the microstructural and printing parameters (i.e. superficial porosity, printing geometry and/or direction, etc.) and the mechanical properties.

Keywords : 3Y-ZrO_2 ceramic material, rapid prototyping technique, microstructural, mechanical characterization, micro-compression tests

Elaboration and characterization of humidity sensors based on micro-carbonized bamboo particles

Ahmed S. Afify[1], Sajjad Ahmad[2], Rao Arsalan Khushnood[3], Pravin Jagdale[1] and Jean-Marc Tulliani[1]

[1]Department of Applied Science and Technology, Politecnico di Torino, INSTM Research Unit PoliTO, LINCE Laboratory, Corso Duca degli Abruzzi 24, 10129 Torino, Italy

[2]Mirpur University of Science and Technology (MUST), 10250 Mirpur, Azad Kashmir, Pakistan

[3]Institute of Civil Engineering (NICE), National University of Sciences and Technology (NUST), 44000 Islamabad, Pakistan

Abstract

In this work, eco-friendly, low cost and efficient humidity sensors were prepared from micro-sized pyrolyzed bamboo. Two types of pyrolized particles were investigated: as such (CB) and annealed ones (CBA). After pyrolysis, the materials were first manually ground and then, attrition milled. The synthesized particles were characterized by using field emission-scanning electron microscopy, Raman spectroscopy, specific surface area (SSA) measurements and thermogravimetric analysis. The sensing materials were screen-printed onto commercial α-alumina substrates with platinum electrodes prior to firing at 300°C for 1 hour. The humidity sensing properties of the pyrolyzed bamboo based sensors were investigated at room temperature in the relative humidity (RH) range from 0.0 to 96.0%. The pyrolyzed bamboo based humidity sensors exhibited an excellent response towards humidity starting from 10% RH, while CBA showed a response starting from 20% RH. These differences can be explained on the basis of SSA results: annealing led to drastic reduction of open porosity. Finally, the response and recovery times were reasonably fast (less than 2 minutes).

Keywords : Bamboo, Pyrolysis, Humidity sensor.

Transparent polycrystalline ceramics for ballistic application

Caroline Gajdowski[1,2], Judith Böhmler[1], Elodie Barraud[1], Sébastien Lemonnier[1], Yannick Lorgouilloux[2], Sophie d'Astorg[2], Anne Leriche[2]

[1]*Institut franco-allemand de recherches de Saint-Louis, 5 rue du Général Cassagnou, 68301 Saint-Louis, France*

[2]*Laboratoire des Matériaux Céramiques et des Procédés Associés, Boulevard du Général de Gaulle, 59600 Maubeuge, France*

Abstract

Against an armour piercing bullet, a transparent armour made of glass and polymer has to be thick to be efficient, implying a substantial increase of mass. By the addition of a transparent polycrystalline ceramic disposed in the front of the armour, significant lightweighting is expected since the ceramic favours the global efficiency through the fragmentation of the projectile and the decrease of the penetration at the impact. The spinel $MgAl_2O_4$ has shown good performances as front face of transparent armours thanks to a unique combination of optical and mechanical properties, such as a cubic structure, a wide range of transmission, a low density and a high toughness. From a material point of view, the ceramic has to present a poreless microstructure without impurities to be transparent. And as a material for ballistic protection, it has to exhibit a high hardness, which can be brought with a fine and homogeneous microstructure. For this study, spinel nanopowders of high purity have been chosen to obtain green bodies by uniaxial and cold isostatic pressing. To avoid grain growth and thus to preserve a fine microstructure and a high mechanical resistance, the samples have been pressureless sintered without any additives nor sintering aids. The residual porosity has been eliminated by hot isostatic pressing. For both thermal treatments, different temperatures and holding times have been investigated. First promising results are observed where samples with a diameter of 21 mm and a thickness of 2 mm present an in-line transmission of 65% in the visible range.

Keywords : Transparent ceramic, armour, pressureless sintering, spinel

Microwave sintering of nuclear ceramics

Jérémy Croquesel[1], Sylvie Pillon[1], François Valdivieso[2] and Sébastien Saunier[2]

[1]CEA Marcoule, DEN/DTEC/SECA/LFC, BP 17171, 30207 Bagnols sur Cèze cedex, France

[2]Ecole des Mines de Saint-Etienne, Laboratoire Georges Friedel CNRS UMR 5307, 158 cours Fauriel, 42023 Saint Etienne, France

Abstract

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. The technical feasibility of UO_2 microwave sintering has already been demonstrated in few studies which also related specific effects on grain growth. Nevertheless, various problems like difficult control of the process (constant heating rate, temperature measurement), heterogeneous heating, cracking of the pellets and arcing were reported. Moreover, sintering of MOX pellets has never been investigated yet and the presence of various elements with different dielectric properties may also be restrictive since the interdiffusion kinetic of U/Pu could be modified compared to conventional sintering. This paper describes the development of an instrumented (pyrometer, optical dilatometry) and automated microwave setup, qualified on fuel surrogates, which will allow reproducible and reliable comparison of nuclear fuels sintering compared to conventional sintering. Finite elements modeling were used to design the setup and a hybrid heating technique with a specific sintering cell (design, type and geometry of insulator and susceptor) in a multimodes furnace has been chosen to obtain uniform and controlled heating of the specimens. This setup allows heating under Ar/4%-H_2, and a specific partial pressure of oxygen, as requested for UO_2 and MOX sintering. The results will be used to evaluate the feasibility of nuclear ceramics microwave sintering and to look at the future development of an industrial furnace.

Keywords : Microwaves, sintering, nuclear fuels

Flash sintering of zirconia and zirconia-alumina composites

E. Bichaud[1,2], M.C. Steil[1], J.-M. Chaix[2] and C.P. Carry[2]

[1]LEPMI, Univ. Grenoble Alpes, CNRS, F-38000 Grenoble, France

[2]SIMAP, Univ. Grenoble Alpes, CNRS, F-38000 Grenoble, France

Abstract

"Flash sintering" is an ultrafast sintering technique which enables the densification of conductive ceramics in less than 5 seconds, using furnace temperatures far below the usual temperatures required by pressureless sintering. This work aims at understanding this phenomenon. Investigations have been focused on zirconia and zirconia-alumina composites, when a constant electric field is applied. They are based on two types of experiments: constant heating rate and isothermal stage sintering. Particular attention was paid to the effective conductivity of the materials. Using the knowledge on conventional sintering of the studied materials and measurements of the evolution of their conductivity with temperature, it is shown that ultrafast sintering is mainly driven by the Joule power dissipated inside the material. This dissipation, coupled with the thermally activated conductivity, leads to thermal and electrical runaway. A single criterion based the product $\sigma_{eff}.E_0^2$ of the effective electrical conductivity by the square of the electric field amplitude is proposed for determining. The threshold value is determined by the balance between Joule heating and heat losses with the surroundings, and therefore depends on the sample shape and experimental device. This analysis is consistent with the whole set of experimental data, which do not need any specific effects of current and/or electrical field to explain the results, although such effects cannot be excluded.

Keywords : Flash sintering, zirconia, alumina-zirconia microcomposites

Field assisted sintering processing of bioinspired ceramic/metal composites

M. Marcinkowska[1,2], E. Maire[1], S. Meille[1], J. Chevalier[1] and S. Deville[2]

[1]*Université de Lyon, INSA-Lyon, MATEIS CNRS UMR5510, Villeurbanne 84306, France*

[2]*Laboratoire de Synthèse et Fonctionnalisation des Céramiques, UMR3080 CNRS/Saint-Gobain, Cavaillon F-69621, France*

Abstract

Many natural composite materials derive properties much better than the intrinsic properties of their constituents through elaborated architectures with features at multiple length scales. The structure controlled on multiple length scales provides integration of properties that were thought to be mutually exclusive, such as strength and toughness. In this work we fabricated composites consisting of aligned alumina platelets and layers of titanium. Sintering material under pressure with field-assisted sintering allows diffusion of titanium into the ceramic matrix, leading to nearly dense metal/ceramic composites. The materials obtained by this approach exhibit a combination of high stress and strain values. In order to explain the improvement of mechanical properties, ex-situ testing methods were combined with three point SEM in-situ bending tests, observing crack deflection mechanisms, platelets pull-out, and crack bridging. Those mechanisms, often present in natural materials, provide extrinsic toughening mechanisms, revealed by R-curve behavior (increase of toughness as the crack propagates). The emergence of those mechanisms in these material helps understand the role of each component and their contributions to global mechanical performance of the composite.

Keywords : architectured materials, alumina/titanium composites, SPS, mechanical properties

Study of the Influence of Different Dopants on the Densification of Spinel by Microwave Heating

R. Macaigne[1,2]; S. Marinel[1], E. Savary[3], G. Riquet[1], C. Meunier[2], S. Saunier[2], D. Goeuriot[2]

[1]CRISMAT Laboratory, UMR 6508 CNRS-ENSICAEN-UCN, 6 Boulevard Maréchal Juin, 14050 Caen cedex, France

[2]Ecole Nationale Supérieure des Mines de Saint-Etienne, 158 Cours Fauriel, 42023 Saint-Étienne, France

[3]CNRS-Délégation Normandie, 6 Boulevard Maréchal Juin, 14050 Caen cedex, France

Abstract

Transparent magnesium aluminate spinel ceramic presents very interesting optical properties, especially related to its excellent optical transmission from 0.2 to 5 µm, and attractive mechanical properties. Widely used in the refractory industry, this oxide is a promising candidate for optical and mechanical applications for structural applications (ballistic materials for instance). However, obtaining transparent materials requires a total elimination of porosity, and so implies a very good control of the sintering process. In this work, we have investigated the microwave sintering process in a view of getting homogenous $MgAl_2O_4$ microstructure, suitable for producing, after HIP, transparent ceramic material. For doing that, it has been developed a specific instrumentation to measure the shrinkage and the sample temperature during microwave sintering, in single mode 2.45 GHz microwave cavity, and in working carefully on the temperature calibration. In this work, it will be presented the shrinkage curves of the spinel material, being microwave sintered with different dopants (MgO, TiO_2 and ZrO_2). Those dopants were selected to promote densification through solid state sintering, in a view of getting optimal microstructure (low and fine inter-granular porosity/small grain size). In doing so, the specific effects of the dopants are investigated. The dilatometric data are also exploited through different phenomenological models (Master Sintering Curves and Constant Heating rate methods) in order to characterize as far as we can the sintering behavior of the spinel doped materials under microwave, in relation with their microstructures.

Keywords : Microwave Sintering, spinel, Shrinkage, Master Sintering Curve

Microwave joining process of an alumino-silicate ceramic material for radioactive waste containers

G. Kalfayan[1,2], D. Goeuriot[1], S. Saunier[1], C. Meunier[1] and N. Texier-Mandoki[2]

[1]Centre Sciences des Matériaux et des Structures, CNRS-UMR 5307, ENS des Mines de St-Etienne, 158 cours Fauriel, 42023 St-Etienne, France

[2]Andra, 1-7 rue Jean Monnet, 92298 Chatenay-Malabry Cedex

Abstract

As part of the long-term nuclear waste repository project (Cigeo project), Andra has been studying for several years ceramic containers for high activity radioactive wastes, which are alternative solutions to low carbon steel. The issue is to develop sealing process between the lid and the body of alumino-silicate ceramic containers. Microwave heating for joining was chosen because it allows a local and moderate heating. Previous researches determined three potential sealing glasses taking into account some constraints: expansion coefficient close to that of the ceramic, low melting point (<800°C), resistance to lixiviation... Test in gamma radiation resistance are running on for this selection. The aim of the study is to deepen the understanding of interactions between microwaves and matter. During microwave processing, it often happens the need of using a microwave-absorbing material that is placed around the piece and allows the heating at medium temperature, in the case of non-coupling of the ceramic with microwaves. To industrialize the process, such a part should be avoided. So the present study explores the manners to obtain heating in this situation: size of pieces, crucible design, microwave power cycle… The addition of microwave-absorbing powder into glass is also explored; the parameters are the nature, morphology and size of the powders. In each case the joining microstructures are examined, and relations between joining microstructure and mechanical properties are determined.

Keywords : Microwave; joining; glass; alumino-silicate; radioactive waste containers

Ceramic substrates for laser induced selective metallization

Philipp Ninz[1], Frank Kern[1], Eugen Ermantraut[2], Hagen Müller[3], Wolfgang Eberhardt[3], André Zimmermann[2,3] and Rainer Gadow[1]

[1]Institut für Fertigungstechnologie keramischer Bauteile (IFKB), University of Stuttgart, Allmandring 7b, D-70569, Stuttgart, Germany
[2]Institute for Microintegration, University of Stuttgart, Allmandring 9b, D-70569, Stuttgart, Germany
[3]Hahn-Schickard-Institute for Microassembly Technology, Allmandring 9b, D-70569, Stuttgart, Germany

Abstract

Ceramic substrates for power electronic devices offer beneficial properties such as high insulation voltage, high temperature resistance and high thermal conductivity in comparison to polymers. These properties allow for higher application temperatures and higher power densities resulting in a wider application range and more efficient devices. Laser activation of ceramics enables selective electroless metal deposition to produce finest conducting paths for the manufacturing of electronic devices. Additionally the process enables the deposition of conducting paths following three dimensional structures. Metal deposition is influenced by ceramic substrate properties as well as laser type, laser process parameters and metallization parameters. This study instigates the influence of substrate material, microstructural and chemical properties as well as surface properties on the metal deposition process. Alumina, ZTA and 3 Y-TZP ceramics were manufactured by hot pressing, slip casting and injection molding. Sintering was performed in reducing and oxidizing atmosphere. Laser activation was performed on untreated, polished and sand blasted sample surfaces and was carried out by a Nd:YVO_4 laser with a wavelength of 532 nm and a pulse length of 10 ps. Electroless plating was performed with commercially available electrolytes. Laser parameters were evaluated and correlations to substrate material, density and microstructure were analyzed. Investigations show that metal deposition strongly depends on substrate material, microstructure and the density of oxygen vacancies. Zirconia showed poor metallization in all cases. Fine grain Alumina and ZTA substrates sintered under reducing conditions enable good metallization, defect free conducting paths and fine pitch.

Keywords : Ceramics, alumina, zirconia, TZA, ceramic injection molding, hot pressing, slip casting, electroless plating, laser activation, 3D interconnect device.

Monday 05/09/2016 afternoon

14h00 Keynote lecture: Fracture toughness testing of ceramics: state of the art and new methods, by Robert Danzer

R. Danzer and T. Lube

Institut für Struktur- und Funktionskeramik, Montanuniversitaet Leoben, Franz Josef Strasse 18, A-8700, Leoben, Austria

14h30 **Mechanical behavior of a highly transformable zirconia-based composite**

I. Touaiher[1], M. Saädaoui[1], J. Chevalier [2], H. Reveron[2]

[1]Université Mohamed V, EMI, LERSIM, Avenue Ibn Sina, 10000 Rabat, Morocco - [2]Université de Lyon, INSA de Lyon, MATEIS CNRS UMR 5510, 20 Avenue Albert Einstein, F-69621 Villeurbanne Cedex, France

14h45 **Towards mussels under stress: "novel concepts to design tough and reliable advanced ceramics"**

R. Bermejo[1],Y. Chang[2], R. Danzer[1] and G.L. Messing[2]

[1]Institut für Struktur- und Funktionskeramik, Montanuniversitaet Leoben, Franz Josef Strasse 18, A-8700, Leoben, Austria - [2]Department of Materials Science and Engineering, The Pennsylvania State University, 121 SteidleBldg, University Park, PA 16802, USA

15h00 **Assessment of hardness and elasticity by micro depth sensing indentation with knoop indenter**

G. Ben Ghorbal[1], A. Tricoteaux[1],A. Thuault[1], D. Chicot[2], G. Louis[3]

[1]LMCPA, Université de Valenciennes, Boulevard Général de Gaulle, 59600 Maubeuge - [2]Univ. Lille, FRE 3723 - LML - Laboratoire de Mécanique de Lille, F-59000 Lille, France - [3]LGCgE, Ecole des Mines de Douai, 59500 Douai, France

15h15 **ZTA-NbC Dispersion Ceramics – Influence of NbC Content on the Mechanical Properties and the ED-Machinability**

U. Schmitt-Radloff, F. Kern and R. Gadow

IFKB, University of Stuttgart, Allmandring 7b, 70567, Stuttgart, Germany

15h30 Highlight lecture: Surface Processing of Zirconia, by Marc Anglada

Department of Material Science and Metallurgical Engineering. Universitat Politècnica de Catalunya. C/Sant Ramon de Penyafort (EEBE), 08019 Barcelona, Spain

16h00 Coffee break

16h30 Highlight lecture: Ceramics and CMC Research at Swansea University: The Legacy of Brian (George) Wilshire, by Martin Bache

Institute of Structural Materials, College of Engineering, Bay Campus, Swansea University, Swansea, SA1 8EN, United Kingdom

17h00 **Thermo-Mechanical Fatigue Behaviour of a SiC_f/SiC Ceramic Matrix Composite**

J.P. Jones[1], M.T. Whittaker[1], M.R. Bache[1], P.J Doorbar[2], P. Jones[1]

[1]Institute of Structural Materials, Swansea University, Fabian Way, Swansea, SA1 8EN, United Kingdom - [2]Rolls-Royce plc, P.O. Box 31, Derby, DE24 8BJ, United Kingdom

17h15 **Progressive Evaluation of Damage in SiC_f/SiC Ceramic Matrix Composite Specimens via X-Ray Computed Microtomography**

Z. Quiney[1], J.P. Jones[1], P. Doorbar[2] and M.R. Bache[1]

[1]Institute of Structural Materials, Swansea University Bay Campus, Fabian Way, Swansea, SA1 8EN, United Kingdom -[2]Rolls-Royce plc, P.O. Box 31, Derby, DE24 8BJ, United Kingdom

17h30 **Low-cost thermostructural fiber-reinforced composites for mass production**

C. Mingazzini[1], P. Fabbri[1], M. Scafè[1], D. Caretti[2], D. Nanni[2], L. Laghi[3], M. Morganti[3]

[1]ENEA Faenza Research Laboratories, via Ravegnana 186, Faenza (Ra), Italy - [2]Bologna University, Dep. of Industrial Chemistry, Viale Risorgimento 4, Bologna, Italy - [3]CertiMaC, Via Granarolo 62, Faenza (Ra), Italy

17h45 **Fiber-Matrix Interface in Carbon Fiber Reinforced Ceramics and Light Metals via LPI Coatings**

R. Gadow, M. Jiménez, P. Weichand

Institut for Manufacturing Technology of Ceramic Components and Composites, University of Stuttgart, Allmandring 7b, D-70569 Stuttgart Germany

18h00 Poster Session

19h00 Poster/Get together (buffet dinner)

Fracture toughness testing of ceramics: state of the art and new methods

Robert Danzer and Tanja Lube

Institut für Struktur- und Funktionskeramik, Montanuniversitaet Leoben,
Franz Josef Strasse 18, A-8700, Leoben, Austria

Abstract

Fracture toughness characterizes a material's resistance against the propagation of cracks. It is a very important property of ceramics. But – compared to the situation of metals - it is not yet a material parameter which is measured routinely in ceramics characterization and which is rarely used for design.

This is mainly caused by two reasons. First, specimens with geometries like those used for metals fracture toughness testing are much too big, complicated and expensive to be machined from ceramics. Second, fracture toughness testing requires a well-defined and sharp pre-crack in the specimen. In metallic materials it can be obtained by fatigue pre-cracking. This is not possible in ceramics, because they are not highly susceptible to cyclic fatigue. A number of alternative methods for pre-crack generation have been developed, leading to controversial discussions about the validity of the results obtained with these different methods.

The state of the art of fracture toughness measurement techniques for ceramics will be presented and reviewed. Furthermore, new methods that can also be applied to small specimens as they are often needed in material's development, will be discussed.

Mechanical behavior of a highly transformable zirconia-based composite

I. Touaiher[1], M. Saädaoui[1], J. Chevalier[2], H. Reveron[2]

[1]Université Mohamed V, EMI, LERSIM, Avenue Ibn Sina, 10000 Rabat, Morocco

[2]Université de Lyon, INSA de Lyon, MATEIS CNRS UMR 5510, 20 Avenue Albert Einstein, F-69621 Villeurbanne Cedex, France

Abstract

Zirconia toughened ceramics (ZTC) are attractive materials for several medical and engineering applications as they exhibit high strength and toughness compared to other oxide ceramics. These superior properties result from the stress induced tetragonal to monoclinic zirconia phase transformation occurring in these materials. The aim of this work is to study the mechanical properties and the slow crack behavior of a highly transformable Ce-TZP/ 8vol% Al_2O_3/ 8vol% $SrAl_{12}O_{19}$ composite with different grain sizes and stabilizers content. Fracture toughness values of more than 10 MPa.$m^{1/2}$ and 17 MPa.$m^{1/2}$, respectively were obtained, with SEVNB and DT tests. A large transformed zone with a marked autocatalytic phase transformation is observed in the specimens subjected to a double torsion test. The influence of the microstructure and the phase transformation on the slow crack growth behavior is discussed.

Keywords : Slow-crack growth, strength, zirconia composite, phase transformation

Towards mussels under stress: “novel concepts to design tough and reliable advanced ceramics”

Raul Bermejo[1],Yunfei Chang[2], Robert Danzer[1] and Gary L. Messing[2]

[1]*Institut für Struktur- und Funktionskeramik, Montanuniversitaet Leoben, Franz Josef Strasse 18, A-8700, Leoben, Austria*

[2]*Department of Materials Science and Engineering, The Pennsylvania State University, 121 SteidleBldg, University Park, PA 16802, USA*

Abstract

The design of ceramic layered composites with weak or strong interfaces can affect the propagation of surface cracks during external loading. The former approach aims to induce crack deflection, thus increasing the failure resistance of the composite. The latter uses residual compressive stresses to arrest or hinder the propagation of cracks. For practical applications, the design of layered-based structural and/or functional ceramic components often requires strong interfaces to guarantee the functionality of the device. In this work, novel concepts are presented which combine different approaches used in layered architectural design with strong interfaces aiming to obtain highly reliable ceramic materials with high fracture resistance. The use of tailored residual stresses in embedded layers within the structure is demonstrated to act as an effective barrier to the propagation of surface flaws, providing a minimum design strength for the material, below which failure cannot take place. In addition, texturing of the microstructure in embedded layers is utilized to provide preferential paths for conducting propagating cracks, thus protecting the underlying structure. The location and thickness of the layers designed with compressive stresses can be optimized to maximize the crack growth resistance of the multilayer system. A combination of experiments and modelling is presented, showing the potential of layered architectures in the design of future ceramic components with spatially resolved strength and toughness.

