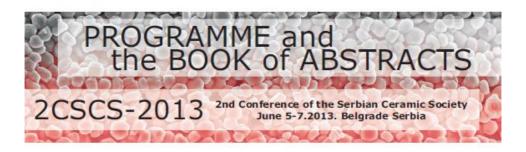
The Serbian Ceramic Society
The Academy of Engineering Sciences of Serbia
Institute for Multidisciplinary Research - University of Belgrade
Institute of Physics - University of Belgrade
Vinča Institute of Nuclear Sciences - University of Belgrade



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Dear ceramists,

On behalf of all committees of the 2nd Conference of The Serbian Ceramic Society (2CSCS-2013), and also on behalf of the co-organizers of this Conference i.e. Academy of Engineering Sciences of Serbia, Institute for Multidisciplinary Research - University of Belgrade, Institute of Physics - University of Belgrade, Vinča Institute of Nuclear Sciences - University of Belgrade, it is our great pleasure to welcome you to Belgrade and Serbia on June 5-7th 2013.

The Serbian Ceramic Society is national society which brings together the scientists and engineers working in the fields of research and application of ceramic materials. There is rather large ceramic community in Serbia since it has long tradition which involves both traditional and advanced ceramic materials. The members of The Serbian Ceramic Society, are professionally dealing with very attractive topics like nanostructured ceramics, ceramics in energy conversion, eco- and bio-ceramics, as well as, ultra high temperature ceramic composites. The activities of The Serbian Ceramic Society include organizing highly interesting lectures for the members, but also Students Meetings, which has taken place in Novi Sad under the sponsorship of the European Ceramic Society each year since 1998. In addition, the Serbian Ceramic Society publishes, since 2007, the Journal "Processing and Application of Ceramics" which is becoming ever more attractive to authors from abroad.

The aim of the 2CSCS-2013 is to bring together the scientists working in the field of ceramic materials for the exchange of attractive results in the areas of the development, characterization and application of ceramic materials as well as, to improve contacts for future scientific cooperation.

The abstracts of the papers that are going to be presented at the 2ndConference of The Serbian Ceramic Society are summarized in this book. They are divided according to topic to which the papers belong, i.e. into:

- Ceramic Powders, Characterization and Processing (chemical routes, hydrothermal synthesis, non-conventional routes, dispersion and processing aids, wet processing, spray-drying, plastic forming, net shape forming and porous products)
- 2. **High Temperature Phenomena, Sintering and Microstructure Design** (high temperature reactions, phase diagrams, densification and grain growth, tailoring microstructure to properties, hard coatings and wear)
- Electro and Magnetic Ceramics (ferroelectric and relaxors, piezoelectric, films, multilayer devices, interfaces, capacitor, microwave ceramics, varistors, conducting ceramics and electrodes, ionic conductors, resistors)
- 4. **Ceramic Composites, Membranes and Multimaterials** (ceramic matrix composites, fibres, nanocomposites and polymer transformation, laminates, biocomposites)
- 5. **Refractories, Cements, Glass and Corrosion** (raw materials and engineering, emission control, environment, recycling)
- 6. Ceramic Heritage

Four plenary lectures, fourteen invited lectures, twenty-two oral and fifty-seven poster presentations will be presented at the Conference. This book contains, as mentioned, all the received abstracts, and some of the papers, after regular peer review will be published in the international journal The Processing and Application of Ceramics.

June 5-7th, 2013. Belgrade, Serbia

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PROGRAMME

Wednesday - June 5, 2013

08.00 - 09.00 h, Registration

09.00 - 09.30 h, Oppening ceremony

09.30 - 10.00 h, Cocktail

Plenary & invited lectures

Chair: Snežana Bošković, Branko Matović

10.00 – 10.35 h, Plenary lecture, P-1

V.D. Krstic, High Fracture Toughness Ceramics and Their Application

10.35 – 10.55 h, Invited lecture, I-1

L. Ćurković, D. Ljubas, H. Otmačić-Ćurković, S. Šegota, S. Babić, Synthesis and Applications of Nanostructured Titania Sol—Gel Films on Glass and Stainless Steel

10.55 – 11.15 h, Invited lecture, I-2

D. Randjelović, T. Novaković, L. Rožić, AFM Studies of Ceramic Based Adsorbents, Catalysts and Composites

11.15 – 11.35 h, Coffee break

Session 1. Ceramic Powders, Characterization and Processing

Chair: Zorana Dohčević-Mitrović, Vladimir Srdić

11.35 – 11.50 h, O-1

J. Pantić, M. Prekajski, M. Dramićanin, N. Abazović, N. Vuković, A. Kremenović, B. Matović, *Synthesis and Characterization of Chrome Doped Sphene Pigments*

11.50 – 12.05 h, O-2

J. Zavašnik, M. Podlogar, A. Rečnik, Sonochemical and Solvothermal Synthesis of Fe-sulphides

12.05 – 12.20 h, O-3

M. Radović, B. Stojadinović, N. Tomić, S. Aškrabić, A. Golubović, B. Matović, Z. Dohčević-Mitrović, *Investigation of Defect Electronic States in CeO*₂ *Nanocrystals Synthesized by SPRT, Hydrothermal and Precipitation Method*

12.20 – 12.35 h, O-4

M. Prekajski, A. Radojković, G. Branković, S. Bosković, H. Oraon, R. Subasri, B. Matovic, *Synthesis, Characterization and Electrical Properties of Ce*_{1-x} $Bi_xO_{2-\delta}$ solid solution

12.40 - 13.40 h, Lunch break

13.00 - 14.00 h, Poster Session 1

Session 2: Ceramic Composites, Membranes and Multimaterials

Chair: Ravi Kumar, Dušan Bučevac

14.00 – 14.20 h, Invited lecture, I-3

J. Lamovec, Comparative Microhardness Analysis of Composite Systems with Various Thin Metallic Multilayer Composite Films

14.20 – 14.40 h, Invited lecture, I-4

- D. Nesheva, N. Nedev, V. Dzhurkov, M. Curiel, E. Manolov, I. Bineva,
- B. Valdez, Amorphous or Crystalline Silicon Nanodots for Memory and Detector Applications

14.40 – 14.55 h, O-5

B. Babić, M. Momčilović, J. Gulicovski, M. Prekajski, J. Pantić, M. Stojmenović, B. Matović, *New Ordered Mesoporous Ceria Synthesized by Templating Procedure*

