



Faculty of Technology  
University of Novi Sad

VIII  
STUDENTS'  
MEETEING

Processing and Application  
of Ceramics

December 2-5, 2009



SM 2009

VIII Students' Meeting - SM-2009

**“The Eighth Students’ Meeting”**  
**PROCESSING AND APPLICATION**  
**OF CERAMICS**

**PROGRAMME**  
**and**  
**BOOK OF ABSTRACTS**



**SM-2009**

**December 2-5, 2009**  
**Novi Sad, Serbia**

**Programme and Book of Abstracts of The Eighth Students' Meeting – SM-2009, “Processing and Application of Ceramics”** publishes abstracts from the field of ceramics, which are presented at traditional international Students' Meeting.

***Editors-in-Chief***

Prof. Dr Vladimir V. Srdić  
Prof. Dr Jonjaua Ranogajec

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## **Preface**

*The Eighth Students' Meeting, SM-2009, "Processing and Application of Ceramics", is organized by the Department of Materials Engineering, Faculty of Technology, University of Novi Sad (December 2-5, 2009).*

*The idea of this kind of meeting appeared in 1998, when group of people from the Department of Materials Engineering gathered together and established the meeting which is today well recognized and greatly appreciated among the students and scientists from all over the Europe. The Students' Meeting started first as a national meeting, but with patient work and strong effort we succeeded to raise the quality up to the standards of today's International Meeting. The main goals of this traditional Meeting are the promotion of the work in the field of ceramics done by young researchers and closer international contacts between students from different universities and institutes, through exchange of knowledge, ideas and experience. We appreciate very much that European Ceramic Society recognized the efforts and the enthusiasm we have put into this idea of building the bridge between young researchers and we truly hope that the European Ceramic Society will support this initiative in the future. Special thanks to the JECS Trust Fund as one of the main sponsors of the Meeting.*

*The quality of presented papers at the Meetings and their scientific significance was inspiration for launching the international journal "Processing and Application of Ceramics". This journal today is a well established journal that among others publishes also the papers from the Students' Meeting.*

*Growing from a small national meeting, the Students' Meeting today gathers students from over 25 countries. The core themes of processing, characterization and specific material families have provided the framework for this Meeting enabling four topics for students and scientists to present ongoing results:*

- *Advanced Ceramics*
- *Ceramics Composites*
- *Traditional Ceramics*
- *Culture Heritage*

*The opening ceremony is followed by a plenary lecture of Prof. Louis Winnubst and we would like to take this opportunity to acknowledge the support and effort he made to welcome us at our Meeting. We wish to express our thanks to the members of the local organizing committee in Novi Sad for their effort and time during preparation of the meeting, and especially to thank our endorsers and sponsors for making this event possible.*

*A warm welcome to all participants of SM-2009 and hope you will have a successful meeting with many interesting discussions. Have a good and fruitful time in Novi Sad!*

*Editors*

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*The Eighth Students' Meeting – SM-2009*

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**PROGRAMME**





**WEDNESDAY, DECEMBER 2, 2009.**

**09.00 – 10.30 h – Registration**

**10.30 – 10.45 h – Opening**

**10.30 – 11.45 h – Section 1A**

*Advanced Ceramics* (Blue Hall)

- |                 |   |
|-----------------|---|
| 10.45 – 11.00 h | <b>A1 – Urban Dosler, et al., Slovenia</b><br>Low-temperature sintering and microwave dielectric properties of MgO-B <sub>2</sub> O <sub>3</sub> -SiO <sub>2</sub> -based glass ceramics                                    |
| 11.00 – 11.15 h | <b>A2 – Lavinia P. Curecheriu, et al., Romania</b><br>Functional properties of multiferroic nanocomposites with core-shell structures   |
| 11.15 – 11.30 h | <b>A3 – Moazzam Ali, et al., Germany</b><br>Chemical vapor functionalization of ZnO nanocrystals  |
| 11.30 – 11.45 h | <b>A4 – Linda Kipsova, et al., Slovakia</b><br>The preparation of MgSiN <sub>2</sub> and LaSi <sub>3</sub> N <sub>5</sub> additives and their influence on thermal conductivity of Si <sub>3</sub> N <sub>4</sub> ceramics |
| 11.45 – 12.00 h | <b>A5 – Balazs Forizs, et al., Hungary</b><br>Nanosize WO <sub>3</sub> for gas sensing and photocatalysis   |

**12.00 – 13.30 h – Welcome Party**

**13.30 – 14.15 h – Plenary lecture – Louis Winnubst, The Netherlands**  
Porous and dense ceramic membranes: Preparation, properties and microstructures

## 14.15 – 15.30 h – Section 2

### *Advanced Ceramics* (Blue Hall)

- 13.30 – 13.45 h **A6 – Radosław Lach, et al., Poland**  
Nanocomposites in the alumina - YAG system
- 13.45 – 14.00 h **A7 – Agnese Pavlova, et al., Latvia**  
Investigation of the electrical properties of vacuum annealed titanium oxide-containing ceramics
- 14.00 – 14.15 h **A8 – Tina Šetinc, et al., Slovenia**  
Hydrothermal synthesis of  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$
- 14.15 – 14.30 h **A9 – Marko Radović, et al., Serbia**  
Hydrothermal synthesis and characterization of ultrafine  $\text{CeO}_2$  and  $\text{Ce}_{0.9}\text{Fe}_{0.1}\text{O}_2$  powders
- 14.30 – 14.45 h **A10 – Stanislav Slavov, et al., Bulgaria**  
Electrical characteristics of bismuth titanate ceramics containing  $\text{SiO}_2$  and  $\text{Nd}_2\text{O}_3$

### *Ceramic Composites & Advanced Ceramics* (Class Room, R10)

- 13.30 – 13.45 h **A11 – Eugene Voynich, et al., Ukraine**  
Comparative estimation of thermal shock resistance of layered ceramics by the method of acoustic emission registration
- 13.45 – 14.00 h **C1 – Lidija V. Trandafilović, et al., Serbia**  
Semiconducting nanoparticles in poly(2-(dimethylamino) ethyl methacrylate-co-acrylic acid copolymer
- 14.00 – 14.15 h **A12 – Anna Maria Janus, et al., Poland**  
Preparation and characterization of bio-ceramics based on pure and glass-reinforced hydroxyapatite of pig origin
- 14.15 – 14.30 h **C2 – Mikhail Shiryaev, et al., Russia**  
Materials for medical applications based on potassium polyphosphate and calcium hydroxyapatite
- 14.30 – 14.45 h **C3 – Olena Sych, et al., Ukraine**  
Structure evolution and properties of biogenic hydroxyapatite-based biocomposite

## 15.30 – 16.00 h – Coffe Break

### 16.00 – 16.45 h – Section 3

#### *Advanced Ceramics* (Blue Hall)

- 16.00 – 16.15 h **A13 – Dmytro Demirskyi, et al., Ukraine**  
Effect of microwave heating on initial stage sintering of copper
- 16.15 – 16.30 h **A14 – Lovro Gorjan, et al., Slovenia**  
Debinding of ceramic parts prepared by low pressure injection molding
- 16.30 – 16.45 h **A15 – Františka Frajkorová, et al., Slovak Republic**  
Model system  $Y_2O_3$ - $SiO_2$ -Ti-NbC for liquid phase sintering of sic with high electrical conductivity
- 16.45 – 17.00 h **A16 – Andrei V. Kapylou, et al., Belarus**  
Effect of thermobaric treatment on properties of nanocrystalline tin and TiN-TiB<sub>2</sub> ceramics
- 17.00 – 17.15 h **A17 – Vaclav Pouchly, et al., Czech Republic**  
Master sintering curves of two different alumina powder compacts
- 17.15 – 17.30 h **A18 – Andraž Kocjan, et al., Slovenia**  
Temperature- and time-dependent mechanisms of AlN powder hydrolysis

#### *Traditional Ceramics* (Class Room, R10)

- 16.00 – 16.15 h **T1 – Milica Hadnadjev, et al., Serbia**  
Clay roofing tiles – able to clean itself?
- 16.15 – 16.30 h **T2 – Oana Catalina Mocioiu, et al., Romania**  
Effect of lead oxide content on the physico-chemical properties of transparent ceramics
- 16.30 – 16.45 h **T3 – Dragan Rajnović, et al., Serbia**  
Mechanical properties and fracture mode of polymer added traditional ceramics
- 16.45 – 17.00 h **T4 – Robert Rekecki, et al., Serbia**  
Effects of the firing atmosphere on the microstructure and material properties in heavy clay bodies
- 17.15 – 17.30 h **T5 – Mine Taykurt Daday, et al., Turkey**  
The effect of zircon and zinc oxide reduction on production stages, final properties and microstructure of fast fired glass-ceramic wall tile glazes
- 17.30 – 17.45 h **T6 – Miloš Vasić, et al., Serbia**  
The influence of mechanical activation on drying process
- 17.45 – 18.00 h **T7 – Dmtar Zorić, et al., Serbia**  
Biological efficiency of photocatalytic TiO<sub>2</sub> coatings on clay roofing tiles substrate

### 20.00 h – Social Event

## THURSDAY, DECEMBER 3, 2009.

### 09.00 – 10.30 h – Section 4

#### *Advanced Ceramics* (Blue Hall)

- 09.00 – 09.15 h **A19 – Piotr Boszkowicz, et al., Poland**  
Studies of the mechanism of A-Si:H layer growth in plasma conditions
- 09.15 – 09.30 h **A20 – Aleksandra Šaponjić, et al., Serbia**  
Carbothermal reduction of diatomaceous earth using various sources of carbon
- 09.30 – 09.45 h **A21 – Vojka Žunič, et al., Slovenia**  
Synthesis of TiO<sub>2</sub> powders with a high surface area
- 09.45 – 10.00 h **A22 – Darja Ilieva, et al., Bulgaria**  
Glass ceramic materials in the system GeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-TeO<sub>2</sub>
- 10.00 – 10.15 h **A23 – Mitar Perušić, et al., Bosnia and Herzegovina**  
Analysis of the single scan kinetic model of dehydration of aluminum-hydroxide
- 10.15 – 10.30 h **A24 – Oksana Kornienko, et al., Ukraine**  
Phase relations in the systems with zirconia, ceria and lanthana

#### *Advanced Ceramics* (Class Room, R10)

- 09.00 – 09.15 h **A25 – Jarosław Kaszewski, et al., Poland**  
ZrO<sub>2</sub>:TB nanopowders for application in photonics
- 09.15 – 09.30 h **A26 – Zoltán Győri, et al., Hungary**  
Synthesis and characterization of concentration gradient based CdSeAB heterostructure quantum dots
- 09.30 – 09.45 h **A27 – Daria Pomykalska, et al., Poland**  
Preparation and properties of zirconia nanopowders in the MnO<sub>x</sub>-Y<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>
- 09.45 – 10.00 h **C4 – Libor Kobera, et al., Czech Republic**  
Sources of structural instability of aluminosilicate inorganic polymers investigated by solid state NMR spectroscopy
- 10.00 – 10.15 h **A28 – Daniel Sibera, et al., Poland**  
Synthesis and characterization ZnO doped with Fe<sub>2</sub>O<sub>3</sub>, CoO and Al<sub>2</sub>O<sub>3</sub>
- 10.15 – 10.30 h **A29 – Milan Nikolić, et al., Serbia**  
Synthesis of core-shell particles for enzyme immobilization

### 10.30-10.45 h – Coffe Break

## THURSDAY, DECEMBER 3, 2009.

### 10.45 – 12.00 h – Section 5

#### *Advanced Ceramics* (Blue Hall)

- 10.45 – 11.00 h **A30 – Tobias Lehnert, et al., Germany**  
Ferroelectric characterization of low temperature sol-gel bonded particle
- 11.00 – 11.15 h **A31 – Elisa Mercadelli, et al., Italy**  
Screen-printed piezoceramic thick films
- 11.15 – 11.30 h **A32 – Henrik Haspel, et al., Hungary**  
Dielectric spectroscopic studies on titanate nanowires
- 11.30 – 11.45 h **A33 – Mária Daranyi, et al., Hungary**  
Properties of carbonaceous thin films containing 1D inorganic nanoobjects
- 11.45 – 12.00 h **A34 – Ines Bračko, et al., Slovenia**  
Hydrothermal synthesis of modified titanate-based one-dimensional nanostructures

#### *Ceramic Composites & Advanced Ceramics* (Class Room, R10)

- 10.45 – 11.00 h **C5 – Felicia Prihor, et al., Romania**  
Mutiferroic properties of BiFeO<sub>3</sub>-based ceramics
- 11.00 – 11.15 h **A35 – Jelena D. Bobić, et al., Serbia**  
Electrical properties of barium bismuth titanate
- 11.15 – 11.30 h **C6 – Katerina V. Kyrylenko, et al., Ukraine**  
Effect of process flowsheet on improving of advanced resistive ceramic composites Si<sub>3</sub>S<sub>4</sub>-ZrC
- 11.30 – 11.45 h **C7 – Dušan K. Božanić, et al., Serbia**  
Fabrication of metal and semiconductor nanoparticles in biopolymer matrices
- 11.45 – 12.00 h **C8 – Sergey Mazhuga, et al., Ukraine**  
Sintering and dielectric properties of BaTiO<sub>3</sub>/Ni nanocomposite

### 13.15-14.30 h – Lunch

### 20.00 h – Social Event

## FRIDAY, DECEMBER 4, 2009.

### 09.30 – 10.45 h – Section 6

#### *Culture Heritage* (Class Room, R10)

- 09.30 – 09.45 h **T8 – Teslim Kolawole Orunsolu, et al., Nigeria**  
Application of ceramic in modern art/fine art
- 09.45 – 10.00 h **H1 – Snežana Petrović, et al., Serbia**  
Weathering process of the eighteenth century wall paintings in the church of Bodjani monastery
- 10.00 – 10.15 h **H2 – Estel Colas, et al., France**  
Potential restoration stone assessment for Strasbourg's cathedral
- 10.15 – 10.30 h **H3 – Jasna Gulan, et al., Serbia**  
Eighteenth century wall paintings of the church of Bodjani monastery
- 10.30 – 10.45 h **H4 – Darko Despotović, et al., Serbia**  
The importance of material characterization in the restoration of polychrome surfaces Case study: 18th century Serbian baroque art

### 10.45 – 11.15 h – Coffe Break

### 11.15 – 12.30 h – Section 7

#### *Advanced Ceramics & Ceramic Composites* (Class Room, R10)

- 11.15 – 11.30 h **A36 – Artur Bradecki, et al., Poland**  
High temperature reactions in  $ZrSiO_4$ - $Al_2O_3$  system
- 11.30 – 11.45 h **C9 – Erika Csehová, et al., Slovak Republic**  
Mechanical properties of  $Al_2O_3$ -SiC nanocomposites
- 11.45 – 12.00 h **A37 – Sanja Martinović, et al., Serbia**  
Synthesis and properties of low cement castable sintered at different temperatures
- 12.00 – 12.15 h **C10 – Maya Markova-Velichkova, et al., Bulgaria**  
Fast synthesis of the polycrystalline materials on the base of  $Zn_3V_2MoO_{11}$  and  $Zn_{2.5}VMoO_8$
- 12.15 – 12.30 h **A38 – Dunja Avramov, et al., Serbia**  
Effect of the thermal schedule treatment on the obtaining of high density alumina

### 12.30 – 14.00 h – Lunch

## FRIDAY, DECEMBER 4, 2009.

### 14.00 – 15.15 h – Section 8

#### *Advanced Ceramics* (Class Room, R10)

- 14.00 – 14.15 h **A39 – Cristian Andronescu, et al., Romania**  
Structure and electric conductivity of (La, Sr)(Ga, Mg)O<sub>3</sub> solid electrolyte
- 14.15 – 14.30 h **A40 – Katarzyna Gdula, et al., Hungary**  
Characterization of BaCe<sub>0.8</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>O<sub>3-δ</sub> proton conductor
- 14.30 – 14.45 h **A41 – Milena Rosić, et al., Serbia**  
Synthesis and crystal structure of Ca<sub>(1-x)</sub>Gd<sub>x</sub>MnO<sub>3</sub>
- 14.45 – 15.00 h **A42 – Adina Magdalena Musuc, et al., Romania**  
Thermal analysis of Sr and Mg doped lanthanum gallate (LSGM) obtained by wet chemistry route
- 15.00 – 15.15 h **A43 – Aleksandra Mielewczyk, et al., Poland**  
Molten salt synthesis and properties of acceptor doped LaNbO<sub>4</sub>

### 15.15 – 15.45 h – Coffe Break

### 15.45 – 17.00 h – Section 9

#### *Ceramics Composites & Advanced Ceramics* (Class Room, R10)

- 15.45 – 16.00 h **C11 – Lachezar Radev, et al., Bulgaria**  
In vitro bioactivity of biphasic calcium phosphate silicate glass-ceramic in CaO-SiO<sub>2</sub>-P<sub>2</sub>O<sub>5</sub> system
- 16.00 – 16.15 h **A44 – Dmitry Petukhov, et al., Russia**  
Gas separation membranes and flow-through catalytic membrane reactors based on porous alumina films
- 16.15 – 16.30 h **C12 – Irina Cherniakova, et al., Ukraine**  
Effect of the cooling rate after hot pressing on electrical conductivity of Si<sub>3</sub>N<sub>4</sub>-TiO<sub>2</sub> (TiH<sub>2</sub>) ceramic composites
- 16.30 – 16.45 h **A45 – Marija Puševac, et al., Serbia**  
Effect of Starting Nanopowders on Densification and Properties of Ceria
- 16.45 – 17.00 h **C13 – Mykhailo Soltys, et al., Ukraine**  
SHF response on both particles size and stoichiometry of metallic refractory compounds

### 17.00 h – Social Event

## SATURDAY, DECEMBER 5, 2009.

### 09.30 – 10.45 h – Section 10

#### *Ceramic Composites & Advanced Ceramics* (Blue Hall)

- 09.30 – 09.45 h **C14 – Igor Khobta, et al., Ukraine**  
Synthesis of TiN-TiB<sub>2</sub> ceramic composite by spark plasma sintering method
- 09.45 – 10.00 h **C15 – Aljoša Maglica, et al., Slovenia**  
The preparation and sintering of silicon nitride powders coated with nano-sized ZrN
- 10.00 – 10.15 h **C16 – Peter Tatarko, et al., Slovak Republic**  
Characterization of rare-earth doped Si<sub>3</sub>N<sub>4</sub>/SiC micro/nano-composites
- 10.15 – 10.30 h **C17 – Oleg A. Tokariiev, et al., Ukraine**  
Wear-resistant multilayer ceramic coatings TiN/ZrN and TiN/TiCrN for drilling application obtained by vacuum-arc deposition
- 10.30 – 10.45 h **A46 – Sebastjan Perko, et al., Slovenia**  
Mechanical properties of porous Y-TZP core-shell nanocomposites

#### *Advanced Ceramics & Ceramics Composites* (Class Room, R10)

- 09.30 – 09.45 h **A47 – Kristine Salma, et al., Latvia**  
Calcium phosphate bioceramics prepared by wet chemical precipitation route
- 09.45 – 10.00 h **C18 – Dmitry Larionov, et al., Russia**  
Different approaches to fabrication of bulk biomaterials based on calcium phosphates
- 10.00 – 10.15 h **A48 – Zoltan Dudas, et al., Romania**  
Hybrid mesoporous matrices and nanocomposites obtained by sol-gel method for bioapplications
- 10.15 – 10.30 h **A49 – Janis Locs, et al., Latvia**  
Development of porous bioceramics
- 10.30 – 10.45 h **A50 – Adela Egelja, et al., Serbia**  
Synthesis of biomorphic SiC and SiO<sub>2</sub> ceramics

### 10.45 – 11.15 h – Coffe Break



## SATURDAY, DECEMBER 5, 2009.

### 11.15 – 13.00 h – Section 11

#### *Ceramic Composites & Advanced Ceramics* (Blue Hall)

- 11.15 – 11.30 h **C19 – Viktor Puchý, et al., Slovak Republic**  
Microstructure and properties of zirconia/carbon nanofiber composites
- 11.30 – 11.45 h **C20 – Lenka Benešová, Czech Republic**  
Composite materials derived from geopolymers reinforced by polymer fibers
- 11.45 – 12.00 h **A51 – Dragana Smiljanić, et al., Bosnia and Herzegovina**  
Technological result of decomposition sodium aluminate solutions depending on the decomposition parameters
- 12.00 – 12.15 h **C21 – Milica Pošarac, et al., Serbia**  
Synthesis and characterization of SiC/cordierite composite materials
- 12.15 – 12.30 h **C22 – Piotr Próchniak, et al., Poland**  
New carbon materials for electrodes for electrochemical capacitors
- 12.30 – 12.45 h **A52 – Olena Kirkova, et al., Ukraine**  
Investigation of thermal shock resistance enhancement for layered ceramics with quartz substrate and two-sided niobium pentoxide coating using computer simulation

#### *Advanced Ceramics* (Class Room, R10)

- 11.15 – 11.30 h **A53 – Katarzyna Berent, et al., Poland**  
Preparation and characterization of PLZT ferroelectric ceramic
- 11.30 – 11.45 h **A54 – Mirjana M. Vijatović, et al., Serbia**  
Barium titanate thick films prepared by screen printing technique
- 11.45 – 12.00 h **A55 – Oana Catalina Mocioiu, et al., Romania**  
Influence of alkali excess on crystallization of lead-free sodium potassium niobate thin films
- 12.00 – 12.15 h **A56 – Ilze Smeltere, et al., Latvia**  
Influence of MnO<sub>2</sub> and WO<sub>3</sub> addition on sintering and properties of lead-free KNN ceramics
- 12.15 – 12.30 h **A57 – Lucacz Kilanski, et al., Poland**  
The role of chemical disorder on magnetism of (CD,ZN)<sub>1-x</sub>Mn<sub>x</sub>GeAs<sub>2</sub> semiconductor
- 12.30 – 12.45 h **A58 – Artem Iatsenko, et al., Ukraine**  
Influence of clay mineral additives on the properties of high porous permeable cellular glassforming materials

### 13.00 h – Closing of the Meeting





*The Eighth Students' Meeting – SM-2009*

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**PROGRAMME**

A1

**LOW-TEMPERATURE SINTERING AND MICROWAVE DIELECTRIC  
PROPERTIES OF MgO-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-BASED GLASS CERAMICS**

Urban Došler, Marjeta Maček Kržmanc, Danilo Suvorov

*Jozef Stefan Institute, Ljubljana, Slovenia*

Due to the current requirements for small, light and integrated electronic devices, researchers are constantly focused on the search for new materials and packaging technologies. Low-temperature co-fired ceramic (LTCC) technology is one of the latest solutions for the fabrication of 3-dimensional multi-material modules. Materials that are used as substrates have to fulfill the dielectric requirements, such as a low permittivity ( $\epsilon_r < 8$ ), low dielectric losses ( $\tan \delta$ ), i.e., a high quality factor (Qxf) and a stable temperature coefficient of resonant frequency ( $\tau_f$ ). In today's LTCC modules less-expensive electrodes are applied (Ag,Cu,Au). Therefore, the sintering temperature must not exceed 920°C, and at the same time a full chemical compatibility between the electrode materials and the ceramics must be obtained. Our previous investigations have shown that glass ceramics based on the MgO-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> system exhibit dielectric and sintering behaviors that are suitable for LTCC substrate applications.

In this work we describe the synthesis conditions for the preparation of low-loss MgO-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-based glass-ceramic materials. We have studied the influence of composition, the addition of Al<sub>2</sub>O<sub>3</sub> and the nucleating agents (TiO<sub>2</sub>, ZrO<sub>2</sub>) on the crystallization of the phases. These MgSiO<sub>3</sub>, Mg<sub>2</sub>B<sub>2</sub>O<sub>5</sub> and Mg<sub>2</sub>Al<sub>14</sub>Si<sub>5</sub>O<sub>18</sub> crystalline phases were determined by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX). The correlations between the type and the amount of a particular crystalline phase on the dielectric properties will be discussed.