Keywords : Hierarchical laminates; residual stresses; textured layers; crack arrest.

Assessment of hardness and elasticity by micro depth sensing indentation with knoop indenter

G. Ben Ghorbal[1], A. Tricoteaux[1], A. Thuault[1], D. Chicot[2], G. Louis[3]

[1]LMCPA, Université de Valenciennes, Boulevard Général de Gaulle, 59600 Maubeuge
[2]Univ. Lille, FRE 3723 - LML - Laboratoire de Mécanique de Lille, F-59000 Lille, France
[3]LGCgE, Ecole des Mines de Douai, 59500 Douai, France

Abstract

Instrumented Knoop indentation is a favorable candidate to investigate mechanical properties of ceramic materials since it is less sensitive to cracking than other indenters. However, it remains little studied since it presents a complex elastic-plastic behavior. The aim of this work is to develop an efficient method to assess mechanical properties of ceramics by instrumented Knoop indentation. Vickers and Knoop tests were carried out with an instrumented micro hardness tester on five different ceramics in order to compare the results between the two methods. Additional measurements of elastic modulus were performed by ultrasonic method to compare the results with those obtained by indentation. It was found that Young's modulus obtained with Knoop indenter is overestimated for all tested materials. Additionally, it was found that Knoop indentation leads to lower hardness numbers than those obtained with Vickers indenter. This is due to the asymmetrical elastic recovery of a Knoop impression occurring along the short axis. This anisotropic elastic recovery is not taken into account in the methodology developed for axisymmetric indenter (as Vickers indenter). An elastic recovery factor based on the measured impressions was proposed in this work to determine the actual plastic area and to evaluate the real hardness and elastic modulus. Knoop indenter can give reliable results of hardness and elastic modulus directly from the loading/displacement curve if the appropriate correction factor is used. Besides, using depth sensing indentation with Knoop indenter can give an idea of elasticity of materials. The Knoop indenter is a favorable candidate to investigate mechanical properties of brittle bulk and thin films materials, particularly heterogeneous materials.

Keywords: Mechanical properties, ceramics, Knoop indentation

ZTA-NbC Dispersion Ceramics – Influence of NbC Content on the Mechanical Properties and the ED-Machinability

U. Schmitt-Radloff, F. Kern and R. Gadow

IFKB, University of Stuttgart, Allmandring 7b, 70567, Stuttgart, Germany

Abstract

ZTA ceramics are well known for their good mechanical properties such as high hardness and bending strength and moderate fracture resistance. By adding an additional electrically conductive phase, such as a transition metal carbide, ceramics can be machined by electrical discharge machining (EDM). In this study the influence of NbC content on the mechanical and electrical properties of electrically conductive ZTA-NbC ceramics was investigated. Therefore the NbC content of the ceramics was varied in 2 vol-% increments between 24 and 32 vol -%. The composition of sub-micron sized ZTA matrix with 17 vol. % zirconia and a stabilizer contend of 1.0 mol-% yttria was kept constant. The starting powders were alloyed by attrition milling. Samples were hot pressed at 1525°C for 2h at 40MPa pressure and prepared for mechanical characterization by lapping and polishing. Hot pressed samples were mechanically characterized in terms of of Young´s modulus, density, Vickers hardness, 4-point bending strength and fracture resistance by ISB- and direct crack length measurement method. It was found that the NbC content effects mechanical properties such as fracture resistance, bending strengh and hardness. A maximum fracture resistance of 5 MPa√m was found at a NbC content of 32 vol-%. Hardness exceeds 1750 HV10 with a maximum of 1930 HV10 at 28 vol-% NbC. Highest 4p-bending strength of 750 ± 80 MPa was found at 28 vol- % NbC.
Electrical conductivity was determined by using the 4 point measurement method. All samples were machined by Wire Electrical Discharge Machining. Surface morphologies and surface roughness of the machined surfaces were studied by SEM and white light interferometry. The cutting speed was determined for benchmarking the ED-machinability of the ZTA-NbC ceramics.

Keywords : EDM, ZTA-NbC, Mechanical Properties.

Surface Processing of Zirconia

Marc Anglada

Department of Material Science and Metallurgical Engineering.
Universitat Politècnica de Catalunya.

Abstract

In the last decades, there has been growing interest in developing ceramic materials with high fracture toughness and strength for structural applications. In particular, zirconia based ceramics have attracted interest because they possess an excellent combination of these two mechanical properties, together with high wear and corrosion resistance, high hardness, low thermal conductivity and excellent biocompatibility. Thus, 3 mol % yttria-doped tetragonal zirconia (3Y-TZP) is the single oxide ceramic with the highest strength and is being used to manufacture several different implants for the human body, from dental crowns and frameworks in dentistry, to femoral heads for total hip replacements.

The high strength is a result of its nearly nanometric grain size (a few hundred of nanometres) and its relatively good fracture toughness, K_{Ic}. This property can be increased by changing the stability of the tetragonal phase at room temperature with the objective of controlling the extent of phase transformation from tetragonal (t) to monoclinic (m) in front of the crack tip (transformation toughening). However, increasing toughening by t-m transformation makes easier the spontaneous t-m transformation of the external surface in contact with moisture present in the environment; such effect is known as hydrothermal degradation or low temperature degradation (LTD) and is accompanied by surface microcracking. These two phenomena, which may not be independent, are the main drawbacks for the wider use of 3Y-TZP and should be considered in the design of prosthesis in contact with body fluids.

As LTD takes place from the surface to the interior one straight approach to try to solve the problem of LTD is to process or modify the zirconia surface in a way compatible with the requirements of the final application order to reach a higher resistance to LTD. In doing so the transformability of the internal parts could be also increased and overall higher fracture toughness achieved.
Different approaches for increasing the resistant to LTD of the surface of 3Y-TZP in which the research group has been working in the last years will be presented and discussed. These include grinding and heat attack, sandblasting, diffusion of Ce, pre-sintering + infiltration, HF attack plus infiltration and laser patterning.

Ceramics and CMC Research at Swansea University: The Legacy of Brian (George) Wilshire

M.R. Bache

Institute of Structural Materials, Swansea University, Swansea, SA1 8EN, United Kingdom

Abstract

Research into the structural integrity of ceramic systems at Swansea University has gained International recognition since the 1970s through the pioneering studies of Brian Wilshire. After establishing a fundamental understanding of creep mechanisms in metallic alloys and the development of associated lifing methodologies, Brian turned his attention to the demands of high temperature characterisation of monolithic ceramics, followed by interest in more advanced ceramic matrix composites. Throughout, his research was recognised by major multi-national sponsors in the materials processing and aerospace sectors in addition to recognition from academic peers. Ever since the incorporation of Wales into the CIEC "family" Brian was a fervent supporter of the network, using the biennial CIEC meeting as a regular vehicle for dissemination and the opportunity to renew professional and social friendships with some of Europe's leading exponents of ceramics research.

The current review will serve as a retrospective of his work focussed upon the mechanical characterisation of ceramics employed for high performance industrial applications. Deformation, damage accumulation and fracture in a range of silicon carbide based materials, including monolithic forms through to fibre reinforced ceramic matrix composites incorporating various fibre variants, will be presented. The impact of this previous research on current creep and fatigue assessments of the latest generation CMCs destined for aero-engine components will then be discussed.

Keywords : Fibre reinforced composites; Creep; Deformation; Fracture; Applications

Prof Brian Wilshire OBE FREng

Thermo-Mechanical Fatigue Behaviour of a SiC_f/SiC Ceramic Matrix Composite

J.P. Jones[1], M.T. Whittaker[1], M.R. Bache[1], P.J Doorbar[2], P. Jones[1]

[1]*Institute of Structural Materials, Swansea University, Fabian Way, Swansea, SA1 8EN, United Kingdom*

[2]*Rolls-Royce plc, P.O. Box 31, Derby, DE24 8BJ, United Kingdom*

Abstract

The structural integrity of silicon carbide fibre reinforced silicon carbide ceramic matrix composites (SiC_f/SiC), when employed for components in a gas turbine engine, is intimately controlled by the evolution of damage through the combination of thermo-mechanical loading and environmental degradation. In particular, the ingress of oxygen and/or water vapour from the ambient atmosphere or internal gas stream may lead to failure of the critical load bearing fibre constituents. To simulate these representative operating conditions in the laboratory can pose significant technical challenges. A state of the art test facility has been developed, incorporating an innovative radiant lamp furnace with non-invasive thermography temperature control. The load controlled LCF system is capable of delivering thermal ramp rates up to 25°C/s^{-1} whilst accurately controlling temperature of the entire gauge section and monitoring strain through side mounted extensometery.

Utilising this facility, the thermomechanical fatigue properties of a CMC material were evaluated employing a 550-800°C temperature cycle and compared to conventional, isothermal low cycle fatigue data generated at the extreme temperatures of the TMF cycle. A detailed three-dimensional assessment of the specimen gauge section both pre and post testing was undertaken using computed tomography (CT) in order to identify sites of damage accumulation. Detailed fractography using optical and scanning electron microscopy (SEM) allowed for correlation between inherent processing artefacts within the composite architecture and subsequent crack initiation sites and crack growth paths.

Keywords : Thermo-mechanical Fatigue, Ceramic, Thermography

Progressive Evaluation of Damage inSiC_f/SiC Ceramic Matrix Composite Specimens via X-Ray Computed Microtomography

Z. Quiney[1], J.P. Jones[1], P. Doorbar[2] and M.R. Bache[1]

[1]*Institute of Structural Materials, Swansea University Bay Campus, Fabian Way, Swansea, SA1 8EN, United Kingdom*

[2]*Rolls-Royce plc, P.O. Box 31, Derby, DE24 8BJ, United Kingdom*

Abstract

A detailed understanding of fatigue damage evolution in SiCf/SiC ceramic matrix composite (CMC) is required if this class of material is to be utilised for advanced engineering components. Although several established techniques allow damage accumulation to be monitored *in situ*, it is challenging to correlate such measurements with precise quantities of internal damage and their form without employing additional, potentially destructive, inspection techniques.

To this end, high resolution X-ray computed microtomography (µCT) was employed to periodically inspect compact tension-type SiC_f/SiC CMC specimens subjected to incremental fatigue cycles in laboratory experiments. Using electrical resistance measurements as the primary monitoring technique, alongside a secondary acoustic emission system, specimens were cycled until a significant change in the resistance value was encountered. At this stage the specimen was removed from the test frame, inspected via µCT to quantify the internal damage, before being returned to the frame for continuation of this process, repeated multiple times until ultimate failure. Progressive changes in resistance and acoustic emission measurements were correlated with internal damage, in particular crack extension. These experiments represent a transition from traditional pre and post-test specimen NDE towards full *in situ* X-ray CT monitored fatigue techniques.

Keywords: Ceramic Matrix Composites (CMCs), X-Ray Computed Microtomography (µCT), Non-Destructive Evaluation (NDE), Structural Health Monitoring (SHM), Damage Progression.

Low-cost thermostructural fiber-reinforced composites for mass production

C. Mingazzini[1], P. Fabbri[1], M. Scafè[1], D. Caretti[2], D. Nanni[2], L. Laghi[3], M. Morganti[3]

[1]ENEA Faenza Research Laboratories, via Ravegnana 186, Faenza (Ra), Italy

[2]Bologna University, Dep. of Industrial Chemistry, Viale Risorgimento 4, Bologna, Italy

[3]CertiMaC, Via Granarolo 62, Faenza (Ra), Italy

Abstract

Basalt reinforced inorganic composites prepared by using Preceramic Prepregs represent a potentially interesting solution for thermostructural applications in transports and constructions up to 800°C, being both cheap and environmentally friendly. For temperature higher than 800°C (e.g. thermal and acoustic insulation fire resistant panels), higher thermostructural properties can be achieved only by exploiting ceramic fibers, with the remarkable disadvantage of costs still too high for mass production. Continuously increasing use of SiC fibers for engine aeronautical applications may, in prospect, reduce ceramic fibers costs, but meanwhile carbon fibers are the only affordable fiber type which may guarantee thermostructural properties up to 1200-1300°C, in incidental conditions (e.g. fire). Carbon fibers weak point is, of course, they cannot withstand oxidant environments above 400°C, but this limitation may be overcome developing suitable multimaterial solutions, where carbon fibers are protected from oxidation. The combination of lamination, warm pressing and use of and Preceramic Prepregs is being studied aiming at affordable processes for high-volume productions: thermomechanical and thermophysical properties (thermal conductivity in particular) may be changed over a wide range, while lamination ensures the possibility to produce both simple and complex shapes. Oxidations tests at various temperatures and subsequent residual mechanical properties are reported. Thermostructural compression tests were also performed.

Keywords : Continuous Fiber Ceramic Composites (CFCC); Preceramic Prepregs; Filament winding

Fiber-Matrix Interface in Carbon Fiber Reinforced Ceramics and Light Metals via LPI Coatings

R. Gadow, M. Jiménez, P. Weichand

Institut for Manufacturing Technology of Ceramic Components and Composites, University of Stuttgart, Allmandring 7b, D-70569 Stuttgart Germany

Abstract

Carbon fibers are an outstanding reinforcement for polymer matrices and moreover are successfully employed in CMC and MMC applications due to their excellent creep and long-term thermochemical and thermomechanical stability. Nevertheless, their poor oxidation resistance requires a suitable protection of the filaments before being exposed to temperatures over 350 °C in air. This fact, in addition to the necessary adjustment of the fiber-matrix interface regarding chemical compatibility and adhesion with ceramics and light metals matrices, justifies the deposition of tailored ultra-thin protective ceramic coatings as technical solution. The continuous liquid polymer impregnation technique (LPI) with subsequent pyrolysis, which applies a Polymer-Derived Ceramic layer on the desized surface of every single carbon fiber filament from coil to coil, is a cost-effective fiber coating alternative to state of the art chemical vapor deposition (CVD) methods. The existing LPI coating plant at the IMTCCC was therefore modified with two main objectives: enhancing its flexibility and reducing the mechanical and thermal stresses, coated fibers suffer during the process. A new concept of coating reactor based on state of the art roving impregnation techniques was introduced and tested. Homogenous ceramic layers derived from phenolic, polysiloxane and polysiloxazane-based resins were deposited. A reduction of the fiber friction in the plant was achieved and proven by mechanical characterization of the coated single filaments. The coated rovings were implemented in SiOC matrices enabling an improvement of tenacity in comparison to the uncoated fibers.

Keywords : Liquid polymer impregnation, ceramic coatings, carbon fibers.

Tuesday 06/09/2016 morning

08h30 Highlight lecture: Processing of Electroactive Components by means of Selective Laser Sintering / Melting (SLS/SLM), by Maurice Gonon

M. Gonon, N. Basile
Materials Institute, UMONS, Rue de l'Epargne 56, 7000, Mons, Belgium

09h00 **Evidence of microwave effects on the sintering of alpha and gamma alumina powder**

J. Croquesel[1], D. Bouvard[1], J.M. Chaix[1], C.P. Carry[1] and S. Saunier[2]
[1]Univ. Grenoble Alpes, CNRS, SIMAP, 38000 Grenoble, France - [2]Ecole des Mines de Saint-Etienne, SMS team, 158 cours Fauriel, 42023 Saint Etienne, France

09h15 **Investigating influences of suspension plasma spray parameters on photocatalytic activity of TiO_2 films**

T.P. Vu[1], N. Otto[2], A. Vogel[1], F. Kern[1], A. Killinger[1], R. Gadow[1]
[1]IFKB, University of Stuttgart, Allmandring 7b, 70569, Stuttgart, Germany - [2]ISWA, University of Stuttgart, Bandtäle 2, 70569, Stuttgart, Germany

09h30 **Reaction bonded boron carbide composite fabricated by microwave heating**

M. Dutto[1,3], D. Goeuriot[1], S. Saunier[1], S. Marinel[2], N. Frage[3] and S. Hayun[3]
[1]Département Mécanique et Procédés d'Elaboration directe, Centre des Science des Matériaux et des Structures, Laboratoire Georges Friedel UMR 5307, Mines Saint-Etienne, 42023 Saint-Etienne Cedex 2, France - [2]CRISMAT Laboratory UMR 6508 CNRS-ENSICaen-UCBN, 6 Boulevard du Maréchal Juin, 14050 Caen Cedex, France - [3]Departement of Materials Engineering, Ben-Gurion University of Negev, P.O. Box 653, Beer-Sheva 84105, Israel

09h45 **Micropatterning of calcium phosphate bioceramics by femtosecond laser**

M. Lasgorceix[1], C. Ott[1], L. Boilet[1], S. Hocquet[1], S. Chamary[2], A. Leriche[2], V. Lardot[1], F. Cambier[1]
[1]CRIBC, 4 Avenue Gouverneur Cornez, 7000 Mons, Belgium - [2]LMCPA, Boulevard Charles de Gaulle, 59600 Maubeuge, France

10h00 **Microstructural damage in zirconia ceramics by laser patterning**

E. Jiménez-Piqué, E. Roitero, A. Bouzo, M. Ochoa, M. Turón, J.J. Roa and M. Anglada
Department of Material Science and Metallurgical Engineering. Universitat Politècnica de Catalunya. C/Sant Ramon de Penyafort (EEBE), 08019 Barcelona, Spain

10h15 **Low temperature degradation of laser patterned zirconia (3Y-TZP)**

E. Roitero[1,2,3], M. Ochoa Sánchez[1], F. Soldera[3], F. Mücklich[3], M. Anglada[1,2], E. Jimenez-Pique[1,2]
[1]Department of Materials Science and Metallurgical Engineering, Universitat Politecnica de Catalunya, Av. Diagonal 647, Barcelona 08028, Spain - [2]Center for Research in Nanoengineering, CRnE, Univerisitat Politècncica de Catalunya, C. Pascual i Vila 15, Barcelona 08028, Spain - [3]Department of Material Science and Engineering, Saarland University, Saarbrucken 66123, Germany

10h30 Coffee break

11h00 Keynote lecture: Zirconia in Dentistry, by Corrado Piconi

Institute of Clinical Orthopedics and Traumatology, Catholic University Sacro Cuore, 240 via Pasquale Ii, 00168, Rome, Italy

11h30 **Evaporation of phosphate groups during calcium phosphate sintering**

N. Döbelin, M. Bohner

RMS Foundation, Bischmattstrasse 12, 2544, Bettlach, Switzerland

11h45 **Suspension flame spraying of bioceramic coatings with antibacterial properties**

P. Krieg[1], R. Gadow[1], A. Killinger[1] and A. Bernstein[2]

[1]Institute for Manufacturing Technologies of Ceramic Components and Composites (IMTCCC), University of Stuttgart, Allmandring 7b,70569 Stuttgart, Germany - [2]Musculoskeletal research lab, Department of Surgery, Clinics of Orthopedics and Trauma Surgery, University of Freiburg - Medical Centre, Hugstetter Straße 55, 79106 Freiburg, Germany

12h00 **3D Printed Hardystonite-Chitosan Scaffolds for Bone Regeneration**

H. Elsayed[1], P. Colombo[1,2], S. Ramirez[3,4], S. Tadier[3], L. Gremillard[3], A. Montembault[4], L. David[4], T. Delair[4]

[1]Department of Industrial Engineering, University of Padova, via Marzolo 9, 35131 Padova, Italy - [2]Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA 16801, USA - [3]Université de Lyon, INSA-Lyon, Université Claude Bernard Lyon 1, MATEIS CNRS UMR 5510, 69621 Villeurbanne Cedex, France - [4]Université de Lyon, Ingénierie des Matériaux Polymères, IMP@Lyon1, CNRS UMR 5223, Université Claude Bernard Lyon 1, 69622 Villeurbanne Cedex, France

12h15 **Mineralization process of chitosan hydrogels by calcium phosphate apatite**

S. Ramirez[1,2], L. David[1], T. Delair[1], E. Maire[2], A. Montembault[1], S. Tadier[2], L. Gremillard[2]

[1]Université de Lyon, Ingénierie des Matériaux Polymères, IMP@Lyon1, CNRS UMR 5223, Université Claude Bernard Lyon 1, 69622 Villeurbanne Cedex, France - [2]Université de Lyon, INSA-Lyon, Université Claude Bernard Lyon 1, MATEIS CNRS UMR 5510, 69621 Villeurbanne Cedex, France

12h30 **Novel bioceramic structures for intelligent neuroimplantation**

R. Gadow, F. Kern, A. Killinger

Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Allmandring 7b, D-70569 Stuttgart, Germany

12h45 Lunch

Processing of Electroactive Components by means of Selective Laser Sintering / Melting (SLS/SLM)

M. Gonon, N. Basile

Materials Institute, UMONS, Rue de l'Epargne 56, 7000, Mons, Belgium

Abstract

The consolidation of ceramics powder by SLS/SLM offers numerous advantages over conventional sintering: parameters optimized to local conditions, low energy consumption, reduced processing time... Two categories of treatments can be identified according to the selectivity required. When using a defocused beam the entire area to be treated is simultaneously irradiated. Times of treatments of several minutes are compatible with diffusion mechanisms. The control of the temperature and homogeneity are easy but selectivity is poor. A high selectivity is obtained by scanning a surface with a focused beam. Control of temperature levels and gradients is difficult and strongly depends on the scan conditions. Moreover times of treatment are very short. This presentation aims at showing the interest at using focused beam SLS/SLM for processing electroceramics encountered in electronic components. To date, the main technique used for manufacturing those components, combines screen-printing of the compounds and conventional sintering. As, all the materials constituting a component have different thermal requirement compromises limiting the performances have to be made. SLS/SLM appears therefore as promising alternative.

The literature review highlights that most of the researches on this topics used defocused laser beam or applies a focused beam for realizing dots or lines patterning. Rare are the examples of treatments using a focused beam for processing an extended area. This presentation will show two examples of researches carried at UMONS on that topic: the melting and crystallization of piezoelectric glass ceramic from a glass powder coating; the selective laser sintering of a $BaTiO_3$ powder coating.

Keywords : Laser, sintering, electroceramics.