14.55 – 15.10 h, O-6

N. Stanković, N. Daneu, A. Rečnik, Topotaxial Transformation of Ilmenite to Rutile and Hematite

15.10 – 15.30 h, Coffee break

Session 3. Ceramic Heritage

Chair: Radmila Jančić, Vesna Maksimović

15.30 – 15.45 h, O-7

A. Došen, A. Radosavljević Mihajlović, M. Prekajski, V. Maksimovic, Characteriztion of the Higly Porous Natural Building Material: Tufa from Pivnice Natural Heritage Monument

15.45 – 16.00 h, O-8

V. Janjić, M. Gajić-Kvaščev, V. Bikić, L. Damjanović, V. Andrić, *Physico-chemical Study of Early Modern Age Painted Pottery from Belgrade, Serbia*

Thursday - June 6, 2013

Plenary lecture

Chair: Vladimir Urbanovich, Miladin Radović

09.00 – 09.35 h, Plenary lecture, P-2

K. Yoshida, Development of High Performance SiC_f/SiC Composites Based on Electrophoretic Deposition Process

Session 4. High Temperature Phenomena, Sintering and Microstructure Design

09.35 – 09.55 h, Invited lecture, I-5

R. Kumar, Understanding the Deformation and Fracture Mechanical Properties of Precursor Derived Ceramics by Indentation

09.55 – 10.15 h, Invited lecture, I-6

B. Matovic, Effect of Sintering Technique on Properties of Monolithic Nanocrystalline SiC Ceramics

10.15 – 10.35 h, Coffee break

Chair: Katsumi Yoshida, Ravi Kumar

10.35 – 10.55 h, Invited lecture, I-7

M. Radovic, Structure and Properties of the MAX Phases

10.55 – 11.10 h, O-9

V. Urbanovich, *The Properties of Nanocrystalline Composite Materials Obtained by High Pressure Sintering*

11.10 – 11.25 h, O-10

S. Drev, A. Rečnik, N. Daneu, Spinel Twins and Spinel-taaffeite Epitaxies in BeO-doped MgAl₂O₄ Ceramics

11.25 - 11.40 h, O-11

M. Radovic, M. Lizcano, M. Westwick, *The Effects of Water Content and Chemical Composition on the Structure and Compressive Strength of Geopolymers*

11.40 – 11.55 h, O-12

D. Bučevac, J. Pantić, V. Maksimović, Fabrication of Accicular Mullite by Controled Oxidation of Molybdenum Disilicide

11.55 – 12.10 h. O-13

D. Zagorac, J. Christian Schön, J. Zagorac, I. Vladimirovich Pentin, M. Jansen, *Zinc Oxide: Connecting Theory and Experiment*

12.10 – 13.10 h, Poster Session 2

12.30 – 13.30 h, Lunch break

13.30 – 16.30 h, Visit to Royal Palaces of Karadjordjevic Dynasty

21.00 h, Conference dinner

Friday – June 7, 2013

Plenary lectures

Chair: Elisabetta Di Bartolomeo, Tatjana Volkov Husović

09.00 – 09.35 h, Plenary lecture, P-3

M. Romagnoli, Geopolymer Materials: Opportunities and Difficulties

09.35 - 10.10 h, Plenary lecture, P-4

E. Traversa, Chemically Stable Proton Conducting Electrolytes for Solid Oxide Fuel Cells Operating at 600°C

10.10 - 10.30 h, Coffee break

Session 5: Electro and Magnetic Ceramics

Chair: Biljana Stojanović, Nina Daneu

10.30 – 10.50 h, Invited lecture, I-8

D. Tripkovic, D. Strmcnik, N.M. Markovic, V.R. Stamenkovic, *Advanced Materials for Electrochemical Applications*

10.50 – 11.10 h, Invited lecture, I-9

E. Di Bartolomeo, A. D'Epifanio, S. Licoccia, *Thin Film Electrolytes for Solid Oxide Fuel Cells*

11.10 – 11.30 h, Invited lecture, I-10

A. Bencan, S. Drnovsek, T. Rojac, G. Trefalt, J. Jouin, J. Pavlic, B. Malič, K.G. Webber, Y. Seo, J. Rödel, D. Damjanovic, *How to Improve the Performance of Lead Zirconate Titanate Piezoelectric Ceramics?*

11.30 – 11.45 h. O-14

B. Jančar, D. Vengust, A. Šestan, V. Bobnar, Z. Kutnjak, D. Suvorov, *Layered Cobaltates as Thermoelectric Materials*

11.45 – 12.00 h, O-15

I. Atkinson, J. Pandele, E. Volceanov, V. Bretan, A.M. Anghel, C. Munteanu, C. Hornoiu, V. Fruth, *Synthesis and Characterization of BiFeO*₃ *Nanorods and Nanotubes*

12.00 – 12.15 h, O-16

O. Jakšić, D. Randjelović, D. Vasiljević-Radović, Z. Jakšić, On Modeling of Adsorption of Large Molecules on Crystalline Ceramic Surfaces

12.15 – 13.15 h, Lunch break

12.40 - 13.40 h. Poster Session 3

Invited lectures

Chair: Enrico Traversa, Victor Fruth

13.40 – 14.00 h, Invited lecture, I-11

S. Gardelis, P. Manousiadis, A.G. Nassiopoulou, Silicon Nanostructures: Optical and Optoelectronic Properties-Potential Applications in 3rd Generation Photovoltaics

14.00 – 14.20 h, Invited lecture, I-12

N. Daneu, N. Novak Gramc, A. Rečnik, M. Maček Kržmanc, S. Bernik, 'Shock-sintering' - A Novel Procedure for the Preparation of ZnO-based Varistor Ceramics

14.20 – 14.40 h, Invited lecture, I-13

Z. Dohčević-Mitrović, Magnetism in Oxide Nanomaterials

14.40 – 14.55 h, O-17

D. Gautam, M. Winterer, *Bulk Nanostructured ZnO for Thermoelectric Applications*

14.55 – 15.10 h, O-18

J. Vukmirovic, B. Bajac, N. Samardzic, B. Mojic, G. Stojanovic, V.V. Srdic, Synthesis and Characterization of BaTiO₃ Sols and their Application for Films Fabrication by Ink-Jet Printing

15.10 – 15.25 h, O-19

B. Bajac, J. Vukmirović, B. Mojic, S. Ognanovic, A. Kukovecz, B. Stojanović, V.V. Srdić, *Preparation and Structural Characterization of NiFe*₂O₄/BaTiO₃ Multilayered Thin Films

15.25 – 15.40 h, O-20

A.C.M. Kuniyil, B. Bajac, J. Vukmirovic, V.V. Srdic, G. Stojanovic, *Synthesis and Characterization of SnO*₂ *Films*