A2

## FUNCTIONAL PROPERTIES OF MULTIFERROIC NANOCOMPOSITES WITH CORE-SHELL STRUCTURES

Lavinia P. Curecheriu<sup>1</sup>, M.T. Buscaglia<sup>2</sup>, V. Buscaglia<sup>2</sup>, P. Postolache<sup>1</sup>,  
L. Mitoseriu<sup>1</sup> and P.Nanni<sup>2,3</sup>

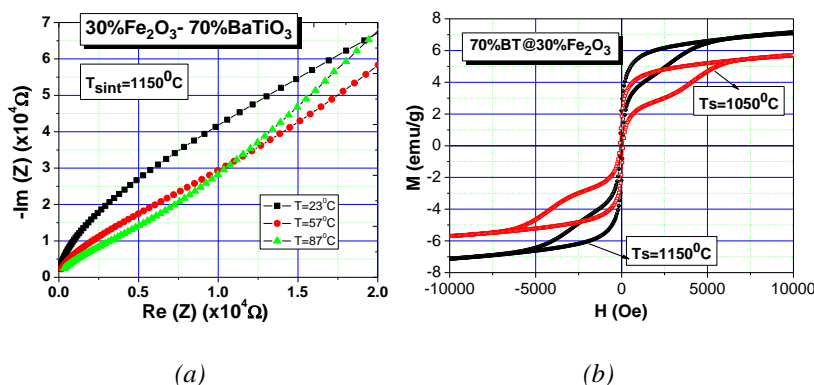
<sup>1</sup>Department of Physics, Al. I. Cuza Univ., Iasi, Romania

<sup>2</sup>Inst. for Energetics & Interphases - CNR, Genoa, Italy

<sup>3</sup>Dept. Chem. & Proc. Eng., Univ. of Genoa, Genoa, Italy

The nanoscale coating of particles of a material with a dissimilar compound is an effective tool to engineer their surface, microstructural characteristics and functional properties. In this work, core-shell multiferroic nanocomposites formed by  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> magnetic core and BaTiO<sub>3</sub> ferroelectric shell were prepared.

Spherical and cubic shaped hematite particles (300-500 nm size) were firstly prepared by precipitation method. The shell of BaTiO<sub>3</sub> was obtained by precipitation of Ti (IV) solution on the hematite cores and of BaCO<sub>3</sub> on their surface followed by thermal treatment to induce the perovskite phase formation. Two compositions with 70% and 50% BaTiO<sub>3</sub> were obtained. Dense and homogeneous di-phase ceramics with fully isolated hematite regions within BaTiO<sub>3</sub> matrix (0-3 connectivity) were further obtained by sintering at different temperatures.



*Fig. 1 Functional properties of composite ceramics sintered from core-shell BaTiO<sub>3</sub>@Fe<sub>2</sub>O<sub>3</sub> composite nanopowders (a) complex diagram of impedance, (b) magnetic properties for same composition sintered at two temperatures.*

The presence of diphase composition is also distinguished from the impedance plot that has more than one component (Fig. 1a). The magnetic properties are in good

correlation with the sample microstructures and were interpreted in relationship with their composition and degree of connectivity (Fig. 1b). The presence of both ferroelectric and magnetic properties at room temperature demonstrates the multiferroic character of the composites.

A3

### CHEMICAL VAPOR FUNCTIONALIZATION OF ZnO NANOCRYSTALS

Moazzam Ali<sup>1</sup>, Marty Donakowski<sup>2</sup>, Markus Winterer<sup>1</sup>

<sup>1</sup>*Nanoparticle Process Technology, University Duisburg-Essen, Duisburg, Germany*

<sup>2</sup>*Department of Chemistry, University of Minnesota, Minneapolis, USA*

In the last few years, the interest in printable electronics has substantially increased. The advantage of printable electronics is the feasibility of large-scale production of finely tuned patterns, even on flexible substrates. The ink required for printing, especially for inkjet printing, plays an important role in the quality and reliability of the final products. Chemical Vapor Functionalization (CVF) is a method, which is used to generate functionalized nanomaterials in the form of inks with a high production rate. In CVF, two reactors are used in series. First reactor consists of a hot quartz tube (600 °C) where ZnO nanocrystals are synthesized in the gas phase from diethylzinc and oxygen. Second reactor, connected at the exit of the first one and kept at lower temperature (400 °C), is used as functionalization chamber. At the connecting point of the two reactors, vapor of organic functionalizing agents are injected which reacts with the surface of ZnO nanocrystals in the vapor phase. ZnO nanocrystals have been functionalized by 1-hexanol, hexanoic acid, hexanal and 1-hexylamine. In-situ analysis of the functionalization has been performed by Quadrupole mass spectrometry. Functionalized ZnO nanocrystals have been characterized by Dynamic Light Scattering, Transmission Electron Microscopy, Diffuse Reflectance Infrared Fourier Transform Spectroscopy and Nuclear Magnetic Resonance Spectroscopy.

A4

**THE PREPARATION OF MgSiN<sub>2</sub> AND LaSi<sub>3</sub>N<sub>5</sub> ADDITIVES AND  
THEIR INFLUENCE ON THERMAL CONDUCTIVITY OF Si<sub>3</sub>N<sub>4</sub>  
CERAMICS**

Linda Kipsova<sup>1</sup>, Zoltán Lenčes<sup>1</sup>, You Zhou<sup>2</sup>, Pavol Šajgalík<sup>1</sup>

<sup>1</sup>*Institute of Inorganic Chemistry, Slovak Academy of Sciences, Bratislava, Slovakia*

<sup>2</sup>*National Institute of Advanced Industrial Science and Technology (AIST),  
Nagoya, Japan*

The presented work deals with preparation and characterization of magnesium and lanthanum silicon nitride based ceramics. MgSiN<sub>2</sub> is considered as potential candidate for substrates in electronics owing to relatively high thermal conductivity and LaSi<sub>3</sub>N<sub>5</sub> is suitable engineering material for applications in harsh environment. Powders of ternary nitrides MgSiN<sub>2</sub> and LaSi<sub>3</sub>N<sub>5</sub> were prepared by reaction synthesis at temperatures 1350 - 1400°C. The thermal conductivities of dense ternary nitrides measured by laser flash method were  $\lambda(\text{MgSiN}_2) = 21,9 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$  and  $\lambda(\text{LaSi}_3\text{N}_5) = 4,1 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$ , respectively. Strength, hardness and fracture toughness were also determined for these materials.

Silicon nitride ceramics with MgSiN<sub>2</sub> and LaSi<sub>3</sub>N<sub>5</sub> were prepared by reaction bonding method and showed excellent thermal conductivity;  $\lambda(\text{Si}_3\text{N}_4/\text{MgSiN}_2) = 135 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$  and  $\lambda(\text{Si}_3\text{N}_4/\text{LaSi}_3\text{N}_5) = 91 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$ .

A5

**NANOSIZE WO<sub>3</sub> FOR GAS SENSING AND PHOTOCATALYSIS**

Balazs Forizs<sup>1</sup>, Imre Miklos Szilagyí<sup>2</sup>

<sup>1</sup>*Department of Inorganic and Analytical Chemistry, Budapest University of Technology  
and Economics, Budapest, Hungary*

<sup>2</sup>*Materials Structure and Modeling Research Group of the Hungarian Academy of  
Sciences, Budapest University of Technology and Economics, Budapest, Hungary*

For applications in gas sensors, catalysis, photoelectrochromical cells and chromogenic (electro-, photo- and thermochromic) devices, tungsten oxides have attracted much attention in the past decades. The most used polymorph of tungsten oxides is monoclinic (m-) WO<sub>3</sub>. In addition, the hexagonal polymorph, h-WO<sub>3</sub>, is also widely researched due to its open-tunnel structure. We intended to study the effect of crystalline structure and composition of tungsten oxides on several applications (gas sensing, photocatalysis).

Monoclinic and hexagonal  $\text{WO}_3$  samples were prepared through annealing hexagonal ammonium tungsten bronze (HATB),  $(\text{NH}_4)_x\text{WO}_{3-y}$ . These samples were analyzed by powder X-ray diffraction (XRD), scanning electron microscopy (SEM), Raman, solid state  $^1\text{H}$ -MAS NMR and X-ray photoelectron (XPS) spectroscopy. By varying the heating temperature, we could control the crystalline structure of tungsten oxides, while by adjusting the annealing atmosphere; we managed to control the average oxidation state of the samples. As a result, we could prepare oxidized and reduced nanosize h- $\text{WO}_3$  and m- $\text{WO}_3$ .

Then, we studied tungsten oxides as gas sensors to several gases (e.g.  $\text{Cl}_2$ ,  $\text{NH}_3$ ,  $\text{H}_2$ , organic vapours). It was found that both the crystalline structure and the composition had strong influence on the gas sensitivity of samples. The samples were also tested as photocatalysts for decomposing organic dyes (e.g. methylene blue, methylene orange). Here it was similarly found that crystalline structure and the composition had impact on the photocatalytic effect of tungsten oxides. The mechanism of how composition and structure influenced the applications was thoroughly analyzed and explained.

A6

### **NANOCOMPOSITES IN THE ALUMINA - YAG SYSTEM**

Radosław Lach and Krzysztof Haberk

*AGH University of Science and Technology, Kraków, Poland*

Particulate composite material in the alumina/YAG system was prepared by the following technique. Within alumina particle suspension yttria precursor was precipitated with ammonium carbonate. Drying and calcination at  $600^\circ\text{C}$  resulted in the mixture of alumina and yttria particles. The latter being much finer than alumina particles. This mixture was additionally homogenized by short attrition milling in aqueous suspension of pH selected on the basis of zeta ( $\zeta$ ) potential measurements. It was found that acidic conditions (realized with  $\text{HNO}_3$ ) result in hard agglomerates. Such powder shows poor sintering ability and not homogenous second particles distribution. Much better properties show powders homogenized under basic condition. They give dense materials composed of YAG inclusions within alumina matrix. YAG particles result from the reaction of  $\text{Y}_2\text{O}_3$  with  $\text{Al}_2\text{O}_3$  during heat treatment. Homogenization of the system mixed at  $\text{pH}=8$ , which correspond to the heteroflocculation conditions, is slightly better than observed at  $\text{pH}=10$ . YAG inclusions (20 vol.%) increase hardness and fracture toughness of the material in comparison to pure alumina polycrystals.



A7

### INVESTIGATION OF THE ELECTRICAL PROPERTIES OF VACUUM ANNEALED TITANIUM OXIDE-CONTAINING CERAMICS

Agnese Pavlova<sup>1</sup>, Janis Barloti<sup>2</sup>, Valdis Tetters<sup>2</sup>, Janis Locs<sup>1</sup>,  
Liga Berzina-Cimdina<sup>1</sup>

<sup>1</sup>*Riga Technical University, Riga Biomaterials Innovation and  
Development Centre, Latvia*

<sup>2</sup>*Riga Technical University, Division of Electronic Equipment, Latvia*

Titanium oxide ceramics were formed using dry extrusion process. Obtained green bodies were thermally treated in two stages – in the air and vacuum atmosphere in temperatures from 1200°C up to 1350°C with 25°C step. Influence of treatment conditions on physicochemical and electrical properties of obtained ceramics was investigated. Physicochemical properties depend not only on vacuum treatment conditions but also on first treatment in air conditions. It was found that increase of temperature during sintering in vacuum leads to increased electrical conductivity. It was found that all samples treated in vacuum are n-type semiconductors. Activation energy ( $\Delta E$ ) was calculated using conductivity of sample dependence on temperature.  $\Delta E$  is in the range from 0,049 to 0,061 eV. The physicochemical properties of the obtained samples were investigated using scanning electron microscopy (SEM) and X-ray diffractometry (XRD).

A8

### HYDROTHERMAL SYNTHESIS OF $\text{Na}_{0,5}\text{Bi}_{0,5}\text{TiO}_3$

Tina Šetinc, B. Jančar, M. Spreitzer, D. Suvorov

*Department for Advanced Materials, Institut Jožef Stefan, Ljubljana, Slovenia*

Sodium bismuth titanate,  $\text{Na}_{0,5}\text{Bi}_{0,5}\text{TiO}_3$  (NBT), is a complex perovskite-type material and a candidate for lead-free piezoelectric ceramics. The relaxor ferroelectric behavior of the NBT material opens up the possibility for applications as a voltage-tunable capacitor. The aim of our work was to obtain nano-sized crystalline NBT particles that could be subsequently employed in the form of a thin film. Recently, hydrothermal synthesis has proven to be a promising route for the production of various compounds. In contrast to other, more conventional, techniques, hydrothermal synthesis offers several advantages, including the synthesis of ultrafine, crystalline, high-purity, uniform powders at lower temperatures from a variety of inexpensive precursors. Furthermore, the hydrothermal medium is attractive for ceramic powder synthesis because of the combined effects of solvent, temperature, and pressure on the ionic reaction equilibria that can stabilize desirable products while inhibiting the formation of

undesirable compounds. The properties of the final product can be controlled by utilizing chemical-process variables, such as temperature, pressure, reactant concentrations, pH, selection of precursors, etc.

The reaction mechanisms for perovskite materials under hydrothermal conditions are poorly understood. In the literature, two basic mechanisms are proposed – the “in-situ” transformation and the dissolution-recrystallization process – depending on the alkali concentration in the reaction solution. Furthermore, in a multicomponent system there are a few possible phases that can be formed. Our study was focused on the influence of the reaction parameters on the synthesis of the NBT and the formation of secondary phases.

The hydrothermal synthesis of the NBT powder was performed in a strong alkaline medium with the use of TiO<sub>2</sub> (anatase) as the titanium precursor, Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O as a source of bismuth ions and NaOH as the sodium precursor and the provider of a highly basic environment. The influence of the processing parameters, such as the NaOH concentration in solution, the concentration of precursors, the reaction temperature and the time, on the formation of the NBT and the presence of secondary phases were investigated. In addition, the effect of the different solvents used was also examined. The obtained powders were characterized by X-ray diffraction analysis (XRD) and transmission electron microscopy (TEM).

A9

#### **HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF ULTRAFINE CeO<sub>2</sub> AND Ce<sub>0.9</sub>Fe<sub>0.1</sub>O<sub>2</sub> POWDERS**

Marko Radović<sup>1</sup>, Zorana Dohčević-Mitrović<sup>1</sup>, Aleksandar Golubović<sup>1</sup>, Sonja  
Aškračić<sup>1</sup>, Branko Matović<sup>2</sup>, Maja Šćepanović<sup>1</sup>, Zoran V. Popović<sup>1</sup>

<sup>1</sup>*Institute of Physics, Center for Solid State Physics and new Materials, Belgrade, Serbia*

<sup>2</sup>*Institute of Nuclear Sciences Vinca, Belgrade, Serbia*

Hydrothermal method has been employed to synthesize pure and 10 mol% Fe<sup>3+</sup> doped CeO<sub>2</sub> nanopowders from Ce(SO<sub>4</sub>)<sub>2</sub>·4H<sub>2</sub>O precursor, with FeCl<sub>3</sub>·6H<sub>2</sub>O as a dopant, in NH<sub>4</sub>OH and NaOH solution. Calcination of the samples was carried out at 140°C and 200°C. The characterization of crystalline structure, vibrational and optical properties was performed using X-ray diffraction, Raman spectroscopy and spectroscopic ellipsometry. Obtained results showed that the Fe-doped samples are solid solutions with different size of nanocrystals, very dependent on the synthesis temperature and type of basic solution. Raman measurements demonstrated electron molecular vibrational coupling and increase of oxygen vacancy concentration whereas doping provokes a small decrease of optical absorption edge in comparison with pure ceria.

The zones with highest level of breaking stresses are determined and the dependencies of extreme equivalent stresses on anvil geometric parameters are obtained

at various states of HPA: “assembly”, “loading” and “unloading” of HPA. It has been shown that due to the changing of the cavity profile, it is possible to decrease the level of stresses in HPA anvils in two times. It allows increasing their service time. It makes it possible to employ the steel anvils instead of the hard alloy ones and provides the expenses decrease for manufacturing silicon nitride ceramic products

A10

**ELECTRICAL CHARACTERISTICS OF BISMUTH TITANATE  
CERAMICS CONTAINING  $\text{SiO}_2$  AND  $\text{Nd}_2\text{O}_3$**

Stanislav Slavov, Elena Kashchieva, Milena Krapchanska, Yanko Dimitriev  
*University of Chemical Technology and Metallurgy, Sofia, Bulgaria*

Bismuthate-titanate ceramics containing  $\text{SiO}_2$  and  $\text{Nd}_2\text{O}_3$  as additives are synthesized by two methods - solid state reaction and melt quenching. The phase composition of the obtained materials is determined by x-ray diffraction analysis (XRD - TUR M62, Cu-K $\alpha$  radiation), energy dispersive spectroscopy (EDS - EDAX 9900). The microstructure is observed by scanning electron microscopy (SEM - 525M, Philips).

The diffraction data show that the main crystalline phase in the produced ceramics is  $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ . The scanning electron microscopy investigations illustrate the texturing of the crystals upon the axis C after appropriate thermal treatment of selected samples. Electrical measurements were performed by means of complex impedance spectroscopy in the 10 – 100 kHz frequency range using Hewlett-Packard HP4192A impedance analyzer. Dependence of the polarization on the applied electric field is studied. The performed electrical measurements show that the methods of synthesis and the type and content of the additives influence on the dielectric parameters.

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A11

**COMPARATIVE ESTIMATION OF THERMAL SHOCK RESISTANCE  
OF LAYERED CERAMICS BY THE METHOD OF ACOUSTIC  
EMISSION REGISTRATION**

Eugene Voynich, Olena Kirkova, Alexandr Frolov

*Frantsevich Institute for Problems of Materials Science of Ukraine, Kiev, Ukraine*

Estimation and comparison of thermal shock resistance of concrete products or samples have large significant in developing ceramic articles. Because the articles with the same shapes and sizes have different properties according to used materials and different methods, they were produced. The possibility to obtain the data of samples made of materials, which possess increased thermal shock resistance, without destruction represents particular interest. Their breaking points cannot be reached with standard test methods. The equipment for comparison estimation of thermal shock resistance of such materials by the method of acoustic emission registration in air cooling conditions has been created.

The equipment consists of the electric furnace, the mechanical device for fast extraction of the sample, and the system of acoustic emission registration. The system allows record the acoustic emission signals and the sample surface temperature data at cooling. Comparative estimation of the thermal shock resistance of layered ceramics and influence of macrostructure of substrate layer on it were established with the represented equipment. Acoustic emission registration carried out by cooling on air samples from temperature of 1000°C. Mathematical simulation was used for research and analyses samples thermal shock resistance. Four kinds of samples which have the shape of plates were used: (1) the plate from continuous quartz ceramics with coating; (2) the plate from continuous quartz ceramics without coating; (3) segmented substrate with sharp angles with coating and (4) segmented substrate with fillet angles. The thermal shock resistance was estimated from the number of AE impulses (Fig. 1). The experimental results have shown that the number of AE impulses decreases by  $\approx 1.5 - 2$  times and their amplitude decreases as well for the samples with a segmented substrate.

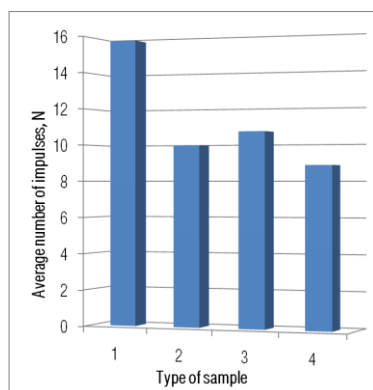


Figure 1. Average number of impulses for different sample types

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A12

### PREPARATION AND CHARACTERIZATION OF BIO-CERAMICS BASED ON PURE AND GLASS-REINFORCED HYDROXYAPATITE OF PIG ORIGIN

Anna Maria Janus, Marek Faryna

*Institute of Metallurgy and Materials Science, Krakow, Poland*

Hydroxyapatite (HAp -  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ) is a major constituent of the mineral phase of human bone. Due to its biocompatibility it is considered an attractive material for filling of bone defects. Hydroxyapatite can be obtained either by synthesis or from natural sources (e.g. from pig and bovine bones or corals). Hydroxyapatite of natural origin, in comparison to synthetic one, is believed to be more bioactive and to exhibit higher osteoconductivity. This is mainly due to the fact that its chemical composition is more similar to that of human bone. Such a similarity can be associated i.a. with the presence of carbonate groups within the structure of hydroxyapatite. This feature however, calls for more sophisticated methods of processing of hydroxyapatite of natural origin. Simple sintering in air of such bio-ceramics would result in partial decomposition of hydroxyapatite and formation of compounds (e.g. of CaO) which negatively influence the material.

This work describes the process of preparation of hydroxyapatite from pig bones. Results of the powder characterization by means of E-SEM, TEM, XRD, BET isotherm and DSC are shown. Furthermore, various methods used for sintering of the material are described. Modification of sintering procedures (to address the possibility of formation of undesirable phases) is presented. As decomposition of carbonates is an equilibrium process, one of the employed preparation methods was sintering of bio-ceramics in the atmosphere of carbon dioxide. Another sintering procedure was based on addition of sintering aid (in the form of phosphate glass of low melting point) to hydroxyapatite ceramics in order to reduce the temperature of sintering and decrease the risk of thermal decomposition of carbonates. The influence of several parameters, sintering atmosphere, temperature and addition of sintering aid on composition and morphology of pure and glass modified dense hydroxyapatite bio-ceramics was analyzed and is described.

A13

### **EFFECT OF MICROWAVE HEATING ON INITIAL STAGE SINTERING OF COPPER**

Dmytro Demirskyi<sup>1,2</sup>, Dinesh K. Agrawal<sup>1</sup>, Andrey Ragulya<sup>2</sup>

<sup>1</sup>*Institute for Problems of Materials Science NAS of Ukraine, Kyiv,*

<sup>2</sup>*Materials Research Institute, The Pennsylvania State University, Pennsylvania, USA*

Microwaves have found a broad application in different branches of industry replacing some conventional heating technologies. Microwave processing provides possibility to achieve homogeneous and fast heating in compacts is through microwave heating.

Traditionally microwaves have been used to sinter ceramics, refractory materials as well as ferrites, but for past decade microwave processing is opening new provability's in materials science. It is well known, that bulk conductors are reflectors of microwaves, though, it was shown that metals too could couple with microwaves, provided the metals are in powder form rather than monolithic. Though there have been attempts to explain microwave heating of metal powders, there is still not a consensus on a comprehensive theory to explain the mechanism.

To clarify this situation series of model experiments were performed on microwave sintering of conductive materials. Such works includes neck growth kinetics during microwave sintering as well as densification kinetics.

A14

### **DEBINDING OF CERAMIC PARTS PREPARED BY LOW PRESSURE INJECTION MOLDING**

Lovro Gorjan, Aleš Dakskobler, Tomaž Kosmač

*Jozef Stefan Institute, Ljubljana, Slovenia*

Binder removal from parts, formed by low pressure injection molding (LPIM) is a delicate and time consuming operation, in which defects can be introduced that remain in the material to the finished product. With the use of high porous wicking powder embedment, a partial thermal debinding can be utilized, which can considerably speed up the process and reduce many debinding flaws.

In this work we report on the kinetics of the partial thermal debinding in the wicking embedment of ceramic parts prepared by the LPIM. High solid content suspension for injection molding was prepared from  $Al_2O_3$  powder and paraffin-based binder. The binder was thermally removed by the different time-temperature controlled procedures. After the process the distribution of the binder inside the samples was investigated.