Evidence of microwave effects on the sintering of alpha and gamma alumina powder

J. Croquesel[1], D. Bouvard[1], J.M. Chaix[1], C.P. Carry[1] and S. Saunier[2]

[1]Univ. Grenoble Alpes, CNRS, SIMAP, 38000 Grenoble, France

[2]Ecole des Mines de Saint-Etienne, SMS team, 158 cours Fauriel, 42023 Saint Etienne, France

Abstract

Although many research works have been conducted on microwave sintering of ceramics, the reasons for specific microwave effects on sintering and even the existence of such effects is still under debate. This is because most of the research works lack systematic and valuable comparison between conventional and microwave sintering and they use limited or inaccurate experimental data. In this context, direct microwave sintering of pure α and γ submicronic alumina powders has been investigated in a 2.45 GHz single mode cavity furnace allowing for an accurate control of the thermal cycle and for a continuous measurement of specimen dimensional changes, so that direct comparison with conventional sintering can be achieved. Special attention has been paid on the influence of particle grain size and of the γ to α phase transformation occurring during heating of γ alumina powder on the sintering behaviour. Experimental data unequivocally showed a significant effect of microwaves on α powders (lower densification starting temperature and smaller activation energy) and on γ powders (lower phase transformation temperature). However the sintering trajectory in grain size vs density diagram is similar in conventional and microwave sintering. Therefore microwave heating was not found to be beneficial for obtaining dense alumina with very fine grains. Microwave effects have been explained through the ponderomotive force induced by the electromagnetic field and acting on diffusion and phase transformation mechanisms.

Keywords : Sintering, Microwaves, Alumina

Investigating influences of suspension plasma spray parameters on photocatalytic activity of TiO2 films

T.P. Vu[1], N. Otto[2], A. Vogel[1], F. Kern[1], A. Killinger[1], R. Gadow[1]

[1]IFKB, University of Stuttgart, Allmandring 7b, 70569, Stuttgart, Germany

[2]ISWA, University of Stuttgart, Bandtäle 2, 70569, Stuttgart, Germany

Abstract

In recent studies, the suspension plasma spray technology (SPS) was shown as a promising method for deposition of photocatalytic TiO_2 coatings. However, only little information about the effects of SPS process parameters on the resulting photocatalytic activity is available. In this study, several suspensions were prepared from different powders and various dispersion mediums successively sprayed. Four titania powders with a proven photocatalytic track record, Evonik P25, Kronoclean 7050, Hombicat UV100 and Sigma Aldrich were used to study the influence of starting powder type. Furthermore, the composition of the dispersing medium, substrate activation and the thickness of coating layer were varied in order to investigate their influence on coating structure, adhesion to substrate and photocatalytic activity. XRD, SEM, optical microscopy and interferometry were used to characterize the properties of the coatings. Degradation of methylene blue was tested as a marker for the photocatalytic activity. At the chosen parameters for plasma spraying (500 A, 40 l/min Ar and 8 l/min H_2), alcohol and water in a ratio of 1:3 was identified as the best composition. Suspensions sprayed with higher alcohol content suffered from the reduction in anatase phase; higher water content reduced the coating adhesion to the substrate. Regarding the initial powders, films made of Evonik P25 and Sigma Aldrich TiO_2 had higher photocatalytic activity than others. While the activation of substrate had an influence on photocatalytic activity of the coating layer, thickness of the coating did not affect to the activity.

Keywords : titania, suspension plasma spray, photocatalysis, water treatment

Reaction bonded boron carbide composite fabricated by microwave heating

Mathieu Dutto[1,3],Dominique Goeuriot[1], Sébastien Saunier[1], Sylvain Marinel[2], Nachum Frage[3] and Shmuel Hayun[3]

[1]Département Mécanique et Procédés d'Elaboration directe, Centre des Science des Matériaux et des Structures, Laboratoire Georges Friedel UMR 5307, Mines Saint-Etienne, 42023 Saint-Etienne Cedex 2, France

[2]CRISMAT Laboratory UMR 6508 CNRS-ENSICaen-UCBN, 6 Boulevard du Maréchal Juin, 14050 Caen Cedex, France

[3]Departement of Materials Engineering, Ben-Gurion University of Negev, P.O. Box 653, Beer-Sheva 84105, Israel

Abstract

Boron Carbide is an attractive material for various applications that require high hardness and neutron absorption. Boron carbide is usually fabricated by hot pressing at temperatures above 2000 °C. The production cost is a major drawback for widespread applications in the fields of defense and nuclear energy. An alternative approach for the fabrication of boron carbide based ceramics is the reaction bonding process that allows significantly reduces the production cost. According to this approach a porous B_4C preform is infiltrated by molten Si or its alloys. This technique was extensively studied using conventional heating, but only limited works on the microwave heating were conducted. In the present study the microstructure and mechanical properties of the reaction bonded B_4C composites (RBBC) using microwave (2.45 GHz) heating will be presented. The experimental results will be discussed and compared with the RBBC fabricated by the conventional approach of heating.

Keywords : Microwaves heating, Boron Carbide, Reaction bonding

Micropatterning of calcium phosphate bioceramics by femtosecond laser

Marie Lasgorceix[1], Cédric Ott[1], Laurent Boilet[1], Stéphane Hocquet[1],
Shaan Chamary[2], Anne Leriche[2], Véronique Lardot[1], Francis Cambier[1]

1 CRIBC, 4 Avenue Gouverneur Cornez, 7000 Mons, BELGIUM
2 LMCPA, Boulevard Charles de Gaulle, 59600 Maubeuge, FRANCE

Abstract

Surface topography of synthetic bone implants is an essential factor which impacts their bioactivity, especially by the presence of micro-patterns likely to generate cells growth guidance. In this study, laser machining technology was employed in order to obtain controlled regular micro-patterns on dense calcium phosphate surfaces, without any contamination. According to literature, many studies with laser micropatterning are performed on metallic alloys, but there are few works dealing with calcium phosphate ceramics because of the unwanted phase transformations induced by the thermal impact of such a process. In the present study, the choice of the source was directed towards a femtosecond pulsed laser, to limit this thermal impact. Substrates with perfectly controlled micropatterning and without any secondary phase were obtained by optimization of the process parameters (laser power, scanning speed, pulse frequency). The phase analyses were performed by XRD and the microstructural characteristics of the micro-patterned surfaces were investigated by microscopy methods (optical, confocal, scanning electron). The benefits of the surface laser treatment on wettability was shown by contact angle assays performed with water and simulated body fluid. Relationships between surface topography and wettability mechanisms were established thanks to a wide variety of micropatterned designs, allowed by the precision of the femtosecond laser process. This technique seems to provide an interesting alternative to conventional surface treatments of calcium phosphates. In order to demonstrate the influence of the micro-patterns on cell behavior, *in vitro* experiments are currently being performed.

Keywords : Calcium phosphate bioceramics; Micropatterning; Femtosecond laser

Microstructural damage in zirconia ceramics by laser patterning

E. Jiménez-Piqué, E. Roitero, A. Bouzo, M. Ochoa, M. Turón, J.J. Roa and M. Anglada

Department of Material Science and Metallurgical Engineering. Universitat Politècnica de Catalunya. C/Sant Ramon de Penyafort (EEBE), 08019 Barcelona (SPAIN)

Abstract

Laser patterning of ceramics is being increasingly used to modify the topography of the surface with the aim of changing the performance of the surface in-service conditions, such as biological response, tribological resistance or the mechanical adhesion enhancement to other materials (e.g. porcelain veneer in dental replacements). This patterning is in the order of 0.1-10 μm in depth and can be made as lines, arrays of spots or other geometries, generating a wide collection of different surface treatments. In addition to its flexibility in patterning, the use of laser is relatively fast and inexpensive. However, during the same process of producing patterning by laser, damage can be introduced in the material. This damage is generally small in size and it is generally ignored. However, ceramics are sensible to damage, and this can affect the reliability of the material, especially after time in aggressive environments.

In this work, we have generated different laser treatments on three zirconia-based ceramics: 3Y-TZP, 12Ce-TZP and Alumina/zirconia composites, using a 355-nm UV ns-pulsed laser, in order to study the damage induced as a function of different laser parameters. Ablation thresholds and topographical changes are recorded as a function of energy per pulse and number of pulses. After laser treatment, the microstructure of the surfaces are characterized by laser scanning optical microscopy, FIB/SEM, DRX and nanoindentation, observing phase transformation, microstructure, microcracks and voids for different laser irradiation conditions. Strength tests are also conducted in selected samples. Obtained results show that laser patterning of surfaces is a sound method to generate controlled topographical features, but at the same time can induce microstructural damage in ceramics due to the high temperature gradient and the formation crystalline defects, which affects the structural integrity of the material.

Keywords : Zirconia, laser, damage

Low temperature degradation of laser patterned zirconia (3Y-TZP)

E. Roitero[1,2,3], M. Ochoa Sánchez[1], F. Soldera[3], F. Mücklich[3], M. Anglada[1,2], E. Jimenez-Pique[1,2]

[1]Department of Materials Science and Metallurgical Engineering, Universitat Politecnica de Catalunya, Av. Diagonal 647, Barcelona 08028, Spain.

[2]Center for Research in Nanoengineering, CRnE, Univerisitat Politècncica de Catalunya, C. Pascual i Vila 15, Barcelona 08028, Spain.

[3]Department of Material Science and Engineering, Saarland University, Saarbrucken 66123, Germany.

Abstract

Doped Zirconia ceramics, especially 3Y-TZP, are being increasingly used in dental applications because of their good mechanical properties (high strength and hardness), bio-inertness and aesthetic outcome. There has been a growing interest in modifying the surface topography in order to influence the biological response to these biomaterials. Among the available methods, laser patterning is one of the most promising because it is a non-contact and therefore not-contaminating technique and it allows great precision. In particular, Direct Laser Interference Patterning (DLIP) allows patterning at the micrometric- and nanometric-scale in a single-step process. Studies on the long term behaviour of laser-treated 3Y-TZP have not yet been performed. This is an issue of paramount importance for 3Y-TZP for biomedical application since it is highly sensitive to Low Temperature Degradation (LTD). Therefore, the objective of this work is to characterize the long-term behaviour of zirconia treated with DLIP and its resistance to LTD.

In this work, the third harmonic of a Q-switched Nd:YAG laser (355nm, 10ns, 10Hz) was employed to pattern the surface of zirconia discs with stripes with a periodicity of 10 μm. Accelerated tests to simulate LTD were performed and the aging kinetics of the laser-treated material was evaluated with XRD and SEM on FIB cross-sections, monitoring the monoclinic-phase content with time.

Results show that DLIP of the surface of 3Y-TZP accelerates the aging kinetic. This originates in the collateral damage produced by laser: micro-cracking, residual stresses, pores and point-defects. Ageing kinetics are compared for different laser parameters and results are discussed in relationship with the damage produced.

Keywords : Zirconia, Laser, Low Temperature Degradation

Zirconia in Dentistry

Corrado Piconi

Institute of Clinical Orthopedics and Traumatology, Catholic University Sacro Cuore, 240 via Pasquale Ii, 00168, Rome, Italy.

Abstract

Zirconia (Y-TZP) was introduced in dentistry by Prof. Sami Sandhaus, who on 1980 started implanting the SIGMA implant (Incermed, Lausanne, CH), that is still in clinical use so far. This zirconia fixture-and-abutment device replaced the alumina dental implants CBS and Cerasand that he developed and used since 1968. So far, zirconia dental implants are manufactured by many companies worldwide, most of them in Germany and Switzerland. In addition, several models of zirconia abutments for titanium fixtures are in clinical use. Besides implants, zirconia is used in the production of blanks for the production by CAD/CAM of the structural part of crowns, bridges, and of full arch dentures also over implants. Two other applications of zirconia in dentistry are endodontic posts and orthodontic brackets. The rationale for using zirconia is not only due to its biological safety and mechanical behavior, but mainly to aesthetics, a factor that is substantial in dental restorations. Namely, using zirconia the dark appearance of gingival tissues surrounding the fixture is avoided, as well as the marginal black lines appearing in case of soft tissue retraction. Moreover, there are evidences of less bacterial adhesion and of plaque formation on zirconia, A further factor driving the growth of the use of zirconia in dentistry is the increasing number of patients complaining for sensitization to metals, then concerned about implanting titanium fixtures. The success of zirconia is fostering the development of composite materials for dental load-bearing applications which are presently in an advanced development stage.

Keywords Zirconia, dentistry, implants, composites

Evaporation of phosphate groups during calcium phosphate sintering

Nicola Döbelin, Marc Bohner

RMS Foundation, Bischmattstrasse 12, 2544, Bettlach, Switzerland

Abstract

The chemical reactivity of α- and β-tricalcium phosphate (α- or β-$Ca_3(PO_4)_2$; α- or β-TCP) can be significantly reduced by a thermal treatment at T > 450°C. This result has been attributed to a decrease of the density of surface defects. However, recent data has revealed that a thermal treatment at 500°C provokes a 20-30% increase of the Ca/P and O/P ratios at the β-TCP surface, suggesting the formation of a calcium oxide layer. The aim of the present study was to shed some light on these results by measuring the surface composition of pressed pellets of either monetite (DCP; $CaHPO_4$) or calcium deficient hydroxyapatite powders (CDHA; $Ca_{(10-x)}(HPO4)_x(PO4)_{(6-x)}(OH)_{(2-x)}$ where x = 0.4) after a thermal treatment at 1100°C. For that purpose, 3 pellets per material were pressed, piled on top of each other, sintered, and the inner and outer pellet surfaces were analyzed by Rietveld refinement of X-ray diffraction (XRD) patterns. Up to 40% β-TCP was detected on the surface of sintered DCP pellets (rest: calcium pyrophosphate; $Ca_2P_2O_7$), whereas only 2% could be found on the inner surface of the top pellet. The CDHA converted to a mixture of β-TCP and hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$). A slight but significant increase of the Ca/P molar ratio was observed at the outer CDHA surface when these pellets were calcined alone in the furnace, whereas a slight but significant decrease was observed when these pellets were calcined in the presence of monetite, suggesting that phosphate evaporation plays an important role during calcium phosphate sintering.

Keywords : Sintering, calcium phosphate, bone substitute

Suspension flame spraying of bioceramic coatings with antibacterial properties

Peter Krieg[1], Rainer Gadow[1], Andreas Killinger[1] and Anke Bernstein[2]

[1]Institute for Manufacturing Technologies of Ceramic Components and Composites (IMTCCC), University of Stuttgart, Allmandring 7b,70569 Stuttgart, Germany

[2]Musculoskeletal research lab, Department of Surgery, Clinics of Orthopedics and Trauma Surgery, University of Freiburg - Medical Centre, Hugstetter Straße 55, 79106 Freiburg, Germany

Abstract

Degradable bioceramic coatings offer a faster osseointegration of endoprosthetic implant devices in the body and can be manufactured through various processing technologies, such as thermal spraying. Among the thermal spraying techniques, a novel way to produce these bioactive coatings is the High Velocity Suspension Flame Spraying (HVSFS). A common problem that occurs after the implantation is the risk of an inflammation in the body which can result in the need of a second operation or even the removal of the implant. In a novel approach, metal ions with known antibacterial properties, such as silver (Ag) or copper (Cu), are incorporated in the suspension flame sprayed coatings to reduce the risk of inflammation. In this study, coatings with these metallic phases produced of hydroxyapatite, tricalcium phosphate, bioglass and a calcium potassium sodium phosphate are evaluated regarding their microstructure and phase composition, as well as their in-vitro behavior.

Metallic particles, as well as metal oxides, were successfully incorporated in the coatings and detected using micro-Raman and scanning electron microscopy. The in-vitro characterization shows no cytotoxicity, despite the presence of metals and metal oxides.

Keywords : Bioceramic coating, antibacterial, thermal spraying

3D Printed Hardystonite-Chitosan Scaffolds for Bone Regeneration

Hamada Elsayed[1], Paolo Colombo[1,2], Silvia Ramirez[3,4], Solène Tadier[3], Laurent Gremillard[3], Alexandra Montembault[4], Laurent David[4], Thierry Delair[4]

[1]Department of Industrial Engineering, University of Padova, via Marzolo 9, 35131 Padova, Italy

[2]Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA 16801, USA

[3]Université de Lyon, INSA-Lyon, Université Claude Bernard Lyon 1, MATEIS CNRS UMR 5510, 69621 Villeurbanne Cedex, France

[4]Université de Lyon, Ingénierie des Matériaux Polymères, IMP@Lyon1, CNRS UMR 5223, Université Claude Bernard Lyon 1, 69622 Villeurbanne Cedex, France

Abstract

Chitosan composite materials have received considerable attention in the field of bone tissue engineering due to their high biocompatibility, biodegradability, suitability for cell ingrowth and osteoconduction. In this work, we investigated a novel approach to develop 3D printed hardystonite-chitosan (HT-CS) scaffolds, which employs direct ink writing (*i.e.* additive manufacturing technology) of a preceramic polymer-filled with fillers to produce regular 3D hardystonite ceramic scaffolds, with engineered porosity in terms of pore size and distribution. Using a silicone preceramic polymer in the ink composition allowed to have suitable rheological properties of the ink, and to form the desired crystalline phases upon heat treatment, by its reaction with the fillers (CaO and ZnO precursors). Highly porous scaffolds (up to 80% total porosity) with regular pore size of about 0.4 mm were produced. The sintered filaments welded perfectly to each other, did not deform during sintering and were themselves highly porous, due to the gas release from the polymer to-ceramic conversion. The porosity (macro and micro) is beneficial, as it allows for a higher degree of attachment of the cells to the scaffold, and allows for the modification of the mechanical and surface properties of the ceramics scaffold by impregnation with chitosan biopolymer. Chitosan was impregnated into the scaffolds, varying different experimental conditions. Pure HT scaffolds without coating had a rather low compressive strength of 1.69 ± 0.11 MPa, which increased up to 3.27 ± 0.08 MPa after coating with CS hydrogels, making the HT-CS scaffolds good candidates for bone regeneration.

Keywords : Bioceramic scaffolds; Hardystonite; Chitosan; Bone tissue engineering

Mineralization process of chitosan hydrogels by calcium phosphate apatite

S. Ramirez[1,2], L. David[1], T. Delair[1], E. Maire[2], A. Montembault[1], S. Tadier[2], L. Gremillard[2]

[1]Université de Lyon, Ingénierie des Matériaux Polymères, IMP@Lyon1, CNRS UMR 5223, Université Claude Bernard Lyon 1, 69622 Villeurbanne Cedex, France
[2]Université de Lyon, INSA-Lyon, Université Claude Bernard Lyon 1, MATEIS CNRS UMR 5510, 69621 Villeurbanne Cedex, France

Abstract

Bone healing is a field of never ending demand for people of all ages. A bioinspired composite made of a physical hydrogel of chitosan (CS) mineralized by hydroxyapatite would combine the necessary healing and angiogenic properties of CS hydrogels with the bioactivity and osteoconductivity of apatite, and present adaptable chemical, architectural, mechanical and biological properties. Therefore, the objective of this work is to set up a protocol for the mineralization of CS physical hydrogels and to understand how the precipitation of apatite is influenced by the surrounding organic phase. Hydrogels were prepared by dissolution of CS powder in acetic acid and then direct contact with a NaOH solution, while apatite was precipitated from $Ca(NO_3).4H_2O$ and $(NH_4)_2HPO_4$ salts. The experimental set up developed in this Ph.D. work permitted to investigate two mineralization routes:

- precipitation of an apatite phase inside a preformed hydrogel,
- concomitant apatite precipitation and CS gelation.

X-ray diffraction and tomography together with SEM observations showed the presence of apatite as a mineral phase on the CS hydrogel surface. Apatite crystals, which were formed, had an imperfect structure, a low crystalline order similar to the inorganic part of bone tissue. This work helped to better understand the physico-chemical interactions between CS hydrogel and calcium phosphate salts. Strategies such as *in-situ* apatite precipitation and CS gelation are feasible, opening the way for a gradient of composition and structural properties of the mineral/hydrogel associations and a platform for additive manufacturing techniques.

Keywords : Apatite; Biocomposite; Mineralization process; Bone tissue

Novel bioceramic structures for intelligent neuroimplantation

R. Gadow, F. Kern, A. Killinger

Institute for Manufacturing Technologies of Ceramic Components and Composites, University of Stuttgart, Allmandring 7b, D-70569 Stuttgart, Germany

Abstract

Deep Brain Stimulation (DBS) is a promising therapy that shows excellent results in the treatment of chronic diseases such as Parkinson's disease, tremor, epilepsy and chronic pain with electric impulses in specific areas of the brain. Due to the fact that the probes are made of metal, their localization during the operation with the help of Magnetic Resonance Imaging (MRI) is not possible. As a consequence, surgeons have to perform test stimulations of the brain in order to localize a specifically targeted section. For these test stimulations, the patient has to be awake which causes a significant amount of stress and anxiety. A novel approach in achieving higher precision settings is the development of MRI compatible DBS probes. The challenge was to develop a MRI compatible, non-metallic probe material with high stiffness and strength characteristics but also sufficient toughness and flexibility. In this work, different compositions of nanosized ceramic multiphase feedstocks with the aim of producing long and dimensionally highly accurate MRI compatible substrates via the extrusion technology were investigated as well as the functionalization of the probe surface for signal transmission. Different PVD and CVD based thin solid films exhibiting sufficient electrical conductivity and minimum magnetical interference in the MRI were evaluated. As a first step in the development of a multi-contact electrode, laser-structuring of these thin film coatings was investigated. In conclusion, an important progress for a less stressful but more patient-friendly and performing DBS therapy has been developed with substantial contribution of ceramic and surface technology research in modern healthcare.