15.40 – 16.00 h, Coffee break

Session 6: Refractories, Cements, Glass and Corrosion

Chair: Marcello Romagnoli, Miroslav Komljenović

16.00 - 16.15 h, Invited lecture, I-14

T. Volkov-Husovic, S. Martinovic, M. Vlahovic, J. Majstorovic, A. Devecerski, B. Matovic, *Nondestructive Evaluation of Degradation Level for Refractory and Polymer Composite Materials in Extreme Conditions*

16.15 - 16.30 h, O-21

E. Volceanov, G. Predeanu, A.T. Abagiu, F. Zăman, L.G. Popescu, A. Volceanov, V. Fruth, C. Andronescu, *Ecoceramics an Alternative for Industrial Silicate Wastes*

16.30 - 16.45 h, O-22

M. Stevic, Domestic Resources and Materials for the Future of Serbia

HIGH FRACTURE TOUGNNESS CERAMICS AND THEIR APPLICATION

Vladimir D. Krstic

Department of Mechanical and Materials Engineering, Queen's University, Kingston, K7L 5H6 Ontario, Canada

The presentation will center on a new generation of laminated ceramics with exceptionally high apparent fracture toughness and on silicon nitride and stabilized zirconia ceramics known to have a combination of high fracture toughness and high fracture strength. New concentric ring laminates with fracture toughness of over 24 MPa.m^{1/2} will be presented and their properties and applications described. The key in this concentric ring structure is the absence of the planes for easy crack propagation and delamination. The presence of planes for easy crack propagation is the largest impediment for wider use of these structures under stress. Silicon nitride and zirconia on the other hand exhibit a combination of high fracture toughness and strength not found in other monolithic and composite ceramics. Some of the commercially available silicon nitride ceramics have fracture toughness of over 10-11 MPa.m^{1/2} and flexural strength of over 1150 MPa, which makes this material highly suitable for high-pressure, high-temperature applications such as race car seals, gas turbine components and crucibles in non-ferrous metal smelting. Partially and fully dense zirconia ceramic is another class of ceramic with a combination of high fracture toughness and strength. Stabilization of zirconia is known to result in ceramics with either high fracture toughness or high fracture strength but not both. However, many structural applications require both high strength and toughness. Recently, a new class of zirconia was developed with fracture toughness of over 15 MPa.m^{1/2} and strength of over 1000 MPa which makes this ceramic highly suitable for applications such as resistant welding in automotive and other industries.

DEVELOPMENT OF HIGH PERFORMANCE SiC_f/SiC COMPOSITES BASED ON ELECTROPHORETIC DEPOSITION PROCESS

Katsumi Yoshida

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Continuous silicon carbide fiber-reinforced silicon carbide matrix composites (SiC_f/SiC) have been expected to be used as components for high-temperature gas turbine, spacecrafts and future nuclear and fusion power reactors. It is important to form the optimum fiber/matrix interface in order to obtain high performance SiC_f/SiC composite. For the practical use of continuous SiC_f/SiC composites as structural parts, the establishment of the fabrication process of high-performance SiC_f/SiC composites that is simpler, environmentally benign and less costly compared with conventional processes has been strongly requested.

Electrophoretic deposition (EPD) is a colloidal process wherein ceramic bodies are shaped directly from a stable colloid suspension by a DC electric field. Our research group paid attention to EPD and proposed the novel fabrication process of SiC_f/SiC composites based on EPD. We demonstrated that the fabrication process based on EPD is an effective way to control the interfaces of SiC_f/SiC composites and to obtain high-performance SiC_f/SiC composites.

In this congress, our novel fabrication process of high performance SiC_f/SiC composites based on electrophoretic deposition (EPD) will be reviewed.

GEOPOLYMER MATERIALS: OPPORTUNITIES AND DIFFICULTIES

Marcello Romagnoli

Università di Modena e Reggio Emilia, Modena, Italy

Geopolymers are inorganic materials consisting of a polymeric Si–O–Al framework, similar to zeolites. They are generating in recent years more and more interest among technologists and scientists. They are not a new materials because their discovery takes place in 50s with the first studies of Viktor Glukovsky or since 70s with Joseph Davidovits. Their use may be even more old. In an its book, Davidovits said that pyramids could be built by Egyptians with geopolymers and not with natural rocks. Also ancient romans used inorganic binders more similar to a geopolymer than to modern concrete.

As every material, geopolymers have pros, cons and weak points that are still unresolved.

In this work, the most interesting, positive aspects and the most important technical-scientific problems for this kind of material are reported.

CHEMICALLY STABLE PROTON CONDUCTING ELECTROLYTES FOR SOLID OXIDE FUEL CELLS OPERATING AT 600°C

Enrico Traversa

King Abdullah University of Science and Technology, Thuwal, Saudi Arabia

The high cost of solid oxide fuel cells (SOFCs), related to their high operating temperatures, hinders the general use of SOFCs and causes long-term stability problems. A step forward towards reducing the SOFC working temperature at 600°C or below can be the use of high temperature proton conductor (HTPC) oxides as electrolytes, due to their lower activation energy for proton conduction (0.3-0.6 eV), with respect to oxygen-ion conducting electrolytes. Moreover, proton conductor electrolytes offer the advantage of generating water at the cathode, and thus the fuel does not become diluted during cell operation. Y-doped barium cerate (BCY) has been considered as the most promising HTPC electrolyte for SOFC use, but its practical deployment has been hindered by its reactivity with water vapour and CO₂. Commercial development needs the use of chemically stable materials are neede, especially for carbon-based fuels, such as ethanol produced from biomasses. Doped BaZrO₃ is an alternative material that offers excellent chemical stability against CO₂ and H₂O reaction, but low conductivity values for sintered pellets are usually reported. We have recently made significant progresses in the electrolyte development by improving the conductivity of Y-doped barium zirconate (BZY). We followed various strategies to improve the BZY conductivity, including the use of co-doping and sintering aids to improve its sinterability, and the fabrication of films by pulsed laser deposition (PLD). The possibility to develop a next generation of SOFCs, though, needs also the development of electrode materials. The recent work in developing tailored cathode and anode materials for BZY electrolytes will be also presented.