We found that debinding takes place in two separate stages. In the first stage, the rate of binder removal depends on capillary extraction of the binder into the surrounding wicking embedment. A characteristic binder distribution forms, with clearly distinct binder-depleted and binder-rich regions. The first is located in the center of the molded part, while the later occupies place between the binder-depleted region and the surface. With the progress of the debinding, the binder-rich region is shrinking until it eventually disappears. Only after it disappears, air can enter the interior and bulk oxidation can occur. At this moment the second phase of debinding starts, in which gaseous transport and reactions of binder degradation into volatile components control the rate of the process. If the temperature is above approximately  $170^\circ C$  a bulk oxidation of binder takes place, which significantly speed up the process. Additionally, the oxidative transformation of remaining binder can improve mechanical properties of debinded parts.

A15

**MODEL SYSTEM  $Y_2O_3$ - $SiO_2$ -Ti-NbC FOR LIQUID PHASE SINTERING OF SiC WITH HIGH ELECTRICAL CONDUCTIVITY**

Františka Frajkorová, Miroslav Hnatko, Pavol Šajgalík  
*Institute of Inorganic Chemistry, Slovak Academy of Sciences,  
Bratislava, Slovak Republic*

Electrical nature of polycrystalline silicon carbide initiates a necessity to modify the compositions in order to increase electrical conductivity for some special progressive applications. Methods leading to this goal are based on a distribution of electrically conductive phase into non-conducting matrix. The present work deals with optimization of the chemical composition of  $Y_2O_3$ - $SiO_2$ -Ti-NbC system aimed at preparing dense SiC material sintered by liquid phase sintering. The purpose of optimization was improvement of the electrical conductivity of system at good mechanical properties preservation.  $Y_2O_3$ - $SiO_2$  systems (sintering additives) with different addition of Ti-NbC phases (electrically conductive phases) were studied at present work. The samples were hot pressed at 1200 and 1650°C respectively in flowing Ar-gas atmosphere for 1 hour, 30 MPa of mechanical pressure was applied. Changes of chemical and phase composition and mechano-physical properties (hardness, fracture toughness, electrical conductivity) of prepared samples were investigated. Electrical conductivity of sample increases with sintering temperature and content of electrically conductive phases. The sample sintered at 1650°C with 30% of conductive phases has shown suitable properties for proposed application: reasonable hardness (HV1 ~ 10GPa), fracture toughness ( $KIC \sim 4 \text{ MPa}\cdot\text{m}^{1/2}$ ), high electrical conductivity ( $\sigma \sim 6 \text{ S}\cdot\text{cm}^{-1}$ ).

**Keywords:** liquid phase sintered SiC, percolation phenomenon, electrical properties

A16

**EFFECT OF THERMOBARIC TREATMENT ON PROPERTIES OF NANOCRYSTALLINE TiN AND TiN-TiB<sub>2</sub> CERAMICS**

Andrei V. Kapyrou, Vladimir S. Urbanovich  
*Scientific-Practical and Materials Research Center, NAS of Belarus, Minsk, Belarus*

Titanium nitride (TiN) and titanium diboride (TiB<sub>2</sub>) are high-melting-point compounds with a high hardness, low electric resistance and good thermal stability. Pure TiN and TiB<sub>2</sub> powders are very difficult to fully densify because of covalent bonding and a very low self-diffusion coefficient. The synthesis of nanosized powders shows great promise of producing dense TiN and TiB<sub>2</sub> parts at lower temperatures and



improving the mechanical properties of sintered compacts. Dense ceramics on the basis of TiN and TiN-TiB<sub>2</sub> having nanocrystalline structure and increased hardness have been obtained using High Pressure Sintering (HPS). HPS is especially effective for fabricating nanoceramics based on high-melting point compounds because dense materials can be produced without any additives while the duration of sintering can be reduced.

In this work, the effect of powder degassing and HPS temperature and time on the densification and properties of nanocrystalline TiN and TiN-TiB<sub>2</sub> has been investigated. For this reason, pure TiN powder and composite TiN-23wt%TiB<sub>2</sub> powder with a mean particle size of 55 nm and 30 nm respectively were sintered under static pressure of 3.5 GPa in a temperature range of 900-1600°C for 45-120 s. Some of the green bodies were degassed in vacuum before sintering. It was shown that samples sintered after vacuum annealing have a density about 1-2 % higher in case of TiN and about 9 % higher in case of TiN-TiB<sub>2</sub> than before degassing. Microhardness and microstructure investigations have shown that recrystallisation of nanocrystalline TiN and TiN-TiB<sub>2</sub> ceramics sintered under the pressure of 3.5 GPa begins at the sintering temperatures of 1100-1200°C. The maximum microhardness obtained was 23.2±1.0 GPa for TiN and 24.9±1.2 GPa for TiN-TiB<sub>2</sub>.

A17

### **MASTER SINTERING CURVES OF TWO DIFFERENT ALUMINA POWDER COMPACTS**

Vaclav Pouchly and Karel Maca

*Department of ceramics and polymers, Brno University of Technology,  
Brno, Czech Republic*

Concept of Master Sintering Curve is a strong tool for optimizing sintering schedule. The sintering behavior can be predicted, and sintering activation energy can be calculated with the help of few dilatometric measurements. In this paper an automatic procedure was used to calculate Master Sintering Curves of two different alumina compacts. The sintering activation energies were determined as 640 kJ/mol for alumina with particle size of 240 nm, and 770 kJ/mol for alumina with particle size of 110 nm. The possibility to predict sintering behavior with the help of Master Sintering Curve was verified.

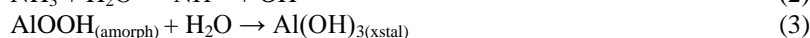
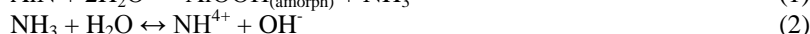
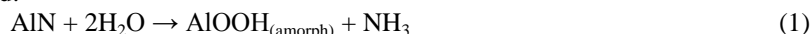
A18

### TEMPERATURE- AND TIME-DEPENDENT MECHANISMS OF AlN POWDER HYDROLYSIS

Andraž Kocjan, Kristoffer Krnel, Tomaž Kosmač

*Jozef Stefan Institute, Ljubljana, Slovenia*

In the presence of water, aluminum nitride (AlN) powder decomposes, forming aluminum hydroxides and ammonia. Although the hydrolysis of AlN powder has been known for a long time – about a century ago it was exploited for the production of ammonia – until recently the kinetics and mechanism had not been extensively studied. For AlN powder hydrolysis at room temperature (RT) the following reaction scheme was proposed:



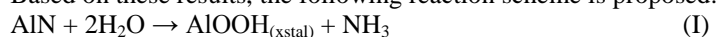
AlN powder first reacts with water to form X-ray amorphous aluminum hydroxide (pseudoboehmite, AlOOH), which later dissolves and recrystallizes as bayerite (Al(OH)<sub>3</sub>) over time. The kinetics of AlN hydrolysis was described using an unreacted-core model and the chemical reaction at the product-layer/unreacted-core interface was proposed to be the rate-controlling step during the initial stage of the reaction. The mechanisms of the aluminium-hydroxides formation during the AlN powder hydrolysis were assumed to be similar to the ageing and crystallization of amorphous aluminium hydroxide gels, but this assumption has not been experimentally confirmed yet. Besides, very little literature data exists describing AlN powder hydrolysis at the elevated temperatures relevant to newly developed ceramic processing techniques, such as the HAS (Hydrolysis Assisted Solidification) process that exploits the hydrolysis at elevated temperature in the solidification of aqueous ceramic suspensions in an impermeable mould. Furthermore, AlN powder hydrolysis at elevated temperatures can also be exploited in the preparation of nanostructured aluminate coatings onto a ceramic substrate immersed in diluted AlN slurry.

The aim of our work was to verify the validity of the anticipated mechanisms during AlN powder hydrolysis at RT and to check the applicability of the existing model at elevated temperatures. The AlN hydrolysis reactions were investigated by measuring the pH and the temperature of a diluted AlN suspension, followed by characterization of the solid reaction products. The hydration temperature varied from RT to 90°C, and the ageing time from a few minutes up to 30 days.

According to the results of our study, poorly crystalline boehmite is the first hydrolysis reaction product formed irrespective of the starting hydrolysis temperature, whereas bayerite is formed by direct precipitation as soon as the extensive boehmite formation is completed. The boehmite-to-bayerite conversion (Eq. 3) via dissolution-

recrystallization is a temperature- and pH-dependent process. It will only occur at lower hydrolysis temperatures, at higher pH values and for prolonged hydrolysis times.

Based on these results, the following reaction scheme is proposed:



A19

## STUDIES OF THE MECHANISM OF a-Si:H LAYER GROWTH IN PLASMA CONDITIONS

Piotr Boszkowicz, Katarzyna Tkacz – Śmiech

*AGH University of Science and Technology, Faculty of Materials Science and Ceramics,  
Kraków, Poland*

Solar cells based on hydrogenated amorphous silicon layers (a-Si:H) have high potential to become competitive ones in photovoltaics market. This is thanks to low raw material needs and low production costs. Such cells can be easily implemented in building constructions. A technology of good quality a-Si:H makes a use of plasma enhanced chemical vapor deposition (PE CVD) technique.

Unfortunately, before passing to a cost-effective production on commercial scale some questions need answers and a few fundamental problems should be solved. The most important ones concern an influence of technological parameters on the layer growth. In the respective studies various physical - chemical phenomena which contribute the layer growth should be considered and the mechanisms of homo- and heterogeneous reactions should be examined in details.

In this work, the following issues will be discussed:

- What is a composition of silane plasma generated with a use of 13,56 MHz (radio frequency) and 2,45 GHz (Microwave) frequencies? What is a role of plasma species in the deposition of a-Si:H layers?
- How do the heterogeneous reactions between silane plasma species and active surface sites go? What is a role of hydrogen in the formation of active surface sites?
- What are the optimal technological conditions necessary to receive the a-Si:H layer of good quality and at effective growth rate?

The discussion will be supported by experimental results gained for the a-Si:H layer series deposited in the PE CVD system. The reaction models will be based on the results from ab initio calculations (Gaussian 03 program).

A20

### CARBOTHERMAL REDUCTION OF DIATOMACEOUS EARTH USING VARIOUS SOURCES OF CARBON

Aleksandra Šaponjić, B. Babić, B. Matović

*“Vinča” Institute of Nuclear Sciences, Materials Science Lab, Belgrade, Serbia*

The possibility of using natural inorganic precursors for the synthesis of silicon carbides and nitrides, and their composites, is investigated. The starting material was diatomaceous earth as a source of  $\text{SiO}_2$  and three different carbon sources: sacharosis, carbon cryogel, and active coal. On the other side, the influence of each individual carbon source on the process of carbothermal reductions and nitrifications and properties of the obtained powders in the course of synthesis of composite powders based on silicon nitride and silicon carbide is also performed. Parameters of the chemical reaction gas-solid (temperature, time, structure, and contents of the reduction substance) have been defined. In the course of synthesis of inorganic powders of silicon carbides and nitrides, different synthesis methods requiring the expensive initial components, high temperatures, and high pressures are applied. In this work, however, the considerably more economical method of carbothermal reduction is applied. During the synthesis of these powders, special attention is paid to the preparation of the starting mixtures in order to achieve a homogenous distribution of initial components in the produced samples. The powders were characterized by X-ray and SEM investigations. It was found that it is possible to produce either mixture of non-oxide powders ( $\text{Si}_3\text{N}_4/\text{SiC}$ ) or pure SiC powders depending on temperature.

**Keywords:** diatomaceous earth, gas-solid reactions, microstructure, X-ray diffraction, scanning electron microscopy

A21

### SYNTHESIS OF $\text{TiO}_2$ POWDERS WITH A HIGH SURFACE AREA

Vojka Žunič, S. Davor Škapin, Marjeta Maček Kržmanc and Danilo Suvorov

*“Jožef Stefan” Institute, Ljubljana, Slovenia*

In the past few decades, heterogeneous metal-oxide photocatalysts have attracted much attention due to their various applications in the purification of water and air, in the photo-induced biodegradation of microbial, for photovoltaic devices, for self-cleaning coatings, for anti-fogging mirrors and for many other applications. Among these oxides,  $\text{TiO}_2$  anatase is one of the most promising and studied materials. For the high photocatalytic activity of anatase powders, a good crystallinity and a high specific

surface area are very important. Furthermore, the use of amphiphilic triblock copolymers as a template can increase the surface area. In order to achieve highly crystalline TiO<sub>2</sub> anatase powders, calcination at a high temperature (300-500°C) is required. The crystallization of TiO<sub>2</sub> during this calcination leads to a collapse and destruction of the organic template structure supporting the TiO<sub>2</sub> framework and this decreases the specific surface area. The thermal stability of TiO<sub>2</sub> can be improved by using phosphoric acid as the catalyst. The incorporation of phosphorous is known to stabilize the TiO<sub>2</sub> framework and increase the surface area, and hydrothermal or solvothermal syntheses are often used to achieve the crystallization of anatase at lower temperatures (100-150°C).

In this presentation we will discuss the synthesis of TiO<sub>2</sub> anatase powders with a high crystallinity and a high surface area. Materials with a high surface-to-volume ratio offer more active sites for carrying out photocatalytic reactions. In the sol-gel method followed by a solvothermal treatment and calcination we synthesized TiO<sub>2</sub> anatase powders by using a P123 amphiphilic triblock copolymer as the template and phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) as the TiO<sub>2</sub> framework stabilizer. The synthesized TiO<sub>2</sub> anatase samples were analyzed by X-ray powder diffraction (XRD), differential thermal analysis (DTA) and scanning electron microscopy (SEM). The specific surface area was measured with the Brunauer-Emmet-Teller (BET) method. We obtained a highly crystalline TiO<sub>2</sub> anatase with a high specific surface area, greater than 250 m<sup>2</sup>/g. The high surface area is most probably the consequence of the P123 triblock copolymer self-assembly and the alkylene oxide complexation of the Ti-alkoxide groups. Moreover, the formation of the well-crystallized anatase TiO<sub>2</sub> powders might be related to the successful control of the crystal growth by using H<sub>3</sub>PO<sub>4</sub> during the applied processing route.

A22

### **GLASS CERAMIC MATERIALS IN THE SYSTEM GeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-TeO<sub>2</sub>**

Darja Ilieva, Elena Kashchieva, Yanko Dimitriev

*University of Chemical Technology and Metallurgy, Sofia, Bulgaria*

Fast quenching method is a very useful technique for obtaining of monolithic porous free polycrystalline materials. In our previous studies [1, 2] the ability was shown for producing glasses and ceramics in the system GeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-TeO<sub>2</sub> in the absence of modifier and ion conducting components. Due to the metastable phase separation in the system GeO<sub>2</sub>-TeO<sub>2</sub> and to the stable phase separation in the TeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub> system, in the ternary system, depending on the composition and thermal history, it is possible to obtain various microheterogeneous structures.

The purpose of the present investigation is to synthesize polycrystalline ceramic materials in the system GeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-TeO<sub>2</sub> with specific dielectric properties and with controlled ratio of amorphous to crystalline phases. The selected compositions contain 40-60 mol% GeO<sub>2</sub>, 10-30 mol% B<sub>2</sub>O<sub>3</sub> and 15-50mol% TeO<sub>2</sub>. From 5 to 10mol% CoO

are introduced in some batch compositions in order to modify the electric parameters. The melting was carried out in alumina crucible at 900-1200°C. The obtained amorphous samples were subjected to additional heat treatment.

The XRD analysis confirms the presence of TeO<sub>2</sub> and GeO<sub>2</sub> microcrystals. The microstructure of amorphous and polycrystalline samples were investigated by TEM and SEM. Heterogeneous droplet-like and elongated microformations ranging in size below 0.5 μm are observed in the amorphous matrix. The electrical measurements show that the synthesized polycrystalline materials possess dielectric behavior.

Thus it is proved that by fast quenching and additional heat treatment dielectric materials could be produced in the system GeO<sub>2</sub>-B<sub>2</sub>O<sub>3</sub>-TeO<sub>2</sub>.

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A23

### ANALYSIS OF THE SINGLE SCAN KINETIC MODEL OF DEHYDRATION OF ALUMINUM-HYDROXIDE

Mitar Perušić<sup>1</sup>, Dragana Stanišić<sup>1</sup>, Radislav Filipović<sup>2</sup>, Branko Pejović<sup>1</sup>,  
Živan Živković<sup>3</sup>

<sup>1</sup>University of East Sarajevo, Faculty of Technology Zvornik, RS, Bosnia & Herzegovina

<sup>2</sup>Factory of Alumina “Birac” Zvornik, RS, Bosnia & Herzegovina

<sup>3</sup>University of Belgrade, Technical Faculty Bor, Serbia

Thermal dehydration of aluminium-hydroxide fares during three main steps. A predominant line of thought is that during heating aluminium-hydroxide decomposes to AlOOH which transforms into a dehydrated aluminium-oxide which suffers a series of phase transformations with increase of temperature to final phase product α-Al<sub>2</sub>O<sub>3</sub>. As is well known the thermal decomposition of solids could be researched by differential thermal analysis (DTA), also using specific kinetic models, it permits calculation of activation energy (E<sub>a</sub>), pre-exponential factor (Z), and rate constant (k) from a single DTA scan. For this research is used Borchardt and Daniels (B/D) kinetic approach. Experimental samples of aluminum-hydroxide were used with different particle sizes: +100 μm, -80+63μm, -30 μm.

**Keywords:** aluminum-hydroxide, DTA, kinetic model

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A24

**PHASE RELATIONS IN THE SYSTEMS WITH ZIRCONIA, CERIA  
AND LANTHANA**

Oksana A. Kornienko, E. R. Andrievskaya

*Frantsevich Institute for Problems of Materials Science, Ukraine NASU, Kiev, Ukraine*

Study of phase equilibria in the systems with zirconia, ceria and lanthana is interesting from both fundamental and practical viewpoints. Materials based on ceria, zirconia and lanthana are perspective for application in medicine, energy production and machine building because of unique combination of their properties. The phase diagrams of the systems are considered the physicochemical background for creation of such materials as solid electrolytes in fuel cells, oxygen sensors, catalyst carriers, refractory for furnaces, thermal barrier coatings alloys and so on.

The objective of this work is to investigate the phase equilibria in the ternary  $ZrO_2$ - $CeO_2$ - $La_2O_3$  system and specify phase relations in the boundary binary system  $CeO_2$ - $La_2O_3$  at 1500 and 1100°C in air in the whole concentration range. Powders of  $ZrO(NO_3)_2$ ,  $CeO(NO_3)_2$  and  $La_2O_3$  (99.99 %) were used as raw materials. The samples were prepared in steps of 1 and 5 mol % from nitrate solutions with their subsequent evaporation and decomposition at 1250°C for 2 h. Thermal treatment was carried out in the furnaces with heating elements based on  $MoSi_2$  (1500°C for 150 h) and H23U5T (1250°C for 12780 h) in air. The phase compositions were investigated by X-ray diffraction, microstructural phase and electron microprobe X-ray analyses.

The results of this investigation showed that the structure of the boundary binary systems defines the phase equilibria in the ternary system. The isothermal

sections at 1500 and 1100°C for the ternary system  $ZrO_2$ - $ZrO_2$ - $CeO_2$ - $La_2O_3$  were developed. The solid solutions based on tetragonal (T)  $ZrO_2$  and cubic with fluorite-type structure (F)  $CeO_2$ , hexagonal (A)  $La_2O_3$ , as well as intermediate phase with pyrochlore-type structure (Py)  $La_2Zr_2O_7$  were formed in this system and their lattice parameters were determined.

The study of solid state reaction of  $CeO_2$  (fluorite-type, F) and  $La_2O_3$  (hexagonal modification of rare-earth oxides, A) at temperatures 1500, 1100°C showed that, in the system  $CeO_2$ - $La_2O_3$ , two types of solid solutions were revealed: cubic symmetry based on fluorite F- $CeO_2$  and hexagonal one A- $La_2O_3$ - based, which separated by two phase field (A+F). The ceria solubility in A- $La_2O_3$  is 25 mol % at 1500°C and 15 mol % at 1100°C. The lanthana solubility in the F- $CeO_2$  was found is about 49 mol % at 1500, 1100°C.

A25

### **ZrO<sub>2</sub>:Tb NANOPOWDERS FOR APPLICATION IN PHOTONICS**

Jarosław Kaszewski<sup>1</sup>, Urszula Narkiewicz<sup>1</sup>, Witold Łojkowski<sup>2</sup>

<sup>1</sup>*Institute of Chemical and Environment Engineering, West Pomeranian University of Technology, Szczecin, Poland*

<sup>2</sup>*Institute of High Pressure Physics, Polish Academy of Sciences, Warszawa, Poland*

Zirconium dioxide is known for its ultimate properties, which can be enhanced by using nanocrystalline instead of microcrystalline material. Zirconia is excellent material for application in photonics due to its hardness, transparency and high refractive index. Zirconium dioxide has three polymorphs in standard conditions: monoclinic, tetragonal and cubic. The monoclinic phase is thermodynamically stable up to 1100°C, and tetragonal phase exists in the temperature range 1100-2370°C. By lowering the size of the zirconia particles below critical diameter, one can obtain stable tetragonal phase in room temperature. Another way to synthesize high temperature phase particles is doping pure zirconium dioxide with some foreign ions. Classic stabilisation dopants are yttrium, scandium, lanthanum and calcium. Terbium is very attractive as a dopant due to efficient green light emission. Several terbium doped materials were commercialised as light conversion phosphors. Solvothermal synthesis has many advantages in comparison with classic calcinations process. Near or supercritical solvent has a number of properties that allow obtaining high quality nanomaterials. Microwave heating in solvothermal process has even more advantages including low temperature gradient in reaction mixture or enhanced process kinetics. Fine  $ZrO_2$ :Tb nanopowders were obtained by microwave solvothermal processing and other methods. We will discuss properties of prepared nanopowders as materials applicable in photonics.



A26

**SYNTHESIS AND CHARACTERIZATION OF CONCENTRATION  
GRADIENT BASED CdSe/AB HETEROSTRUCTURE QUANTUM DOTS**

Zoltán Győri, Ákos Kukovecz, Zoltán Kónya

*Department of Applied and Environmental Chemistry, University of Szeged,  
Szeged, Hungary*

Quantum Dots (QDs) are semiconductor nanocrystals. They are intensely studied today because of their unique, size-dependent optical and electronic properties originating from the quantum confinement effect. Because of their extraordinary optical properties, there are many efforts to use them as light-emitting diodes, lasers or as markers in biological imaging.

In order to utilize their good optical properties it is necessary to protect the surface of the nanocrystals, therefore, core/shell QDs are often preferred to bare dots. CdSe QDs are often investigated in practical application oriented works because their emitted wavelengths cover the whole visible spectral range. These nanocrystals can be coated by a wider bandgap semiconductor shell which not only protects the core but also reduces the nonradiative recombination which results in brighter emission.

The main synthesis method of core/shell dot production is the so-called two-step mechanism: first, semiconductor nanocrystals are synthesized and then these are coated with several atomic layers of a wide band semiconductor material. If the chemical reactivities of the different constituents are favourable it is possible to create core/shell QDs with the quicker and simpler „one-pot” synthesis which results in QDs with a concentration gradient core-shell structure.

We studied the properties of  $Cd_xZn_{1-x}Se_ySy_{1-y}$  heterostructured QDs. These concentration gradient based QDs were characterized using TEM, PL spectroscopy, UV-VIS and XRD. We were able to get dot-shaped nanocrystals with good size uniformity and tunable emission wavelength that depends on composition and crystal growth time.