Keywords : Coatings on implants, Deep brain stimulation DBS therapy, ceramic MRI precision probes, extrusion of zirconia ceramics

Tuesday 06/09/2016 afternoon

14h00 Highlight lecture: Advanced multiphase zirconia ceramics, by Frank Kern
IFKB, Universität Stuttgart, Allmandring 7b, D-70569 Stuttgart, Germany

14h30 **Can (Mg,Y)-PSZ be a valuable alternative to 3Y-TZP for dental application?**
G. de Portu[1], J. Chevalier[2], T. Douillard[2], L. Gremillard[2]
[1]Institute of Science and Technology for Ceramics (ISTEC-CNR), Faenza, Italy - [2]UMR CNRS 5510 (MATEIS), INSA-Lyon, Villeurbanne, France

14h45 **Bioceramic manufacturing process based on the impregnation of three-dimensional printed wax molds**
B. Charbonnier, C. Laurent, D. Marchat
Ecole Nationale Supérieure des Mines, CIS-EMSE, INSERM U1059, F-42023 158 cours Fauriel Saint-Etienne cedex 2, France

15h00 Highlight lecture: Alumino-silicate wastes from ornamental stones: a new resource for construction materials, by Paola Palmero
P. Palmero[1], A. Formia[1], P. Antonaci[2] and J.-M. Tulliani[1]
[1]DISAT, Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Torino, Italy - [2]DISEG, Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Torino, Italy

15h30 **Solidification of a liquid earth binder via polymer gelation: towards poured earth concretes**
A. Pinel[1,2], Y. Jorand[1], E. Fleury[2], C. Olagnon[1] and A. Charlot[2]
[1]Univ Lyon, INSA-Lyon, MATEIS CNRS UMR 5510, Université Claude Bernard Lyon 1, F-69621 Villeurbanne Cedex, France - [2]Univ Lyon, INSA-Lyon, Ingénierie des Matériaux Polymères CNRS UMR 5223, Université Claude Bernard Lyon 1, F-69621 Villeurbanne Cedex, France

15h45 **Innovative cementitious and geopolymer materials as a sustainable alternative to traditional binders**
D. Ziegler, J.-M. Tulliani, Paola Palmero
Department of Applied Science and Technology, Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Italy

17h00 Confluence Museum visit

20h00 Conference dinner

Advanced multiphase zirconia ceramics

Frank Kern

IFKB, Universität Stuttgart, Allmandring 7b, D-70569 Stuttgart, Germany

Abstract

Zirconia materials, owing to their excellent mechanical properties such as strength and fracture resistance have become standard materials for a wide range of applications at ambient or slightly elevated temperatures. Transformation toughening, the mechanism responsible for these properties is a martensitic, stress induced phase transformation of metastable tetragonal to monoclinic which is associated with volume expansion and shear. The metastable tetragonal phase is retained by addition of metal oxides of the alkaline earth or rare earth group. Applications range from mechanical engineering to biomedical. Standard zirconia materials have today reached a high degree of maturity. New developments in zirconia based materials are based on either on enhancements of existing materials, e.g. Y-TZP powders have been continuously improved concerning homogeneity, sinterability and resistance to low temperature degradation, biomedical grade ZTA today has reached strength and toughness comparable to the best Y-TZP materials however at much higher hardness and ageing resistance. New material concepts include the use of new technologies to introduce the stabilizers and dopants, new or combined stabilizer systems and exploit the potential of composite materials by addition of second phases such as various oxides (alumina, spinel or hexaaluminates). New materials for predominantly mechanical engineering applications have been generated by including electrically conductive transition metal compounds making the composites electrically conductive and electric discharge machinable. Recently new composite systems including carbon nanotubes and graphene have been developed. In the talk the state of the art will be briefly reviewed and new developments, with a focus on material concepts elaborated at university of Stuttgart will be discussed.

Keywords : Ceramics, zirconia, mechanical properties, microstructure

Can (Mg,Y)-PSZ be a valuable alternative to 3Y-TZP for dental application?

G. de Portu[1], J. Chevalier[2], T. Douillard[2], L. Gremillard[2]

[1]*Institute of Science and Technology for Ceramics (ISTEC-CNR), Faenza, Italy*

[2]*UMR CNRS 5510 (MATEIS), INSA-Lyon, Villeurbanne, France*

Abstract

Twenty years old samples of (Mg,Y)-PSZ - $MgAl_2O_4$ spinel composites have been characterized in order to verify the microstructure and the mechanical properties after such a long period of natural aging.

The amount of different phases has been determined by XRD while a deep observation of the microstructural features has been carried out by Focus Ion Beam Microscopy. Bending strength, hardness and toughness have been also assessed. Translucency exhibited by this material has been qualified by measuring the contras ratio (CR). Comparing the results obtained in this study with those obtained twenty year ago, the materials reveals an optimum phase stability , superior to that exhibited by conventional 3Y-TZP, a good strength and hardness and a remarkable toughness. The value of CR is comparable with those exhibited by the zirconia monolithic ceramics commonly used in dentistry, which is very good for a composite. In addition, the fact that the material is mostly a modified version of Mg-PSZ leads to a price very competitive, since only few yttrium is used. For all these reasons, 20 years after their initial development, (Mg,Y)-PSZ based composites could be proposed as valid alternative to 3Y-TZP for dental applications.

Keywords : (Mg,Y)-PSZ; Zirconia; Dental Application

Bioceramic manufacturing process based on the impregnation of three-dimensional printed wax molds

Baptiste Charbonnier, Coralie Laurent, David Marchat

Ecole Nationale Supérieure des Mines, CIS-EMSE, INSERM U1059, F-42023
158 cours Fauriel Saint-Etienne cedex 2, France

Abstract

Nowadays, new engineering developments that combine computational methods and additive manufacturing (AM) are able to overcome the current design limitations and sample-to-sample variations inherent in conventional ceramic manufacturing methods. In this context, a robust bioceramic manufacturing process based on the impregnation of three-dimensional (3D) printed wax mold was developed. The innovative and creative potential of this process is based on the ability to manufacture and then to impregnate homogeneously molds with complex structural designs. Results indicate that this process preserves hydroxyapatite phase biocompatibility, permits homogeneous shrinkage of the biomaterial during heat treatments, and allows reproducible and precise manufacturing of custom architectures (e.g., 5 µm widths and high cusps for channels of 200 µm with a 6 µm printer resolution). Apart from the influence of the wax mold printing strategy (e.g., orientation, resolution) on the micro- and macro- architectural features, the bioceramic quality is also determined by the slurry formulation and drying rate in contact with the mold for suspension viscosity less than or equal to 80 mPa s. The development of porous application-specific bioceramics with controlled and standardized architectures using this process will enable biological assessments in a reproducible way to advance bone tissue regeneration.

Keywords : Additive manufacturing, Bioceramic, Calcium phosphates, Slurry.

Alumino-silicate wastes from ornamental stones: a new resource for construction materials

Paola Palmero[1], Alessandra Formia[1], Paola Antonaci[2] and Jean-Marc Tulliani[1]

[1]*DISAT, Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Torino, Italy*

[2]*DISEG, Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Torino, Italy*

Abstract

Impressive amounts of wastes are produced yearly by the extraction and transformation processes of natural stone. In particular, this study addresses the finest fraction of these wastes, named *mud*, and specifically those having siliceous composition. Currently, these muds are disposed in landfills and often dumped directly into ecosystems, without adequate treatment. This poses serious challenges, since they can damage the environment, create necrotic conditions for flora and fauna and endanger human health.

In this work, a mud composed by quartz, feldspars, biotite and dolomite, is used for producing dense and foamed materials through an innovative and simple process, in which a mixture of mud and alkaline solution (plus foaming agent, when needed) is produced, cast in moulds and cured at 80°C for 48 hours. A comparative study with a second mineral powder, having a similar composition but carbonates-free, proved the key role of alumino-silicates in producing hardened materials with excellent mechanical properties. In fact, compressive and flexural strength ranged between about 30-35 MPa and 12-14 MPa, respectively, depending on the starting mineral powder. Also the foamed samples showed good mechanical properties (compressive and flexural strength of about 6.5 and 3.1 MPa, respectively, for samples with density of about 0.8 g/cm^3) besides low thermal conductivity (about 0.3 W/mK). These results demonstrate that alumino-silicate wastes could serve as innovative source for producing materials suitable for dense and cellular building elements, thus opening new re-employment opportunities and providing an effective alternative to mud disposal and related issues.

Keywords : Stone wastes; alumino-silicate materials; mechanical properties

Solidification of a liquid earth binder via polymer gelation: towards poured earth concretes

A. Pinel[1,2], Y. Jorand[1], E. Fleury[2], C. Olagnon[1] and A. Charlot[2]

[1]Univ Lyon, INSA-Lyon, MATEIS CNRS UMR 5510, Université Claude Bernard Lyon 1, F-69621 Villeurbanne Cedex, France

[2]Univ Lyon, INSA-Lyon, Ingénierie des Matériaux Polymères CNRS UMR 5223, Université Claude Bernard Lyon 1, F-69621 Villeurbanne Cedex, France

Abstract

Developing raw earthen construction is nowadays a major environmental issue, due to its low embodied energy. A solution would be to use a similar process as cement-based concrete, by molding into formworks that can be removed within one day. Nevertheless, this is limited by technical difficulties, owing to the fact that earth is not a hydraulic binder. In that perspective, this work focuses on inducing a liquid-solid transition to an earth suspension without waiting for drying, similar to cement based materials. A way to solve this challenge can be inspired from the field of advanced ceramics, in which some forming techniques rely on a liquid-solid transition via polymers gelation.

First experiments, using only a sieved soil, water and the selected polymer showed that it was possible to obtain a liquid mixture which was able to solidify within 24 hours. Experiments have then been conducted at the mortar scale. Samples made by sand added to the former mixture were obtained by molding. Compression tests performed on several sample series of different compositions showed that a resistance near 0.1 MPa could be reached within only 24 hours and without any drying. This would be a sufficient resistance for a wall to stand under its own weigh, allowing a rapid removal of the formworks. A good workability was also ensured during tens of minutes. The work done proved the potential of the selected polymer and the very promising way of solving the challenge exposed. Testings at the concrete scale are currently being performed.

Keywords : Earthen construction, concrete, gelation, polymers, composite

Innovative cementitious and geopolymer materials as a sustainable alternative to traditional binders.

Daniele Ziegler, Jean-Marc Tulliani, Paola Palmero

Department of Applied Science and Technology, Politecnico di Torino,
Corso Duca degli Abruzzi, 24, 10129, ITALY

Abstract

The term “geopolymer” is used to define a new class of inorganic alkali-activated alumino-silicate materials. Indeed, geopolymerization indicates a chemical reaction between alumino-silicates and alkali metal silicates, under strong alkaline conditions. The recent literature underlines the excellent mechanical strength, the fire resistance and low thermal conductivity of geopolymers. Due to such unique properties, they are being investigated for the development of a variety of cost-effective products, such as construction materials. Indeed, due to the environmental impact of Portland cement production, the construction industry is targeting its rational use through the development of more efficient binders, like geopolymers. In this work, innovative cementitious as well as geopolymers pastes and mortars have been developed as sustainable alternative to Portland cement. First, cements were prepared starting from an aqueous mixture of flue dust and fly ash, by-products from the cement industry and coal combustion, respectively. Second, geopolymers were prepared by mixing class F fly ash and rice husk ash with a potassium hydroxide solution. Cast samples were cured under wet and dry conditions for cements and geopolymers, respectively, up to 90 days. Lightened samples were also prepared, by adding different amounts of foaming agent to the mixture, before casting.
Dense and porous samples were submitted to density measurements, three-point bending and compression tests, X-Ray diffraction and FESEM analysis. In addition, the thermal conductivity of foamed samples was measured. Such innovative binders showed comparable properties and lower CO_2 footprint as respect to a traditional “green” cement based on blast furnace slag, here used as a reference, opening a practical avenue towards truly sustainable concretes.

Keywords : green binders, geopolymers, dense and lightened samples, mechanical properties.

Wednesday 07/09/2016 morning

09h00 Keynote lecture: Can mechanical properties of ceramics be improved by nanotubes reinforcement?, by Arturo Dominguez-Rodriguez

A. Domínguez-Rodríguez[1], B.M. Moshtaghioun[1] and D. Gómez García[1,2]

[1]Department of Condensed Matter Physics, University of Seville, P. O. 1065, 41080 Sevilla, Spain - [2]Institute of Materials Science, University of Seville-CSIC, Spain

09h30 **Study of crystallization, structure and piezoelectric properties of Sr-fresnoite and Sr-Ba fresnoite glass-ceramics**

M.-S. Renoirt, M. Gonon

Materials Institute, Faculty of Engineering- University of Mons, Rue de l'Epargne 56, 7000, Mons, Belgium

09h45 **The effects of SiO_2 content on the dielectric properties of $BaTiO_3$ ceramics and statistical approaches on the dielectric properties**

U. Şen[1], K. E. Öksüz[2], M. Torman[1] and Ş. Şen[1]

[1]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 54187, Sakarya, Turkey. - [2]Cumhuriyet University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 58140, Sivas, Turkey

10h00 **Processing and properties of ceramics with structured porosity for Oxygen Transport Membranes.**

J. Seuba[1], C. Guizard[2], A. J. Stevenson[3]

[1]Université de Lyon, INSA-Lyon, MATEIS CNRS UMR5510, F-69621 Villeurbanne, France - [2]Institut Européen des Membranes, Université de Montpellier 2, Place Eugene Bataillon, 34095 Montpellier Cedex 5, France - [3]Laboratoire de Synthèse et Fonctionnalisation des Céramiques, UMR3080 CNRS/Saint-Gobain, F-84306 Cavaillon, France

10h15 **Oxidation of ZrB_2 and HfB_2 based ceramics at very high temperature under steam condition**

V. Guérineau, A. Jankowiak

ONERA - The French Aerospace Lab, F-92322 Châtillon, France

10h30 Coffee break

11h00 Highlight lecture: Measurement of residual stresses in cemented carbides for cutting tools and finite element simulations of stress partition. by Daniele Mari

LCMP, IPHYS, EPFL, Station 3, 1015, Lausanne, Switzerland

11h30 **Synthesis and characterization of a new $Ti_3(Al,Cu)C_2$ MAX phase solid solution**

M. Nechiche[1,2], V. Gauthier-Brunet[2], T. Cabioc'h[2], A. Joulain[2], V. Mauchamp[2], X. Milhet[2], S.Azem[1], S. Dubois[2]

[1]Laboratoire Elaboration, Caractérisation des Matériaux et Modélisation (LEC2M), Université Mouloud MAMMERI de Tizi-Ouzou. BP17, 15000. Algérie - [2]Institut PPRIME, CNRS/Université de Poitiers/ENSMA, UPR 3346, BP 30179, 86962 Chasseneuil du Poitou-Futuroscope Cedex, France.

11h45 **Effect of small additions of calcia on the grain size and mechanical properties of ceria-stabilized tetragonal zirconia**

M. Turon-Vinas[1,2], M. Anglada[1,2]

[1]CIEFMA-Department of Materials Science and Metallurgical Engineering, ETSEIB, Universitat Politècnica de Catalunya, 08028 Barcelona, Spain - [2]CRnE, Campus Diagonal Sud, Edifici C', Universitat Politècnica de Catalunya, 08028 Barcelona, Spain

12h00 **Machinability in Sepiolite Based Glass-Ceramics containing LiF_2 (AlF_3)**

G. Bayrak[1], E. Ercenk[2] and S. Yilmaz[2]

[1]Welding Technology Department: Vocational School of Arifiye, Sakarya University, Fatih Mh. Esit Sk. No:7/A, 54580, Arifiye, Sakarya, Turkey - [2]Metallurgical and material science engineering: Faculty of Engineering, Sakarya University, M7 Building, 54187, Serdivan, Sakarya, Turkey

12h15 **Development of a computer aided method for the characterization of refractory materials microstructure.**

N. Preux[1], P. Pilate[1], V. Lardot[1], F. Cambier[1], E. Brochen[2], C. Dannert[2], F. Holleyn[3], O. Krause[3]

[1]BCRC (Belgian Ceramic Research Centre, member of EMRA), avenue Gouverneur Cornez, 4, 7000 Mons Belgium - [2]Forschungsgemeinschaft Feuerfest e.V.,Rheinsstrasse 58, 56203, Höhr-Grenzhausen, Germany - [3]Hochschule Koblenz, WesterWald Campus, Rheinstrasse 56, 56203, Höhr-Grenzhausen, Germany

12h30 **The diamond and its industrial applications in dies and special tools**

A. Cellard, N. Barthod

BALLOFFET Company, 53 Route du Charveyron, 01150, Lagnieu, France

12h45 Lunch and end of the conference

14h00 Optional: visit of SETARAM facilities

Can mechanical properties of ceramics be improved by nanotube reinforcement?

Arturo Domínguez-Rodríguez[1], Bibi Malmal Moshtaghioun[1] and Diego Gómez García[1,2]

[1]Department of Condensed Matter Physics, University of Seville, P. O. 1065, 41080 Sevilla, Spain

[2]Institute of Materials Science, University of Seville-CSIC, Spain.

Abstract

Since Iijima discovered carbon nanotubes (CNTs), all different forms of CNTs such as single wall carbon nanotubes (SWCNTs) and multi wall carbon nanotube (MWCNTs) have attracted scientific interest due to their unique combination of properties. Concerning mechanical properties, there are several reviews showing contradictory results. In order to understand these discrepancies, and before showing if the nanotubes can or cannot improve the mechanical properties, it is necessary to consider the nature of CNTs, their dispersion within the matrix, the processing conditions and their damage during this process to obtain a novel engineering ceramic matrix composite (CMCs). These points will be analyzed in this presentation.

Keywords : nanotubes, mechanical properties, nanotube-reinforced ceramics processing

Study of crystallization, structure and piezoelectric properties of Sr-fresnoite and Sr-Ba fresnoite glass-ceramics

M.-S. Renoirt, M. Gonon

Materials Institute, Faculty of Engineering- University of Mons, Rue de l'Epargne 56, 7000, Mons, Belgique

Abstract

Despite exhibiting much lower electromechanical performances than ferroelectrics, non-ferroelectric piezoelectrics are potential candidates for high temperature applications. Indeed, non-ferroelectrics piezoelectrics do not present depolarization with time or under increasing temperature. For such non-ferroelectric compounds, a preferential structural orientation of dipolar moments is induced during processing, leading to piezoelectric properties (single crystal or textured polycrystals). In her PhD thesis, N. Maury demonstrated that a preferential orientation of piezoelectric Sr-fresnoite crystals ($Sr_2TiSi_2O_8$) is possible through glass-ceramics processing, by surface nucleation mechanism during an isothermal crystallization treatment on suitable glass composition.

Using N. Maury's optimal parent glass composition at different temperatures of isothermal crystallization treatments, the present work shows a -180° dipolar moment inversion between the opposite crystal faces. This result highlights that the dipoles direction is conditioned during crystallization by the free surfaces. In addition, the expansion coefficient mismatch between the fresnoite crystals and the residual glass is shown by d_{33} charge coefficient measurements and crystal lattices parameters determination by XRD analysis. According to the correlation of these two measurements, the expansion coefficient mismatch generates internal stresses and results in a decreasing of piezoelectric performances.

To discuss these results, changes in the parent glass composition have been made in order to modify the crystal (Sr/Ba- fresnoite) or the residual glass properties. Piezoelectric measurements and XRD analyses have been completed by mechanical characterization of the final glass ceramic and acoustic emission during thermal cycles.

Keywords: Glass-ceramics, Piezoelectricity, Crystallization

The effects of SiO_2 content on the dielectric properties of $BaTiO_3$ ceramics and statistical approaches on the dielectric properties.

Uğur Şen[1], Kerim Emre Öksüz[2], Merve Torman[1] and Şaduman Şen[1]

[1]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 54187, Sakarya, Turkey.

[2]Cumhuriyet University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 58140, Sivas, Turkey

Abstract

In this study, dielectric properties of SiO_2 doped barium titanate based ceramic materials were investigated depending on SiO_2 content and applied frekance. In this process, 0%, 0.1%, 0.2% and 0.4% SiO_2-doped $BaTiO_3$ ceramic powder substitution was prepared by solid state mixed oxide technique. X-ray diffraction analysis showed that the main phases formed in the sintered samples are $BaTiO_3$. LCR meter was used for examinations of the dielectric properties of the sintered samples. Lorentzian, Gaussian and Paraboloid equations were used for regression analysis of the prediction of the SiO_2 doped $BaTiO_3$ ceramics. Lorentzian equation was to give the best results among the regression equations.

Keywords : Barium titanate, Grain Growth, Kinetic, Activation Energy, Sintering.

Processing and properties of ceramics with structured porosity for Oxygen Transport Membranes

Jordi Seuba[1], Christian Guizard[2], Adam J. Stevenson[3]

[1]*Université de Lyon, INSA-Lyon, MATEIS CNRS UMR5510, F-69621 Villeurbanne, France*

[2]*Institut Européen des Membranes, Université de Montpellier 2, Place Eugene Bataillon, 34095 Montpellier Cedex 5, France*

[3]*Laboratoire de Synthèse et Fonctionnalisation des Céramiques, UMR3080 CNRS/Saint-Gobain, F-84306 Cavaillon, France*

Abstract

Based on mixed ionic and electronic conducting ceramics, oxygen transport membranes (OTMs) are on the verge of commercialization for important energy related applications including oxycombustion, syngas production, and catalytic reactors. However, the processing costs and performance limitations of these membranes currently limit their market potential.

In this presentation, I will detail our efforts to improve properties and performance of these systems by engineering the structure of the porous support. In particular, I will address the effects of pore morphology on strength and gas flow using ice-templated ZrO_2 as a model system. Measured mechanical properties are compared to Ashby's models of strength in porous ceramics. Permeability measurements are also compared to available models. Finally, the tradeoffs between mechanical stability and permeability are discussed in the context of the structured pore morphologies and the OTM application.

Keywords : Porous ceramics, oxygen transport membranes, mechanical properties, freeze-casting

Oxidation of ZrB_2 and HfB_2 based ceramics at very high temperature under steam condition

Vincent Guérineau, Aurélie Jankowiak

ONERA - The French Aerospace Lab, F-92322 Châtillon, France

Abstract

The Ultra-High Temperature Ceramics, such as ZrB_2-SiC or HfB_2-SiC composites, are of growing interest for applications like hypersonic flights, re-entry vehicles or propulsion. These materials are expected to sustain very high temperatures (>2000°C) in very corrosive and oxidizing atmospheres thanks to the development of a multi-oxide scale composed of a MeO_2 skeleton and a glassy layer. Few studies focus one the role of H_2O during the oxidation of UHTC which is a common byproduct in combustion environment. The aim of this thesis is to better understand and to model the oxidation mechanisms in H_2O environments at temperatures as high as 2000°C. Fully densified samples of ZrB_2-20vol%SiC (ZS20), ZrB_2-7.5vol%SiC (ZS7.5), HfB_2-20vol%SiC (HS20) and HfB_2-20vol%SiC-3vol%Y_2O_3 (HSY) were manufactured using a spark plasma sintering device (INSAVALOR, France). A home-made Oxidation Laser Bench (BLOX) is used to quickly heat samples between 1200 and 2400°C, under controlled atmosphere (1 atm, 30 vol. % H_2O) thanks to a high power CO_2 laser (2 kW). Oxidation mechanisms are studied through scanning electronic microscopy examinations coupled with energy dispersive spectroscopy and X-ray diffraction analyses. Thicknesses of the different layers allow calculating recession rates. For all samples, several layers are observed and the nature of the oxides depends on the temperature of the test. At temperature above 1900°C, recession rate is strongly increased due to layer spallation, porosity and cracks formation. Beyond 2000°C, the top layer is always spalled off the material underneath. First results indicate that HS and HSY are the most promising samples.