SYNTHESIS AND APPLICATIONS OF NANOSTRUCTURED TITANIA SOL-GEL FILMS ON GLASS AND STAINLLESS STEEL

Lidija Ćurković¹, Davor Ljubas¹, Helena Otmačić-Ćurković², Suzana Šegota³, Sandra Babić²

¹Faculty of Mechanical Engineering and Naval Architecture, Zagreb, Croatia ²Faculty of Chemical Engineering and Technology, Zagreb, Croatia ³Ruđer Bošković Institute, Zagreb, Croatia

Nanostructured titania (TiO_2) films were deposited on borosilicate glass and AISI 304 stainless steel substrates by sol-gel process and dip-coating technique. For the preparation of two colloidal suspensions or sols (sol 1 and sol 2) titanium isopropoxide was used as a precursor, propanol as a solvent, with addition of nitric acid as a catalyst and acetylacetone for peptization. Both of the prepared sols contained the same amount of mentioned components, the only difference was in the addition of polyethylene glycol (PEG) as organic/polymer additive to sol 2. After preparation of solutions, deposition of coatings by dip-coating technique was made, followed by drying and calcining at the temperature of 550 $^{\circ}$ C.

The deposited films were characterized by X-ray diffraction (XRD), thermal gravimetry (TG), differential scanning calorimetry (DSC), atomic force microscopy (AFM), scanning electron microscopy (SEM) equipped with EDS detector, UV–Vis spectroscopy and glow-discharge optical emission spectrometry (GD-OES).

The photocatalytic activity of the coated glass substrates was evaluated by the photocatalytic degradation of Congo red dye (CR) and sulfonamide antibiotic sulfamethazine. Photocatalytic activity of the films was monitored by means of UV/VIS spectrophotometry.

Electrochemical corrosion behavior of the coated stainless steel substrates was evaluated in simulated marine environment in 3 wt. % aqueous NaCl solution by electrochemical impedance spectroscopy (EIS) and in 0.5 M aqueous HCl solution by potentiodynamic polarization.

The addition of PEG to the TiO_2 solution resulted in the changes in the film surface morphology, and affected the ratio of anatase-rutile crystal phases and the investigated properties of TiO_2 (photocatalytic activity on glass substrate and corrosion behaviour on stainless steel substrate).

The present study shows that sol-gel TiO_2 films with smooth surfaces may be applied for corrosion protection, while the films with higher surface roughness can improve the photocatalytic activity.

Acknowledgement This work has been supported by the Croatian Ministry of Science, Education and Sports Projects: 120-1201833-1789, 120-1253092-3021, 125-1252973-2572, 125-1253008-1350, 098-0982934-2744.

AFM STUDIES OF CERAMIC BASED ADSORBENTS, CATALYSTS AND COMPOSITES

<u>Danijela Randjelović</u>¹, Tatjana Novaković², Ljiljana Rožić²

¹IHTM – Centre of Microelectronic Technologies and Single Crystals, University of Belgrade, Belgrade, Serbia ²IHTM – Department of Catalysis and Chemical Engineering, University of Belgrade, Belgrade, Serbia

Atomic force microscopy (AFM) is a powerful nondestructive technique for characterization of different classes of materials, including metals, ceramics, polymers and composites. Unlike many other techniques, AFM measurements does not require complicated sample preparation, like coating with conductive film, which can affect the validity of obtained sample image. On the other hand, the force exerted on the sample by the AFM probe is extremely low and as such does not influence the surface topography of the samples. AFM offers possibility of 2D and 3D presentation of the sample morphology, line or area analysis of the topography, estimation of the dimensions of specific features on the sample, roughness analysis etc. Results of AFM study of the following samples will be presented: 1) alumina gels, 2) active porous alumina coatings, 3) polymer/alumina composites, 4) acid-activated bentonite powders, 5) acid-activated bentonite powders with various loadings of HPW (HPW/AAB powders). Two AFM systems were applied for characterization of these samples, AutoProbe CP-Research SPM (TM Microscopes-Bruker) and NTEGRA Prima AFM (NT-MDT).

COMPARATIVE MICROHARDNESS ANALYSIS OF COMPOSITE SYSTEMS WITH VARIOUS THIN METALLIC MULTILAYER COMPOSITE FILMS

Jelena Lamovec

Institute of Chemistry, Technology and Metallurgy, University of Belgrade, Belgrade, Serbia

In comparison with the corresponding pure metals, composite structures consisting of alternating metallic layers may present improved mechanical, optical, electrical or magnetic properties which are important for applications in micromechanical systems. Layered structures consisting of two or more soft metals may exhibit very high strengths when the layer thickness values are reduced into nano-range. Among these structures, copper/nickel system has received significant attention, because copper and nickel have similar crystal structure and a small lattice mismatch which allow the growth of coherent and semicoherent epitaxial layers. The large elastic modulus mismatch imposes considerable image forces on dislocations near the interface and contributes to the overall strength of copper/nickel multilayers.

Electrodeposition has been shown to be a viable method to produce layered metallic structures.

Composite systems of alternately electrodeposited nanocrystalline Ni and Cu films on cold-rolled polycrystalline copper substrates were fabricated. Nanostructured nickel films were deposited from two different electrolytes: lab-made sulphamate and commercial sulphate "Slotonik 20, Schloetter", and copper films from lab-made sulphate bath.

Chosen deposition parameters for experimental investigation were the next: the current density values were maintained at 10mAcm^{-2} and 50 mAcm^{-2} , which resulted in variations in microstructures and mechanical properties and deposition time was determined according to plating surface and projected thickness of sublayers (from 75nm to 300nm) and total thickness of the films (5 and $10\text{ }\mu\text{m}$).

The indentation properties of different composite systems were determined with standard Vickers microhardness testing, with loads ranging from 0.049N to 1.96N. Dependence of Vickers microhardness on sublayer thickness, Ni/Cu sublayer thickness ratio and total thickness of the film was investigated. In order to extract the composite-film hardness from the composite system hardness, composite model of Korsunsky will be examined. With increasing the sublayer thickness ratio, the composite microhardness value also increases. It gives the possibility of "tailoring" the mechanical properties of the films.

AMORPHOUS OR CRYSTALLINE SILICON NANODOTS FOR MEMORY AND DETECTOR APPLICATIONS

Diana Nesheva¹, Nikola Nedev², V. Dzhurkov¹, Mario Curiel², Emil Manolov¹, Irina Bineva¹, Benjamin Valdez²

¹Institute of Solid State Physics Bulgarian Academy of Sciences, Sofia, Bulgaria
²Institute of Engineering, Autonomous University of Baja California,
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Despite of the huge commercial success, conventional floating gate devices have their limitations. The self-assembling Si nanocrystals in a silicon dioxide matrix represent an alternative material, which is compatible with the existing conventional microelectronic technology. Structures of the type metal-insulator-nanocrystals-insulator-semiconductor have been fabricated in which the process of direct tunnelling to charge and discharge the nanoparticles is used. This new concept allows reduction of the size of the memory device and improvement of their reliability and lifetime. Similar structures can be used for registration of ionizing radiation which remains one of the most important problems in the field of control of nuclear wastes, nuclear power stations, in medicine and space investigations.