A27

**PREPARATION AND PROPERTIES OF ZIRCONIA NANOPOWDERS  
IN THE  $MnO_x$ - $Y_2O_3$ - $ZrO_2$**

Daria Pomykalska, Mirosław M. Bućko

*AGH – University of Science and Technology, Faculty of Materials Science and  
Ceramics, Kraków, Poland*

Fully stabilized zirconia solid solutions (s.s.) containing manganese seem to be promising materials for intermediate layers between electrolytes and cathodes in solid

oxide fuel cells. When manganese is present in the zirconia solid solutions, they start to exhibit electronic conductivity due to the formation of some electronic defects related with variable valence of manganese and, in consequence, activation polarization losses that accompany the transfer of charge between electrolyte and cathode may be reduced. Powders of (Mn,Y,Zr)O<sub>2</sub> materials were prepared by means of the co-precipitation-calcination method and then sintered for 2h at 1500°C. Two series of zirconia solid solution were prepared. In the first series manganese ions were substituted for yttrium ions and in the second series for zirconia ones. The compositional dependence of the lattice parameters did not exactly comply with the Vegard rule what suggested variability of manganese valency. The bulk and grain boundary conductivity of the samples, determined using impedance spectroscopy, were strictly dependent on their chemical composition. The measurements of the electromotive force of galvanic cells involved prepared samples as electrolyte revealed their electronic conductivity.

A28

### **SYNTHESIS AND CHARACTERIZATION ZnO DOPED WITH Fe<sub>2</sub>O<sub>3</sub>, CoO AND Al<sub>2</sub>O<sub>3</sub>**

Daniel Sibera<sup>1</sup>, Urszula Narkiewicz<sup>1</sup>, Jan Mizeracki<sup>2</sup>, Adam Presz<sup>2</sup>,  
Witold Łojkowski<sup>2</sup>

<sup>1</sup>*Institute of Chemical and Environment Engineering, West Pomeranian University of  
Technology, Szczecin, Poland*

<sup>2</sup>*Institute of High Pressure Physics, Polish Academy of Science, Warszawa, Poland*

The aim of the work was to synthesize and characterize a series of nanosized ZnO powders doped with various concentration (from 5 to 70 wt.%) of transition metal and alumina. The effect of chemical composition on morphology and phase composition of the samples was determined. The samples were prepared by coprecipitation and calcination processes from nitrate solutions using ammonia. The obtained hydroxides were filtered, dried and calcined at 300°C for 1 hour. The phase composition of the samples was determined using X-ray diffraction measurements. For ZnO doped with Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> nanocrystalline samples, the hexagonal ZnO, and/or cubic ZnFe<sub>2</sub>O<sub>4</sub> and ZnAl<sub>2</sub>O<sub>4</sub> phases were identified. In case of ZnO doped with CoO the hexagonal ZnO, Co<sub>3</sub>O<sub>4</sub> and spinel ZnCo<sub>2</sub>O<sub>4</sub> were identified. The morphology of the received materials was characterized using scanning electron microscopy. Depending on chemical composition, different structures of agglomerates were observed.

A29

### SYNTHESIS OF CORE-SHELL PARTICLES FOR ENZYME IMMOBILIZATION

Milan Nikolić<sup>1</sup>, Konstantinos P. Giannakopoulos<sup>2</sup>, Mirjana Antov<sup>1</sup>,  
Vladimir V. Srdić<sup>1</sup>

<sup>1</sup>*Faculty of Technology, University of Novi Sad, Novi Sad, Serbia*

<sup>2</sup>*Institute of Materials Sciences, NCSR “Demokritos”, Athens, Greece*

Particles of core-shell structure were synthesized and used for immobilization of lipase from *Candida rugosa*. Silica core particles were initially prepared using tetraethylorthosilicate and used for assembly of zinc ferrite and silica nanoparticles on their surface. External surface of the silica cores was in addition modified to enable attractive electrostatic interaction between oppositely charged surfaces of silica cores and shell silica particles. The pore structure and thickness of mesoporous shell were tuned by changing the processing parameters. Transmission electron microscopy revealed that the synthesized particles had well defined core which was evenly coated with shell. It was shown that core particles were uniform spheres with average diameter ~600 nm having very low total pore volume while the silica shell, obtained using a sodium silicate as precursor, had mesoporous structure with average pore size up to 20 nm, high surface area and high total pore volume. On the other side, the zinc ferrite shell had small pores below 5 nm and insufficiently developed crystal structure. Lipase was immobilized inside mesoporous silica shell and amount of bound enzyme depended on the shell thickness and pore structure.

A30

### FERROELECTRIC CHARACTERIZATION OF LOW TEMPERATURE SOL-GEL BONDED PARTICLES

Tobias Lehnert, P. Herbeck-Engel, J. Adam, M. Veith

*INM - Leibniz Institute for New Materials, Saarbruecken, Germany*

Small isolated particles with implied good ferroelectric properties are highly demanded for achieving fine-grained ferroelectric ceramics as well as for the preparation of flexible piezoelectric ceramic/polymer composites. While a large variety of synthetic methods offers ferroelectric particles with different particle sizes, crystallographic phases, structures, defect densities and chemical compositions, a simple correct measurement system to determine the ferroelectric properties of such as synthesized particles is lacking. We report on a method (Double Pulse Switching Current Method) to correctly characterize ferroelectric films made of such as synthesized particles.

Commercially available BaTiO<sub>3</sub> particles were glued together by an inorganic sol gel system. This inorganic binding system promotes a higher dielectric constant between the particles and therefore higher electric fields acting on the particles themselves are possible compared to a structure with solely air surrounding the particles. The achieved remanent polarization is still much lower (~ 15 %) than for a sintered ceramic and at the same time a higher electric field has to be applied on the sample due to the described dielectric contrast in the this films.

A31

### SCREEN-PRINTED PIEZOCERAMIC THICK FILMS

Elisa Mercadelli, Alessandra Sanson, Carmen Galassi

*Institute of Science and Technology for Ceramics, National Research Council,  
CNR-ISTEC, Faenza, Italy*

The attractive piezoelectric properties of Nd doped lead zirconate titanate (PZTN,  $\text{Pb}_{0.988}(\text{Zr}_{0.52}\text{Ti}_{0.48})_{0.976}\text{Nb}_{0.024}\text{O}_3$ ) ceramics have led to the extensive use of these materials in different technological applications. In recent years, the drive toward device miniaturization has created a strong interest in PZT thick-film technology. With thicknesses in the range 5–80  $\mu\text{m}$ , screen-printed PZT thick films fill an important technological gap between thin-film and bulk ceramics offering the advantage of miniature scale and direct integration into hybrid electronic packages.

On the other hand, the screen printing process presents potential thermal matching and chemical compatibility problems between the functional film, the substrate and the electrodes at the processing temperatures used.

Different PZTN inks have been optimized using terpineol as solvent. Their rheological and thermal behavior, were thoroughly characterized before printing them on different substrates. Among the ones generally used, silicon and alumina were selected as substrates for this study. Silicon for its applications in microelectromechanical systems (MEMS) and alumina because is a widely used cheap substrate for the electronics industry.

By optimizing the ink formulations and adjusting the screen printing setup, homogeneous single or multiple thick films with excellent printing resolution on both substrates were obtained.

Thermal analysis of PZTN inks was carried out to define the burnout process necessary to obtain cracks-free films. Sintering tests showed the massive PbO diffusion into the alumina substrate already at 900°C whereas, crack-free PZTN thick films with thickness ranging from 5 to 20  $\mu\text{m}$  were obtained on silicon.

A32

### **DIELECTRIC SPECTROSCOPIC STUDIES ON TITANATE NANOWIRES**

Henrik Haspel, Ákos Kukovecz, Zoltán Kónya

*Department of Applied and Environmental Chemistry, University of Szeged,  
Szeged, Hungary*

One-dimensional nanostructures such as nanotubes, nanowires and nanobelts have been actively studied recently in order to understand their novel physical properties and develop nanoscale electronic devices. A series of 1D nanostructures, such as ZnO, TiO<sub>2</sub>, In<sub>2</sub>O<sub>3</sub>, and SnO<sub>2</sub> nanowires/nanorods/nanobelts, have been successfully synthesized and applied in chemical sensors. Humidity sensors represent a particularly important field in chemical sensing due to their important practical applications in environment detection or monitoring.

Here we report on our dielectric spectroscopic studies on the sensing properties of hydrothermally synthesized titanate nanowires. Earlier impedance measurements performed at various frequencies, relative humidities and temperatures showed the dependence of the electrical conductivity on these parameters. We varied the water content (11-98 RH%) and temperature (275-307 K) of the titanate samples independently, and recorded the dielectric spectra in the 10<sup>-2</sup>-10<sup>7</sup> Hz frequency regime.

In order to get a better insight into the sensing feature and extract all the information carried by the measured spectra, we used different representations of dielectric data, e.g. permittivity and electric modulus. We identified the dielectric relaxations, the conductivity and electrode polarization contributions in the spectra and determined the describing parameters by non-linear fitting. Further conclusions could be drawn by analyzing the dependence of these parameters on the temperature and humidity.

A33

### **PROPERTIES OF CARBONACEOUS THIN FILMS CONTAINING 1D INORGANIC NANOOBJECTS**

Mária Daranyi, István Sarusi, Ákos Kukovecz, Zoltán Kónya

*Department of Applied and Environmental Chemistry, University of Szeged,  
Szeged, Hungary*

Carbon nanotubes (CNTs) have unique mechanical, thermal and electrical properties such as good thermal stability, exceptional electric conductivity and large elastic modulus. The combination of these advantageous properties renders CNTs

promising candidates for use as fillers in polymer nanocomposites. The incorporation of CNTs can make polymer matrices conductive and the presence of embedded CNTs can induce the enhanced growth of graphitic crystals in carbonized polymers [1.]

In this paper we report the effect of nanoparticles on the morphology, structure and electrical properties of a carbonized MWCNT/PAN composite thin layer. We examined the decomposition of a multiwall carbon nanotube (MWCNT)/polyacrylonitrile (PAN) composite layer. PAN, N,N-dimethylformamide and a predetermined amount of MWCNT were used to prepare the PAN solution. Polymer films of approx. 2-3  $\mu\text{m}$  thickness were cast on glass and quartz substrates by spin coating. The polymeric coatings were heated in N<sub>2</sub> flow (200-1000°C). The thickness of the carbon layers at different pyrolysis parameters was measured by scanning electron microscopy (Hitachi S-4700 Type II cold field emission SEM instrument). The conductivity of the samples was measured by a purpose-built two-probe apparatus utilizing a PicoScope ADC-216 oscilloscope with 16 bit A/D resolution and a Metex MS9150 power supply. The thermal decomposition was carried out by heating from 30 to 1000°C in helium atmosphere. The evolved gases were analysed by a Pfeiffer QMS 200 quadropole mass spectrometer. On the basis of our results it will be possible to fine-tune the properties of MWCNT-embedded carbon films.

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A34

### HYDROTHERMAL SYNTHESIS OF MODIFIED TITANATE-BASED ONE-DIMENSIONAL NANOSTRUCTURES

Ines Bračko, Boštjan Jančar, Danilo Suvorov

*Advanced Materials Department, “Jožef Stefan” Institute, Ljubljana, Slovenia*

Layered titanates with the general formula  $A_2Ti_nO_{2n+1}$  ( $A = H, Na, K$  and  $n = 3, 4, 5$ ) and  $A_xTi_{2-x/4}\square_{x/4}O_4$  ( $A = Na, H, Cs$ ,  $\square = \text{vacancy}$ ) exhibit a layered-type structure composed of edge-shared  $TiO_6$  octahedra and alkali metal cations in the interlayer space. The bulk-layered titanates have been particularly intensely investigated due to their ion-exchange ability and the possible applications arising from their structure. Furthermore, their layered structure and the ion-exchange/intercalation ability have made them interesting for the preparation of various one-dimensional (1D) nanostructures. Namely, the hydrothermal treatment of crystalline or amorphous  $TiO_2$  in a highly alkaline solution of NaOH provides an effective synthesis route for the formation of various 1D titanate-based nanostructures, such as nanotubes and nanobelts. The titanate-based 1D nanostructures inherit the intercalation ability of their parent titanates. Therefore, the intercalation of cations, molecules, nanoparticles, etc. leads to the preparation of various

1D nanostructured materials (1D perovskites) and enables the modification of the optical, electrical and magnetic properties of titanate-based 1D nanostructures. In particular, the modification of titanate-based 1D nanostructures by the transition metals is of great interest due to the possible applications of as-modified nanostructures in photocatalytic processes, waste-water purification and catalysis.

We synthesized one-dimensional, nanostructured alkaline titanates by the hydrothermal treatment of crystalline  $\text{TiO}_2$ , anatase in a 10-M NaOH solution at various temperatures and for various times. The modification of the hydrothermally synthesized 1D nanostructures was performed under ambient and hydrothermal conditions. For the characterization of the crystal structure, the morphology and the composition of the products we used x-ray powder diffraction and transmission electron microscopy (HRTEM, EDS, and electron diffraction).

A35

### ELECTRICAL PROPERTIES OF BARIUM BISMUTH TITANATE

Jelena D. Bobić<sup>1</sup>, M. M. Vijatović<sup>1</sup>, S. Greičius<sup>2</sup>, J. Banys<sup>2</sup>, B. D. Stojanović<sup>1</sup>

<sup>1</sup>*Institute for Multidisciplinary Researches, Belgrade, Serbia*

<sup>2</sup>*Faculty of Physics, Vilnius University, Vilnius, Lithuania*

Family of bismuth oxides was discovered more than 50 years ago by Aurivillius [1]. Recently, there has been renewed interest in the properties of the Aurivillius phases as temperature-stable ferro-piezoelectrics [2]. Several bismuth-layered crystal structures and their properties have been investigated in detail. However, a lot of aspects of the preparation and properties of barium bismuth titanate,  $\text{BaBi}_4\text{Ti}_4\text{O}_{15}$  [BBiT] remain unexplored, whereas being promising candidate for high-temperature piezoelectric applications, memory application and ferroelectric nonvolatile memories (Fe-RAM).

	$T_c$ (°C)	$\Delta T_{\text{relaxor}}$	$\epsilon$	$\tan\delta$	$\gamma$
BBiT - 1	417 - 402	15	1188-1546	~ 0.07	1.97

In present work, BBiT was prepared by solid state reaction from mixture of oxide:  $\text{BaO}$ ,  $\text{TiO}_2$  and  $\text{Bi}_2\text{O}_3$  which was, previously, milled for 6 h. Mixture was heated at 750°C for 4 h and after that sintered at 1130°C for 1 h. The values of  $T_c$ , dielectric constant ( $\epsilon$ ), the loss tangent ( $\tan\delta$ ), degree of relaxation behavior ( $\Delta T_{\text{relaxor}}$ ) and degree of diffuseness of the material ( $\gamma$ ) are summarized in Table below. The degree of relaxation behavior is found to be only 15 K, which suggests the shift of  $T_c$  with frequency is not much distinct in BBiT ceramics. A modified Curie-Weiss relationship is used to study the diffuseness behavior of a ferroelectric phase transition where value of  $\gamma$  indicates the degree of diffuseness of BBiT material. The dielectric relaxation rate follow the Vogel-Fulcher relation with activation energy = 0.013eV, relaxation

frequency =  $2.09 \times 10^8$  Hz, and freezing temperature =  $378^\circ\text{C}$ . All these parameters indicate that BBiT is a relaxor ferroelectric.

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A36

### HIGH TEMPERATURE REACTIONS IN $\text{ZrSiO}_4\text{-Al}_2\text{O}_3$ SYSTEM

Artur Bradecki, Stanisława Jonas

*AGH University of Science and Technology, Faculty of Materials Science and Ceramics  
Kraków, Poland*

The mullite-zirconia composite possesses high fire and heat shock resistance as well as good mechanical and chemical resistance at high temperatures. In this work solid state reactions between  $\text{ZrSiO}_4$  and  $\text{Al}_2\text{O}_3$  leading to formation of mullite and zirconia composite material have been investigated. The oxide composition was specified according to a stoichiometry of the reaction equation  $3\text{Al}_2\text{O}_3 + 2\text{ZrSiO}_4 \rightarrow 3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 + \text{ZrO}_2$ . Data were obtained at temperatures ranging from  $1400^\circ\text{C}$  to  $1600^\circ\text{C}$  for a period of reaction time ranging from 30 minutes to 60 hours. Samples were examined with X-ray diffraction and electron scanning microscopy with energy dispersive X-ray analysis (SEM+EDX). Obtained results indicate that the first stage of the reaction is decomposition of  $\text{ZrSiO}_4$  into oxide components, then a two-way diffusion of  $\text{Si}^{+4}$  and  $\text{Al}^{+3}$  ions goes on followed by nucleation and crystalline mullite grows. Reaction rate at the temperature  $1500^\circ\text{C}$  is very low. Decomposition of  $\text{ZrSiO}_4$  is the fastest stage of reaction at the temperature of  $1600^\circ\text{C}$ .



A37

**SYNTHESIS AND PROPERTIES OF LOW CEMENT CASTABLE  
SINTERED AT DIFFERENT TEMPERATURES**

Sanja Martinović<sup>1</sup>, Jelena Majstrović<sup>2</sup>, Velislav Vidojković<sup>1</sup>,  
Tatjana Volkov-Husović<sup>3</sup>

<sup>1</sup>*Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia*

<sup>2</sup>*University of Belgrade, Faculty of Mining and Geology, Belgrade, Serbia*

<sup>3</sup>*University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia*

Low cement high alumina castable studied in this paper was synthesized and treated at different sintering temperatures. Since any inhomogeneity introduced during the castable preparation can remain inside the material degrading its properties and therefore quality during service life, particular attention was given to the processing procedure in order to produce material with the optimal characteristics. Composition of the castable regarding particle size distribution was adjusted according to the Andreassen’s packing model. Samples were sintered at 1100, 1300 and 1600°C for three hours. Influence of different sintering temperatures on the castable properties will be discussed. Compressive and flexural strengths were determined by classic method of destructive testing while water immersion method was used for determination of bulk density and apparent porosity. Changes of elastic properties and microstructure were observed by non-destructive testing methods, ultrasonic measurements and image analysis. Based on the results, it can be concluded that sintering temperature has strong influence on the properties of the low cement castable. Exceptionally good properties was obtained for the sample sintered at 1600°C, but it should be highlighted that the samples treated at 1100 and 1300°C were provided good properties, too. This should not neglect because of the energy saving importance.

A38

**EFFECT OF THE THERMAL SCHEDULE TREATMENT ON THE  
OBTAINING OF HIGH DENSITY ALUMINA**

Dunja Avramov<sup>1</sup>, Nikolina Pavlović<sup>1</sup>, Vaclav Pouchly<sup>2</sup>, Ljubica M. Nikolić<sup>1</sup>,  
Karel Maca<sup>2</sup>

<sup>1</sup>*University of Novi Sad, Faculty of Technology, Department of Materials Engineering,  
Novi Sad, Serbia*

<sup>2</sup>*Department of ceramics and polymers, Brno University of Technology, Brno,  
Czech Republic*

Obtaining nanocrystalline alumina with average grain size below 100 nm and high relative density is still one of the most challenging objectives in modern ceramic technologies. This kind of research is directed towards obtaining a transparent alumina ceramics with great possibilities of application in various optical devices. However, for the successful obtaining of such alumina it is necessary to solve many problems, starting from preparing suitable green bodies, as a critical precondition for obtaining dense, defect and pore-free ceramics, to the choice of appropriate temperatures and regimes of sintering. According to literature data, in order to achieve submicrometer grain size in alumina various regime of sintering, mostly pressure-assisted techniques, are usually applied with all their disadvantages and limitations. The main goal of this work is to apply and compare effects of the different sintering conditions on production of high density alumina. Two types of powders (one prepared from Bayer liquor and the other commercial) were subjected after cold isostatic pressing to three different procedures of sintering: single step sintering up to 1500°C, two step sintering and three step sintering as recently reported new method. Various temperatures of individual sintering steps were chosen on the basis of dilatometric measurements. Obtained alumina samples were characterized using XRD, SEM, BET, FTIR spectroscopy and DLS methods. Apart from achieved relative density of alumina samples up to 99.68%TD, all obtained results confirmed that further experimental work is needed for better optimization of all process parameters.

A39

### **STRUCTURE AND ELECTRIC CONDUCTIVITY OF (La, Sr)(Ga, Mg)O<sub>3</sub> SOLID ELECTROLYTE**

Cristian Andronescu, S. Preda, C. Hornoiu, A. Musuc, E. Volceanov, V. Fruth

*Institute of Physical Chemistry “Ilie Murgulescu” of the Romanian Academy,  
and “Politehnica” University of Bucharest, Bucharest, Romania*

Sr and Mg doped lanthanum gallate perovskites (La<sub>1-x</sub>Sr<sub>x</sub>Ga<sub>1-y</sub>Mg<sub>y</sub>O<sub>3-δ</sub>) shortened as LSGM are promising electrolyte materials for intermediate temperature solid oxide fuel cells (IT-SOFCs).

The evolution of secondary phases, such as LaSrGa<sub>3</sub>O<sub>7</sub> has been identified to be a problem in the preparation of LSGM. Actually, no matter what technical preparation route was adopted (wet-based or solid state-based method) the presence of the secondary phases, accompanying the main phase LSMG were reported.

In this work we try to obtain high pure Sr and Mg doped LaGaO<sub>3</sub> ceramics using a specific annealing treatment and to characterized this material. Thermal analysis (DTA/TG) on the stoichiometric raw mixture was realized. Morphological and structural aspects were investigated by mean of X-ray diffractometry, FT-IR spectrometry and scanning electron microscopy. The electrical behaviour of the obtained ceramic was characterized by impedance spectroscopy.

A40

### **CHARACTERIZATION OF BaCe<sub>0.8</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>O<sub>3-δ</sub> PROTON CONDUCTOR**

Katarzyna Gdula, Aleksandra Mielewczyk, Sebastian Molin\*, Bogusław Kusz,  
Maria Gazda

*Faculty of Applied Physics and Mathematics – Solid State Physics Department, \*Faculty  
of Electronics, Telecommunications and Informatics – Department of Biomedical  
Engineering, Gdansk University of Technology, Gdansk, Poland*

Intermediate temperature solid oxide fuel cells (IT-SOFCs) are very promising devices of energy conversion. Proton conducting ceramics may be applied as electrolytes in the intermediate temperature solid oxide fuel cells. Protonic ceramic fuel cells (PCFC) are especially attractive for operation at intermediate temperatures of 600°C and below. Many rare-earth doped perovskite materials have been studied as electrolytes for PCFCs because of their high protonic conductivity at about 600°C and their stability in reducing atmospheres. This work presents the structure, microstructure and electrical properties of BaCe<sub>0.8</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>O<sub>3-δ</sub> proton conducting ceramics produced by molten salt synthesis (MSS) method using KCl as a salt in 1100°C. Initial perovskite structure BaCeO<sub>3</sub> was

doped by yttrium to increase protonic conductivity and zirconium was applied to improve chemical stability of this material. Structure and microstructure were investigated by X-ray diffraction. Electrical properties of the samples were studied with Impedance Spectroscopy (IS).

A41

#### **SYNTHESIS AND CRYSTAL STRUCTURE OF $\text{Ca}_{(1-x)}\text{Gd}_x\text{MnO}_3$**

Milena Rosić, Branko Matović, Biljana Babić, Adela Egelja,  
Aleksandar Devečerski, Ana Radosavljević - Mihajlović

*Institute of Nuclear Sciences "Vinca", Laboratory for Material Science, Belgrade, Serbia*

This paper deals with  $\text{Ca}_{(1-x)}\text{Gd}_x\text{MnO}_3$  ( $x = 0.1; 0.2; 0.3$ ) synthesized by sucrose-nitrate procedure (SNP). SNP is combustion method in which sucrose  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$  was used as fuel while calcium nitrate tetrahydrate  $\text{Ca}(\text{NO}_3)_4 \cdot \text{H}_2\text{O}$ , manganese (II) nitrate hydrate  $\text{Mn}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ , gadolinium (III) nitrate hexahydrate  $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  were used as oxidants. Obtained powders  $\text{CaMnO}_3$ ,  $\text{Ca}_{0.9}\text{Gd}_{0.1}\text{Mn}$  and  $\text{Ca}_{0.8}\text{Gd}_{0.2}\text{Mn}$  were calcinated at temperature between 600 - 800°C. The structural parameters of the synthesized samples and structural changes were investigated by X-ray analysis and Raman spectroscopy. Williamson - Hall plots were used to separate the effects of size and strain in the nano powders. Specific area was measured by BET method.