Keywords : UHTC, Steam, ZrB_2, HfB_2, SiC

Measurement of residual stresses in cemented carbides for cutting tools and finite element simulations of stress partition

Daniele Mari

LCMP, IPHYS, EPFL, Station 3, 1015, Lausanne, Switzerland

Abstract

Thermal residual stresses play a major role in the mechanical properties of cemented carbides. Their magnitudes are high (e.g. 2 GPA in cobalt and 0.5 GPa in WC) and their distribution is complex. The magnitude depends on thermal expansion coefficients, binder fraction and particle size. The response of WC based cemented carbides to uniaxial compression is deeply influenced by thermal residual stresses. Experiments on the systems WC-Co and WC-Ni were performed using neutron diffraction as a mean to measure residual stresses in each bulk phase. The response to loading-unloading cycles gives an insight into the role of thermal residual stresses in the toughness of cemented carbides. The binder deforms plastically at relatively low stress in the direction transverse to load axis due high thermal stress that are relaxed by this effect. Therefore, these studies have an important impact to practical application in cutting tools. Several Finite Element Models of the morphology of WC – 10wt%Co can be used to reproduce the build-up of thermal residual stresses as well as the phase specific strain during loading-unloading in compression. The different models differ only in their geometry of the interpenetrating skeletons of WC and Co. They all respect the given volume proportion of each phase. Thermo-elasticity is considered for the brittle WC, while also plasticity is included to model the Co binder phase. We compare the predictions of our FEM models with phase specific strain obtained by in-situ neutron diffraction and discuss the model.

Keywords : WC-Co, WC-NI, compression, thermal stresses, toughness.

Synthesis and characterization of a new $Ti_3(Al,Cu)C_2$ MAX phase solid solution

M. Nechiche[1,2], V. Gauthier-Brunet[2], T. Cabioc'h[2], A. Joulain[2], V. Mauchamp[2], X. Milhet[2], S.Azem[1], S. Dubois[2]

[1]*Laboratoire Elaboration, Caractérisation des Matériaux et Modélisation (LEC2M), Université Mouloud MAMMERI de Tizi-Ouzou. BP17, 15000. Algérie.*

[2]*Institut PPRIME, CNRS/Université de Poitiers/ENSMA, UPR 3346, BP 30179, 86962 Chasseneuil du Poitou-Futuroscope Cedex, France.*

Abstract

Ti_3AlC_2 is a member of the MAX phase compounds having the general formula $M_{n+1}AX_n$ (n = 1-3), where M is an early transition metal, A is an A group element (from IIIA to VIA), and X is either carbon or nitrogen. In these materials, the $M_{n+1}X_n$ layers, characterized by strong covalent M-X bonds, are interleaved with A layers through weak M-A bonds. This inherent nanolayered structure provides a unique combination of metal-like and ceramic-like properties. In this work, Ti_3AlC_2 MAX phase powders were first prepared at 1450°C for 2 hours from 1.9TiC:1.05Al:1.0Ti reactant mixture. High-energy milling of Ti_3AlC_2 and Cu (40vol.%) powders was then performed to form large aggregates containing alternative lamellas of both compounds. Finally, a new $Ti_3(Al,Cu)C_2$/Cu metal matrix composite has been produced by sintering Ti_3AlC_2 and Cu co-milled powders. Using XRD and TEM-EDXS, it has been demonstrated that Cu can enter the crystallographic structure of the Ti_3AlC_2 MAX phase, whereas a Cu(Al) solid solution is formed during thermal treatment. TEM-EELS analyses have demonstrated that Cu is mainly located on the A site of the MAX phase. The composition of the MAX phase solid solution, determined after selective chemical etching of the Cu(Al) matrix, by analyzing the filtrate and the solid phase using ICP-OES end EDXS methods respectively, is $Ti_3(Al_{0.5}Cu_{0.5})C_2$.

Keywords : MAX phase, solid solution, powder metallurgy, microstructural characterization.

Effect of small additions of calcia on the grain size and mechanical properties of ceria-stabilized tetragonal zirconia

M. Turon-Vinas[1,2], M. Anglada[1,2]

[1]*CIEFMA-Department of Materials Science and Metallurgical Engineering, ETSEIB, Universitat Politècnica de Catalunya, 08028 Barcelona, Spain.*

[2]*CRnE, Campus Diagonal Sud, Edifici C', Universitat Politècnica de Catalunya, 08028 Barcelona, Spain*

Abstract

Yttria, with a concentration of 3 mol% is the most common dopant used for stabilizing the tetragonal phase of zirconia. The resulting material, which is referred to as 3Y-TZP, has attracted much attention in restorative dentistry because of its biocompatibility, aesthetical appearance, high hardness and strength, and good fracture toughness. Besides the constrained tetragonal monoclinic transformation under stress which takes place in the crack process zone, Y-TZP may also transform spontaneously to monoclinic in humid environments, and this phenomenon is referred to as low temperature degradation (LTD), and it is detrimental for the near surface since it induces surface microcracking and grain pullout.

Ceria is also a common dopant for stabilizing the tetragonal phase of zirconia, which in concentrations higher than about 10% molar makes zirconia much more resistant to LTD. Ce-TZP ceramics can also have higher fracture toughness than 3Y-TZP, but lower hardness and fracture strength, which is partly associated to its higher grain size. In the literature, it has been reported that small additions of solutes to Ce-TZP may reduce the grain size. Thus, with the aim to obtain a ceramic resistant to LTD with a better compromise between fracture toughness, hardness and strength, disc-shaped samples of Ce-TZP were prepared by suspension drying technique with the addition of 0.5 mol% to 5 mol% CaO_2 and its influence on the microstructure and mechanical properties has been studied. Initial results show that low concentrations of CaO_2 can significantly reduce the grain size and increase the hardness of Ce-TZP. Other mechanical properties are currently analysed.

Keywords : Zirconia, ceria, processing

Machinability in Sepiolite Based Glass-Ceramics containing LiF_2 (AlF_3)

Gunhan Bayrak[1], Ediz Ercenk[2] and Senol Yilmaz[2]

[1]*Welding Technology Department: Vocational School of Arifiye, Sakarya University, Fatih Mh. Esit Sk. No:7/A, 54580, Arifiye, Sakarya, TURKEY*

[2]*Metallurgical and material science engineering: Faculty of Engineering, Sakarya University, M7 Building, 54187, Serdivan, Sakarya, TURKEY*

Abstract

At the present study, sintering behavior and machinability of sepiolite based glass-ceramics were investigated. The sepiolite based glass-ceramics which has SiO_2 and other glass making oxides were produced by sintering method. Some additives were added to this natural raw material for increase machinability and sintering properties as B_2O_3, K_2O SiO_2, Al_2O_3 and LiF_3. Initial materials were mixed by ball milling for 2 h using alumina media. The mixed and milled powders were sieved to grain sizes smaller than 75 µm and pressed at 100 MPa. The pressed samples were sintered at 900 -1200 °C for 1 h in an electric furnace using a heating rate of 5 °C/min. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were performed for characterization studies. In addition drilling test were applied on sintered samples to determine machining ability. The results indicated that all samples exhibits good sintering and machinability properties.

Keywords : Sepiolite, glass-ceramic, sintering, machinability

Development of a computer aided method for the characterization of refractory materials microstructure.

N. Preux[1], P. Pilate[1], V. Lardot[1], F. Cambier[1], E. Brochen[2], C. Dannert[2], F. Holleyn[3], O. Krause[3]

[1]BCRC (Belgian Ceramic Research Centre, member of EMRA), avenue Gouverneur Cornez, 4, 7000 Mons Belgium

[2]Forschungsgemeinschaft Feuerfest e.V.,Rheinsstrasse 58, 56203, Höhr-Grenzhausen, Germany

[3]Hochschule Koblenz, WesterWald Campus, Rheinstrasse 56, 56203, Höhr-Grenzhausen, Germany

Abstract

Refractory materials exhibit complicated microstructure. Microscopic observations are limited to some restricted areas showing only some grains or boundaries. This is not sufficient to investigate and to quantify the whole microstructure and eventually degradation. Systematic investigation needs imaging of large areas and a good contrast between phases. The purpose of this work was to investigate possibilities to describe and to quantify the whole microstructure of alumina castables and their degradation after thermal shock. Castables were prepared through a classical way. Microscopy samples preparation was optimized and some special treatments were realized to highlight the microstructure. After surface grinding and polishing, optical and electronical microscopy was used for image recording. A software was selected to obtain large area images. It combines a great number of small pictures. This treatment allows to obtain large area pictures and to keep the accuracy of the microscopy. Some methods and tricks were developed to improve picture quality in optical microscopy. With ink impregnation, large dense particles remain white while porous parts become black. Vacuum impregnation with fluorescent resin and UV lighting leads to enhance some microstructure features. Dark field was used to obtain pictures with better definition of grain boundaries. Image analysis softwares were tested. The main operation is thresholding. This method is not achievable on materials with low contrast but can be applied after some treatments. A software proposes a “modulus” detecting grain boundaries in standard condition. First results are promising, images and their analysis are related to the known features of the materials.

Keywords : Refractories, microstructure, microscopy

The diamond and its industrial applications in dies and special tools

A. Cellard, N. Barthod

BALLOFFET Company, 53 Route du Charveyron, 01150, Lagnieu, France

Abstract

The shape, colour, purity, composition, internal and crystalline structure, nature and distribution of structural defects are governed by diamond growth processes. The strong covalent bondings of diamond are responsible for its outstanding properties such as exceptional hardness and tribological properties. For extreme industrial applications such as wire, tube and profile drawing, a fundamental particularity of monocrystalline diamond is its oriented cristallographic structure, essential to a perfect product finish (roundness, surface finish). Showing great properties such as high wear resistance, exceptional thermal conductivity, this remarkable material allows to produce high precision and cost effective tools for the industry.

The French Company Balloffet, created in 1870, manufactures a wide range of high precision tools in natural and synthetic diamond (monocrystalline and polycrystalline) from 0.007mm up to 33mm : drawing, stranding, tubing dies, shaped dies, diamond coated dies, as well as a wide variety of wear parts and special diamond tools with drilled diamond inserts such as waterjet cutting and cleaning nozzles, diamond guides, pinholes, diamond injectors … all designed to give the best quality, performance, accuracy and a long tool life. To match customer requirements, every stage of manufacturing process (diamond choice, flattening, mounting, laser drilling, ultrasonic shaping, preadjusting, mirror polishing, wire adjusting) are carefully monitored. Balloffet manufactures its own ultrasonic and wire shaping and polishing machines, and a wide range of tool cleaning and measuring equipment.

We would be pleased to introduce this fascinating field of diamond with these specific and unusual industrial applications.

Keywords : Diamond, Diamond machining, Diamond wire drawing die, Special diamond tools, Wear parts.

LIST OF POSTERS

1 **The temperature gradients inside an alumina elaborate by spark plasma sintering process**

Y. Achenani[1], M. Saädaoui[2], A. Cheddadi[1], G. Bonnefont[3], G. Fantozzi[3]

[1]Mohammadia School of Engineers, ERSTER Team, Mohammed V University In Rabat, Avenue Ibn Sina, P.O. Box 765-Agdal, Rabat, Morocco - [2]Mohammadia School of Engineers, ERSIM Team, Mohammed V University In Rabat, Avenue Ibn Sina, P.O. Box 765-Agdal, Rabat, Morocco – [3]INSA Lyon, MATEIS Laboratory, UMR 5510 CNRS, Lyon University, 20 Avenue Albert Einstein, F-69621 Villeurbanne Cedex, Lyon, France

2 **Hybrid sol-gel coating containing ceramic particles for mild-steel protection**

P. Aubry[1], C. Delmotte[1], S. Wallon[2], E. Lachery[3], A. Nicolay[4], M. Poelman[3], M.-G. Olivier[4], M. Wenkin[2], V. Lardot[1] and F. Cambier[1]

[1]Belgian Ceramic Research Centre (membre of EMRA), 4 Avenue Gouverneur Cornez, 7000 Mons, Belgium - [2]Coatings Research Institute - CoRI, 21 avenue Pierre Holoffe, 1342 Limelette, Belgium - [3]Materia Nova (membre of EMRA), 56 rue de l'Epargne, 7000 Mons, Belgium - [4]Service Science des matériaux, Université de Mons, 56 rue de l'Epargne, 7000 Mons, Belgium

3 **Elaboration and characterization of mulitte obtained by reaction sintering of Algerian kaolin and aluminum hydroxide**

M.L. Bella[1,2,3], M. Hamidouche[1,2], L. Gremillard[3]

[1]Emerging Materials Research Unit, University Ferhat ABBAS Setif 1, 19000, Setif, Algeria – [2]Optical and precision mechanics institute, University Ferhat ABBAS Setif 1, 19000, Setif, Algeria. - [3]MATEIS UMR CNRS 5510, INSA-Lyon, Bat Blaise pascal, 7 Av. Jean Capelle, Villeurbanne, 69621, Lyon, France.

4 **Spark Plasma Sintering mechanisms of Spinel ($MgAl_2O_4$) nanopowders**

S. Benaissa[1,3], A. Zegadi[2,3], M. Hamidouche[2,3], M. Kolli[2,3], G. Bonnefont[4], G. Fantozzi[4]

[1]Optical Research and Photonics Unit, CDTA, Ferhat Abbas University Setif 1, Algeria - [2]Emerging Materials Research Unit, Ferhat Abbas University Setif 1, Algeria - [3]Optics and Precision Mechanics Institute, Ferhat Abbas University Setif 1, Algeria - [4]MATEIS Laboratory, INSA Lyon, France

5 **A comparative study of different strategies to obtain quantitative chemical information from hyperspectral data based on the Fourier Transform Infrared Spectroscope (FTIR)**

T. Bonnal, G. Foray, S. Tadier, E. Prud'homme

MATEIS, INSA Lyon, 20 Avenue Albert Einstein, 69621 Villeurbanne

6 **Direct-ink writing of high-strength dense ceramics**

E. Camposilvan[1, 2], V. Garnier[1], L. Gremillard[1] and J. Chevalier[1]

[1]Université de Lyon, INSA de Lyon, MATEIS CNRS UMR5510, 7 Av. Jean Capelle, 69621 Villeurbanne, France - [2]Department of Materials Science and Metallurgical Engineering, Universitat Politècnica de Catalunya, Av. Diagonal 647, Barcelona, Spain

7 **Wear Behaviour of Cordierite doped with ZrO_2 Produced from Zeolite**

B. Çitak, A. Ş. Demirkiran

Sakarya University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

8 **Experimental study of electromagnetic field impact on surface diffusion in zinc oxide ceramic**

A. Ducoulombier, F. Valdivieso, J. Bruchon, S. Saunier, P. Ganster, C.Meunier
LGF CNRS-UMR 5307, Mines de St-Etienne, 158 cours Fauriel, 42023 St-Etienne, France

9 **Processing and characterization of zirconia (3Y-TZP) composites with graphene nanoplatelets**

A. Gallardo-López[1], I. Márquez-Abril[1], A. Morales-Rodríguez[1], A. Muñoz[1], R. Poyato[1,2]
[1]Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain - [2]Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. AméricoVespucio 49, 41092, Sevilla, Spain

10 **A Choline Chloride/DMSO solvent for the direct synthesis of diformylfuran from carbohydrates in the presence of heteropolyacids**

W. Ghezali[1,2], K. De Oliveira Vigier[1], R. Kessas[2] and F. Jerome[1]
[1]UMR 7285 CNRS, IC2MP, Université de Poitiers, 1 rue Marcel Dore TSA 41105-86 073 POITIERS Cedex 9, France - [2]Université des Sciences et de la Technologie d'Oran – Mohamed Boudiaf, BP 1505 El-M'Naouar, Oran, Algérie

11 **Low temperature degradation and mechanical properties of (Y,Ce-) co-stabilized zirconia**

A. Gommeringer, F. Kern and R. Gadow
Institut für Fertigungstechnologie keramischer Bauteile, Universität Stuttgart, Allmandring 7b, 70569 Stuttgart, Germany

12 ***In situ* TEM Nano-Compression and Mechanical Analysis of MgO and Al_2O_3 nanoparticles**

I. Issa[1,2], L. Joly-Pottuz[1], J. Réthoré[2], C. Esnouf[1], V. Garnier[1], J. Amodeo[1], J. Morthomas[1], J. Chevalier[1] and K. Masenelli-Varlot[1]
[1]INSA-Lyon, MATEIS CNRS UMR5510, 69621 Villeurbanne, France - [2]INSA-Lyon LaMCoS CNRS UMR5259, 69621 Villeurbanne, France

13 **Nanostructured Zirconia Composites with High Ageing Resistance**

M. Johannes[1], J. Schneider[2], S. Begand[1]
[1]Fraunhofer Institute for Ceramic Technologies and Systems, Department of Oxid- and polymer-ceramic Components, Michael-Faraday-Str. 1, 07629 Hermsdorf/Germany - [2]Bauhaus University, Faculty of Construction Chemistry and Polymers, Coudraystr. 11a, 99423 Weimar/Germany

14 **Dielectric Properties of Zirconium Modified $(Na_{0.5}Bi_{0.5})TiO_3$ Ceramics by Mechanical Activation**

D. Kırsever and H. Ö. Toplan
Sakarya University, Metallurgy and Materials Engineering, Sakarya, Turkey

15 **Effect of Mechanical Activation on the Structural and Electrical Properties of Mn doped $(Na_{0.5}Bi_{0.5})TiO_3$ Ceramics**

D. Kırsever and H.Ö. Toplan
Sakarya University, Metallurgy and Materials Engineering, Sakarya, Turkey

16 Surface modification of zirconia-toughened alumina bioceramics for orthopedic applications

Q. Flamant[1], C. Caravaca[2], S. Meille[2], L. Gremillard[2], J. Chevalier[2], K. Biotteau-Deheuvels[3], M. Kuntz[3], R. Chandrawati[4], I. K. Herrmann[4], C. D. Spicer[4], M. M. Stevens[4] and M. Anglada[1]

[1]Department of Materials Science and Metallurgical Engineering, Universitat Politècnica de Catalunya, Av. Diagonal 647, 08028 Barcelona, Spain - [2]Université de Lyon, INSA-Lyon, MATEIS UMR5510, 7 avenue J. Capelle, 69621, Villeurbanne Cedex, France - [3]CeramTec GmbH, 73207 Plochingen, CeramTec-Platz 1-9, Germany - [4]Department of Materials, Department of Bioengineering and Institute of Biomedical Engineering, Imperial College London, London SW7 2AZ, United Kingdom.