A brief review of the results of other research groups on MOS structures containing Si nanoparticles for non-volatile memory and detector applications is made. Then a description of the most important results obtained during the last 6-7 years by the authors and their collaborators in the field of development of MOS structures with dielectric film containing amorphous and crystalline silicon nanoparticles is presented. Short information is given about the preparation by thermal evaporation of silicon monoxide in vacuum of SiO_x films of two different compositions (x=1.15 and 1.3) as well as for the annealing conditions used to grow amorphous and crystalline Si nanoparticles. Transmission Electron Microscopy, infra red transmission and Raman Scattering data prove the nanoparticle formation. A newly developed technique for preparation of MIS structures containing amorphous or crystalline Si nanoparticles suitable for non-volatile memory application is described. Data from high frequency (1 MHz) capacitance-voltage measurements in various ranges are given. They prove that the information recording is related to nanoparticle charging. Second, energy saving approach of MOS structure preparation is described which uses a single film deposition process and a two step postdeposition annealing. Results on testing some MOS structures for detection of gamma and X-ray radiation are shown. A comparison with results of other groups using conventional floating gate device is made and some advantages of the proposed sensors are indicated.

UNDERSTANDING THE DEFORMATION AND FRACTURE MECHANICAL PROPERTIES OF PRECURSOR DERIVED CERAMICS BY INDENTATION

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Precursor derived ceramics produced by the solid state thermolysis (SST) of polymeric precursors have gained immense interest in recent years due to its remarkable thermo-mechanical properties at exceedingly high temperatures. These ceramics are considered in recent times for sustaining contact loads in the form of coatings for high temperature tribological applications electromechanical systems (MEMS). Hence, a critical understanding of the elastoplastic behavior upon contact loading will be of scientific and technological interest. Conventional techniques such as tensile testing seldom assist in understanding the elasto-plastic behavior due to the brittle nature of these materials under tension. However, under contact loading they tend to exhibit limited plastic deformation and the flow stress upon deformation was observed to depend upon the hydrostatic pressure. Hence, a depth sensing nanoindentation coupled with microindentation could be considered as effective tools in understanding the deformation behavior. We have investigated the indentation response of several ternary and quaternary Sibased ceramics and understood the deformation mechanisms and characterized the fracture mechanical properties. Apart from the fact that all the precursor derived ceramics show a large elastic recovery, they seem to also exhibit anomalous deformation behavior which is evident from the crack evolution patterns. The talk aims at unraveling some of the interesting aspects deformation response of these classes of ceramics which have not been reported so far.

EFFECT OF SINTERING TECHNIQUE ON PROPERTIES OF MONOLITHIC NANOCRYSTALLINE SIC CERAMICS

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The effect of sintering technique on density, microstructure and mechanical properties of monolithic nanocrystalline SiC ceramics was studied. Cubic SiC nanopowder synthesized by sol-gel process with the average particle size of 15 nm was densified by three different sintering techniques. The main difference between these techniques was the mechanical pressure which was applied during sintering in order to fabricate dense SiC samples. The first technique was pressureless sintering which was conducted without the help of mechanical pressure whereas the other two techniques were pressure assisted. One was spark plasma sintering which provides relatively modest mechanical pressure of ~ 30 MPa and the other technique was pressure sintering assisted with very high mechanical pressure of ~ 4 GPa. Mechanical properties of the sintered samples such as hardness, fracture toughness and Young's modulus were determined and correlated to the microstructural features. It was found that the increase in applied mechanical pressure increases the density of sintered samples and improves mechanical properties particularly hardness and Young's modulus. The best results were measured in samples obtained by pressure sintering using high-pressure (4 GPa) "anvil-type with hollows" apparatus. Relatively dense samples were obtained even at 1500 °C whereas fully densified samples (> 99%) were obtained after sintering at 1900 °C for only 1 minute. These samples exhibited micro-hardness and Young's modulus of elasticity of 33 GPa and 450 GPa, respectively.

STRUCTURE AND PROPERTIES OF THE MAX PHASES

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In the last 15 years or so, a new class of ceramics has emerged that has challenged their typical description as materials that are hard, difficult to machine, and susceptible to damage and thermal shock. This new class of 60+ members known as the MAX phases - have formula: $M_{n+1}AX_n$ - where n = 1, 2, or 3, where M is an early transition metal, A is an A-group element (a subset of group 13-16 elements) and X is C and/or N. This family of layered machinable ternary carbides and nitrides has many chemical and physical properties in common with their binary counterparts. However, they also show some unique properties that are more typical of metals than ceramics. Some of the MAX phases – notably Ti₂AlC and Ti₃SiC₂ are refractory and stiff, but readily machinable, excellent thermal and electrical conductors, thermal shock and damage tolerant, oxidation and creep resistant. Therefore, the MAX phases are a new and exciting class of carbides and nitrides that bridge the gap between properties typical of metals and ceramic, while offering fundamentally new directions in tuning the structure and properties of ceramics for emerging applications. The purpose of this talk is to give an overview of the more salient structural, physical and mechanical properties of the MAX phases and where our current understanding stands. Some of their potential applications will be also highlighted.

ADVANCED MATERIALS FOR ELECTROCHEMICAL APPLICATIONS

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The major barriers to broad commercialization of technologies such as fuel cells, metal-air batteries and electrolyzers are the cost, performance and durability of currently employed materials. Improvements in materials design at atomic molecular level are expected to bring substantial change in their functional properties. It has been demonstrated that tuning of surface and bulk properties such as composition, structure, and electronic properties are critical aspects toward novel materials with unique performance. An increase in catalytic activity of almost two orders of magnitude compared to the conventional Pt/C catalyst was reported for well-ordered single crystalline surfaces. This finding inspired subsequent nanoscale catalyst design aiming to synthesize more active, but also durable catalysts. However, another component, high surface area support is also critical in the design of practical materials. A synergy between well-defined systems and novel synthetic routes for nanomaterials incorporated in different supports will be presented as a potent approach in development of functional materials for electrochemical applications.

THIN FILM ELECTROLYTES FOR SOLID OXIDE FUEL CELLS

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Alternative energy production is one of the major problems for future sustainable development. Solid oxide fuel cells (SOFCs) are a promising technology for this aim, but their mass development is hindered by high costs and stability issues related to high operation temperature (1000 °C). Significant research efforts are now being devoted to decreasing the SOFC operation temperature in the intermediate range (below 700 °C, IT). Aim of our research is to push the limit of SOFC operation temperature to the lowest possible value, developing highly performing nanostructured materials and innovative device architectures to fabricate SOCFs operating at 450-700 °C.