A42

#### **THERMAL ANALYSIS OF Sr AND Mg DOPED LANTHANUM GALLATE (LSGM) OBTAINED BY WET CHEMISTRY ROUTE**

Adina Magdalena Musuc, Jose Calderon Moreno, Cristian Andronescu, Silviu Preda, Adriana Rusu and Victor Fruth

*Romanian Academy, "Ilie Murgulescu" Institute of Physical Chemistry,  
Bucharest, Romania*

Sr and Mg substituted  $\text{LaGaO}_3$  (LSGM) is a promising solid electrolyte for intermediate temperature solid oxide fuel cells (IT-SOFC). Phase purity of this material and bulk high densities were a subject of investigation for a long time. Thermal behavior of intermediate phases and final LSGM phase were investigated by means of differential scanning calorimetry (DSC) and DTA/TG analysis combined with FTIR spectrometry. The thermodynamic parameters of intermediary phases were evaluated. Also the kinetics of chemical reactions was investigated. The powders and sintered bodies were characterized using scanning electron microscopy-energy dispersive analysis (SEM-

EDAX), and X-ray diffraction (XRD), FTIR spectroscopy. On the basis of thermal analysis combined of FTIR spectrometry a detailed reaction mechanism was proposed.

A43

**MOLTEN SALT SYNTHESIS AND PROPERTIES OF ACCEPTOR  
DOPED LaNbO<sub>4</sub>**

Aleksandra Mielewczyk<sup>1</sup>, Katarzyna Gdula<sup>1</sup>, Sebastian Molin<sup>2</sup>, Bogusław Kusz<sup>1</sup>,  
Maria Gazda<sup>1</sup>

<sup>1</sup>*Faculty of Applied Physics and Mathematics and* <sup>2</sup>*Faculty of Electronics,  
Telecommunications and Informatics, Gdansk University of Technology,  
Gdansk, Poland*

For breakthrough development in solid oxide fuel cell, novel materials should be obtained. Proton conducting materials like acceptor doped LaNbO<sub>4</sub> are promising in relation to their future technical applications in SOFC. In this work several acceptor doped LaNbO<sub>4</sub> electrolytes, with different stoichiometries, were synthesized using molten salt synthesis method (MSS). Molten salt synthesis seems to be promising method for obtaining single phase and oriented samples in lower temperatures in comparison with solid state synthesis. In this method nominal amounts of oxides were mixed with KCl salt in 1:2 ratio and milled in agate mortar. The powder was calcinated at 1100°C for 3h. After cooling to room temperature the KCl was removed by washing with hot deionised water. After drying, the obtained powder was uniaxially pressed into pellets which were calcinated at various temperatures from 900-1200°C. The properties of synthesized samples were investigated using X-ray diffraction (XRD) and Impedance Spectroscopy (IS) methods.

A44

**GAS SEPARATION MEMBRANES AND FLOW-THROUGH  
CATALYTIC MEMBRANE REACTORS BASED ON POROUS  
ALUMINA FILMS**

Dmitry Petukhov, Dmitry Buldakov, Ruslan Aziev, Andrei Eliseev,  
Yuri Tretyakov

*Materials Science Department of Moscow State University, Moscow, Russia*

One of the most important trends of modern industry is separation of gas and liquid mixtures and developing methods for their purification away from different

pollutions. Polymeric membranes are widely using for separation and purification processes today. However polymeric membranes have some disadvantages: low thermal and chemical stability. Ceramic membranes are deprived of these disadvantages. Ceramic membranes can be used for high temperature gas separation and as supports for catalytic membrane reactors. So the main aim of this work is to study possibility using anodic alumina films for gas separation.

Anodic alumina films were prepared by two-step anodization of aluminum in 0.3M oxalic acid solution at 40V or by one step anodization of aluminum using “hard anodization” approach (in 0.3M oxalic acid solution at constant voltage in range from 130 to 160V). According to SEM prepared films have inner pore diameter in range from 36 nm to 190 nm, depending on anodization conditions. Film thickness is in range from 70 to 200  $\mu\text{m}$ , depending on anodization duration.

Obtained films have high thermal stability porous structure is stable up to 1000°C, and crystallization of amorphous alumina into  $\theta\text{-Al}_2\text{O}_3$  occurs at 840°C.

According to gases permeability measurement the membranes permeance is very high up to 170  $\text{m}^3/(\text{m}^2 \cdot \text{bar} \cdot \text{hour})$  for nitrogen at 25°C. The main mechanism of gas diffusion through membranes is the Knudsen diffusion (the quantities of gas flow linearly depend on inverse square root of gases molecular weights and linearly depend on inverse square root of gas temperature). Consequently anodic alumina films have high permeance and Knudsen selectivity, so it can be used for high temperature gas separation based on Knudsen diffusion.

Moreover in our work we try to use anodic alumina films as a support for asymmetrical membrane Pd/Al<sub>2</sub>O<sub>3</sub>. The thin layer of Pd was sputtered on alumina film by thermal sputtering. According to gas permeability measurements sputtering of Pd film lead to drastically increasing of membrane selectivity to hydrogen (ideal separation factor H<sub>2</sub>/CO<sub>2</sub> 27, hydrogen permeance 7.2  $\text{m}^3/(\text{m}^2 \cdot \text{bar} \cdot \text{hour})$  at 450°C).

Another prospective application of anodic alumina films are supports for flow-through catalytic membrane reactors. In this work we obtain Pt nanoparticles on pore walls by incipient wetness impregnation of alumina membrane with 0,025M H<sub>2</sub>PtCl<sub>6</sub> solution and further annealing in hydrogen atmosphere. Propylene hydrogenation was used as a model reaction for study catalytic activity of catalytic membrane reactor. According to our measurement full conversion of propylene into propane take place at 70°C.

A45

### **EFFECT OF STARTING NANOPOWDERS ON DENSIFICATION AND PROPERTIES OF CERIA**

Marija Puševac<sup>1</sup>, Slavica Zec<sup>1</sup>, Snežana Bosković<sup>1</sup>, Branko Matović<sup>1</sup>,  
Fritz Aldinger<sup>2</sup>, Slavko Mentus<sup>3</sup>

<sup>1</sup>*Institute of Nuclear Sciences "Vinča", Belgrade, Serbia,*

<sup>2</sup>*Max-Planck Institute, PML, Stuttgart, Germany,*

<sup>3</sup>*Faculty of Physical Chemistry, University of Belgrade, Belgrade, Serbia*

Ceria based nanopowders were prepared in two different ways by applying modified glycine/nitrate (MGNP) and self propagating room temperature (SPRT) methods. Nanopowders differed in properties with particle size of SPRT being an order of magnitude lower in comparison with powders obtained by MGNP procedure. Two different compositions were synthesized:  $Ce_{0.8}Y_{0.18}Gd_{0.01}Sm_{0.01}O_{2-\delta}$  and  $Ce_{0.8}Nd_{0.01}Sm_{0.015}Gd_{0.025}Dy_{0.04}Y_{0.05}Yb_{0.06}O_{2-\delta}$ , as well as pure ceria for comparison. Completely different dilatometric behavior was observed whereby SPRT powders underwent very fast sintering at much lower temperatures in comparison with MGNP powders. This behavior as well as the properties of samples was discussed in our paper.

A46

### **MECHANICAL PROPERTIES OF POROUS Y-TZP CORE-SHELL NANOCOMPOSITES**

Sebastjan Perko, Ales Dakskobler, Tomaz Kosmac

*“Jozef Stefan” Institute, Ljubljana, Slovenia*

Porous ceramics are gaining increasing interest because of their wide array of applications, such as filters, sensors, catalysts supports and biomaterials. Since these materials exhibit poor mechanical properties, any improvement should have a significant effect on their applicability. In order to increase the strength while keeping high porosity, several approaches are being pursued including mixing of starting powders with different particle size distributions.

In our work, the core-shell concept was adopted in order to produce high strength porous Y-TZP ceramics. Materials were prepared using homoaggregation resulting in a homogeneous distribution of nano-sized particles attached to the surface of micron-sized particles. After slip casting, green pellets were biscuit-sintered at various temperatures in an ambient air atmosphere. The flexural strength of these core-shell nanocomposites progressively increased with the fractional density, starting from 80 MPa at 55% of theoretical density (TD) and reaching a plateau of 670 MPa at 70% of

TD. This value is approximately two times higher than that obtained with the conventional biscuit-sintered Y-TZP ceramics exhibiting similar fractional density.

A47

### **CALCIUM PHOSPHATE BIOCERAMICS PREPARED BY WET CHEMICAL PRECIPITATION ROUTE**

Kristine Salma, Liga Berzina-Cimdina, Natalija Borodajenko

*Riga Biomaterials Innovation and Development Centre, Riga Technical University,  
Riga, Latvia*

Due to excellent biocompatibility with living body and bioactivity, calcium phosphate (CaP) ceramics (hydroxylapatite [HAp,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ],  $\beta$ -tricalcium phosphate [ $\beta$ -TCP,  $\text{Ca}_3(\text{PO}_4)_2$ ], biphasic calcium phosphates [BCP,  $\beta$ -TCP + HA]) are widely used as biomedical implant materials. Biomaterials and bioceramic made of calcium phosphates are produced in different forms: granules, blocks (dense or porous), injectable compositions, self-hardening cements, coatings on metallic implants, composites from polymers, and so forth.

Various synthesis routes have been developed for the preparation of calcium phosphates, which must present desirable characteristics, such as, stoichiometry of synthesis product, crystallinity, particle size, morphology and specific surface area. Calcium phosphate characteristics significantly influence CaP bioceramic properties, such as, definite phase and chemical composition, sinterability, thermal stability, fine and homogenous microstructure, biomechanical properties and then cellular response in living body.

In this work calcium phosphates were synthesized by modified wet chemical precipitation route. Contrary to the conventional chemical precipitation route calcium hydroxide was homogenized with planetary mill to obtain homogenous synthesis product without impurities such as CaO.

The aim of this work was to study the influence of main processing parameters of wet chemical precipitation synthesis product and to control not only the phase and chemical composition, but also thermal stability and microstructure of calcium phosphate bioceramics.

The results showed that it is possible to obtain calcium phosphates with different and reproducible phase compositions (HAp,  $\beta$ -TCP and HAp/ $\beta$ -TCP) by modified wet-chemical precipitation route. The  $\beta$ -TCP content in HAp powder is found to be highly dependent on the changes in technological parameters and it can be controlled with ending pH, synthesis temperature, aging time of suspension and thermal treatment. Pure, crystalline and highly thermally stable (up to 1300°C) HAp bioceramic with homogenous grainy microstructure, grain size up to 200-250 nm and high open



porosity can be successfully synthesized by elevating synthesis temperature to 70°C, stabilizing ending pH at 9 and aging time of suspension at 20 h.

A48

### HYBRID MESOPOROUS MATRICES AND NANOCOMPOSITES OBTAINED BY SOL-GEL METHOD FOR BIOAPPLICATIONS

Zoltan Dudas<sup>1,2</sup>, Corina Enache<sup>1</sup>, Eugenia Cosma-Fagadar<sup>1</sup>, Gabriela Preda<sup>2</sup>,  
Adrian Chiriac<sup>2</sup>, Cecilia Savii<sup>1</sup>

<sup>1</sup>*Institute of Chemistry of the Romanian Academy, Timisoara, Romania*

<sup>2</sup>*West University of Timisoara, Timisoara, Romania*

The preferred methods developing new materials for industrial and medical applications should be easy to handle, low cost, besides they should offer a safe and suitable technology [1]. The sol-gel technology fulfills all this conditions [2-3].

In this work the sol-gel technique was used to generate silica matrices by acid hydrolysis of silane precursors as tetraethoxysilane (T), methyltrimethoxysilane (M), isobuthyltriethoxysilane (iB) dimethyldiethoxysilane (DM), in the presence and absence of additives such as polyvinylalcohol (PVA) and ionic liquids.

Some organic/inorganic hybrid SiO<sub>2</sub>-matrix composites were prepared by using mixtures of above mentioned precursors in the following mol ratios: M/T in the 3/1 and 1/3 range; DM/M/T: 4/1/1 and 3/1/1; DM/T in the 3/2 and 1/3 range and iB/T: 1/1.

Physicochemical characterizations were made by nitrogen adsorption/desorption isotherms, FT-IR spectroscopy, SEM, TEM, thermal analysis. All obtained amorphous matrices presented mesoporous morphology with the surface areas ranging between 7 and 535 m<sup>2</sup>/g.

The obtained matrixes were used to immobilize enzymes, porphyrins and Eu<sup>3+</sup>. The best results were obtained in case of matrixes prepared at DM/T : 3/2 molar ratio, in presence of PVA for Alcalase immobilization, iBT 1/1 molar ratio in presence of ionic liquid for Eu<sup>3+</sup> and 5,10,15,20-tetrakis(4-allyloxyphenyl) porphyrin entrapment.

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A49

### DEVELOPMENT OF POROUS BIOCERAMICS

Janis Locs, Rita Serzane, Liga Berzina-Cimdina, Romans Sadretdinovs  
*Riga Technical University, Riga Biomaterials Innovation and Development Center,  
Riga, Latvia*

Hydroxyapatite (HAp) is known as a highly bioactive and biocompatible inorganic material. There are several ways to fabricate porous bodies, such as foaming, solvent casting and particulate leaching, rapid prototyping applications like 3D printing, freezing technique, electro-spinning, etc. Our work mostly concerns from this methods casting/foaming, pressing with burn-out additives, plastic forming and polymer foam infiltration. The porosity and microstructure of the ceramics obtained are mainly influenced by manufacturing technology and additives used. The aim of this work is develop porous bioceramic materials with adequate pore structure and appropriate mechanical properties for bone ingrowth and regeneration based on HAp, which are used on a large scale for biomedical applications such as dentistry, bone replacement and tissue engineering. In current research commercial HAp and on site in Riga Technical University, Riga Biomaterials innovation and development centre synthesized HAp was used. Ceramics made from on site synthesized HAp has presented good results in previous in vivo and clinical studies. Samples obtained were investigated using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and mechanically tested. The pore size and the pore microstructure characteristics were determined using scanning electron microscopy (SEM). Beside, the porosity analysis was evaluated out using an Archimedes principle.

A50

### SYNTHESIS OF BIOMORPHIC SiC AND SiO<sub>2</sub> CERAMICS

Adela Egelja, Branko Matović, Biljana Babić, Aleksandar Devečerski,  
Milena Rosić  
*Institute of Nuclear Sciences "Vinča", Laboratory for Material Science, Belgrade, Serbia*

Tilia wood was transformed by pyrolysis into carbon preform. This porous carbon preform was infiltrated with TEOS (Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>), as a source of silica, without pressure at 298K. An in situ reaction between the silica and the carbon template occurred in the cellular wall at a high temperature. Depending on the employed atmosphere, non-oxide (SiC) or oxide (SiO<sub>2</sub>) ceramics were obtained. Scanning electron microscopy (SEM), X-ray diffraction (XRD), infra red (IR) spectroscopy, mercury porosimetry and BET measurements were employed to characterize the phases and crystal structure of

crystal structure of biomorphic ceramics. The experimental results showed that the biomorphic cellular morphology of the wood maintained in both the SiC and SiO<sub>2</sub> ceramics, which consisted of β-SiC with trace of α-SiC, and SiO<sub>2</sub>, respectively.

A51

### TECHNOLOGICAL RESULT OF DECOMPOSITION SODIUM ALUMINATE SOLUTIONS DEPENDING ON THE DECOMPOSITION PARAMETERS

R. Smiljanić<sup>1</sup>, Dragana Smiljanić<sup>1</sup>, R. Filipović<sup>1,2</sup>, M. Sudar<sup>2</sup>

<sup>1</sup>Alumina factory “Birac“, Zvornik, RS, Bosnia and Herzegovina

<sup>2</sup>Faculty of Technology, Zvornik, RS, Bosnia and Herzegovina

One of the main indicators of the efficiency of decomposition sodium aluminate solution was presented with the degree and the specific results of decomposition. As the value of these two indicators increased the specific consumption of energy is less. Tests carried out in this paper were aimed to demonstrate at which conditions specific result of decomposition sodium aluminate solution are the largest and how its increase reflects the quality of the obtained aluminum hydroxide. The results of these tests showed that correcting the parameters of decomposition, without changing the essential process of decomposition, with the same power consumption can be achieved increasing the degree and specific results of decomposition for 10 to 15%, and that the quality of the obtained aluminum hydroxide remains the same.

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A52

**INVESTIGATION OF THERMAL SHOCK RESISTANCE  
ENHANCEMENT FOR LAYERED CERAMICS WITH QUARTZ  
SUBSTRATE AND TWO-SIDED NIOBIUM PENTOXIDE COATING  
USING COMPUTER SIMULATION**

Olena Kirkova, Eugene Voynich, Alexander Frolov

*Frantsevich Institute for Problems of Materials Science of Ukraine, Kiev, Ukraine*

For investigation of thermal shock resistance enhancement of layered ceramics with quartz substrate and two-sided protective niobium pentoxide coating the mathematical modeling has been conducted. In the work [1] a substrate layer was proposed to fabricate from separate segments connected using the coating material for further increase in thermal shock resistance. Thus, samples in the form of plates with both continuous and segmented quartz substrates with niobium pentoxide junctures between segments were examined using computer simulation.

The simulation target was to research the effect of the difference in the linear thermal expansion coefficients for coating and substrate materials on the stress initiation in the article. Therefore, the temperature was assumed to be the same throughout the sample bulk for calculation simplicity. Since the experimental investigations showed that the most intense sample cracking occurred at about 800°C during cooling from 1000°C, the calculations were performed for this temperature range. The calculations were made using a thermoelastic model based on the finite element method. To estimate stresses arising in the plate during its cooling, the Mises stresses were used. As the developed articles consisted of long plane elements, the calculations were performed for a plane strain model. The calculation results showed that the average Mises stresses in quartz substrate and over the niobium pentoxide/quartz boundary decreased with dividing the substrate material into segments. Furthermore, the simulation revealed that the presence of sharp angles on the substrate segments led to the stress concentration while near angles with fillet maximum Mises stresses were lower. To determinate the influence of the juncture thickness on the arisen stresses, the calculation of the maximum stresses was performed for a juncture with different thickness. The simulation showed that the maximum Mises stresses for niobium pentoxide juncture decreased with increasing its thickness. Therefore, in designing articles the juncture were proposed to fabricate with thickness more than a half of the substrate segment thickness for thermal shock resistance enhancement.

The results of experiments, which were carried out using the equipment for AE registration, confirmed thermal shock resistance enhancement for segmented samples and samples without sharp angles.

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A53

### PREPARATION AND CHARACTERIZATION OF PLZT FERROELECTRIC CERAMICS

Katarzyna Berent<sup>1</sup>, Marek Faryna<sup>1</sup> and Małgorzata Płońska<sup>2</sup>

<sup>1</sup>*Polish Academy of Sciences, Institute of Metallurgy and Materials Science, Kraków,  
Poland*

<sup>2</sup>*University of Silesia, Faculty of Computer and Materials Science, Department of  
Materials Science, Sosnowiec, Poland*

Microstructural evolution and dielectrical properties of  $(\text{Pb}_{1-x}\text{La}_x)(\text{Zr}_{0.65}\text{Ti}_{0.35})\text{O}_3$  [designated as PLZT x/65/35] ceramics with the increasing La content have been investigated. The series compositions have been chosen because these compositions are across the economically important morphotropic phase boundary (MPB) area in the phase diagram of the PLZT solid – solution system. PLZT powders have been prepared by two different production routes: by the calcined mixed oxide method and the sol – gel method, they also underwent consolidation by means of the hot uniaxial pressing method. Sintering process was carried out at 1200°C.

The samples were characterized by X-ray Diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The dielectric properties of the ceramics were investigated by measurements of dielectric constant and dielectric loss as a function of temperature at a fixed frequency. For all samples the effect of La content induced changes in the material properties. Presented results of the research give a detailed account of the relationships between preparation method and physical properties of obtained materials.

**Keywords:** PLZT, ferroelectric ceramics, calcined mixed oxide, sol – gel

A54

### BARIUM TITANATE THICK FILMS PREPARED BY SCREEN PRINTING TECHNIQUE

Mirjana M. Vijatović<sup>1</sup>, J. D. Bobić<sup>1</sup>, B. D. Stojanović<sup>1</sup>, B. Malič<sup>2</sup>

<sup>1</sup>*Institute for Multidisciplinary Research, Belgrade, Serbia*

<sup>2</sup>*Institute Jozef Stefan, Ljubljana, Slovenia*

Pure and doped with lanthanum and antimony barium titanate (BaTiO<sub>3</sub>-BT, BTL and BTS) powders were prepared by the polymeric organometallic precursors method (Pechini process-PPM) starting from citrate solutions.

For the preparation of thick films was used screen printing technique. The paste was prepared from suspension of organic material (resin, organic solvent and additives to improve rheological behavior of paste) and barium titanate powders obtained by Pechini procedure, using a ratio 30:70. To get better adhesion between paste and substrate it was added a low temperature melting glass in powder form. The electrodes were prepared from the silver/palladium mixture (Ag/Pd – 70/30). The bottom electrode was deposited on the Al<sub>2</sub>O<sub>3</sub> support and in the middle was screen-printed dielectric layer of (1) pure barium titanate, (2) barium titanate doped with 0.3 mol% La or (3) barium titanate doped with 0.3 mol% Sb. On the top was deposited the upper electrode. The layer-sandwich was sintered at 850°C during 1h in air flow atmosphere.

Measurements of thickness and roughness of barium titanate thick films (BT, BTL and BTS) were performed as well as electrical measurements such as dielectric constant and dielectric losses vs. temperature. The value of dielectric constant was very low and the possible reason could be that the electrode material could diffuse from the surface of BT layer through intergranular pores and affect on changing the dielectric properties of films. This measurement also can point out that dopants may have the influence on dielectric constant. Dopants in this case are decreasing dielectric constant value. Measurement of hysteresis was also performed.

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A55

### INFLUENCE OF ALKALI EXCESS ON CRYSTALLIZATION OF LEAD-FREE SODIUM POTASSIUM NIOBATE THIN FILMS

Oana Catalina Mocioiu<sup>1</sup>, Barbara Malic<sup>2</sup>, Alja Kupec<sup>2</sup>, Marija Kosec<sup>2</sup>,  
Maria Zaharescu<sup>1</sup>

<sup>1</sup>*Ilie Murgulescu Institute of Physical Chemistry of Romanian Academy,  
Bucharest, Romania*

<sup>2</sup>*“Jožef Stefan” Institute, Ljubljana, Slovenia*

Alkaline niobates comprise a group of piezoelectric materials which could replace lead-based perovskites, such as commercially used  $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$  (PZT) ceramics, because they are lead-free and therefore environment friendly. Alkali niobate thin films could find applications in microelectronics and microelectromechanical systems (MEMS), however there are only rare reports on chemical solution deposition of such films.

The  $\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3$  coating solution with the stoichiometric (K+Na)/Nb ratio was obtained by reacting alkali acetates and niobium ethoxide in 2-methoxyethanol as a reactive solvent. The obtained xerogel was characterised by thermal analysis (TG/DTA) and infrared spectroscopy.