17 Characterization of in-situ ZrB_2-Al_2O_3 composites Produced by Powder Metallurgy

D. Koroğlu[1], B. Citak[1], O. Ozdemir[1], A.Ş. Demirkiran[2]

Sakarya University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

18 Reinforcement of Hydroxyapatite Bioceramic by Addition of ZrO_2 and Al_2O_3

S. Laasri[1], M. Essadik[2], M. Taha[2], A. Laghzizil[3], E-K. Hlil[4], A. Hajjaji[1]

[1]Laboratoire des Sciences de l'Ingénieur pour l'Energie, Ecole Nationale des Sciences Appliquées d'El Jadida, BP 1166, EL Jadida Plateau 24002, Morocco - [2]Laboratoire de Thermodynamique et Energétique, Université Ibn-Zohr, Faculté des Sciences, BP 8106 Agadir Morocco - [3]Laboratoire de Chimie Physique Générale, Faculté des Sciences, Université Mohammed V, BP.1014 Rabat Morocco - [4]Institut Néel, CNRS et Université Joseph Fourier, BP 166, 38042 Grenoble, France

19 Role of inclusions on the diffusion after spark-plasma sintering of Ce:YAG for optical applications

E. Lachaud[1,2,3], A. Vivet[3], V. Garnier[1], Y. Guyot[2], G. Fantozzi[1], L. Bonneau[3], G. Boulon[2], G. Bonnefont[1]

[1]MATEIS, INSA de Lyon, avenue Jean Capelle, Villeurbanne - [2]ILM, Université Claude Bernard Lyon 1, Domaine scientifique de la Doua, bâtiment Kastler, Villeurbanne - [3]Baikowski, les Marais Noirs, Poisy

20 Assembly of transparent polycrystalline Er :YAG ceramic for Laser application

M. Lagny[1,2], A. Katz[1,2], J. Boehmler[1], S. Lemonnier[1], É. Barraud[1], S. D'Astorg[2], Y. Lorgouilloux[2], A. Leriche[2]

[1]ISL, 5 rue du Général Cassagnou, 68300 SAINT-LOUIS - [2]LMCPA, Pôle universitaire de Maubeuge, Boulevard Charles de Gaulle, 59600 MAUBEUGE

21 Effect of microstructure on electron trapping and conduction in polycrystalline ceramics under defocused electron beam irradiation in SEM

O. Mekni[1,2], D. Goeuriot[1], G. Damamme[3]

[1]Laboratoire Georges Friedel CNRS UMR 5307, Ecole des Mines de Saint Etienne, 158 coursFauriel, 42023 Saint-Etienne, France - [2]Laboratory of Materials, Organization and Properties LR99ES17, University of Tunis El Manar, Campus Universities El Manar, 2092 Tunis, Tunisia - [3]CEA / Gramat, BP80200, 46500 Gramat, France

22 **Thermomechanical properties and fracture of resin-bounded-sand cores. Application in aluminum foundry**

C. Menet[1], P. Reynaud[1], G. Fantozzi[1] and A. Laforet[2]

[1]MATEIS, INSA Lyon, 7 avenue Jean Capelle, 69621, VILLEURBANNE, France - [2]Montupet, rue Jean-François Champollion, 36130, DIORS, France

23 **Corrosion and low temperature degradation of zirconia dental ceramics**

A. Nowicka, D. Galusková, S. Mikušinec and D. Galusek

Vitrum Lauguricio – Joint Glass Center of the IIC SAS, TnU AD, and FChFT STU, Študentská 2, 911 50 Trenčín, Slovakia

24 **Grain growth kinetics and regression analysis of the PbO-doped $BaTiO_3$ ceramics**

K.E. Öksüz[1], Ş. Şen[2] and U. Şen[2]

[1]Cumhuriyet University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 58140, Sivas, Turkey - [2]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 54187, Sakarya, Turkey

25 **Zirconia behavior after surface laser treatment**

R. S. F. Pereira[1], C. G. Moura, F[2]. S. Silva[2] B. Henriques[1] and M. C. Fredel[1]

[1]CERMAT, Universidade Federal de Santa Catarina, Florianópolis, Brazil - [2]Center for Micro-Electro Mechanical System, University of Minho, Braga, Portugal

26 **Mechanical behaviour of a randomly porous β-TCP sample: study of the fracture path by X-ray tomography**

C. Petit, S. Meille, E. Maire, S. Tadier and J. Adrien

INSA de Lyon MATEIS CNRS UMR5510 Université de Lyon, 20 avenue Albert Einstein, 69621 VILLEURBANNE, France

27 **Effect of processing on microstructure and room temperature mechanical and electrical properties of 3YTZP/2.5 vol% MWNT ceramic nanocomposites**

R. Poyato[1,2], Á. Gallardo-López[2], A. Morales-Rodríguez[2], F. Gutiérrez-Mora[2], A. Muñoz[2], A. Domínguez-Rodríguez[2]

[1]Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. Américo Vespucio 49, 41092, Sevilla, Spain - [2]Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain

28 **Producing conductive ceramics for electro discharge machining**

R. Poyato[1,2], J. Guzmán-Mínguez[2], A. Morales-Rodríguez[2], A. Muñoz[2], A. Gallardo-López[2]

[1]Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. Américo Vespucio 49, 41092, Sevilla, Spain - [2]Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain

29 **Analysis and 3D microstructural characterization for the Ce, Y and Zr elements through the grain boundaries of 12Ce-ZrO_2/3Y-ZrO_2 composites as a function of the sintering temperature**

J.J. Roa[1,2], H. Aboulfadl[3,4], J. Barrirero[3,4], M. Turon-Viñas[1,2], F. Mücklich[3,4] and M. Anglada[1,2]

[1]Dept. Materials Science and Engineering, ETSEIB-Universitat Politècnica de Catalunya, Avda. Diagonal 647, 08028, Barcelona, Spain - [2]Centre for Research in NanoEngineering, Universitat Politècnica de Catalunya,C/Pascual i Vila 15,08028, Barcelona, Spain - [3] Chair of Functional Materials, Saarland University, Campus D3 3, D-66123, Saarbruecken, Germany - [4]Materials Engineering Center Saarland, Steinbeis Research Center, Campus D3 3, D-66123, Saarbruecken, Germany

30 **Comparison of Properties CAS Based Ceramics and Glass-Ceramics Produce From Pure and Waste Raw Materials**

N. Toplan, Z.Y. Merkit

Sakarya University, Metallurgy & Materials Engineering, 54187 Sakarya, Turkey

31 **Inkjet printing of relative humidity resistive sensors**

P. Aubry[1], Traianidis[1], V. De Wolf[2], E. Grivei[2], J.M. Tulliani[3], V. Lardot[1] and F. Cambier[1]

[1]Belgian Ceramic Research Center (BCRC), avenue Gouverneur Cornez 4, B-7000 Mons, Belgium - [2]NANO-4, Av. N. Copernic 1 , B-7000 Mons, Belgium - [3]Dipartimento Scienza, Applicata e Tecnologi (DISAT), Politecnico Di Torino, Corso Duca degli Abruzzi 24, 10129 Torino, Italy

32 **Gamma Attenuation Properties of Bi_2O_3 doped-CMAS glass ceramics enameled steel**

A.B. Tugrul[1], B. Buyuk[1], E. Demir[1], E. Altuncu[2], H.O. Toplan[2] and N. Toplan[2]

[1]Istanbul Technical University, Energy Institute, Nuclear Researches Division, Ayazaga Campus, 34469, Sarıyer, Istanbul, Turkey - [2]Sakarya University, Engineering Faculty, Metallurgy and Materials Engineering Department, Esentepe Campus, 54187, Sakarya, Turkey

33 **Gamma Attenuation Properties of PbO doped-CMAS glass ceramicsenameled steel**

A.B. Tugrul[1], N. Toplan[2], B. Buyuk[1], E. Demir[1], E. Altuncu[2] and H.O. Toplan[2]

[1]Istanbul Technical University, Energy Institute, Nuclear Researches Division, Ayazaga Campus, 34469, Sarıyer, Istanbul, Turkey - [2]Sakarya University, Engineering Faculty, Metallurgy and Materials Engineering Department, Esentepe Campus, 54187, Sakarya, Turkey

34 **The effect of additives on the thermal properties and sealing characteristic of basalt based glass-ceramics for solid oxide fuel cell application**

S. Yılmaz, A. Ates and E. Ercenk

Sakarya University, Engineering Faculty, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

35 **Enhanced magnetoelectric properties of 3SCNO-4CFO-3SCNO laminated composite**

P. Zachariasz[1], M. Sikora[2] and P. Guzdek[1]

[1]Institute of Electron Technology - Cracow Division, ul. Zabłocie 39, 30-701 Kraków, Poland - [2]AGH University of Science and Technology, Academic Center for Materials and Nanotechnology, Al. A. Mickiewicza 30, 30-059 Kraków, Poland

36 **Fabrication of $MgAl_2O_4$ spinel from synthesized nanopowders**

A. Zegadi[1,3], S. Benaissa[2,3], , M. Hamidouche[1,3], M. Kolli[1,3], G. Bonnefont[4], G. Fantozzi[4]

[1]Emerging Materials Research Unit, Ferhat Abbas University Setif 1, Algeria - [2]Optical Research and Photonics Unit, CDTA, Ferhat Abbas University Setif, Algeria - [3]Optics and Precision Mechanics Institute, Ferhat Abbas University Setif 1, Algeria - [4]MATEIS Laboratory, INSA Lyon, France

37 **Apparent Fast Interdiffusion and Localized Doping Produced in Bulk Nanocrystalline Ceria and Zirconia by Spark Plasma Sintering**

I.G. Tredici, G. Spinolo, U. Anselmi-Tamburini

Department of Chemistry, University of Pavia, V.le Taramelli 12, 27100 Pavia, Italy

The temperature gradients inside an alumina elaborate by spark plasma sintering process

Youssef Achenani[1], Malika Saädaoui[2], Abdelkhalek Cheddadi[1],
Guillaume Bonnefont[3], Gilbert Fantozzi[3]

[1]Mohammadia School of Engineers, ERSTER Team, Mohammed V University In Rabat, Avenue Ibn Sina, P.O. Box 765-Agdal, Rabat, Morocco

[2]Mohammadia School of Engineers, ERSIM Team, Mohammed V University In Rabat, Avenue Ibn Sina, P.O. Box 765-Agdal, Rabat, Morocco

[3]National Institute of Applied Sciences, MATEIS Laboratory, UMR 5510 CNRS, Lyon University, 20 Avenue Albert Einstein, F-69621 Villeurbanne Cedex, Lyon, France

Abstract

Spark Plasma Sintering (SPS) is a consolidation technique which combines the effects of pulsed electrical current and uniaxial pressure to the graphite die containing the material powder. Compared to other conventional methods, SPS offers exceptional benefits, including rapid heating rate and reduced sintering temperature and holding time, which allow to limit grain growth and to improve mechanical or physical properties of the final products. The sample temperature is of particular importance to the SPS process, since it determines the structure and properties of the material and their homogeneity. However, it is difficult to control due to inherent experimental difficulties and the large number of the involved parameters. Finite element modeling with consideration of the thermoelectric phenomena involved during SPS sintering played a crucial role in understanding this process. However, most of the numerical studies lack of experimental validation and their accuracy depends on the simplifying assumptions. In this study, standard sintering experiments of alumina were performed and their results were used to develop faithful simulations with limited approximations. Particular attention was given to the thermal boundary conditions to reproduce the experimental temperature measurements. The influence of various experimental parameters on both radial and axial temperature distributions was then investigated: sample dimensions, contacts between the sample and punches, die location.

Keywords : Spark Plasma Sintering, Temperature gradients, Finite element modeling

Hybrid sol-gel coating containing ceramic particles for mild-steel protection

Philippe Aubry[1], Cathy Delmotte[1], Simon Wallon[2], Emeline Lachery[3], Arnaud Nicolay[4], Mireille Poelman[3], Marie-Georges Olivier[4], Mireille Wenkin[2], Véronique Lardot[1] and Francis Cambier[1]

[1]Belgian Ceramic Research Centre (membre of EMRA), 4 Avenue Gouverneur Cornez, 7000 Mons, Belgium
[2]Coatings Research Institute - CoRI, 21 avenue Pierre Holoffe, 1342 Limelette, Belgium
[3]Materia Nova (membre of EMRA), 56 rue de l'Epargne, 7000 Mons, Belgium
[4]Service Science des matériaux, Université de Mons, 56 rue de l'Epargne, 7000 Mons, Belgium

Abstract

Since the early 90s, progressive ban of compounds containing hexavalent chromium has led to extensive researches to develop alternative treatments against steel corrosion, including sol-gel coating as an encouraging way. Siloxan protective layers are the most promising alternative. There are indeed a wide variety of precursors comprising one or more organic groups for obtaining hybrid coatings, combining the advantages of those components. The inorganic part contributes to the adhesion of the layer as well as its mechanical and chemical resistances. The organic part can increase the flexibility of the final layer, improve the adhesion of final organic coatings and so, its protective role.

Our work has focused on adding a self-healing property to the sol-gel matrix in case of defect or damage. It was shown that corrosion inhibitors may be incorporated into nanocontainers dispersed in sol-gel layer. If there is a scratch, the active compounds may be released on the specific location of the attack to play their protective action. Hybrid sol-gels were formulated from mixing alkoxysilanes with clay particles as nanocontainers for non-toxic inhibitors. Coatings on mild steel substrates were obtained by dip-coating and compared in terms of covering effect and thickness by SEM observations. Electrochemical Impedance Spectroscopy (EIS) was used for the evaluation of sol-gel coatings performances. Mechanical adhesion and resistance of the sol-gel barrier combined with a final organic layer will also be presented. These results were obtained in the framework of CLEARZINC project supported by FEDER and Wallonia.

Keywords : Coating, sol-gel, self-healing, mild steel.

Elaboration and characterization of mulitte obtained by reaction sintering of Algerian kaolin and aluminum hydroxide

M.L. Bella[1,2,3], M. Hamidouche[1,2], L. Gremillard[3]

[1]*Emerging Materials Research Unit*, University Ferhat ABBAS Setif 1, 19000, Setif, Algeria

[2]*Optical and precision mechanics institute, University Ferhat ABBAS Setif 1, 19000, Setif, Algeria.*

[3]*MATEIS UMR CNRS 5510, INSA-Lyon, Bat Blaise pascal, 7 Av. Jean Capelle, Villeurbanne, 69621, Lyon, France.*

Abstract

Kaolin is commonly used in the production of ceramic materials. This natural material has the advantage of being abundant with affordable cost. The main mineral component of kaolin is kaolinite ($Al_2O_3.2SiO_2.2H_2O$). During heating, kaolinite undergoes many structural and microstructural transformations leading to the formation of mullite ($3Al_2O_3.2SiO$) and silica (SiO_2), according to the following reaction: 3 ($Al_2O_3.2SiO_2.2H_2O$) → $3Al_2O_3.2SiO_2$ + $4SiO_2$ + $6H_2O$ In this work, we developed a mullite from Algerian kaolin and commercial aluminum tri-hydroxide. Kaolin comes from 3 referenced sites (DD1, DD2 and DD3) located at Djebel Debbagh (north-East of Algeria). The ores are extracted from open sky mines as friable rocks.. The raw powders are analyzed using different analytical technics (DTA / TGA, XRD, SEM …). Then, crude kaolin is reduced to an average particle size similar to that of $Al(OH)_3$ (D_{50}~2 µm) and mixed with $Al(OH)_3$ (in proportions such that the formation of SiO_2 is avoided), using different milling technics. Then the composite powder is dried and processed into a green body, either using uniaxial pressure or slip casting. Finally, the green bodies are sintered in air. All steps of this preparation are conducted aiming at a simple, robust and cheap process. The prepared samples under different conditions were finally characterized. The most important properties were measured and studied, such as: microstructure (grain shape (acicular), grain size (0.42µm), pore size (0.6µm)), density (2.97 ~ 3g / cm3), porosity (6-7 %), wear resistance, strength and toughness. These results were analyzed and discussed.

Keywords : Kaolin, mullite, aluminum hydroxide, sintering, acicular ceramics.

Spark Plasma Sintering mechanisms of Spinel ($MgAl_2O_4$) nanopowders

S. Benaissa[1,3], A. Zegadi[2,3], M. Hamidouche[2,3], M. Kolli[2,3], G. Bonnefont[4], G. Fantozzi[4]

[1]*Optical Research and Photonics Unit, CDTA, Ferhat Abbas University Setif 1, Algeria*

[2]*Emerging Materials Research Unit, Ferhat Abbas University Setif 1, Algeria*

[3]*Optics and Precision Mechanics Institute, Ferhat Abbas University Setif 1, Algeria*

[4]*MATEIS Laboratory, INSA Lyon, France*

Abstract:

Magnesium aluminate spinel (MgAl2O4) is considered as one of the most promising optical ceramics. It has been under development for many years; the primary focus has been especially on transparent armor, high temperature windows, infrared windows and advanced electromagnetic (EM) windows, etc. It has received a great deal of attention because of its attractive properties such as high melting point, low thermal expansion, high resistance to chemical attack, good mechanical strength, low dielectric constant and good transparency in the visible and IR wavelength ranges .

To be transparent, MgAl2O4 microstructure should be optimized since light transmittance is sensitive to microstructure, such as pores, grain boundaries and impurities, etc. Several reports have shown the effect of such features on the light transmission properties of various polycrystalline materials.

In this work, transparent spinel ceramics MgAl2O4 were made from spinel nanopowders by Spark Plasma Sintering (SPS) at different sintering temperatures. The optical and the mechanical properties of the fabricated spinel are determined as a function of the sintering temperature (optical transmission, refractive index, optical gap, hardness, elastic modulus, toughness and friction coefficient).

Keywords : SPS, Spinel, transparent.

A comparative study of different strategies to obtain quantitative chemical information from hyperspectral data based on the Fourier Transform Infrared Spectroscope (FTIR)

Thomas Bonnal, Geneviève Foray, Solène Tadier, Elodie Prud'homme

MATEIS, INSA Lyon, 20 Avenue Albert Einstein, 69621 Villeurbanne

Abstract

Over the past twenty years, techniques for hyperspectral data acquisition have been deeply improved. Today, Fourier Transform Infrared Spectroscope (FTIR) allows chemical imaging by recording the spectroscopic pattern with a micrometric spatial resolution. Such chemical imaging produces quantitative information on ceramics aging, which is crucial for better material design. Hyperspectral analysis is linked to the local spectra deconvolution in pure spectra per component. Beer-Lambert law leads to an overdetermined system. Calibration step and self-modelling curve resolution techniques (SMCR) are investigated to solve this system. Two model materials are elaborated (i) calcium phosphate with inclusions (ii) limestone filler binded by an organic additive. Both are analyzed to evaluate the quality of the deconvolution. Several parameters have been examined: roughness, component size and degradation type.

- Increasing surface roughness leads to higher signal/noise ratio characterized by a heteroscedastic and endogenous noise.
- Penalized regressions have shown accurate capacity to deal with higher signal/noise ratio and a heteroscedastic noise. But they require a second spectral technique to perform the calibration step.
- Without any *a priori* information, SMCR lead to a reliable mapping of components. Main limitations are component size and noise.

It has been shown that the quality of aging imaging is linked to the degradation. A protocol was established to select the most convenient technique. It was first evaluated on two degradations and will be extended to other materials.

Keywords : Fourier Transform Infrared Spectroscopy, Chemical Imaging, Hyperspectral Imaging, Multivariate Analysis, Calibration problem, Aging Ceramics

Direct-ink writing of high-strength dense ceramics

E. Camposilvan[1, 2], V. Garnier[1], L. Gremillard[1] and J. Chevalier[1]

[1]*Université de Lyon, INSA de Lyon, MATEIS CNRS UMR5510, 7 Av. Jean Capelle, 69621 Villeurbanne, France*

[2]*Department of Materials Science and Metallurgical Engineering, Universitat Politècnica de Catalunya, Av. Diagonal 647, Barcelona, Spain*

Abstract

Direct-ink writing -also called “robocasting”- has shown in recent years the capability for creating 3D complex ceramic bodies with controlled porosity. By extruding a self-setting ceramic paste in form of filament, the desired shape is first created in a layer-by-layer fashion and then densified at high-temperature.

When attempting to shape dense ceramic bodies, this technique has not yet been capable for reproducing the mechanical properties obtained by conventional processing, especially for high-strength materials. Printing precision, pattern design, layer-to-layer spacing and substrate adhesion are more critical for ceramics than for other classes of materials.

The current study is focused on the role of printing parameters and paste rheology on the presence of defects and mechanical properties in final dense parts, showing that a perfect control of the process allows to attain high-strength products.

Keywords : Direct-ink writing, additive manufacturing, yttria-stabilized zirconia, robocasting

Wear Behaviour of Cordierite doped with ZrO_2 Produced from Zeolite

Betül Çitak, Ayşe Şükran Demirkiran

Sakarya University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

Abstract

Cordierite ($2MgO.2Al_2O_3.5SiO_2$) that is known as a high technology ceramics was produced from mechanically activated powder mixture with the intention of increasing the reactivity of substance. The powder mixture consisted of zeolite that was used as a source of silica, pure MgO, Al_2O_3 and as dopant ZrO_2. During powder preparation, mechanical activation duration, speed of main disc and ball to mass ratio was kept constant at 60 min, 600 rpm and 20:1, respectively. After compacting of the mechanically activated powder mixture, the samples were sintered at 1250°C for 1 hour. Friction and wear tests were carried out in dry test conditions under the 1, 3 and 5 N loads at the (0,3) m/s sliding speeds.

Experimental study of electromagnetic field impact on surface diffusion in zinc oxide ceramic.

A. Ducoulombier, F. Valdivieso, J. Bruchon, S. Saunier, P. Ganster, C. Meunier

LGF CNRS-UMR 5307, Mines de St-Etienne, 158 cours Fauriel, 42023 St-Etienne, France

Abstract

Sintering is a key step to obtain a dense material from powders. Compared to conventional heating, microwave heating is a promising way to obtain a faster densification, and at lower temperature. Controlling this process includes a well understanding of the elementary phenomena which occurs during microwave sintering, and especially the electromagnetic wave coupling with matter which would affect diffusion mechanisms and consequently sintering kinetics.

Based on V.W. Mullins works, several studies have shown the possibility to determine the coefficient of diffusion by measuring at a set of temperatures the time evolution of scratch profiles realized on samples. The aim of this work is to study sintering mechanism comparing the kinetic of surface diffusion by two heating ways.

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) with a high purity ZnO powder. The coefficient of surface diffusion and the activation energy associated will be then determined at low temperature (below 700°C). In this aim, a specifical method has been set up to produce samples with high densification, large grain size, and very low rough surfaces. First, the surfaces are scratched using FIB technics. Then, the levelling of these scratches and their widths during heat treatment, are followed by AFM observations from which the diffusion coefficient would be deduced.

Next step of this study will be to determine the different kinds of diffusion (surface, grain boundary, bulk diffusion) which are expected at higher temperature.

Keywords : sintering, microwaves and conventional heating, atomic diffusion, microstructural characterizations

Processing and characterization of zirconia (3Y-TZP) composites with graphene nanoplatelets

A. Gallardo-López[1], I. Márquez-Abril[1], A. Morales-Rodríguez[1], A. Muñoz[1], R. Poyato[1,2]

[1]*Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain*

[2]*Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. AméricoVespucio 49, 41092, Sevilla, Spain.*

Abstract

The addition of graphene to ceramics is relatively recent and has as major objectives reinforcing and enhancing mechanical properties of the resulting composites and making them electrically conductive. In order to obtain the best composites performance, fabrication has to be optimized focusing in preserving graphene and avoiding its agglomeration in the ceramic matrix. With these objectives, composites of 3 mol% yttria tetragonal zirconia (3Y-TZP) containing 1 vol% graphene platelets and monolithic 3Y-TZP ceramic have been prepared by colloidal processing and sintered by spark plasma (SPS), using different processing and sintering parameters. During processing and after sintering microstructure has been analyzed to determine distinct phases in the ceramic and to assess graphene dispersion. Hardness and toughness studies have been also carried out both on graphene / 3Y-TZP and on monolithic 3Y-TZP ceramic to estimate the influence of graphene on the room temperature mechanical properties of these composites, and finally their electrical conductivity has been measured.

Keywords : graphene, composites,processing, microstructure.

A Choline Chloride/DMSO solvent for the direct synthesis of diformylfuran from carbohydrates in the presence of heteropolyacids

W. Ghezali[1,2], K. De Oliveira Vigier[1], R. Kessas[2] and F. Jerome[1]

[1]UMR 7285 CNRS, IC2MP, Université de Poitiers, 1 rue Marcel Dore TSA 41105-86 073 POITIERS Cedex 9, France

[2]Université des Sciences et de la Technologie d'Oran – Mohamed Boudiaf, BP 1505 El-M'Naouar, Oran, Algérie.

Abstract

Here we demonstrate that choline chloride has a beneficial effect on the direct conversion of fructose and inulin to DFF in the presence of Mo-V containing Keggin heteropolyacid. ChCl have two beneficial effects : 1) the enhancement of the conversion of fructose to HMF and 2) a higher conversion of HMF to DFF. A yield to DFF of 84% was obtained from fructose in the optimized conditions. In this mixture inulin was also converted to DFF.