Proton and anion conducting electrolytes, characterized by high conductivity at IT, were developed in thin film form to reduce their ohmic resistance. One of the main factors impairing the electrolyte conductivity is the presence of blocking grain boundaries. The strategies adopted in our laboratory to solve this problem are the fabrication of monocrystalline or highly textured films using pulsed laser deposition (PLD) and the modification of the grain boundary nature using nanostructured materials prepared by wet-chemistry methods to control the sintering procedures. Pulsed laser deposition (PLD) is particularly promising amongst the different film deposition techniques because of its ability in reproducing complex target compositions onto the film.

Highly-textured, epitaxially oriented $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2}O_3$ (LSGM) and $BaZr_{0.8}Y_{0.2}O_3$ (BZY) films were grown by pulsed laser deposition on different substrate (e.g. MgO, NGO, LAO). The study of the microstructure/transport properties correlation was the main aim of the present work. In both cases films with good crystalline quality with improved electrochemical properties were obtained.

Our results demonstrated that optimizing the control of the crystallographic quality reducing the mismatch between the substrate and the deposited film is a key issue to achieve electrolyte thin films with optimized electrical performances.

HOW TO IMPROVE THE PERFORMANCE OF LEAD ZIRCONATE TITANATE PIEZOELECTRIC CERAMICS?

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Piezoelectric ceramics based on the lead zirconate titanate solid solution [(Pb,Zr)TiO₃] with the composition at the morphotropic phase boundary are commercially important due to their excellent electromechanical properties. Unfortunately, as all ceramics, these materials are brittle, which imposes severe limitations for some piezoelectric applications. A possible way to toughen the originally brittle (Pb,Zr)TiO₃ ceramic is by introducing zirconia particles.

We investigated the effect of introducing tetragonal yttria-stabilized zirconia (TZ) particles in the "soft" [Pb_{0.98}Ba_{0.01}][(Zr_{0.53}Ti_{0.47})_{0.98}Nb_{0.02}]O₃ (PZT) ceramics. The PZT- xTZ, (x = 0, 2, 5, 10, 20 vol%) composites were prepared by the solid state synthesis and sintered at 1275° C, 2h. We have shown that zirconium diffuses into the PZT, which resulted in a shift of the matrix phase composition towards the zirconia-rich rhombohedral phase. The addition of zirconia hinders the matrix grain growth and changes the fracture mode. The obtained dielectric and piezoelectric properties of the composites were related to the observed changes in the phase composition and microstructure. [1] Furthermore, we have shown that the addition of zirconia leads to transformation toughening and reduced ferroelastic toughening of PZT-TZ composites. [2,3]

By examining the microstructures of the composites we found, however, that the second phase zirconia particles coalesced within the PZT matrix, forming agglomerates that could affect the mechanical properties of the composites. In order to achieve a homogeneous distribution of the TZ grains within the PZT matrix, we used a modified solid state synthesis procedure, which included pre-milling, pH adjustment and modification of the surface of the TZ powder. The homogeneity of the TZ distribution in the PZT-TZ ceramic composites prepared from the heteroagglomerated particles was evaluated using the Voronoi-diagram analysis. At the end of the presentation, we will address the impact of the distribution of the TZ particles in the PZT matrix on the fracture behavior of the PZT-TZ composites.

I-11

SILICON NANOSTRUCTURES: OPTICAL AND OPTOELECTRONIC PROPERTIES-POTENTIAL APPLICATIONS IN 3rd GENERATION PHOTOVOLTAICS

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Nanostructured silicon in the form of porous silicon (PSi) or in the form of silicon nanocrystals (SiNCs) embedded in dielectric matrices, such as silicon dioxide or silicon nitride, has unique optical and electrical properties which make it a very useful material for nanoelectronics, photonics and solar cell applications. Generally, silicon nanostructures with sizes smaller than the Bohr radius of excitons in crystalline silicon, show quantum size effects. This results in the widening of the energy band gap and the enhancement of the oscillator strength of absorption and light emission due to the breakdown of the k-conservation rule. This makes silicon nanostructures a very promising absorber material for 3rd generation solar cells.

Motivated by the potential use of SiNCs in Si-based solar cell designs, we have grown single and multilayered nanocrystalline Si (nc-Si) films containing SiNCs of controlled sizes on quartz and crystalline silicon substrates by low pressure chemical vapour deposition (LPCVD) of Si and subsequent oxidation at high temperatures. In this talk, we review our results regarding the structural properties, electrical transport, optical and optoelectronic properties of these films and examine the possibility of using such films in 3rd generation photovoltaics.

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I-12

'SHOCK-SINTERING' – A NOVEL PROCEDURE FOR THE PREPARATION OF ZnO-BASED VARISTOR CERAMICS

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ZnO-based varistor ceramics is characterized by their excellent nonlinear current-voltage (I-V) characteristic. It is prepared by sintering ZnO with small amounts of dopants. The addition of Bi₂O₃ is crucial for the development of varistor effect, since it melts during sintering and forms thin resistive layers (Schottky barriers) at ZnO-ZnO grain boundaries. Other dopants are added to tailor the breakdown voltage (grain growth modifiers like Sb₂O₃, TiO₂) enhance the nonlinearity (transition metal oxides) suppress the leakage current or improve the energy absorbing capability of the varistor. Grain growth modifiers are known to trigger the formation of dopant-rich inversion boundaries (IBs) in ZnO grains. This can be used to effectively control the ZnO grain growth via the so-called IB-induced grain growth mechanism. In the nucleation stage of grain growth IBs are formed as dopant-rich planar defects in ZnO grains. The affected ZnO(IB) grains start to grow rapidly in the direction of the inherent IB. Anisotropic growth in this direction is continued until impingements of ZnO(IB) grains. In the next phase ZnO(IB) grains thicken, following the Ostwald's ripening mechanism, until most of the normal ZnO grains are consumed. If the initial number of ZnO(IB) nuclei is low, the final microstructure will be coarse-grained and vice-versa. While the formation of IBs in Sb₂O₃-doped ZnO is abundant and the final microstructures are usually fine-grained and suitable for high-voltage applications, coarse-grained microstructures for lowvoltage varistor ceramics are usually obtained by the addition of TiO₂ or Bi₄Ti₃O₁₂. However, during conventional sintering regime, usually only a few ZnO(IB) grains form and these grains become much larger compared to the normal ZnO grains.