We prepared thin films from the solutions with the stoichiometric (K+Na)/Nb ratio and with 5 - 10 mol% excess by spin coating on Pt/Si substrates, drying, pyrolysis and rapid thermal annealing. In the presentation we discuss the effect of alkali excess on microstructure, phase composition and electrical properties of  $\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3$  thin films.

A56

### INFLUENCE OF $\text{MnO}_2$ AND $\text{WO}_3$ ADDITION ON SINTERING AND PROPERTIES OF LEAD-FREE KNN CERAMICS

Ilze Smeltere, Maris Livinsh, Marija Duncs, Vismants Zauls

*Institute of Solid State Physics University of Latvia, Riga, Latvia*

Piezoelectric ceramics are used for producing transducers, actuators, sensors etc. Usually lead based ceramics (PZT, PMN-PT) are used for this purpose. Hence lead is toxic it is an urgent task to search for lead free piezoelectric ceramics with electrical properties comparable to those of commercial PZT. One of the most promising candidates for replacing lead containing ferroelectric ceramics with perovskite structure is potassium sodium niobate. However the high volatility of alkali niobates during the sintering hinders the formation of dense ceramics. In this study we have investigated modified  $(\text{K}_{0.5}\text{Na}_{0.5})\text{NbO}_3$  solid solution systems.  $\text{MnO}_2$  and  $\text{WO}_3$  were used as dopants

with a purpose to promote the sintering and densify ceramic sample body. The influence of 0.5 – 1.5 wt% MnO<sub>2</sub> and WO<sub>3</sub> addition on the sintering, structure and electrical properties of (K<sub>0.5</sub>Na<sub>0.5</sub>)NbO<sub>3</sub> has been investigated. Optimal sintering temperatures of KNN ceramics were observed to be in the narrow interval: 1090-1110°C for MnO<sub>2</sub> doped KNN; 1140-1160°C for pure KNN and doped with WO<sub>3</sub>. Moreover a little shift from optimal temperature ( $\pm 10^\circ\text{C}$ ) can change the properties noticeably. XRD patterns showed that all the samples have single perovskite structure. Microstructure of ceramics was changed by using additives. Compared with undoped (K<sub>0.5</sub>Na<sub>0.5</sub>)NbO<sub>3</sub>, the addition of MnO<sub>2</sub> or WO<sub>3</sub> lead to improvement in the dielectric permittivity, piezoelectric charge coefficient d<sub>33</sub> as well remanent polarization Pr.

A57

### THE ROLE OF CHEMICAL DISORDER ON MAGNETISM OF (Cd,Zn)<sub>1-x</sub>Mn<sub>x</sub>GeAs<sub>2</sub> SEMICONDUCTOR

Lucacz Kilanski<sup>1</sup>, M. Górski<sup>1</sup>, W. Dobrowolski<sup>1</sup>, E. Dynowska<sup>1</sup>, M. Wójcik<sup>1</sup>,  
B. J. Kowalski<sup>1</sup>, J. R. Anderson<sup>2</sup>, C. R. Rotundu<sup>2</sup>, D. Maude<sup>3</sup>, S. A. Varnavskiy<sup>4</sup>,  
S. F. Marenkin<sup>4</sup>

<sup>1</sup>*Institute of Physics, Polish Academy of Sciences, Warsaw, Poland*

<sup>2</sup>*Department of Physics & Center for Nanophysics and Advanced Materials,  
University of Maryland, USA*

<sup>3</sup>*Laboratoire National des Champs Magnétiques Intenses, Grenoble, France*

<sup>4</sup>*Kurnakov Institute of General and Inorganic Chemistry RAS, Moscow, Russia*

We present systematic studies of structural, magnetic, and transport properties of (Cd,Zn)<sub>1-x</sub>Mn<sub>x</sub>GeAs<sub>2</sub> diluted magnetic semiconductors with chemical composition varying between 0.05 < x < 0.2 grown using a direct fusion method.

The magnetic properties of the studied system were studied by means of SQUID and AC magnetometers. The performed measurements showed that the studied system undergoes a paramagnet-ferromagnet phase transition at temperatures higher than 300 K. In order to explain the physical mechanisms responsible for the observed high temperature ferromagnetism we performed series of measurements including scanning electron microscopy accompanied with energy dispersive spectrometer, high resolution x-ray diffraction and nuclear magnetic resonance. All the performed measurements led to the same conclusion that the presence of MnAs phase separations was responsible for the observed room temperature ferromagnetism. The performed measurements gave for the first time in the studied group of alloys direct experimental proofs about the reason for the existence of a ferromagnetic ordering at high temperatures.

The transport studies revealed that electrical properties of the studied crystals depend strongly on their chemical composition. Depending on the cation type we obtained both hole and electron conductivity with Hall carrier concentrations controlled



by means of Mn content in a broad range of values between  $1018 \div 1020 \text{ cm}^{-3}$ . The high field measurements in magnetic fields up to 24 T revealed the presence of both positive and negative magnetoresistance with large magnitudes. We identified the existence of large nonsaturating positive magnetoresistance with values of the order of 100% at room temperatures. The transport properties were strongly correlated with the magnetic properties of the alloy.

A58

### **INFLUENCE OF CLAY MINERAL ADDITIVES ON THE PROPERTIES OF HIGH POROUS PERMEABLE CELLULAR GLASSFORMING MATERIALS**

Artem Iatsenko, Boris Kornilovych

*National Technical University of Ukraine, “Kyiv Polytechnical Institut”, Kyiv, Ukraine*

High – porous cellular materials (HPCM) are promising line of development of carriers for mass-transfer processes. It has high specific surface area, high permeability due to large open porous cells. That is why HPCM are perfect for use as substrates, catalysts, sorbents and filling for ion-exchange filters and for creation of filtration barriers for solving environmental problems. Such materials can be prepared by various methods. The most promising is duplication of polymer matrix. Polyurethane foams various polymer compositions are widely used materials for matrix. The sole and significant disadvantage of such cellular materials is low strength. The strength of filling must be 2.5-7.0 MPa and higher for use in areas indicated above. The strength of known from literature materials reached 1.4 – 2.7 MPa. Therefore, there is necessity to improve strength and keep high permeability and specific surface area.

The purpose of this study was to investigate the influence of various active additives on the final properties of highly porous materials obtained based on the silicate glass with the addition of montmorillonite.

Commercial open porous polyurethane foam with pore size of 3-5 mm was used for samples preparation. Slurry mass for coating was prepared based on powder of silicate glass with dominate fraction of 20 – 50  $\mu\text{m}$ . The additions 5 % (in addition to glass powder) of activate Na – bentonite was used as thixotropic agent that prevents sedimentation of glass particle in slurry. Polyvinyl alcohol (PVA) (0 – 0.5 %) and water-soluble sodium silicate (0 – 0.5 %) was used for investigation of additives influence.

The effect of microquantities of active additives on the rheological properties of glass - clay slurry was study. Moreover, the quality of prepared coating on polyurethane foam matrix and strength property of sintered HPCM was studied.

It was established that final material with microquantities of complex additives PVA and sodium silicate and without additives had strength 4.7 – 5.0 MPa and 2.4 – 2.5 MPa, respectively.





**CERAMICS COMPOSITES**

C1

**SEMICONDUCTING NANOPARTICLES IN  
POLY(2-(DIMETHYLAMINO)ETHYL METHACRYLATE-  
CO-ACRYLIC ACID COPOLYMER**

Lidija V. Trandafilović<sup>1</sup>, Vladimir Djoković<sup>1</sup>, Nataša Bibić<sup>1</sup>,  
Michael K. Georges<sup>2</sup>, Thottackad Radhakrishnan<sup>2</sup>

<sup>1</sup>*Vinča Institute of Nuclear Sciences, Belgrade, Serbia*

<sup>2</sup>*Department of Chemistry, University of Toronto at Mississauga, Mississauga,  
Ontario, Canada*

Cadmium sulfide, cadmium selenide, lead sulfide, silver sulfide semiconductors with poly(2-(dimethylamino)ethyl methacrylate-co-acrylic acid) co-polymers were prepared and characterized using structural and optical methods. Co-polymer matrices were synthesized in three different mol ratios of PDMAEMA:AA monomer units (1:2, 1:1, 2:1). Transmission electron microscopy analysis show presence of spherical nanostructured semiconductors incorporated in co-polymer matrices p(DMAEMA-co-AA). In the case of nanocomposites with cadmium sulfide and cadmium selenide particles, the optical measurement showed shifts of the onset of the optical absorption towards lower wavelengths. The presence of semiconductor nanoparticles with CdS, CdSe, PbS, Ag<sub>2</sub>S crystal phases was confirmed by XRD measurement. It was also shown that the size of the synthesized CdS nanoparticles depends on the initial molar content of the co-monomers in the copolymer matrix. Since PDMAEMA:AA has not been synthesized so far, the part of the study is concentrated on the physical properties of the copolymer itself.

C2

**MATERIALS FOR MEDICAL APPLICATIONS BASED ON  
POTASSIUM POLYPHOSPHATE AND CALCIUM  
HYDROXYAPATITE**

Mikhail Shiryaev, Tatiana Safronova, Valery Putlyaev

*Department of Materials Science, Moscow State University, Moscow, Russia*

In search of new materials for bone implants significant number of various substances has been already tested. Calcium phosphates are the most perspective materials bearing in mind their content and the role in human organism. Calcium hydroxyapatite (HAP) constitutes an inorganic part of bone tissue, being biocompatible, although, bioresistive and non-resorbable when implanted. Up to now, other phosphates such as calcium pyrophosphate, calcium orthophosphates, mixed alkaline metals-calcium

phosphates have been studied. However the way to an ideal bone substitute material is still unclear. Such a material has to be bioactive, bioresorbable enough to be substituted with ingrowing native, to resemble bone tissue in sense of structure and mechanical properties, and, in addition, it should demonstrate osteoinductivity, i.e. to provoke bone tissue growth. Polyphosphates (condensed phosphates) are significant biological regulators and take part in various enzymatic systems of human organism. So far polyphosphates have not been widely accounted for bone substitute materials in spite of their prominent osteoinductive properties.

The aim of this work was to fabricate and study biocompatible composite materials based on bioresistive HAP and potassium polyphosphate. Powders of HAP (Riedel-de Haën) and potassium dihydrophosphate (LabTeh) of chemical grade purity were used. The mixtures contained HAP and potassium dihydrophosphate in amount of 25, 50, 75 and 100 % with respect to HAP. The mixtures were preliminary examined by TGA to find optimal conditions for thermal treatment. Compacted samples were annealed at 400, 500, 600, 700, 800 and 850°C. XRD study of thus treated samples revealed dihydrophosphate polymerization, as well as HAP-dihydrophosphate interaction leading to phases of  $K_2CaP_2O_7$  (400°C) and two potassium polyphosphate forms (400 and 600°C).

Variability of phase composition and porosity of the samples under study allowed us to create the materials with pre-determined bioresorption rate for non-load-bearing implants. It must be mentioned that potassium polyphosphates with different degrees of polymerization possess variable solubility in fluid media of human organism. Taking this fact as a starting point, perspective bone substitute materials can be elaborated on the basis of polyphosphates.

C3

### **STRUCTURE EVOLUTION AND PROPERTIES OF BIOGENIC HYDROXYAPATITE-BASED BIOCOMPOSITE**

Olena Sych, Nataliya Pinchuk, Liana Ivanchenko

*Frantsevich Institute for Problems of Materials Science of National Academy of Sciences  
of Ukraine, Kyiv, Ukraine*

Hydroxyapatite (HA) – based materials are widely recognized as promising materials for bone tissue restoration. Numerous researches have been devoted to develop ideal biomaterials. Today HA and bioglass are the most attractive biomaterials. Implants made of HA can promote fast bone regeneration and direct bonding to the regenerated bone (without intermediate connective tissue). HA manufactured from bovine bone retains the chemical composition and pore structure of the bone mineral. Silicate-based glasses are also capable of enhancing new bone formation and bonding to the surrounding tissue. Development of HA/glass composites allows one to unite the

advantages of the individual components. In view of the importance of prognostication of biocomposite properties, and production of materials with required characteristics, it is very interesting to study the evolution of the structure and sintering parameters of the composite.

The aim of this study was to investigate the structure evolution and properties of the biogenic HA/glass composite, containing 50 wt% Na<sub>2</sub>O-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-based glass as a sintering aid.

The investigation was two-staged: (i) Study of the sintering and structure of biogenic HA/glass composite charge. (ii) Study of the sintering and structure of biogenic HA/glass composite samples prepared via two steps.

At stage (i) the crucible with biogenic HA/glass composite charge was put in a blast oven and heated in air up to 1000°C with a heating rate of 4-5deg/min. The oven construction included the asbestine cover plate with a “window” made of quartz glass, which was used for visual observation over thermal processing of charges. All visual changes were registered with a digital camera. Stage (ii) meant sample preparation via two steps, i.e. after sintering the material was uniaxially compacted in a stainless steel mold and sintered again at 800°C. Micro- and macrostructural observations of the sintered ceramics were conducted. The phase composition was determined as well. In addition, the volume shrinkage, density and porosity changes during the secondary sintering and the pyknometric density were measured.

It was established that the structure significantly changed in going from the charge to two-step sintered biogenic HA/glass composite. Herein, the pyknometric density increased from 2.49 to 2.73 g/cm<sup>3</sup>. The volume, density and porosity changes achieved 10 %. The borosilicate glass was able to facilitate the sintering process due to the viscous glass flow and to stabilize the HA phase against thermal decomposition.

C4

#### **SOURCES OF STRUCTURAL INSTABILITY OF ALUMINOSILICATE INORGANIC POLYMERS INVESTIGATED BY SOLID STATE NMR SPECTROSCOPY**

Libor Kobera, Jiří Brus, Martina Urbanová

*Institute of Macromolecular Chemistry, Academy of the Czech Republic,  
Prague, Czech Republic*

Layered aluminosilicate find applications in many professional human disciplines. Therefore, aluminosilicate materials take an important place in our world. Among others these materials are exploited in the preparation of zeolite (catalysts) and in the preparation of Alumino-silicate inorganic polymers (AIP`s) that exhibit large chemical and structural potential. AIP`s are usually prepared through reaction of alkali solutions and calcined aluminosilicate layered minerals at the room temperature and

atmospheric pressure. Ideally they should have non-crystalline structure, which keeps molecular water inside 3D-framework. However, during the time, these materials can undergo gradual transformation to zeolite structures at some circumstances. These transformations are accompanied by loss of water and mechanical properties. Stability of AIP’s is the main factor for keeping of mechanical properties. This is the reason why our current work is focused on the deep investigation of the role of water during these processes.

We prepared the stable and unstable systems of AIP’s and focused to changes into structure before- and after- hydrothermal treatment, between mentioned systems. For such task we used very powerful method – solid -state NMR. This is analytical method exploiting interaction of atomic nuclei (with nonzero nuclear spin) with magnetic field. The solid-state NMR spectroscopy with recent methodological developments became an inherent tool in the characterization of various materials. To gain structural information provided by solid-sate NMR we used not only simple one-dimensional experiments on various nuclei like  $^1\text{H}$ ,  $^{23}\text{Na}$ ,  $^{27}\text{Al}$ ,  $^{29}\text{Si}$  but we also used two-dimensional multiple-quantum experiments that were modified to indirectly detect water molecules that are closed in structural units. We tested large scale of NMR techniques involving also  $^1\text{H}$ - $^1\text{H}$  correlation experiments, cross-polarization transfer as well as REDOR experiments to locate and describe properties of clusters of water molecules.

C5

### MUTIFERROIC PROPERTIES OF $\text{BiFeO}_3$ –BASED CERAMICS

Felicia Prihor<sup>1</sup>, P. Postolache<sup>1</sup>, I. Dumitru<sup>1</sup>, L. Mitoseriu<sup>1</sup>, A. Ianculescu<sup>2</sup>

<sup>1</sup>*Department of Solid State and Theoretical Physics, Iasi, Romania*

<sup>2</sup>*Polytechnics University of Bucharest, Bucharest, Romania*

$\text{BiFeO}_3$  is one of the few single-phase multiferroics, showing antiferromagnetic and ferroelectric ordering. Since the dielectric properties in the ceramic state of the pure  $\text{BiFeO}_3$  were rather poor and in order to stabilize the perovskite state and to induce ferromagnetism at room temperature, it was adopted the strategy of doping with rare earth or forming solid solutions. Doping with small amounts of La demonstrated to improve the dielectric and ferroelectric properties and allowed to determine for the first time the piezoelectric characteristics in such compounds [1]. On the other hand, substituting Fe with Mn in  $\text{BiFeO}_3$ -based compounds is supposed cause better properties in terms of leakage current density and also to induce changes in the magnetic order of the system [2].

In the present paper, the effect of Mn substitution on the dielectric and magnetic properties of the  $\text{Bi}_{0.9}\text{La}_{0.1}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$  (BLFM) ceramics has been studied. Homogeneous samples from microstructural point of view were obtained for all the

compositions analyzed. The magnetic properties are strongly affected by the presence of Mn ions. The possible origin of these behaviour is discussed in terms of phase purity, grain size and grain boundary phenomena. The extrinsic properties are impossible to be fully controlled by normal ceramic processing. By controlling the extrinsic contributions to the dielectric properties, the ceramic system might be a valuable multiferroic material for magnetoelectric applications. At room temperature the ceramic is a multiferroic, since it is ferroelectric and magnetically-ordered.

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C6

### EFFECT OF PROCESS FLOWSHEET ON IMPROVING OF ADVANCED RESISTIVE CERAMIC COMPOSITES $\text{Si}_3\text{N}_4\text{-ZrC}$

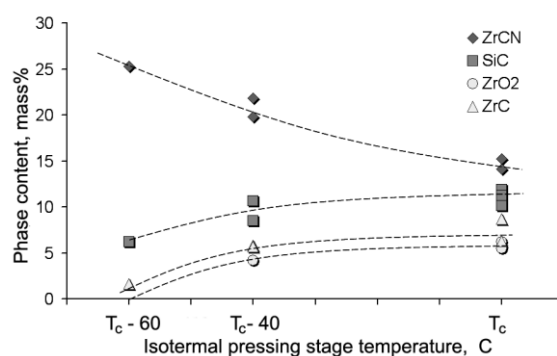
V. Ya. Petrovsky, Katerina V. Kyrylenko

*Fransevich Institute for Problems of Materials Science of NASU, Kiev, Ukraine*

The reliability of resistive ceramic composites has large importance at development of the electrothermal converters for high-intensity heat exchange. The performance of such composites depends on microstructure and phase composition evolution during sintering. This paper covers results of study focused on the influence of initial powders dispersion and concentrations in the blend as well as the effect of isothermal pressing stage temperature on and on phase composition, porosity and specific resistance of the hot-pressed resistive ceramics  $\text{Si}_3\text{N}_4\text{-ZrC}$  with MgO or  $\text{Al}_2\text{O}_3$  being sintering activators.

When employing fine or polydispersed conducting additive an agglomeration of small particles of powder in the course of mixing was observed; the agglomeration pattern depended on ZrC concentration. The inclusion of ZrC in hot-pressing resistive composites  $\text{Si}_3\text{N}_4\text{-ZrC}$  constituted both single particles and agglomerates thereof with the phase containing Si, Al, O and Zr located in between. The general pattern of microstructure evolution was unaffected by the type of activator.





During the sintering process new phases affecting electrophysical properties appeared in the composite namely: dielectric phase  $ZrO_2$ , semiconducting phase SiC. It was established that the  $ZrC_xN_{1-x}$  content is depended linearly on ZrC content in the composite, had only weak dependence on dispersion of components and adopted activating agent while it decreased with the rise of isothermal pressing stage temperature  $T_{is}$ . At the same time caused the increase in the content of SiC and  $ZrO_2$ . It results in deviation of concentration dependence of specific resistance of composite from that expected from the percolation theory.

At the isothermal pressing stage temperatures below certain critical temperature  $T_c$ , a reduction of specific resistance with growth of temperature, accompanied linear reduction porosity and losses of mass was observed. At the temperatures  $T_{is} > T_c$  specific resistance increases with the rise of isothermal pressing stage temperatures, while mass losses and porosity were decreasing. The dependence of specific resistance on conducting phase concentration showed a tendency to assuming the two-threshold shape. That was probably caused by agglomeration of both conducting and dielectric phases.

C7

### FABRICATION OF METAL AND SEMICONDUCTOR NANOPARTICLES IN BIOPOLYMER MATRICES

Dušan K. Božanić<sup>1</sup>, T. Radhakrishnan<sup>2</sup>, V. Djoković<sup>1</sup>

<sup>1</sup>*Institute of Nuclear Sciences “Vinča”, Belgrade, Serbia*

<sup>2</sup>*Department of Chemistry, University of Toronto at Mississauga, Mississauga,  
Ontario, Canada*

The preparation of polymer nanocomposites containing metal and semiconductor nano-crystals has evolved into a large research area due to characteristic electronic, optical and catalytic properties of these nanoparticles, often different from

those of the respective bulk phase. Since a polymer can be easily designed into almost any shape required by a particular application, these properties can be successfully utilized by incorporation of the nanoparticles into polymer matrices. Also, polymers can significantly influence the growth and spatial arrangement of the nanoparticles within the matrix and therefore are convenient templates for the preparation of nanoparticles of different morphologies. Because of possible applications in biotechnology and environmental protection, biopolymers and particularly polysaccharides were recently employed as host materials for metal and semiconductor nanoparticles. The macromolecular chains of these biopolymers possess a large number of hydroxyl groups and they can complex well with metal ions, which in turn enables a good control of size, shape and dispersion of the nanoparticles formed. Here we summarize the modes of preparation and present the results on the structure and the optical properties of metal (Ag, Au), semiconductor (CdSe, PbS, Fe<sub>2</sub>O<sub>3</sub>, Ag<sub>2</sub>S) and composite (Ag-Ag<sub>2</sub>S) nanoparticles synthesized in various polysaccharide matrices such as chitosan, glycogen, alginate and starch.

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C8

### SINTERING AND DIELECTRIC PROPERTIES OF BaTiO<sub>3</sub>/Ni NANOCOMPOSITE

Sergey Mazhuga<sup>1</sup>, Valentina Klymenko<sup>1</sup>, Anastasiya Kotlyarchuk<sup>1</sup>,  
Clive A. Randall<sup>2</sup>, Andrey Ragulya<sup>1</sup>

<sup>1</sup>*Institute for Problems of Materials Science NAS of Ukraine, Kyiv, Ukraine*

<sup>2</sup>*Center for Dielectric Studies, Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania*

In this work dense ceramic nanocomposite barium titanate – nickel with different nickel contents was obtained. The dielectric properties of this composite were investigated. Comparison with dielectric properties of green pellets and sintered pellets was made. Percolation threshold of sintered composite differs from green samples. Percolation threshold changes with densification of samples. High dielectric properties of dense ceramic are discussed.

C9

### MECHANICAL PROPERTIES OF $\text{Al}_2\text{O}_3$ -SiC NANOCOMPOSITES

Erika Csehová<sup>1</sup>, Ján Dusza<sup>1</sup>, Apichart Limpichaipanit<sup>2</sup>

<sup>1</sup>*Institute of Materials Research, Slovak Academy of Sciences, Košice, Slovak Republic*

<sup>2</sup>*Department of Materials, University of Oxford, United Kingdom*

The influence of the SiC addition to the monolithic alumina on the hardness, indentation fracture toughness and fracture of  $\text{Al}_2\text{O}_3 + \text{SiC}$  composites has been investigated. The Vickers hardness was measured at different applied loads and the fracture toughness was determined by „indentation fracture” method. The hardness of the composites was higher at all applied loads in comparison to the hardness of alumina, however, the differences at low loads were minimal. The fracture toughness is slightly increased with the SiC addition. The fracture mode changed from intergranular to transgranular by addition of SiC nanoparticles to the  $\text{Al}_2\text{O}_3$ .