Keywords : Diformylfuran, biomasse, hétéropolyacides.

Low temperature degradation and mechanical properties of (Y,Ce-) co-stabilized zirconia

Andrea Gommeringer, Frank Kern and Rainer Gadow

Institut für Fertigungstechnologie keramischer Bauteile, Universität Stuttgart, Allmandring 7b, 70569 Stuttgart, Germany

Abstract

TZP materials stabilized by either yttria or ceria offer adverse properties such as high strength and moderate toughness and low temperature degradation resistance in case of Y-TZP and high toughness and ageing resistance at a limited level of strength for Ce-TZP. In this study aiming at a new material concept for e.g. dental implants a co-stabilization of TZP with both yttria and ceria was investigated to obtain a material with high strength, toughness and ageing resistance. A submicron size zirconia powder was coated with 1 mol.% yttria via the nitrate route and afterwards blended with 4 to 8 mol.% nanoscale ceria by mixing and milling. 2 vol.-% alumina were added in all cases. Samples were shaped by slip casting in plaster molds and subsequently sintered in air at 1450 °C – 1550 °C at 2h dwell. Mechanical properties such as 4-pt bending strength, hardness and fracture resistance were determined. The microstructure was studied by SEM. Low temperature degradation resistance (LTD) was determined by measuring the monoclinic and tetragonal content of the zirconia phase before and after ageing in saturated water vapor at 134 °C for 1 to 100 hours by XRD.

The characterization of the materials showed variations of mechanical properties over a broad range, depending on compositional and processing variables. Y-Ce-TZP can reach high strength up to 1110 MPa and high fracture toughness (K_{IC}) up to 13 MPa$\sqrt{m}$. As expected low temperature degradation decreases with increasing ceria content. At high ceria costabilizer contents > 6 mol-% the transformed fraction stays below 3 % after ageing for 10 hours.

Keywords : Y-Ce-TZP, co-stabilized Zirconia, LTD, ageing behavior.

In situ TEM Nano-Compression and Mechanical Analysis of MgO and Al_2O_3 nanoparticles

I. Issa[1,2], L. Joly-Pottuz[1], J. Réthoré[2], C. Esnouf[1], V. Garnier[1], J. Amodeo[1], J. Morthomas[1], J. Chevalier[1] and K. Masenelli-Varlot[1]

[1]*INSA-Lyon, MATEIS CNRS UMR5510, 69621 Villeurbanne, France*

[2]*INSA-Lyon LaMCoS CNRS UMR5259, 69621 Villeurbanne, France*

Abstract

Nanometer-sized objects (micropillars, thin films and nanowires) are attracting large attention nowadays due to their breaking through mechanical properties such as high hardness, crack propagation resistance and high elastic limit in comparison to their bulk counterparts. Moreover, these nano-objects exhibit large plastic deformation under high load. The origin of this plastic deformation is still not very well defined. The mechanisms proposed are size-dependent and report this behavior to dislocation nucleation at surfaces and slipping on certain planes depending on the crystal orientation with respect to the solicitation direction. In this study, we propose an innovative mechanical observation protocol of nanoparticles in the 100nm size range. It consists of *in situ* TEM nano-compression tests of isolated nanoparticles. Load–real displacements curves, obtained by Digital Image Correlation, are analyzed and these analyses are correlated with Molecular Dynamics simulations. A constitutive law with the mechanical parameters (Young modulus, Yield stress...) of the studied material at the nano-scale can be obtained. In-situ TEM nano-compression tests were performed on ceramic MgO nanocubes and Al_2O_3 nanoparticles. Magnesium oxide is a model material and its plasticity is very well known at bulk. The MgO nanocubes show large plastic deformation, more than 50% of plastic strain without any fracture.

In situ TEM nano-compression tests of isolated alumina nanoparticles reveal plastic deformations of the nanoparticles. To monitor the behavior of nanoparticles during compaction process, alumina nanoparticles were compacted and thin foils of these compacted nanoparticles were prepared by FIB for HRTEM observations. Their analysis reveals a preferred orientation for the deformation.

Keywords : *In situ*, TEM, deformation mechanism, dislocations

Nanostructured Zirconia Composites with High Ageing Resistance

M. Johannes[1], J. Schneider[2], S. Begand[1]

[1]Fraunhofer Institute for Ceramic Technologies and Systems, Department of Oxid- and polymer-ceramic Components, Michael-Faraday-Str. 1, 07629 Hermsdorf/Germany

[2]Bauhaus University, Faculty of Construction Chemistry and Polymers, Coudraystr. 11a, 99423 Weimar/Germany

Abstract

Dense-sintered ceramic bodies of 3Y-TZP and ATZ (90%Y-TZP) were prepared from commercial powders by means of slip casting, sintering and hot isostatic pressing. The powders were processed in an optimized milling procedure. The properties of the milled particles were determined with diffusive light scattering, Rietveld refinement, BET and SEM. The sintered ceramics were characterized in term of density, microstructure, bending strength, hardness and fracture toughness. The ageing behavior war investigated by using hydrothermal treatment at 134°C up to 200 hours. The phase composition of the sintered ceramic was measured before and after ageing. The enhanced sintering activity allows a decrease in sintering temperature by more than 100 K. After hot isostatic pressing samples reached at least 99.5 % of the theoretical density with a median grain size of 150 nm for 3Y-TZP and 143 nm for ATZ. The bending strength measurement of nanostructured Y-TZP resulted in 1100 MPa and 1700 MPa for ATZ. In terms of fracture toughness, no significant difference was observed. The phase composition of these nanostructured ceramic is not influenced by low-temperature degradation during hydrothermal treatment. The monoclinic zirconia phase is less than 1 wt% even after 200 hours of ageing. In conclusion, it could be proven that a new generation of nanostructured Y-TZP and ATZ ceramic with high resistance against low temperature degradation was manufactured.

Keywords : Zirconia, Nanostructured ceramic, ageing resistance

Dielectric Properties of Zirconium Modified $(Na_{0.5}Bi_{0.5})TiO_3$ Ceramics by Mechanical Activation

Derya Kırsever and H. Özkan Toplan

Sakarya University, Metallurgy and Materials Engineering, Sakarya, Turkey

Abstract

Piezoelectric Zr modified $(Na_{0.5}Bi_{0.5})TiO_3$ (NBT) powders have been prepared by conventionally mixed-oxide method and mechanical activation of BiO_2, Na_2CO_3,TiO_2 and ZrO_2. Non-activated and activated Zr modified NBT powders were characterized by using X-Ray diffraction (XRD), scanning electron microscopy (SEM). Also, mechanical activated Zr modified NBT powders were uniaxially pressed and all pellets were sintered at 1000, 1100, 1200°C for 2 hour in air atmosphere. The microstructure and phase analysis of sintered pellets was investigated by using SEM and XRD. Dielectric properties measurement also were reported.

Keywords : lead free piezoelectric ceramics, mechanical activation, sodium bismuth titanate

Effect of Mechanical Activation on the Structural and Electrical Properties of Mn doped $(Na_{0.5}Bi_{0.5})TiO_3$ Ceramics

Derya Kırsever and H. Özkan Toplan

Sakarya University, Metallurgy and Materials Engineering, Sakarya, Turkey

Abstract

Piezoelectric Mn doped $(Na_{0.5}Bi_{0.5})TiO_3$ (NBT) powders have been prepared by conventionally mixed-oxide method and mechanical activation of BiO_2, Na_2CO_3,TiO_2 and MnO. Non-activated and activated Mn doped NBT powders were characterized by using X-Ray diffraction (XRD), scanning electron microscopy (SEM) and differential thermal analysis (DTA). Also, mechanical activated Mn-doped NBT powders were uniaxially pressed and all pellets were sintered at 1000, 1100, 1200°C for 2 hour in air atmosphere. The microstructure and phase analysis of sintered pellets was investigated by using SEM and XRD. Results of dielectric property measurement was also reported.

Keywords : lead free piezoelectric ceramics, mechanical activation, sodium bismuth titanate

Surface modification of zirconia-toughened alumina bioceramics for orthopedic applications

Quentin Flamant[1], Carlos Caravaca[2], Sylvain Meille[2], Laurent Gremillard[2], Jérôme Chevalier[2], Katia Biotteau-Deheuvels[3], Meinhard Kuntz[3], Rona Chandrawati[4], Inge K. Herrmann[4], Christopher D. Spicer[4], Molly M. Stevens[4] and Marc Anglada[1]

[1]Department of Materials Science and Metallurgical Engineering, Universitat Politècnica de Catalunya, Av. Diagonal 647, 08028 Barcelona, Spain.
[2]Université de Lyon, INSA-Lyon, MATEIS UMR5510, 7 avenue J. Capelle, 69621, Villeurbanne Cedex, France.
[3]CeramTec GmbH, 73207 Plochingen, CeramTec-Platz 1-9, Germany
[4]Department of Materials, Department of Bioengineering and Institute of Biomedical Engineering, Imperial College London, London SW7 2AZ, United Kingdom.

Abstract

Due to their outstanding mechanical properties and excellent biocompatibility, the use of Zirconia Toughened Alumina (ZTA) ceramics in total joint replacements has grown rapidly over the last decade. Nevertheless ZTA is a bioinert material. In the absence of adequate surface modification, this can lead to poor osseointegration and subsequent aseptic loosening. For this reason, in current hip replacement systems, a metal shell with an osseointegrative surface needs to be placed between the acetabular bone and the external surface of the ceramic liner, which restricts the maximal head diameter because of the limited anatomical space. It would thus be beneficial to develop surface modification processes that enable the implantation of ZTA monoblock components in direct contact with bone. Furthermore, periprosthetic joint infections are now the leading cause of failure for joint arthroplasty prostheses.

To address both issues, an improved surface design is required. In this study, two surface modification processes were developed for this purpose: injection molding to tailor the micro-topography and selective etching to induce nano-roughness and interconnected nanoporosity. The porosity allowed loading of an active therapeutic cargo, and the impact of selective etching on mechanical properties and hydrothermal stability was shown to be limited. The combination of injection molding and selective etching thus appears promising for fabricating a new generation of ZTA components implantable in direct contact with bone.

Keywords: Surface modification, Roughness, Topography, Bioceramic, Drug delivery

Characterization of in-situ ZrB_2-Al_2O_3 composites Produced by Powder Metallurgy

Damla Koroğlu, Betül Citak, Ozkan Ozdemir, A. Şükran Demirkiran

Sakarya University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

Abstract

Zr-Al-B based composites having properties such as high strength, corrosion resistance, wear resistance and low density are promising materials. In this study, aluminum powder (99% purity, 15 μm), ZrO_2 powder (98.8% purity, less than 1 μm) and B_2O_3 (99.99% purity, less than 38 μm) were used as raw materials to produce ZrB_2-Al_2O_3 composite by aluminatermic reduction. The powder mixtures were prepared according to $3ZrO_2$-$3B_2O_3$-10Al stoichiometry. After compacting with cold isostatic pressing (CIP), the samples were sintered in an electrical resistance furnace with 6 °C/min heating rate in the Ar atmosphere at 1350°C for 180 min period. Microstructural characterization of the sintered samples was performed by XRD and SEM-EDS analysis. The XRD analysis results showed the present of ZrB_2, Al_2O_3 and Al_3Zr. The wear behaviour of the samples was examined under different loads and the specific wear rate and the friction coefficient values were determined.

Keywords : Sintering; alumina-termic reduction; ZrB_2-Al_2O_3 composites

Reinforcement of Hydroxyapatite Bioceramic by Addition of ZrO_2 and Al_2O_3

S. Laasri[1], M. Essadik[2], M. Taha[2], A. Laghzizil[3], E-K. Hlil[4], A. Hajjaji[1]

[1]*Laboratoire des Sciences de l'Ingénieur pour l'Energie, Ecole Nationale des Sciences Appliquées d'El Jadida, BP 1166, EL Jadida Plateau 24002, Morocco*

[2]*Laboratoire de Thermodynamique et Energétique, Université Ibn-Zohr, Faculté des Sciences, BP 8106 Agadir Morocco*

[3]*Laboratoire de Chimie Physique Générale, Faculté des Sciences, Université Mohammed V, BP.1014 Rabat Morocco.*

[4]*Institut Néel, CNRS et Université Joseph Fourier, BP 166, 38042 Grenoble, France*

Abstract

The development of new biomaterials with enhanced mechanical and physical properties and biocompatibility has become a major challenge in biomaterials community. In this case, biomaterials play a very important role. Among different categories of biomaterials such as hydroxyapatite HAP is responsible for biomineralization, osteoinduction, and osteointegration .and has good biocompatibility and bioactivity. Pure HAP shows poor mechanical properties: low strength (<120MPa) and low fracture toughness, therefore, different inorganic additives have been introduced in HAp matrix in order to improve these properties. Among them, we selected zirconia ZrO_2 and alumina Al_2O_3 as reinforcing agents.
Hydroxyapatite powder has been synthesized from $Ca(OH)_2$ and (NH_4) H_2PO_4 solutions by the neutralization method. Similar method of the preparation in the presence of different concentrations of Zr and Al precursors. In present work, we investigated the effects of ZrO_2 and Al_2O_3 addition on the mechanical and physical properties of HAP. The thermal stability of the prepared composites was studied via calcining in the wide temperature range and analyzed by FT-IR, XRD and SEM. The structural parameters, as well as ZrO_2 / Al_2O_3 and HAP coexistence, promise good mechanical properties of the co-precipitated composite.

Keywords : hydroxyapatite ; Zirconia ; Alumina ; mechanical and physical properties

Role of inclusions on the diffusion after spark-plasma sintering of Ce:YAG for optical applications

Etienne Lachaud[1,2,3], Aurélien Vivet[3], Vincent Garnier[1], Yannick Guyot[2], Gilbert Fantozzi[1], Lionel Bonneau[3], Georges Boulon[2], Guillaume Bonnefont[1]

[1]MATEIS, INSA de Lyon, avenue Jean Capelle, Villeurbanne

[2]ILM, Université Claude Bernard Lyon 1, Domaine scientifique de la Doua, bâtiment Kastler, Villeurbanne

[3]Baikowski, les Marais Noirs, Poisy

Abstract

Transparent polycrystalline ceramics represent nowadays a topic of great interest. Ce:YAG is mainly used for his luminescence properties, since its combination with a blue LED, classically an InGaN semiconductor, allows the production of a bright white LED. Compared to the traditional polymer-ceramic composite, a full ceramic device can reach higher temperatures avoiding thermal degradation. In this study, a transparent polycrystalline ceramic has been obtained by SPS from a Ce:YAG commercial powder supplied by Baikowski. Powder characteristics are a key parameter for these applications since they have a strong influence on densification. After sintering, the samples have been characterized by SEM and their transparency properties have been evaluated. A statistic study of the results has revealed the effect of both secondary phases and porosity on the Real In-line Transmission (RIT). These results suggest the influence of nature and size of the inclusions on the light diffusion.

Keywords : Transparency, diffusion, SPS, YAG.

Assembly of transparent polycrystalline Er :YAG ceramic for Laser application

M. Lagny[1,2], A. Katz[1,2], J. Boehmler[1], S. Lemonnier[1], É. Barraud[1], S. D'Astorg[2], Y. Lorgouilloux[2], A.Leriche[2]

[1]ISL, 5 rue du Général Cassagnou, 68300 SAINT-LOUIS

[2]LMCPA, Pôle universitaire de Maubeuge, Boulevard Charles de Gaulle, 59600 MAUBEUGE

Abstract

The laser developed at the ISL is a Solid State Heat-Capacity Laser (SSHCL): the gain medium is an Erbium-doped YAG single crystal (Er :YAG) which emission at 1.6µm is «eye-safe ». The major drawbacks of this laser are its reduced power of solicitation and lasing time, both due to an accumulation of heat in the laser cavity during use. Because of its homogenous doping, single crystal causes an overheating phenomenon which limits the lasing time. In that context, Er:YAG transparent polycrystalline ceramics present a real interest: thanks to their tunable fine microstructure, they have better thermomechanical properties than single crystals. They also permit the conception of massive transparent pieces with a controlled doping profile, which leads to a better repartition of heat in the material. In order to integrate the polycrystalline ceramic in a SSHC laser, one must focus on two aspects: i) a scale-up to the ideal dimension for a SSHCL use, ii) the development of high-doping gradient ceramics, allowing a better repartition of heat in the solid medium.

The development of a stacked composite made of sintered pellets is a solution for both scaling up and doping gradient issues. The challenge here is to develop an interface with high mechanical, thermomechanical and optical properties without damaging the ones already achieved in each single pellet. *This approach takes* two steps: first, the synthesis of SPS sintered ceramic. Secondly, a couple of samples are mirror polished and co-stacked: this last step favours the development of porosity-free interfaces.

Keywords : Er:YAG, co-stacked, assembly, SPS sintered

Effect of microstructure on electron trapping and conduction in polycrystalline ceramics under defocused electron beam irradiation in SEM

Omar Mekni[1,2], Dominique Goeuriot[1], Gilles Damamme[3]

[1]*Laboratoire Georges Friedel CNRS UMR 5307, Ecole des Mines de Saint Etienne, 158 coursFauriel, 42023 Saint-Etienne, France*

[2]*Laboratory of Materials, Organization and Properties LR99ES17, University of Tunis El Manar, Campus Universities El Manar, 2092 Tunis, Tunisia*

[3]*CEA / Gramat, BP80200, 46500 Gramat, France*

Abstract

Ceramics are widely used as electrical insulating materials such in high voltage electric energy transport, microelectronics and aerospace. Their choice is related to their breakdown voltage which limits their use and causes some reliability problems. To improve the resistance to dielectric breakdown it is necessary to understand and control the cause of this damage process. The microscopic damage processes in insulating materials under electrical stress are indisputably related to energy localization on defects. Indeed, electron trapping causes localization of energy which is relaxed after detrapping, causing a great energy release leading to breakdown and making lose to ceramic its electrical insulation character. In fact, the charge injection and the dielectrics characterization by electron beam techniques are particularly suited to the damage process study. In this context, the purpose of this work is to develop a technique (Induced Current Method) using a specific configuration in the Scanning Electron Microscope (Carl Zeiss SUPRA 55 VP) chamber in order to measure separately and simultaneously the influence and the conduction currents and to trace back to the trapped charge dynamic during and after electron irradiation. This technique improves understanding of trapping phenomenon, transport and stability of trapped charges. The studied ceramics are polycrystalline α-alumina and yttria stabilized zirconia. The grains size and MgO doping effects and the microwave sintering effect on electrical properties of these ceramics are studied and discussed. Via the developed technique, the microstructure - dielectric strength correlations could be well justified.

Keywords : Polycrystalline ceramics, Dielectric breakdown, Electron localization, Induced Current Method, Charge trapping.

Thermomechanical properties and fracture of resin-bounded-sand cores. Application in aluminum foundry.

C. Menet[1], P. Reynaud[1], G. Fantozzi[1] and A. Laforet[2]

[1]MATEIS, INSA Lyon, 7 avenue Jean Capelle, 69621, VILLEURBANNE, France
[2]Montupet, rue Jean-François Champollion, 36130, DIORS, France

Abstract

Automobile cylinder heads are casted with a semi-permanent mold casting process because of their complex shapes. A “sand core” permits the casting of internal cavities. Sand cores are composed of bounded-sand grains. A binder makes bridges between the sand grains and could be made of organic resin or inorganic polymer (alkali silica). During the casting of the liquid aluminum (near 700°C), binder-bridges are thermally damaged, that will make easier the decoring step, in other words the removing of the sand through the pipes of the cylinder head after hammering and vibration of the item.

This kind of granular material has specific properties but has received little attention. Its breakage resistance must be high enough to allow handling and bearing of the weight of the aluminum alloy. However, this resistance must also be low enough to make the decoring step possible. This presentation will deal with the mechanical properties, the damage and breaking mechanisms of a sand core depending on its thermal history and also on the kind of mechanical load. The influence of the binder is primordial and though modifications in its nature, weight percent or intrinsic properties will change deeply the behavior of the material. The degradation of the binder due to the high temperatures of the casting process is the key of the mechanical properties bivalence.

A correlation is indeed possible between the mechanical properties, the weight loss induced by the binder damage and the fracture surface observed under a MEB. Various cycled or monotonous mechanical tests have been performed in order to examine the response of the material subject to compressive, bending or shear stress. A particular attention has been paid to crack propagation. In particular, some compressive tests have been followed in-situ by X-ray tomography, permitting to determine the evolution of the strain field during the test.

Keywords : Granular material, Sand-core, Aluminum casting, mechanical properties, fracture

Corrosion and low temperature degradation of zirconia dental ceramics

Aleksandra Nowicka, Dagmar Galusková, Stanislav Mikušinec and Dušan Galusek

Vitrum Lauguricio – Joint Glass Center of the IIC SAS, TnU AD, and FChFT STU, Študentská 2, 911 50 Trenčín, Slovakia

Abstract

Ceramic materials have been widely studied in recent years as part of a strategy aimed at avoiding intraoral metals in dental implants, and also due to their excellent osseointegration and favorable mechanical properties. Ceramic made of zirconium oxide polycrystals stabilized in their tetragonal form by the addition of 3 mol% of yttrium oxide (denoted as 3Y-TZP) is a material of choice in advanced dental applications and dental implantology. However, as shown in hip implants made of 3Y-TZP, the ceramics are vulnerable to low temperature hydrothermal degradation (LTD). Another factor influencing the properties of dental implants is corrosion caused by excessive consumption of acidic food and beverages combined with poor mouth hygiene, or some health issues associated with excessive production of gastric acid. The aim of this work was study of corrosion in acidic media and its influence on ion leaching, phase composition, micromechanical and tribological properties of a commercial 3Y-TZP dental ceramic. Our last un-published results indicate that acidic corrosion of 3Y-TZP dental ceramics is associated with leaching of yttrium from zirconia ceramics, resulting in partial destabilization of tetragonal zirconia and measurable increase of the content of monoclinic phase at the surface. The influence of corrosive attack on low temperature degradation of a commercial 3Y-TZP dental ceramic has been also studied with the use of an accelerated ageing test (AAT).

Keywords : Zirconia dental ceramics, corrosion, low temperature degradation.