In our most recent work we demonstrated that nucleation of ZnO(IB) grains and further microstructure development in ZnO-BIT system depends mostly on the heating rate. We have shown that one of the essential conditions for the development of homogeneous microstructure is rapid release and efficient distribution of TiO₂, which is required for the formation of Ti-rich (tail-to-tail) IBs within ZnO grains. This can be achieved via the so-called 'shock'-sintering procedure, when the samples are inserted directly into pre-heated furnace. This induces immediate decomposition of BIT to TiO₂-rich Bi₂O₃ liquid phase above 1200°C and leads to abundant nucleation of ZnO(IB) grains. In contrast to conventional sintering, where erratic

nucleation of IBs leads to bimodal grain size distribution, the 'shock'-sintering regime produces microstructures with uniform coarse-grain sizes, required for low-voltage varistor ceramics.

I-13

MAGNETISM IN OXIDE NANOMATERIALS

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The existence of room-temperature ferromagnetism in undoped thin films and nanoparticles of otherwise non-magnetic metal oxides remains controversial. Influence of defects and transition metal dopant ions on ferromagnetic properties of nano-oxides is still unclear. Different models like defect induced magnetism, bound magnetic polaron or F center exchange mechanisms are proposed to explain magnetic ordering in nano-oxides.

In the present work will be demonstrated influence of intrinsic defects and dopant cations (3d, 4d and 4f elements) on magnetic properties of CeO₂ and HfO₂. Different mechanisms responsible for ferromagnetic ordering exist in these systems and are very dependent on the crystal and electronic structure of these materials.

I-14

NONDESTRUCTIVE EVALUATION OF DEGRADATION LEVEL FOR REFRACTORY AND POLYMER COMPOSITE MATERIALS IN EXTREME CONDITIONS

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Non-destructive evaluation of degradation level in extreme condition caused by: thermal shock and cavitation erosion for refractory concrete (LCC) and durability of sulfur concrete in aggressive environment were goal of our investigation. Non-destructive testing methods such are image analysis and UPVT were used.

Thermal stability of refractory LCC samples was tested using standard laboratory procedure of water quench test (ICS 81.080 SRPS B.D8.308 former JUS B. D8. 306). Program for image analysis was applied for monitoring destruction at the surface and bulk of the sample before and during testing. Ultrasonic pulse velocity testing (UPVT) was applied to measure ultrasonic velocities changes during testing.

Cavitation damage ratio was monitored using mass loss during experiment as well as image analysis of the photographs of the samples before and during testing. Image analysis using different software allowed to measure ratio of the damaged surface during cavitation erosion. Results were presented as surface erosion ratio.

In order to predict service life of the sulfur–polymer composite, the samples were subjected to the induced destruction using 10% hydrochloric acid solution. Sulfur–polymer composite showed limited mechanical strength and mass loss, while physico-mechanical properties of Portland cement composite regressed rapidly. The Image Pro Plus software was used for surface destruction monitoring.

Key words: refractory concrete, sulfure concrete, thermal shock, cavitation erosion, durability testing, image analysis, UPVT

SYNTHESIS AND CHARACTERIZATION OF CHROME DOPED SPHENE PIGMENTS

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Mechanical activation of precursors has been used for the preparation of Crdoped sphene ceramic pigments (CaTi_{1-y}Cr_ySiO₅, Cr/Ti atomic ratio = 0.03, 0.05, 0.1, 0.15, 0.35 and 0.5). Ceramic material has been prepared from a powder mixture of CaCO₃, TiO₂, SiO₂ and Cr(NO₃)·9H₂O using vibro-milling for homogenization and precursor activation. The mechanochemical process initially yielded amorphous powders which, on further calcination, crystallized to yield Cr-doped sphene ceramic pigment. Phase evolution in CaTi_{1-y}Cr_ySiO₅ composition with thermal treatment was investigated by X-ray powder diffraction (XRPD). Texture properties and particle size distribution were analyzed by scanning electron microscopy (SEM) and laser diffraction, respectively. UV/vis reflectance spectra are used to determinate the behavior of the chromium ion. The color efficiency of pigments was evaluated by colorimetric analysis (CIE L*a*b system). Photoluminescence measurements were also performed.

SONOCHEMICAL AND SOLVOTHERMAL SYNTHESIS OF Fe-SULPHIDES

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Interest in the Fe-sulphide group arises from their occurrence in metallurgical process, their properties, and their common occurrence in many ore deposits [1-3]. A better understanding of pyrrhotite group is of commercial interest for mineral processing industry [4-5] and for wastewater treatment [6-8]; Fe-sulphides are used in Li-FeS₂ batteries [9] and cation-deficient pyrrhotites have potential application as part of phase-change magnetic memory device [10]. FeS phases are also main obstacle in producing high-quality semiconducting FeS₂ thin films as a substitute for Si-based solar cells [11].

Our motivation was to investigate a new route of preparing nano-sized FeS and FeS_2 by a 2-step sonochemical and solvothermal synthesis with a diethanolamyne (DEA) as an organic precursor, known from natural-gas treatment process [12].

Fe-chloride, sulphur and DEA in stoichiometric ratio Fe+S: DEA = 10:1 were diluted with EtOH_{abs}, mixed in PTFE vessel and sonificated in ultrasonic bath for 10 minutes. Immediately after adding DEA to solid reactants the surface of Fe-chloride turns black; after mixing all of the solution turns pitch-black in a minute. After sonification samples from the solution were taken on copper-lacey grid and immediately transferred to the TEM to avoid oxidation. The products were up 50 nm big particles of FeS, with average size of 10-20 nm (Fig. 1a), usually with minor sulphur residue and without Fe-oxides. The remaining product was transferred to autoclave for 18 hours at 200°C; the solution has a strong H₂S odour and dark-brown colour, while the particles are big enough to precipitate from the suspension. Samples were again inspected in TEM: the particles of FeS are bigger (150 nm) and their size is more uniform (Fig. 1b), often are clustered due to weak magnetic attraction forces. Platy six-folded crystals are composed of alternating hcp and ccp sequences, with cubic phase prevailing. Intermediate product was further mixed with sulphur flakes, diluted with alcohol and heated in autoclave for 18h and again inspected in TEM. The majority of products after second stage of solvothermal treatment were nanosized FeS₂ in size of 2-5 nm developed on FeS cores, sometimes still preserved. Size of remaining FeS crystals is the same as after the first stage of solvothermal treatment, indicating only pyrite developing during heating (Fig. 1c). XRD spectra showed presence of only pyrite, with no marcasite peaks detected, which also correlate with electron-diffraction pattern we collected. The solution with the samples was diluted with absolute alcohol and stored for 90 days in closed plastic flasks, with measured pH 5-6. The samples do not show any oxidation or structural changes when exposed to air, indicating a high stability, uncommon for Fe-sulphides (Fig. 1d,e).