**Keywords:**  $\text{Al}_2\text{O}_3$ -SiC, nanocomposites, microstructure, mechanical properties, toughness

C10

### FAST SYNTHESIS OF THE POLYCRYSTALLINE MATERIALS ON THE BASE OF $\text{Zn}_3\text{V}_2\text{MoO}_{11}$ AND $\text{Zn}_{2.5}\text{VMoO}_8$

Maya Markova-Velichkova, Reni Iordanova

*Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences,  
“Acad. G. Bonchev”, Sofia, Bulgaria*

In our study we applied two different techniques for the preparation of  $\text{Zn}_3\text{V}_2\text{MoO}_{11}$  and  $\text{Zn}_{2.5}\text{VMoO}_8$  polycrystalline materials - melt quenched method (up-down) and mechanochemical synthesis (down-up). These compounds belong to the family of materials with general formula  $\text{M}_{2.5}\text{VMoO}_8$  ( $\text{M} = \text{Zn}, \text{Mg}, \text{Mn}, \text{Co}$ ). They are potential candidates as catalysts in processes of selective oxidation of hydrocarbons. Until now, these two compounds were obtained by conventional solid state reaction. Using infrared spectroscopy and X-ray diffraction we are proven that the melt quenched technique is a very appropriate method for the synthesis of bulk  $\text{Zn}_3\text{V}_2\text{MoO}_{11}$  phase. Mechanochemical activation is more appropriated for the preparation of nonosized  $\text{Zn}_{2.5}\text{VMoO}_8$  powder. It was established that the melt quenched technique and mechanochemical activation are faster, in comparison with conventional ceramic methods for the synthesis.

C11

**IN VITRO BIOACTIVITY OF BIPHASIC CALCIUM PHOSPHATE  
SILICATE GLASS-CERAMIC IN CaO-SiO<sub>2</sub>-P<sub>2</sub>O<sub>5</sub> SYSTEM**

Lachezar Radev<sup>1</sup>, Vladimir Hristov<sup>1</sup>, Maria Helena V. Fernandes<sup>2</sup>,  
Isabel Miranda M. Salvado<sup>2</sup>

<sup>1</sup>*University of Chemical Technology and metallurgy, Sofia, Bulgaria*

<sup>2</sup>*University of Aveiro and CICECO, Aveiro, Portugal*

A novel calcium phosphate silicate glass-ceramic in CaO-SiO<sub>2</sub>-P<sub>2</sub>O<sub>5</sub> system have been synthesized via polystep sol-gel technique. The starting materials used for the synthesizing procedure were Ca(OH)<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub> and TEOS with Ca/P+Si (mol. ratio) equal to 2.3.

The phase formation and structure evolution of the synthesized and thermal treated at 1200°C for 2h sample was examined by using of XRD, 29Si MAS NMR, FTIR and SEM/EDS.

X-ray diffraction analysis proved the presence of Ca<sub>15</sub>(PO<sub>4</sub>)<sub>2</sub>(SiO<sub>4</sub>)<sub>6</sub> and Ca<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>(SiO<sub>4</sub>)<sub>6</sub>. SEM depicts the morphology of doped with silicon calcium phosphates. EDS depicted that Ca/P+Si ratio (at. %). According to 29Si MAS NMR it was founded that in the sample the chemical shifts of -72, -88 and -90 ppm, characteristic of Q<sup>0</sup> (=Si(O<sup>-</sup>)<sub>4</sub>) and Q<sup>2</sup> (=Si(OSiO<sub>3</sub>)<sub>2</sub>O<sup>-</sup>) units, respectively were presented. FTIR spectrum was representative for the silicon substituted hydroxyapatite structures.

In vitro bioactivity of the synthesized sample was evaluated by using of 1.5 SBF. Thermal treated sample was pressed at 50Mpa with PVA to disk (12x2mm) specimen and immersed in 1.5 SBF for different periods of time. FTIR spectra showed that the increases of the intensity of CO<sub>3</sub><sup>2-</sup> were associated with carbonate containing apatite formation on the surface of the prepared ceramic material. SEM images demonstrated that the apatite phase covered the entire surface after immersion in Kokubo solution.

C12

**EFFECT OF THE COOLING RATE AFTER HOT PRESSING ON  
ELECTRICAL CONDUCTIVITY OF  $\text{Si}_3\text{N}_4\text{-TiO}_2$  ( $\text{TiH}_2$ ) CERAMIC  
COMPOSITES**

Irina Cherniakova, Svitlana Zdolnik, Vitaly Petrovsky

*Fransevich Institute for Problems of Materials Science of NASU, Kiev, Ukraine*

During hot pressing (HP) of  $\text{Si}_3\text{N}_4\text{-TiO}_2$  ( $\text{TiH}_2$ ) ceramic composites different defective structures can be formed, such as: solid solutions of Ti and  $\beta\text{-Si}_3\text{N}_4$ , thin films of amorphous Si,  $\text{SiO}_2$  (or  $\text{Si}_2\text{ON}_2$ ) on the crystallite grain boundaries. However the influence of cooling rate ( $V_c$ ) on the crystallization and amorphization processes is not established. The aim of this work is to study electrical response of  $\text{Si}_3\text{N}_4\text{-TiO}_2$  ( $\text{TiH}_2$ ) ceramics to the structure defects formation during HP with different cooling rate.

Investigated samples were obtained by hot pressing with the variation of  $V_c$  in the range of 10-90 °K/min. The DC conductivity was measured in two directions - along and across the direction of HP in the range of temperatures 20-600°C. Electrical parameters was measured during heating and cooling of the samples with the rate of  $4 \pm 0,5$  K/min.

It has been established that  $V_c = 40$  K/min is the critical rate for the crystallization-amorphization processes.

The best combination of properties possess composites with mono-trap defective level  $0,8 \pm 0,05$  eV which is supposedly pertinent to the thin layer of amorphous Si. It is obtained in  $\text{TiH}_2$  additives presence with  $V_c > 50$  K/min and in  $\text{TiO}_2$  additives presence with  $V_c = 30$  K/min. The most defective structures are obtained in  $\text{TiH}_2$  additives presence with  $V_c = 30$  K/min, and with the  $\text{TiO}_2$  additive and  $V_c = 20$  K/min. Structures with the high density of defect states at the depth 0,4 - 0,7 eV from the bottom of the conduction band are formed with the low technological cooling rate ( $V_c = 10\text{-}20$  K/min). However, the influence of the additives type is observed with the high  $V_c$ .

Thus cooling rate after hot pressing governs the processes of electrical conductivity and charge storage in  $\text{Si}_3\text{N}_4\text{-TiO}_2$  ( $\text{TiH}_2$ ) ceramic composites which are perspective as substrates for the large capacity micro assemblies by Flip-Chip technology. Investigation of the temperature behavior of DC conductivity can be used as the method of non-destructive study of the grain-boundary composition of the advanced ceramic composites.

C13

## SHF RESPONSE ON BOTH PARTICLES SIZE AND STOICHIOMETRY OF METALLIC REFRACTORY COMPOUNDS

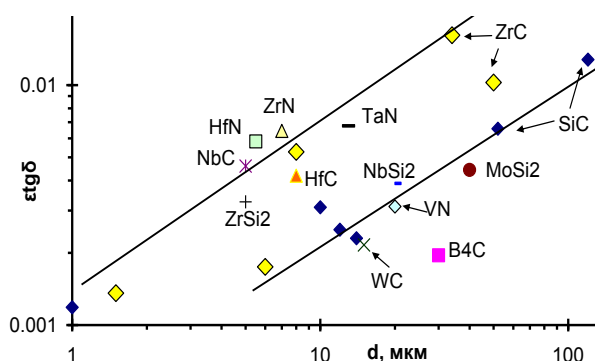
Mykhailo Soltys, Andrii Shpylko, Vitaly Petrovsky

*Frantsevich Institute for Problems of Materials Science of NASU, Kiev, Ukraine*

High efficiency of interaction of electromagnetic waves with substance, absence of near-electrode space charge, makes it possible to use microwaves for not destroying control of foodstuff quality; making of the modern devices, based on periodic multilayer absorbing structures; household and industrial technological processes.

The object of paper is a research of microwave absorption efficiency by powders of metallic refractory compounds depending on the size, morphology, stoichiometry of particles and development of not destroying quality control method of feed stock for bodies of indirect heating.

In the course of paper the phase structure, the particles morphology and grain structure have been tested with the standard methods. Powders absorbing capacity  $p \approx E^2 \cdot \omega \cdot \varepsilon' \cdot \varepsilon_0 \cdot \operatorname{tg} \delta$  was tested by the developed method in two ways: 1) in high fields – heat-metric method; 2) in law fields – Q value change of the short-circuited resonator on frequency 9,53 GHz). The factor of electromagnetic waves absorption  $\alpha$  is tested by permittivity  $\varepsilon'$ , dielectric loss  $\varepsilon''$ , and conductivity  $\sigma$ .



By absorbing characteristics the analyzed powders have been divided in two groups: strong-absorbing (NbC, HfN, ZrN, ZrC, ZrSi<sub>2</sub>, TaN, HfC) and low-absorbing (SiC, NbSi<sub>2</sub>, WC, B<sub>4</sub>C, VN, MoSi<sub>2</sub>, Pb).

It is ascertained that  $\varepsilon'$  is defined by compound electronic structure and does not depend on the size of particles. The maximum value  $\varepsilon'$  is observed in p-type semiconductor materials with a low whole mobility and a high negative value EMS. The loss factor  $\varepsilon'' = \varepsilon' \cdot \operatorname{tg} \delta$  depends on the size of grain by power law, and the morphology doesn't have an influence on absorbing properties of powders compounds. One can mark

out two groups of materials: 1) n-type paramagnetics (high absorption efficiency); 2) p-type diamagnetics (low absorption efficiency). Fine-dispersed powders absorb equally badly because of the big contribution of a surface.

By violation of powder stoichiometry the decrease of microwave absorption efficiency is observed, it could be connected with vacancies formation by non-metal and increase of permittivity.

C14

#### **SYNTHESIS OF TiN-TiB<sub>2</sub> CERAMIC COMPOSITE BY SPARK PLASMA SINTERING METHOD**

Igor Khobta<sup>1</sup>, Aleksandr Petukhov<sup>1</sup>, Andrey Ragulya<sup>1</sup>, Aleksandr Derev'yanko<sup>1</sup>,  
Y. Sakka<sup>2</sup>

<sup>1</sup>*Frantsevich Institute for Problems of Materials Science of NASU, Kiev, Ukraine*

<sup>2</sup>*National Institute of Materials Science, Tsukuba, Japan*

At the present work the research of synthesis processes of ceramic composite material (CCM) on the basis of TiN – TiB<sub>2</sub> as the material for cutting tools by processing of the powder mixture TiH<sub>x</sub> – BN under spark plasma sintering method was carried out. TiN – TiB<sub>2</sub> composites (practically clean) with relative density 64-99 % were synthesized in graphite die under direct passing of superposition of direct electric current and alternating one (SPS machine ERAN 2/1, IPMS of Ukraine) and pulsating direct current (1050 SPS machine, Sumitomo, NIMS of Japan). At the synthesized composite the phase proportion of TiN:TiB<sub>2</sub> was changed in the wide range (from 20% to 80% each phases) by means to addition of active boron or inert titanium nitride in initial powder mixtures. The microstructure, physical and mechanical properties of synthesized composites was investigated. Application of tetragonal titanium hydride as starting material is more preferably in comparison with metallic titanium. The samples have higher properties with TiB<sub>2</sub> content 36-60 wt. %. The composite with TiB<sub>2</sub> content 60 wt.% and properties: hardness HV = 25,9 GPa, fracture toughness K<sub>1C</sub> = 5,8 MPa×m<sup>1/2</sup> was obtained.

C15

**THE PREPARATION AND SINTERING OF SILICON NITRIDE  
POWDERS COATED WITH NANO-SIZED ZrN**

Aljoša Maglica, Kristoffer Krnel, Tomaž Kosmač

*“Jožef Stefan” Institute, Ljubljana, Slovenia*

In past decades much work has been done on the production of electroconductive composites based on  $\text{Si}_3\text{N}_4$  and SiAlON materials with incorporated electrically conductive particles such as  $\text{MoSi}_2$  or WC. Since these conductive particles are usually coarse, it is necessary to add a significant amount to achieve the percolation threshold. Composites with a higher amount of secondary phase are, however, often porous, have lower densities and worse mechanical properties. In order to preserve the good mechanical properties of the matrix phase much attention was devoted to reducing the amount of conductive phase in the composite.

In this work the possibility of preparing  $\text{Si}_3\text{N}_4$ -based materials with a lower amount of electrically conductive nano-sized ZrN particles will be discussed. The silicon nitride particles coated with ZrN nano-particles were prepared by coating the surface of the mixture of  $\alpha$  and  $\beta$ - $\text{Si}_3\text{N}_4$  particles with  $\text{ZrO}_2$ , using a gel-precipitation technique from an aqueous zirconium (IV) acetate solution containing  $\alpha$  and  $\beta$ - $\text{Si}_3\text{N}_4$  particles by the addition of ammonium hydroxide, which was followed by nitridation and/or sintering. The efficiency of the coating was examined by TEM, electron diffraction and EDXS analysis. During nitridation and/or sintering above  $1600^\circ\text{C}$  in nitrogen the  $\text{ZrO}_2$  reacted with  $\text{Si}_3\text{N}_4$  to form ZrN. In the presence of oxide sintering additives, the  $\text{Si}_3\text{N}_4/\text{ZrN}$  composites were prepared at atmospheric pressure in nitrogen. The microstructures of the sintered samples were investigated using XRD and SEM and the mechanical properties and electrical conductivity of the composites were measured. It will be shown that composites with attractive mechanical and electrical properties can be prepared by the sintering of  $\text{ZrO}_2$  or ZrN-coated  $\text{Si}_3\text{N}_4$  powder and choosing the right amount and composition of sintering additives.



C16

### CHARACTERIZATION OF RARE-EARTH DOPED $\text{Si}_3\text{N}_4/\text{SiC}$ MICRO/NANO-COMPOSITES

Peter Tatarko<sup>1</sup>, Štefánia Lojanová<sup>2</sup>, Ján Dusza<sup>1</sup>, Pavol Šajgalík<sup>2</sup>

<sup>1</sup>*Institute of Materials Research, Slovak Academy of Sciences, Kosice, Slovak Republic*

<sup>2</sup>*Institute of Inorganic Chemistry, Slovak Academy of Sciences, Bratislava,  
Slovak Republic*

Influence of various rare-earth oxide additives ( $\text{La}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{Yb}_2\text{O}_3$  and  $\text{Lu}_2\text{O}_3$ ) on the fracture toughness of hot-pressed silicon nitride and silicon nitride/silicon carbide micro/nano-composites have been investigated. The SiC particles in the composite materials were produced by carbothermal reduction of  $\text{SiO}_2$  (“in situ”) during the sintering process. The bimodal character of microstructures was observed in all studied materials where elongated  $\beta\text{-Si}_3\text{N}_4$  grains were embedded in the matrix of much finer  $\text{Si}_3\text{N}_4$  grains. The fracture toughness values increased with decreasing ionic radius of rare-earth elements either in monolithic  $\text{Si}_3\text{N}_4$  or  $\text{Si}_3\text{N}_4/\text{SiC}$  micro/nano-composites. The good relationship between fracture toughness values and aspect ratio of  $\beta\text{-Si}_3\text{N}_4$  grains was observed in both kinds of material. Materials with higher aspect ratio of the  $\text{Si}_3\text{N}_4$  grains (Lu or Yb additives) exhibited crack deflection more frequently compared to the materials exhibiting lower aspect ratio of  $\beta\text{-Si}_3\text{N}_4$  grains, which was responsible for the higher fracture toughness. It was observed that microstructures with the finest and coarsest elongated  $\beta$ -grains had the maximum fracture toughness, whereas the microstructures with the mixture of elongated thin and thick  $\beta$ -grains had lower fracture toughness. The fracture toughness of composites was always lower than monoliths due to the finer  $\text{Si}_3\text{N}_4/\text{SiC}$  microstructures and due to less toughening mechanisms during the crack propagation in this type of material. It means that a beneficial effect of SiC particles on the fracture toughness resulting from the residual stresses around the particles was not observed in the present work. Similarly, the hardness and bending strength values increased with decreasing ionic radius of rare-earth elements either in monoliths or composites. The hardness was higher in composite materials due to their finer microstructures. On the other hand, the positive influence of finer microstructure of the composites on strength was not observed due to the present defects in the form of SiC clusters and non-reacted carbon areas whose negative effect was more significant than the influence of finer microstructure. Significantly improved creep and oxidation resistance was observed in case either of composite materials or materials with smaller ionic radius of  $\text{RE}^{3+}$ .

C17

**WEAR-RESISTANT MULTILAYER CERAMIC COATINGS TiN/ZrN  
AND TiN/TiCrN FOR DRILLING APPLICATION OBTAINED BY  
VACUUM-ARC DEPOSITION**

Oleg A. Tokariev, A. V. Demchyshyn, G. A. Avtonomov

*Frantsevich Inst. for Problems of Materials Science of NASU, Kyiv, Ukraine*

The main purpose of this science work is obtaining and adoption for industrial application of multilayer ion-plasma ceramic coatings TiN/ZrN and TiN/TiCrN. The work is based on physical-mechanical properties of coatings which had been determined before and the dry drilling test data was added to it. Multilayer coatings were deposited on carbide drills (diameter 1,483 mm) and we conducted an experiments using dry drilling onto austenitic stainless steel plate (thickness 5mm).

The results have shown that TiN (H = 23GPa) coated drill’s durability 250% more than uncoated one. In its turn TiN/ZrN (H = 45GPa) coated drills have durability in 30% higher than TiN coated drills, and TiN/TiCrN (H = 40GPa) in 45% higher respectively.

C18

**DIFFERENT APPROACHES TO FABRICATION OF BULK  
BIOMATERIALS BASED ON CALCIUM PHOSPHATES**

Dmitry Larionov, Valery Putlyaev, Alexander Kuznetsov, Alexander Veresov

*Department of Materials Science, Moscow State University, Moscow, Russia*

The extensive progress of medicine during last years requires the emergence and development of new interdisciplinary approaches capable to solve different biomedical problems. It is widely accepted that biomaterials for replacement of damaged bone are one of the key directions in both medicine and materials science. Bone tissue is a composite material made up of organic and inorganic components, where the last one consists of calcium phosphates. According to this, bone implants should be similar to bone in structure and chemical composition. Meeting this demand can be done in different ways but requires a good knowledge in the areas of chemistry, biomechanics and biology.

The aim of current work is to elaborate methods which combine chemical, mechanical and biological approaches to preparing biomaterials. Two different techniques to obtain nanostructured composites were pursued:

1) The use of  $\alpha$  – TCP (alpha-tricalcium phosphate) with low – temperature technique of chemical bonding.  $\alpha$  – TCP can be used as an individual component in cement compounds in order to obtain hydroxyapatite with the reaction of hydrolysis. It is known that  $\alpha$  – TCP is a metastable phase that’s why we faced with the problem of

chemical stabilization. We tried to solve this problem by modifying its chemical composition via doping with  $\text{SiO}_4^{4-}$  anion; the last one has also significant biological impact. The development of the technique of compaction of  $\alpha$  – TCP paste according to our issue allowed us to increase compressive strength of the materials under study by means of porosity reduction up to the values close to that one of cortical bone (higher than 100 MPa).

2) Another method of preparing biomaterials using obtaining microporous plaster of Paris (gypsum) samples and metathesis reaction aimed at the replacement of  $\text{SO}_4^{2-}$  with  $\text{CO}_3^{2-}$  and  $\text{PO}_4^{3-}$  anions in order to prepare anion-modified calcium phosphates (or carbonate) based materials.

Thus obtained materials were characterized by XRD, SEM, and dissolution experiments, mechanical and in vitro tests. The work was supported by RFBR (grants #№07-08-00576, 09-03-01078) and Federal Program “R&D in priority directions of science and technology in Russia for the period of 2007-2012” (state contract #02.513.12.3008).

C19

### **MICROSTRUCTURE AND PROPERTIES OF ZIRCONIA/CARBON NANOFIBER COMPOSITES**

Viktor Puchý<sup>1</sup>, Ján Dusza<sup>1</sup>, Gurdial Blugan<sup>2</sup>, Jerzy Morgiel<sup>3</sup>, Jakob Kuebler<sup>2</sup>,  
Fawad Inam<sup>4</sup>, Ton Peijs<sup>4</sup>, Michael J. Reece<sup>4</sup>

<sup>1</sup>*Institute of Materials Research, Slovak Academy of Sciences, Košice, Slovak Republic*

<sup>2</sup>*Empa, Swiss Federal Laboratories for Materials Testing and Research, Laboratory for High Performance Ceramics, Duebendorf, Switzerland*

<sup>3</sup>*Institute of Metallurgy and Materials Science of Polish Academy of Sciences, Krakow, Poland*

<sup>4</sup>*Nanoforce Technology Ltd and Centre for Materials Research, Queen Mary University of London, London, UK*

Zirconia/carbon nanofiber composites were prepared by hot pressing and spark plasma sintering. The effect of the sintering route and the carbon nanofiber additions on the microstructure, fracture/mechanical and electrical properties of the CNF composites was investigated. The microstructure of the  $\text{ZrO}_2$  and  $\text{ZrO}_2$ -CNF composites consisted of a small grain sized matrix (approximately 120 nm), with relatively well dispersed carbon nanofibers in the composite. All of the composites showed significantly higher electrical conductivity compared to the monolithic  $\text{ZrO}_2$ . The spark plasma sintered composites exhibited higher densities, hardness and indentation toughness but lower electrical conductivity compared to the hot pressed composites. The improved electrical conductivity of the composites is caused by CNFs network and by thin disordered

graphite layers at the  $ZrO_2/ZrO_2$  boundaries. The tribological properties of selected systems have been investigated, too.

**Keywords:** carbon nanofiber, hot press, spark plasma sintering, microstructure, fracture, electrical conductivity

C20

## COMPOSITE MATERIALS DERIVED FROM GEOPOLYMERS REINFORCED BY POLYMER FIBERS

Lenka Benešová

*Institute of Macromolecular Chemistry, AS CR, v.v.i., Prague, Czech Republic*

Geopolymers (GP) are inorganic polymers based on aluminosilicate network prepared from calcinated kaolin or fly-ash. GP synthesis occurs by alkaline activation. It develops into two steps: The first step is the reordering of silica and alumina units by alkaline ions and the second step is polycondensation of these units into a three-dimensional network.

Geopolymers have been intensively studied as promising structural materials due to their high compressive strength.

The aim of this work is the enhancement of mechanical properties of GP by addition of short polypropylene (PP) fibers. Two different types of GPs were prepared, the first one was based on a calcinated kaoline (dehydroxylated kaolinite – “metakaolinite”) (GP – M) and the second one based on fly-ash (GP – F). Fibers were added in the amount of 5, 10 and 20 wt %.

Samples of composite materials were prepared by mixing in a laboratory mixer at ambient temperature. The specimens for compression testing were molded in cylindrical moulds (diameter 10 mm, height 30 mm), specimens for flexuring testing were molded in bar moulds (10 x 10 x 40 mm<sup>3</sup>). After ageing (7, 28 and 90 days, respectively), mechanical properties were measured by a universal testing machine Instron 6025R5800. The matrix of the specimens was investigated by X-ray diffraction (XRD) and NMR spectroscopy.

It was found that compression strength of GP – M and GP – F specimens without fibers increases with increasing ageing time. The compression strength of fiber-containing GPs is lower in comparison to the non-reinforced specimens. The addition of lower fiber content to the matrix (5, 10 %) causes a certain decrease in the compression strength followed by a marked increase at the fiber concentration of 20 wt %. In fact, the compression strength of GP with 20 wt % of PP fibers is comparable to the values of the non-reinforced GP. On the other hand, the addition of PP fibers causes a steady increase in flexural strength in the whole concentration range.