Grain growth kinetics and regression analysis of the PbO-doped $BaTiO_3$ ceramics

Kerim Emre Öksüz[1], Şaduman Şen[2] and Uğur Şen[2]

[1]*Cumhuriyet University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 58140, Sivas, Turkey.*

[2]*Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, 54187, Sakarya, Turkey.*

Abstract

In this study, grain growth kinetics and sintering behavior of PbO doped barium titanate based ceramic materials were investigated depending on sintering temperature and time. In this process, PbO-doped $BaTiO_3$ ceramic powder substitution was prepared by solid state mixed oxide technique. X-ray diffraction analysis showed that the main phases formed in the sintered samples are $BaTiO_3$. Atomic force microscope (AFM) images were used for examinations of the grain structure and size of the sintered samples. Grain growth kinetic equation $Gn=K.t.exp(-Q/RT)$ was used for determining the activation energy of the PbO-doped $BaTiO_3$ ceramic materials. Lorentzian, Gaussian and Paraboloid equations were used for regression analysis of the prediction of the PbO doped $BaTiO_3$ ceramics. Classical kinetic equation and regression analysis compared with the experimental data. Regression analysis was to give better results than that of the classical kinetic results.

Keywords : Barium titanate, Grain Growth, Kinetic, Activation Energy, Sintering.

Zirconia behavior after surface laser treatment

Rafael S. F. Pereira[1], C. G. Moura, F[2]. S. Silva[2] B. Henriques[1] and M. C. Fredel[1]

[1]CERMAT, Universidade Federal de Santa Catarina, Florianópolis, Brazil
[2]Center for Micro-Electro Mechanical System, University of Minho, Braga, Portugal.

Abstract

Zirconia is a widely used ceramic material because of its high mechanical properties, achieved by a martensitic phase transformation. In addition to mechanical properties, zirconia is also a biocompatible material. However, surface modifications have been applied to this material, which leads to an increase of cells growth in contact to the oxide, developing a body friendly material. One of these modifications is promoted by a laser beam, which causes microcracks and residual stress on the material. Furthermore, zirconia degrades when in wet environments, as human body, reducing and making hard to predict its lifetime, especially with cracks. In order to analyze the influence of these treatments in color, aging and mechanical behavior, in this work zirconia samples were exposed to two different YAG laser energy power.

Keywords: Zirconia, Aging, laser, YAG.

Mechanical behaviour of a randomly porous β-TCP sample: study of the fracture path by X-ray tomography

Clémence Petit, Sylvain Meille, Eric Maire, Solène Tadier and Jérôme Adrien

INSA de Lyon MATEIS CNRS UMR5510 Université de Lyon, 20 avenue Albert Einstein, 69621 VILLEURBANNE, France

Abstract

Macroporous β-TCP samples were fabricated by a relatively simple sacrificial template method using polyethylene beads as porogens. Two types of porogens were used to create pores with two different sizes: macro-pores with a size between 100 and 300 µm and smaller pores with a size from 10 to 30 µm. These latter pores were intentionally added to generate defects in the solid phase of the macroporous samples. X-ray tomography was used to characterize the samples at the scale of the architecture and at the scale of the solid phase. For this finer scale, the samples were scanned thanks to “local tomography”, in which the specimen is placed close to the X-ray source. Then, an *ex situ* compression test was performed to follow the crack initiation and propagation by X-ray tomography. The 3D images show that the sample exhibits a typical cellular behaviour characterized by the propagation of many small cracks in different directions. Additionally, the images evidence the importance of the clusters of macro-pores on the initiation of crack propagation. For this latter point, the use of X-ray tomography confirms previous results about the mechanical behaviour in compression of highly porous ceramics.

Keywords : Porous ceramic, X-ray tomography, Mechanical behaviour

Effect of processing on microstructure and room temperature mechanical and electrical properties of 3YTZP/2.5 vol% MWNT ceramic nanocomposites

R. Poyato[1,2], Á. Gallardo-López[2], A. Morales-Rodríguez[2], F. Gutiérrez-Mora[2], A. Muñoz[2], A. Domínguez-Rodríguez[2]

[1]Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. Américo Vespucio 49, 41092, Sevilla, Spain.

[2]Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain.

Abstract

In this work, composites of 3 mol% Y_2O_3 -ZrO_2 with 2.5 vol% multiwall carbon nanotubes (MWNT) have been prepared by colloidal processing and spark plasma sintering (SPS). Different colloidal powder processing routines have been tested with the aim of achieving an homogeneous distribution of the MWNTs in the ceramic, eliminating agglomerates but also minimizing CNT damage during processing. Modifications of the acid treatment applied to the nanotubes, application time of high energy ultrasonic agitation during MWNT dispersion or pH during composite powder mixing have been approached. Also, liofilization of the composite powder mixed suspension versus drying on a hot plate has been

Characterization of the composites phases and microstructure was carried out by means of X-ray diffraction and scanning electron microscopy (SEM). Possible damage of the MWNTs during processing was analyzed by Raman spectroscopy. Hardness and fracture toughness, together with electrical conductivity at room temperature have been also obtained and correlated to the different microstructures obtained in the composites prepared with the different processing routines.

Keywords : processing, composites, carbon nanotubes.

Producing conductive ceramics for electro discharge machining

R. Poyato[1,2], J. Guzmán-Mínguez[2], A. Morales-Rodríguez[2], A. Muñoz[2], A. Gallardo-López[2]

[1]*Instituto de Ciencia de Materiales de Sevilla, CSIC-Univ. de Sevilla, Avda. Américo Vespucio 49, 41092, Sevilla, Spain.*

[2]*Departamento de Física de la Materia Condensada, ICMSE CSIC, Universidad de Sevilla, P.O. Box 1065, Sevilla, Spain*

Abstract

One of the limitations of advanced ceramics is the difficulty to produce complex shapes, which would be necessary for emerging applications in different fields as biomedical implants, pieces for turbine engines, rotors, or different devices. The high values of hardness and wear resistance of ceramics make processes such as cutting and machining very long and costly. Electro discharge machining (EDM) could be a good alternative to conventional mechanical machining, but it requires materials with electrical conductivity higher than 1 Sm^{-1}. Therefore, this technique is usually not suitable for ceramics, which are electrically isolating. However, the recent development of composite technology with carbon nanostructures indicates that it is possible to greatly enhance electrical conductivity of ceramics, allowing EDM to be used to manufacture intricate parts. In this work, composites of Al_2O_3 with different amounts of carbon nanotubes and graphene have been prepared by colloidal processing and spark plasma sintering, and their microstructure and electrical conductivity have been characterized. Achieving an homogeneous distribution of the nanostructures in the ceramic, eliminating agglomerates and minimizing possible damage during processing are key points in order to reach the high conductivity required for EDM.

Keywords: carbon nanostructures, composites, electrical conductivity

Analysis and 3D microstructural characterization for the Ce, Y and Zr elements through the grain boundaries of 12Ce-ZrO_2/3Y-ZrO_2 composites as a function of the sintering temperature

Joan Josep Roa[1,2], Hisham Aboulfadl[3,4], Jenifer Barrirero[3,4], Miquel Turon-Viñas[1,2], Frank Mücklich[3,4] and Marc Anglada[1,2]

[1]Dept. Materials Science and Engineering, ETSEIB-Universitat Politècnica de Catalunya, Avda. Diagonal 647, 08028, Barcelona, Spain
[2]Centre for Research in NanoEngineering, Universitat Politècnica de Catalunya,C/Pascual i Vila 15,08028, Barcelona, Spain
[3]Chair of Functional Materials, Saarland University, Campus D3 3, D-66123, Saarbruecken, Germany
[4]Materials Engineering Center Saarland, Steinbeis Research Center, Campus D3 3, D-66123, Saarbruecken, Germany

Abstract

Polycrystalline tetragonal zirconia stabilized with 3 mol % yttria (3Y-ZrO_2) is extensively employed for dental applications due to its excellent mechanical properties, biocompatibility, and esthetics. If ceria is used instead of yttria, as in 12Ce-ZrO_2, higher fracture toughness and resistance to low temperature degradation (LTD) together with more tolerance to damage by contact loading is achieved. However, the details of the mechanisms responsible for these changes in mechanical as well as in chemical behavior are not well understood.

Hence, the present work conducts a systematic analysis and 3D microstructural study by means of Transmission Electron Microscopy and Atom Probe Tomography (APT) for investigating the distribution and segregation of elements such as Ce, Y and Zr through the grain boundaries (GBs) of 12Ce-ZrO_2/3Y-ZrO_2 composites sintered at 1450 and 1600ºC. Ce was found to be a common segregating element along the GB interfaces for both study temperatures while the Zr content remained unchanged. The detection of Y has been only possible in specimens sintered at 1600ºC; which might be attributed to the tendency to phase partition in Yttrium rich (cubic) and Yttrium poor (tetragonal) phases. To sum up, the APT observations highlight that the Ce segregation to the GB as well as the different yttria concentration content across the grains are some factors which may help to explain the different LTD behavior of 12Ce-ZrO_2/3Y-ZrO_2 with respect to conventional 3Y-ZrO_2.

Keywords : 12Ce-ZrO_2/3Y-ZrO_2 composite, chemical composition analysis, transmission electron microscopy – electron energy loss spectroscopy, atom probe tomography, 3D microstructure characterization

Comparison of Properties CAS Based Ceramics and Glass-Ceramics Produce From Pure and Waste Raw Materials

Nil Toplan, Zafer Yavuz Merkit

Sakarya University, Metallurgy & Materials Engineering, 54187 Sakarya, Turkey

Abstract

In order to produce CAS based ceramics pure raw materials such as $CaCO_3$, Al_2O_3, and SiO_2 were used while CAS based glass-ceramic produced from natural and waste resource as pumice, marble dust and Al_2O_3. CAS based ceramics batch formed at pressing following at homogenization proses. However, CAS glass-ceramic formed as poured into graphite mould after melting proses. Thermal treatment applied between the range to 1000-1200°C temperature and 1-5 hour for both of ceramics and glass. Phase analyses investigation with the aid of XRD and microstructure properties displayed with SEM. The crystalline phases that wollastonit determined at ceramic materials also glass-ceramic. Furthermore, anortit crystallization identified at glass-ceramics more immensely. Density result shows that glass-ceramic more dens than ceramics and according to chemical durability test into the acidic solution showed that glass-ceramics behaved more stability.

Keywords : CAS (CaO-Al_2O_3-SiO_2) ceramic/ glass-ceramic, corrosion, pumice, marble dust

Inkjet printing of relative humidity resistive sensors

P. Aubry[1], M. Traianidis[1], V. De Wolf[2], E. Grivei[2], J.M. Tulliani[3], V. Lardot[1] and F. Cambier[1]

[1]Belgian Ceramic Research Center (BCRC), avenue Gouverneur Cornez 4, B-7000 Mons, Belgium.

[2]NANO-4, Av. N. Copernic 1 , B-7000 Mons, Belgium

[3]Dipartimento Scienza, Applicata e Tecnologi (DISAT), Politecnico Di Torino, Corso Duca degli Abruzzi 24 , 10129 Torino, Italy

Abstract

A commercial sol-gel ink (Nano-4, Mons, Belgium) was used to fabricate inkjet-printed relative humidity sensors. The Dimatix DMP-2831 inkjet printer used in this work allows the control of jetting parameters for each of the 16 nozzles leading to the fine tune of coating characteristics. Up to 35 µm thick uniform coatings have been obtained. Textured coatings were achieved with a similar global thickness. The sensors performance was tested under different temperature and atmospheric conditions, using electrochemical impedance spectroscopy and a resistive sensor's signal conditioning circuit.

Interest of texturing will be discussed as well as attractive aspects of inkjet printing in comparison with screen printing.

Keywords : Inkjet printing, relative humidity sensors, texturing, resistive sensor, electrochemical impedance spectroscopy

Gamma Attenuation Properties of Bi_2O_3 doped-CMAS glass ceramics enameled steel

A. Beril Tugrul[1], Bulent Buyuk[1], Ertugrul Demir[1], Ekrem Altuncu[2], H. Ozkan Toplan[2] and Nil Toplan[2]

[1]*Istanbul Technical University, Energy Institute, Nuclear Researches Division, Ayazaga Campus, 34469, Sarıyer, Istanbul, Turkey*

[2]*Sakarya University, Engineering Faculty, Metallurgy and Materials Engineering Department, Esentepe Campus, 54187, Sakarya, Turkey*

Abstract

There have been many researches on glass-ceramic materials in the quaternary system CaO–MgO–Al_2O_3–SiO_2 (CMAS) during previous years; it has focused on materials containing diopside as the main crystalline phase. CMAS glass-ceramics with diopside as a major crystalline phase generally have high strength together with high chemical resistance and have a wide variety of applications. According to researches, the glass-ceramics can be used as sealants for fuel cell applications, glazes for floor tiles, artificial bones, dental roots and crowns.

In this study, the Bi_2O_3 doped CaO-MgO-Al_2O_3-SiO_2 glass system was prepared by melting method. Glass batches (200 g) were mixed thoroughly, and then melted in a alumina crucible at 1400 °C for 2 h in an electric furnace. The melts were then poured into graphite molds and annealed at 600 °C for 60 min to relax internal stress. After the bulk glasses were dry-milled and enameled on the stainless steel. Cs-137 and Co-60 were used as a gamma radiation sources. The linear attenuation coefficients of the Bi_2O_3 doped CMAS enameling steel were carried out for gamma radiation sources. The experimental results were compared with the theoretical lineer attenuation coefficients which were calculated by using XCOM computer code. The samples have been characterized by X-ray powder diffraction and scanning electron microscope. In conclusion, the gamma shielding property of the stainless steel substrates increased by enameled for nuclear shielding applications.

Keywords : CMAS glass-ceramics, Bi_2O_3, Lineer attenuation coefficients

Gamma Attenuation Properties of PbO doped-CMAS glass ceramics enameled steel

A. Beril Tugrul[1], Nil Toplan[2], Bulent Buyuk[1], Ertugrul Demir[1], Ekrem Altuncu[2] and H. Ozkan Toplan[2]

[1]*Istanbul Technical University, Energy Institute, Nuclear Researches Division, Ayazaga Campus, 34469, Sarıyer, Istanbul, Turkey*

[2]*Sakarya University, Engineering Faculty, Metallurgy and Materials Engineering Department, Esentepe Campus, 54187, Sakarya, Turkey*

Abstract

Glass-ceramic materials, prepared by the controlled crystallization of glasses, have a variety of established uses that depend on their uniform reproducible fine-grained microstructures, absence of porosity and other wide-range of properties, which can be tailored by an adjustment of composition and the heat treatment procedure applied. A number of new glass-ceramic glazes with nominal compositions in the CaO-MgO-Al_2O_3-SiO_2 (CMAS) quaternary system have been reported in the last years; it has focused on materials containing diopside as the main crystalline phase. This phase generally have high strength together with high chemical resistance and have a wide variety of applications.

In this study, the PbO doped CaO-MgO-Al_2O_3-SiO_2 glass system was prepared by melting method. After the bulk glasses were dry-milled and enameled on the stainless steel. Cs-137 and Co-60 were used as gamma radiation sources. The linear attenuation coefficients of the samples were carried out for gamma radiation sources. The experimental results were compared with the theoretical lineer attenuation coefficients which were calculated by using XCOM computer code. The enameled steel have been characterized by X-ray powder diffraction and scanning electron microscope. In conclusion, the gamma shielding property of the stainless steel substrates increased by enameled for nuclear shielding applications.

Keywords: CMAS glass-ceramics, PbO, Lineer attenuation coefficients.

The effect of additives on the thermal properties and sealing characteristic of basalt based glass-ceramics for solid oxide fuel cell application

Senol Yılmaz, Anıl Ates and Ediz Ercenk

Sakarya University, Engineering Faculty, Department of Metallurgical and Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

Abstract

In the current study, novel sealing glasses were developed for solid oxide fuel cell (SOFC) applications from the volcanic rock basalt. Three different glass compositions were prepared according to basalt, alkaline oxide and boron oxide content. Crushed, milled and sieved to less than 100 μm basalt powders were modified by some additives such as Na_2CO_3, K_2CO_3, $CaCO_3$, SiO_2, $MgCO_3$ and B_2O_3. Basalt based glass-ceramics were prepared by melting at 1500 °C in alumina crucible and poured into cold water to obtain glass granules. These glasses granules were milled, sieved to less than 25 μm. The fine glass powders were characterized to use for seal applications by heating microscopy and differential thermal analysis (DTA) for thermal properties such as sintering, softening, sphere, half sphere, melting and glass transition temperature. The powders were applied on the 430 stainless steel interconnector material by spin coating and heat treatment was carried out. The interface properties of the glass-ceramic layer and 430SS were investigated by using X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) depending on glass composition. The results showed that no interactions, cracks formation, or failure were observed at the 430SS/glass-ceramic sealant interface for all glass composition. Thermal properties of glass composition had changed to basalt and other additives.

Keywords : Basalt, Glass-ceramic, 430 SS, Sealant material, thermal properties

Enhanced magnetoelectric properties of 3SCNO-4CFO-3SCNO laminated composite

P. Zachariasz[1], M. Sikora[2] and P. Guzdek[1]

[1]*Institute of Electron Technology - Cracow Division, ul. Zabłocie 39, 30-701 Kraków, Poland*

[2]*AGH University of Science and Technology, Academic Center for Materials and Nanotechnology, Al. A. Mickiewicza 30, 30-059 Kraków, Poland*

Abstract

New materials that expose simultaneous orderings in one single phase are mostly preferable in modern research and innovative technical applications, e.g., opto-electronics, spintronics, information technology or computer engineering. Generally, single-phase multiferroics such as Cr_2O_3 or $BiFeO_3$ reveal a slight natural magneto-electric coupling. Therefore, heterostructure composites with two separate ferro-magnetic and ferroelectric subsystems are considering, in practice. Syntheses of $CoFe_2O_4$ ferrite and $Sr(Cu_{1/3}Nb_{2/3})O_3$ relaxor were performed by the conventional solid state reactions. Two-component heterostructures with a planar structure were prepared by a tape-casting method and co-sintering. Magnetization measurements were carried out in a temperature range from RT to 1200 K by a vibrating sample magnetometer (VSM) in a magnetic field up to 10 kOe. The SCNO-CFO ceramic exhibits the magnetic hysteresis typical of hard magnetic materials, indicating that the investigated composite are magnetically ordered. However, a discrepancy in FC and ZFC curves below 800 K indicates antiferro-magnetic transition of ferrite phase, and considerable decrease of magneto-crystalline anisotropy confirms the rearrangement of Fe atoms that finally reduce the overall magnetization of ferromagnetic phase. A comparison of the linear voltage coefficients for both forms of SCNO-CFO (particulate composite and 3-segmented laminate) reveals an increase in value of αME for composite with a planar structure. This results in the appearance of mechanical stress transferred between ferromagnetic and ferroelectric phases through piezo- and magnetostriction. In conclusion, the 3SCNO-4CFO-3SCNO laminate is showing lower coercivity, higher saturation magnetization and a higher magnetoelectric coefficient than the particulate SCNO-CFO ceramic.

Keywords : multiferroic materials, electroceramics, planar structure, tape casting

Fabrication of $MgAl_2O_4$ spinel from synthesized nanopowders

A. Zegadi[1,3], S. Benaissa[2,3], M. Hamidouche[1,3], M. Kolli[1,3], G. Bonnefont[4], G. Fantozzi[4]

[1]*Emerging Materials Research Unit, Ferhat Abbas University Setif 1, Algeria*
[2]*Optical Research and Photonics Unit, CDTA, Ferhat Abbas University Setif, Algeria*
[3]*Optics and Precision Mechanics Institute, Ferhat Abbas University Setif 1, Algeria*
[4]*MATEIS Laboratory, INSA Lyon, France*

Abstract:

Spinel $MgAl_2O_4$ has been became a technologically very attractive material due to its important physic-chemical, optical, and mechanical proprieties (high melting point 2135°C, high hardness >16GPa, good thermal shock resistance, high strength 180MPa, good resistance to corrosion, good optical transmission in UV, visible, IR wavelength ranges), if fabricated from nano powders.

The conventional synthesis in the solid state allows making $MgAl_2O_4$ spinel by the reaction between MgO an Al_2O_4 as raw materials, unfortunately the temperature required for this reaction is very high. Various wet chemical techniques have been applied for the synthesis of pure spinel powders at relatively low temperatures, but the mastery of preparation of these powders (high purity, chemical homogeneity, control of stoichiometry, fine particles size) is critical.

SPS is considered as one of the most sintering method to get transparent spinel. The transparency is strongly influenced by grain size, presence of porosity, cracking and grains boundaries.

In this current work, powders of $MgAl_2O_4$ spinel are synthesized by sol/gel chemical route. Then we expect to achieve nanostructured ceramics from these powders by SPS. Also commercial spinel nanopowders are used to fabricate massive spinel. Optical and mechanical properties (transmission, scattering, hardness…..) are determined.

Keywords : Spinel, Sol/Gel, SPS, Transparence.

Apparent Fast Interdiffusion and Localized Doping Produced in Bulk Nanocrystalline Ceria and Zirconia by Spark Plasma Sintering

I.G. Tredici, G. Spinolo, U. Anselmi-Tamburini

Department of Chemistry, University of Pavia, V.le Taramelli 12, 27100 Pavia, Italy

Abstract

In the last few years Spark Plasma Sintering (SPS) has emerged as the method of choice for the synthesis of ceramic bulk materials presenting nanocrystalline grain size. The very short sintering times typical of this technique allow obtaining other unusual characteristics. An example is represented by materials presenting a doping limited to a very small region in proximity of the grain boundary. These materials can be obtained through the sintering of nanopowders whose surface has been decorated with salts of the doping element. The extent of the doping process can be characterized analyzing the modification in ionic conductivity and in phase composition. Surprisingly, the rate of the cationic interdiffusion appear to be several order of magnitude faster than predicted by conventional spherical diffusion model. The source of this apparently anomalous interdiffusion is discussed and a possible alternative explanation, based on a more complex diffusion geometry, involving only surface diffusion – instead of bulk diffusion - processes, is presented. The potential offered by this approach for obtaining the doping of ceramic materials in situations where the usual methods for the synthesis of doped nanoparticles are difficult to pursue are also presented. Finally, it will be evidenced how the study of this process offers a unique opportunity to characterize the kinetics of the interdiffusion process in oxides at the nano scale, a process that, despite its exceptional importance in nanoscience and nanotechnology, has been poorly characterized experimentally.

Keywords: Sintering, bulk nanocristalline, Spark Plasma Sintering