NMR spectroscopy ( $^{27}\text{Al}$  and  $^{29}\text{Si}$  MAS) supplied very useful information of the GP matrix. It was revealed that locally ordering networks (rings) formed. Additionally, XRD was employed to describe the mineral phases of the matrix. While GP – M were found to be X-ray amorphous, a small content of zeolite phase was detected in GP-F matrix. Nevertheless GP-F is also generally X-ray amorphous.

C21

### **SYNTHESIS AND CHARACTERIZATION OF SiC/CORDIERITE COMPOSITE MATERIALS**

Milica Pošarac<sup>1</sup>, Tatjana Volkov-Husović<sup>2</sup>, Marija Dimitrijević<sup>2</sup>,  
Branko Matović<sup>1</sup>

<sup>1</sup>*Institute of Nuclear Sciences “Vinca”, Belgrade, Serbia*

<sup>2</sup>*Faculty of Technology and Metallurgy, Belgrade, Serbia*

SiC/cordierite ceramic composites with weight percent ratio of 70:30 were prepared by a mechanical mixing of commercial SiC powder and cordierite obtained from two different precursors (commercially available spinel or Mg-exchanged zeolite A). Mixture of spinel, quartz ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) corresponding to a cordierite stoichiometry was attrition milled for 4 hours, using  $\text{Al}_2\text{O}_3$  balls and ethyl alcohol as media. Mixture of Mg-exchanged zeolite A, quartz ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) corresponding to a cordierite stoichiometry was attrition milled for 4 hours, using  $\text{Al}_2\text{O}_3$  balls and ethyl alcohol as media. Different content (10, 20 and 30 wt%) of graphite powder was used as the pore former to manage the porosity of the specimens. The mixtures were heated in air at different temperatures in order to obtain composites and to burn graphite out. Phase composition and microstructure was investigated with XRD and SEM. Pore size and pore distributions were determined by Image Pro Plus program for image analysis. Vickers hardness test was performed and influence of porosity on hardness of composite materials was investigated. Young's modulus of samples was calculated using measured values of ultrasonic velocities obtained by ultrasonic pulse velocity technique.

C22

## NEW CARBON MATERIALS FOR ELECTRODES FOR ELECTROCHEMICAL CAPACITORS

Piotr Próchniak

*Department of Power Engineering and Electrochemistry, Lublin University of  
Technology, Lublin, Poland*

Electrochemical capacitors (EDLC, supercapacitors) utilise capacity of electric double-layer at the electrode/electrolyte interface. Leading electrode materials for these devices are carbons obtained from natural and synthetic precursors. Carbon conducts electricity and is able to display very high specific area (up to 2000 m<sup>2</sup>/g) to form electric double layer. The present work describes a new type of carbon used as electrode. These templated mesoporous carbons with tailored pore size distribution are very promising material. Carbon monoliths were made by an inverse replica technique using silica as templates and polyfurfuryl alcohol as the carbon precursor. Silica matrixes were made by sol-gel method. After impregnation and polymerization of carbon precursor in silica matrix at 80°C, composites were carbonised at 850°C. Then silica was removed by HF treatment. Controlled synthesis of the starting silica templates together with adjusting time and temperature of carbonization enable to control the pore size of carbon bodies. The structure of the carbon was studied by low-temperature nitrogen adsorption and by thermal analysis. The carbon monoliths were used as electrodes in electrochemical capacitors with 30% H<sub>2</sub>SO<sub>4</sub> in water and 1M Et<sub>4</sub>NBF<sub>4</sub> in acetonitrile and propylene carbonate as the electrolytes. Electrochemical performances of capacitors were studied by cyclic voltammetry, galvanostatic charge–discharge measurements and impedance spectroscopy. The new carbon materials have high BET surface (up to 1300 m<sup>2</sup>/g) and very low resistivity (in a range of 10<sup>-3</sup> Ωm). The best shows capacity of 70 F/g with aqueous electrolyte.



**TRADITIONAL CERAMICS**

T1

### **CLAY ROOFING TILES – ABLE TO CLEAN ITSELF?**

Milica Hadnadjev, Radmila Marinković-Nedučín, Jonjaua Ranogajec

*Faculty of Technology, University of Novi Sad, Novi Sad, Serbia*

Heterogeneous photocatalysis phenomenon is applied in the degradation process of many organic contaminants on solid surfaces. Photocatalysis is based on the excitation of the semiconductor by irradiation with supraband gap photons and the migration of electron-hole pairs to the surface of photocatalysts leading to the reaction of the holes with adsorbed  $\text{H}_2\text{O}$  and  $\text{OH}^-$  to form hydroxyl radicals. Photocatalytic active semiconductor titanium dioxide ( $\text{TiO}_2$ ) has attracted a great deal of attention due to its potential for decomposition of organic compounds as well as UV-sensitive hydrophilic surface conversion agent.  $\text{TiO}_2$  is a leading photocatalyst in environmental decontamination for numerous organics, viruses, bacteria, fungi, algae and cancer cells that can completely degrade and mineralize mentioned species to  $\text{CO}_2$ ,  $\text{H}_2\text{O}$  and harmless inorganic anions. Due to the stability and photosensitivity of  $\text{TiO}_2$ , this semiconductor is of great interest from the ecological and industrial point of view, and therefore was used for this investigation in the field of building material. Besides various applications in air, water-and wastewater-treatment, heterogeneous photocatalytic oxidation is of great importance when it comes to self-cleaning materials.

Clay roofing tiles due to their long-term exploitation are subjected to physical, chemical and biological degradation that lead to deterioration. These ceramic systems have high percentage of total porosity and taking to consideration their non-tolerance for organic coating, the use of surface active materials (SAM) that induce the porosity in the  $\text{TiO}_2$  coatings is of great significance. Photocatalytic coatings were applied onto clay roofing tiles in industrial conditions and designed by the variation of the  $\text{TiO}_2$  quantity ( $\text{mass}/\text{cm}^2$ ) on the tile surface (thin and thick  $\text{TiO}_2$  layer). Despite numerous investigations concerning  $\text{TiO}_2$  coating application, there are only few scientific studies that deal with the complexity related to the manufacturing of clay roofing tiles with  $\text{TiO}_2$  active layer.

The aim of this study is to investigate the connection between UV-activated  $\text{TiO}_2$  layers applied onto clay roofing tiles and their photocatalytic characteristics in comparison to their hydrophilicity and photocatalytic activity after irradiation leading to self-cleaning properties.



T2

**EFFECT OF LEAD OXIDE CONTENT ON THE PHYSICO-CHEMICAL  
PROPERTIES OF TRANSPARENT CERAMICS**

Oana Catalina Mocioiu, Irina Atkinson, Maria Zaharescu  
*Ilie Murgulescu Institute of Physical Chemistry of Romanian Academy,  
Bucharest, Romania*

The properties of lead oxide glasses are interesting for optical and optoelectronic devices. In glasses the lead oxide can be a former or a modifier of the network. The influence of lead content on the physical, optical and chemical properties of glasses in different oxide systems was established in this work.

In order to investigate the material structure, the infrared spectroscopy was used for some systems. The results show that the glass structure is formed mainly from pairs and chains of SiO<sub>4</sub> tetrahedra and that the amount of PbO changed the network ordering. The optical properties were determined by UV-VIS spectroscopy. The physical properties were calculated and some of them were experimentally verified. The behavior of this materials in different alkali, acid and aqueous media was studied.

The obtained data were correlated in order to understanding the influence of lead oxide content on the properties of the final material as well as the behavior of lead in glasses of different compositions.

T3

**MECHANICAL PROPERTIES AND FRACTURE MODE OF POLYMER  
ADDED TRADITIONAL CERAMICS**

Dragan Rajnović<sup>1</sup>, Sebastian Balos<sup>1</sup>, Leposava Šidjanin<sup>1</sup>, Jonjaua Ranogajec<sup>2</sup>

<sup>1</sup>*Faculty of technical Sciences, University of Novi Sad, Novi Sad, Serbia*

<sup>2</sup>*Faculty of Technology, University of Novi Sad, Novi Sad, Serbia*

The object of this paper was to characterize the fracture mode and mechanical properties of traditional ceramics mixed with small amount of polymer. The purpose of polymer addition was to achieve submicron porosity of dry swept grinded raw materials in order to obtain better water absorption. It was expected that enhanced water absorption will improve the production process of traditional clay products reducing the needed time for re-wetting of the raw material. The increase of the plasticity of clay material will be obtained as well as mechanical (flexural strength, hardness), frost and bio-corrosion resistance and self cleaning properties.

The systems studied were the mixtures of dry swept grinded domestic clays and polymer materials. Three materials model systems were produced in green and fired

condition: 0 - control system without polymer addition; 1 - system with 5% mass addition of PEG (polyethylene glycol) and 2 - system with 1% mass addition of PEG. The samples were extruded, air dried and finally fired at 960°C for 1 hour. Two different firing procedures were used: A - heat rate of 400°C/h without intermediate holding times, and B - heat rate of 400°C/h with holding at 150 and 300°C for 1 hour. Three point bend test was carried out to obtain flexural strength. The fracture surfaces were examined visually and by means of SEM (JEOL JSM 6460LV) operating at 20 kV.

It was found that flexural strength of green samples was in the range from 8.2 to 9.1 MPa for the systems 0 to 2, respectively. On the other hand, flexural strength of fired samples was at the level of 30 MPa with a slight increase in the case of polymer added samples compared to the system without polymer addition. Flexural strength and fracture mode were not significantly influenced by different firing procedures. The microscopic examination of green systems showed that a significant level of microcracks was present in all samples, with the most pronounced cracks present in the system 1 (5% of PEG). In the fired samples a slight increase of cracks compared to green was noticed. Furthermore, the highest amount of cracks was present in the system 1 (5% of PEG), followed by the system 2 (1% of PEG), while the system 0 (no polymer added) had the lowest amount of cracks. However, the flexural strength of the polymer added samples remained at the same level, or was slightly increased, in spite of the larger amount of microcracks.

T4

#### **EFFECTS OF FIRING ATMOSPHERE CHARACTERISTICS ON THE PROPERTIES OF CLAY ROOFING TILES**

Robert Rekecki, Jonjaua Ranogajec

*Faculty of Technology, University of Novi Sad, Serbia*

The effects of firing atmosphere on physical properties and microstructural characteristics of clay roofing tiles were studied. The research included: dilatometric investigation of the firing behavior of Kanjiza clay during thermal treatment in oxidizing and reducing atmosphere and the firing of laboratory and industrial samples in both atmospheres, as well as their characterisation. It is concluded that the obtained benefits in the reducing atmosphere (lower porosity, higher frost resistance, better rain tightness) were the functions of the compaction process parameters during the firing process. The microstructure of the samples emphasized higher glassy phase and secondary crystalline phase content in comparison with the ones produced in oxidizing atmosphere.

**Keywords:** clay roofing tiles, oxidizing and reducing atmosphere, microstructure, glassy phase, porosity, mechanical characteristics

T5

**THE EFFECT OF ZIRCON AND ZINC OXIDE REDUCTION ON  
PRODUCTION STAGES, FINAL PROPERTIES AND  
MICROSTRUCTURE OF FAST FIRED GLASS-CERAMIC  
WALL TILE GLAZES**

Mine Taykurt Daday<sup>1,2</sup>, Emrah Dolekcekic<sup>1</sup>, Figen Aydin Egri<sup>2</sup>

<sup>1</sup>*Anadolu University, Department of Materials Science and Engineering,  
Eskisehir, Turkey*

<sup>2</sup>*Toprak Ceramic Company, Eskisehir Tile Plant, Eskisehir, Turkey*

Opaque, glossy white wall tile glazes have been produced by using frit-based glazes that contain high amount of zirconium and zinc oxide. The aim of this study is to reduce the amount of these two expensive components by producing alternative frit compositions. The possibility to define the amount and ratio of the oxides K<sub>2</sub>O-MgO-CaO-ZnO-B<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> the paper studied. Specific investigation was dedicated to the opacity missing and to the surface defects. These phenomena were related with the reduction of zircon and zinc oxide, respectively. The effect of replacement of CaO, MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> mixture by ZrO<sub>2</sub> in order to obtain alternative opacifying phases was studied as well as the replacement of Na<sub>2</sub>O, K<sub>2</sub>O and B<sub>2</sub>O<sub>3</sub> by ZnO in order to organize high temperature viscosity of the glaze. A commercially used frit was used in both cases. This conventionally prepared frit-based glaze slurry was applied on pre-engobed wall tile bodies. Results of the industrially fired specimens pointed out promising surface properties. The developed crystalline phases in the glazed surfaces were identified by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) techniques. Sintering behavior of the glazes, colour and gloss analyses were made with a hot stage microscopy, spectrophotometer and glossmetre, respectively. Decreasing zircon and zinc oxide from frits and consequently, from glazes give a chance to produce approximately 27-30 % cheaper products with intensified opacity and glaze properties.

**Keywords:** zircon, zinc oxide, frit, opacity, wall tile

T6

### THE INFLUENCE OF MECHANICAL ACTIVATION ON DRYING PROCESS

Miloš Vasić, Zagorka Radojević

*“Institute for Testing Materials”, Belgrade, Serbia*

The results of parallel scientific research about behavior of masonry clay prepared by classical procedure and mechanically activated clay have been presented in this paper. One representative sample of masonry clay from the locality Banatski Karlovac has been prepared according to classical procedure by wet grinding to the size granulation below 1 mm. The other representative sample, from the same locality was mechanically activated for 30 minutes in Fritch mill. Prepared samples characterization has been done by using X-ray, thermal characterization (DTA and TGA, DIL), granulometric test and by using some technological characterization. The obtained results show significant differences in structure, morphology and final properties of the obtained samples. The method of multi-factorial experimental design has been used for determination of the samples properties after the drying process. The mathematical relationship as a function of temperature, humidity and the velocity of drying medium was set up. The influence of the same factors on drying time duration in order to reach the critical point was also analyzed. Obtained outputs are very significant for the optimization of masonry clay product drying process and for establishing the fast drying regimes of the same products.

T7

### BIOLOGICAL EFFICIENCY OF PHOTOCATALYTIC TiO<sub>2</sub> COATINGS ON CLAY ROOFING TILES SUBSTRATE

Dmitar Zorić<sup>1</sup>, S. Markov<sup>1</sup>, M. Radeka<sup>2</sup>, J. Ranogajec<sup>1</sup>

<sup>1</sup>*Faculty of Technology, University of Novi Sad, Serbia*

<sup>2</sup>*Faculty of Technical sciences, University of Novi Sad, Serbia*

Photocatalytic titanium dioxide coatings on clay roofing tiles substrate have been extensively studied for possible antimicrobial application. The wide acceptance of photocatalytic TiO<sub>2</sub> to be antimicrobial efficient has led to attempts of different coating processes. This research involves a spray technique as the method for applying two different TiO<sub>2</sub> coatings: mesoporous TiO<sub>2</sub> coating based on PEG-UV light sensitive system and TiO<sub>2</sub> coating (based on commercial suspension) sensitive to visible light radiation. Photocatalytic performances of the deposited specimens are tested with model pollutants - methylene blue solution (UV light sensitive system) and rhodamin B (visible

light sensitive system). Antimicrobial efficiency is evaluated through the deposition of *Aspergillus niger* (fungus) and suspension of cells *Pseudomonas aeruginosa* (bacteria). The antimicrobial efficiency of the TiO<sub>2</sub> coatings was determined by counting fungus colonies that grew on the photocatalytic surfaces. The colony number (CFU) was obtained by using a light microscope and pour plate method. The results of antimicrobial efficiency were correlated with photocatalytic activity, roughness and water activity of photocatalytic titanium dioxide coatings.

T8

### **APPLICATION OF CERAMIC IN MODERN ART/FINE ART**

Teslim Kolawole Orunsolu, Olayemi John Akeredolu

*University of Ilorin, Dugbe Ibadan, Nigeria*

Advanced ceramics and processes have found potential applications in many fields ranging from heat engines to communication from coating and it is used in automotive industries and energy transmission. In this paper, the evolution of ceramic technology is introduced, and an Advanced Ceramics Application branch is developed to illustrate current and future potential application areas.

During the event I will also shed light on area of application of ceramic on the following area that are yet to be used by experts in the industry.

Also, I will also share with prospective delegates what the use of ceramics is like in Nigeria decorative industries but more emphasis will be on industrial use of ceramics, such as:

1. Bearing, seals and Valves.
2. Technology of application of ceramic coatings onto the elements made of aluminum and zircon sand and their alloys.
3. Artworks.





**CULTURE HERITAGE**

H1

## WEATHERING PROCESS OF THE EIGHTEENTH CENTURY WALL PAINTINGS IN THE CHURCH OF BODJANI MONASTERY

Snežana Petrović<sup>1</sup>, Jonjaua Ranogajec<sup>1</sup>, Miroslava Radeka<sup>2</sup>, Slavica Vujović<sup>3</sup>,  
Jasna Gulan<sup>3</sup>

<sup>1</sup>*Faculty of Technology, University of Novi Sad, Novi Sad, Serbia*

<sup>2</sup>*FTN Department for Civil Engineering, Novi Sad, Serbia*

<sup>3</sup>*The Provincial Institute for the Protection of Cultural Monuments of the Autonomous of  
Vojvodina, Petrovaradin, Serbia*

Weathering is a group of processes that leads to decay or degradation of materials upon their exposure to the surrounding environmental conditions. The most dominant factors in the environmental damage of the buildings are water and soluble salts. The object of our investigation was the examination of the historical materials collected from the church of Bodjani Monastery, North of Serbia – 18<sup>th</sup> centuries, where high level of humidity, above 90%, and salt crystallization, are the dominant degradation parameters. The geographic location along the Danube river contributes to the weathering process due to the fact that underground water possesses high sulphate content which in turn reacts with the wall elements components.

Experimental research was carried out in order to investigate the existing connection between raw materials composition and their decomposition based on water absorption – moisture action. On the base of the used methods (X-ray diffraction, pH – value measurements, SEM-EDS, DTA/TG and Hg-porosimetry) and historical data of the church, the degree of structure damage of the examined materials was determined. The mineralogical composition of the raw materials, the firing temperature, the environments to which they had been exposed, the action of humidity and of the microorganisms as well as the reigning environmental conditions were the basic parameters of the weathering process of the examined materials. Our work presents a contribution to a better understanding of the salt crystallization mechanisms (transition between thenardite and mirabilite and thenardite reprecipitation) and durability decrease of the porous ancient bricks, the construction material of the church of Bodjani Monastery. This approach could be one of the ways for defining the appropriate restoration treatments for the church of the Bodjani Monastery.

**Keywords:** cultural heritage, historical bricks, salt crystallization, moisture



H2

## POTENTIAL RESTORATION STONE ASSESSMENT FOR STRASBOURG'S CATHEDRAL

Estel Colas<sup>1,2</sup>, Jean-Didier Mertz<sup>1</sup>, Celine Thomachot<sup>2</sup>, Vincent Barbin<sup>2</sup>

<sup>1</sup>*Laboratoire de Recherche des Monuments Historiques, Champs/Marne, France*

<sup>2</sup>*Groupe d'Etude sur les Géomatériaux et Environnements Naturels, Anthropiques et  
Archéologiques, Université Reims Champagne-Ardenne, Reims, France*

Strasbourg's cathedral is an important historical, architectural and religious monument in Alsace, East of France. The rocks used for the building construction are Vosgien and Meules quartzic sandstones from Middle and Upper Buntsandstein formations (GROSS, 1978; JEANNETTE, 1981, 1997; THOMACHOT, 2002). Nevertheless, Meules sandstone is the major sandstone type used because of the fine-grained particle allowing high quality of sculpture. Limited volume of exploitable rock materials and mineralogical composition variability, such as clay minerals and iron oxides/hydroxides, from one quarry to another one, lead to constant prospection of new compatible and durable sandstone for restoration works.

Exposed Meules sandstones on the monument present different alteration rate which are more reliable to petrophysical properties than environmental and weathering conditions.

The present study is based on five sandstone types from French and German quarries and two exposed stones from the monument. The first one dating from the XIII<sup>th</sup> century is less decayed than the second one dating from the beginning of the XX<sup>th</sup> century. The study of relevant parameters related to these two exposed stones on the monument highlights the best parameters for durability and an appropriate selection from the five potential stones for restoration works.

Meules and Vosgien sandstones are generally composed of more than 80% of quartz, K-feldspar, micas and various clays and oxides associated in a coating matrix.

Mineralogical, structural and hydrodynamical properties are assessed.

This work focuses on clay fraction properties, quantity and reactivity, compared with structural parameters and pore size distributions in order to reach significant parameters of the macroscopical behaviour i.e. swelling/shrinkage under controlled hydric stresses.

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H3

### **EIGHTEENTH CENTURY WALL PAINTINGS OF THE CHURCH OF BODJANI MONASTERY**

Jasna Gulan<sup>1</sup>, Siniša Zeković<sup>1</sup>, Slavica Vujović<sup>1</sup>, Jonjaua Ranogajec<sup>2</sup>,  
Snežana Petrović<sup>2</sup>

<sup>1</sup>*The Provincial Institute for the Protection of Cultural Monuments of the Autonomous  
Province of Vojvodina, Petrovaradin, Serbia*

<sup>2</sup>*Faculty of Technology, University of Novi Sad, Serbia*

Fresco paintings in the church of Bođani Monastery stand as one of the most significant painting complexes in Serbia from the first half of the 18th century. Valuable baroque paintings are in jeopardy due to capillary moisture, atmospheric and condensation humidity. Over the last six decades different conservation interventions have been carried out with the goal of stopping the capillary moisture from entering the walls. For that purpose, a number of procedures were undertaken: air isolation of the walls in 1954, electro-osmosis in 1974, draining procedure in 2000, vapour-permeable mortar in 2001. Unfortunately, none of the interventions has given complete and permanent results.

During 2007 and 2008, as a part of the Development project of integrative conservation of the cultural heritage of Bač: the Provincial Institute for the Preservation of Cultural Monuments, Faculty of Technology in Novi Sad, the Gallery of Matica Srpska, with the partners from Italy have conducted systematic and multidisciplinary researches of the fresco paintings and the architecture of the church of Bođani Monastery. The goal of the researches was to determine and document the state of the church and fresco painting, as well as to conduct the monitoring of the microclimate in the church. The investigation was done in order to form an idea for the future interventions.

For successful restoration of painted walls a number of physical and chemical analyses were done with varying methods. During the research, special attention was paid to the analysis of the palette of Hristofor Žerafović. The following methods were employed in characterization of the materials: X-ray fluorescent spectroscopy (XRF), Raman spectroscopy (RS), Optical microscopy and microscopic analysis of the cross section (OM and CS), scanning electronic microscopy (SEM) and Energy dispersion

spectrophotometry (EDS). Based on the methods that were used, the pigment palette, techniques and technologies of fresco painting of Bođani monastery were determined.

**Keywords:** pigment palette, technique and technologies of fresco painting

H4

**THE IMPORTANCE OF MATERIAL CHARACTERISATION IN THE  
RESTORATION OF POLYCHROME SURFACES CASE STUDY: 18<sup>th</sup>  
CENTURY SERBIAN BAROQUE ART**

Darko Despotović, Daniela Korolija  
*Gallery of the Matica srpska, Novi Sad, Serbia*

The period of 18 century was the period of great changes in Serbian art. Based on byzantine tradition, in the region of Vojvodina it came into contact with European baroque art, and in few decades new influences could have been very well observed on the paintings and other polychrome objects. Later period brought multiple reconstructions, overpaintings and restorations of these rich polychromes. Together with the effect of weathering of original artist materials, the original appearance of many items from the 18th century is not visible today.

In order to provide more insight into their original appearance the technical examination results were combined with art historical and documentary sources. This presentation will bring few examples of such approach and the final results of conservation work.

**Keywords:** weathering of artist materials, conservation, restoration



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