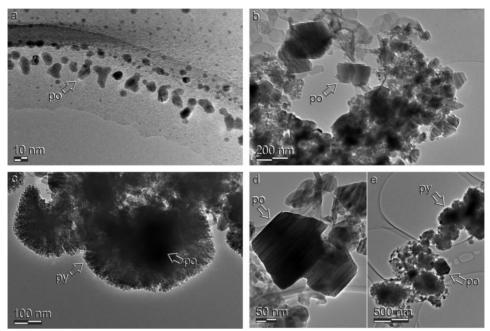


Fig 1: (a) nanocrystals of FeS, already showing contrast streaks due to alternation of hcp and ccp stacking. No residue of reagents were found on TEM of XRD already after 10 minutes of sonification; (b) FeS crystals after first stage of solvothermal treatment; representative six-fold morphology is already visible; (c) pyrite nanocrystals are growing atop of FeS without crystallographic relation to host FeS; (d, e) aging has little effect on oxidation of Fe-sulphides. The samples of FeS (d) and pyrite (e) collected after 90 days of storage in alcohol shows no surface oxidation or alternation.

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INVESTIGATION OF DEFECT ELECTRONIC STATES IN CeO₂ NANOCRYSTALS SYNTHESIZED BY SPRT, HYDROTHERMAL AND PRECIPITATION METHOD

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The present work examines the influence of various synthesis methods on the formation of defect electronic states in the band structure of CeO_{2-v} nanocrystals. Characterization of structural properties and determination of average crystallite size was performed using X-ray diffraction spectroscopy. Raman spectroscopy technique provides an insight into the concentration of oxygen vacancies and vibrational properties of ceria nanocrystals. Scanning tunneling microscopy and scanning tunneling spectroscopy measurements were performed on the CeO_{2-y} nanocrystals and the measurements were compared with available literature data for the electronic band structure of cerium dioxide. The differences in defect electronic states within the band gap were detected among the differently prepared CeO_{2-v} nanocrystals. Optical properties of CeO_{2-v} nanocrystals were investigated by spectroscopic ellipsometry. Through the critical points analysis of ellipsometric data we were able to establish direct relationship between observed variations in electronic structure and optical transitions. This study revealed that synthesis process strongly influences the formation of different oxygen vacancy complexes which, on the other side, have dominant influence on optical, transport and magnetic properties of ceria based materials. In order to reach full potential of these materials it is of great importance to elucidate which type of synthesis process provides better ceria performances.

SYNTHESIS, CHARACTERIZATION AND ELECTRICAL PROPERTIES OF $Ce_{1-x}Bi_xO_{2-\delta}$ SOLID SOLUTION

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A series of nanocrystalline solid solutions $Ce_{1-x}Bi_xO_{2-\delta}$ (x = 0.1 - 0.5) were synthesized by self-propagating room temperature method (SPRT). The products were characterized by X-ray diffraction (XRPD), transmission electron microscopy (TEM), scanning electronic microscope (SEM) and Raman spectroscopy. Results show that the solubility limits of Bi₂O₃ in ceria using SPRT method were as high as 50 mol %. XRPD data showed that all solid solutions crystallized in single-phase cubic fluorite type structure. The average grain sizes of synthesized powders were less than 5 nm. Thermal stability of these solid solutions with different concentration of Bi³⁺ cation was investigated at various temperatures up to 1400 °C by applying thermogravimetric analysis (DTA/TG). It was revealed that all samples with higher concentration of bismuth are unstable during thermal treatment, resulting in Bi leaving the structure of ceria and formation of β -Bi₂O₃ as second phase. Moreover, at a certain temperatures bismuth begins to evaporate. Dense samples were obtained by applying microwave sintering. The ionic conduction measured by impedance spectroscopy showed that the solid solutions with lower dopant content exhibited primarily the grain boundary conduction, whereas for the sintered Ce_{0.5}Bi_{0.5}O_{2.8}, there is only the bulk. In both cases resistance decreased dramatically with increasing temperature.

NEW ORDERED MESOPOROUS CERIA SYNTHESIZED BY TEMPLATING PROCEDURE

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The new ordered mesoporous CeO₂ materials were obtained by templating procedure using ordered mesoporous carbon (FDU) as a matrix. The ordered mesoporous carbon was synthesized by using Pluronic F-123, under acidic conditions. CeO2/carbon composites were obtained by sol-gel polycondenzation of resorcinol and formaldehyde and subsequent pyrolysis, in presence of different amount of Ce(NO₃)₃. The carbon template is eliminated by thermal treatment in air. were characterized nitrogen adsorption-desorption materials by measurements, X-ray diffraction and scanning electron microscopy (SEM). It was revealed that samples have high specific surface, developed mesoporosity and amorphous structure. Porous structure is a function of the Ce/carbon ratio and could be controlled by concentration of starting solution.

0-6

TOPOTAXIAL TRANSFORMATION OF ILMENITE TO RUTILE AND HEMATITE

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In nature, hydrothermal alteration of ilmenite often leads to the formation of multiply twinned, sagenitic rutile [1,2]. Because of its fractal branching twinned rutile could find applications as a support for separation processes and catalysis, while on the other hand this mechanism is important for understanding leaching of ilmenite in the production of TiO₂ powders under acidic conditions. While having potential technological implications this transformation is quite challenging from a structural point of view. A question arises how trigonal and tetragonal minerals match to the point to form intergrowths. The key reason for this lies in their common, hexagonal close-packed, oxygen sublattice. While trigonality of ilmenite

and hematite are produced by distribution of cations, tetragonality of rutile is caused by an additional distortion of the oxygen sublattice.

To study this structural transformation rutile-hematite pseudomorphs after ilmenite from Mwinilunga deposit in Zambia were investigated in basal and prism orientations by optical microscopy, X-ray powder diffractometry (XRD), scanning and transmission electron microscopy (SEM, TEM). Macroscopic examination of the samples reveals that rutile crystals overgrow hosting hematite in a highly ordered manner. Crystals of rutile are twinned by (101) and (301) planes, but also single crystals are observed, oriented in six different directions with respect to hematite, wherefrom crystallographic relationship of $\langle 010 \rangle \cdot (101)_{RUT} \parallel \langle 001 \rangle \cdot \{110\}_{HEM}$ can be deduced. This relationship is confirmed by optical microscopy of (001)_{HEM} thinsection, where rutile domains form 6-fold daisy-like domains, with the extinctions repeated at $\sim 60 \pm 2.8^{\circ}$ under polarized light. The observed deviations from 60° between (001)_{RUT} and (110)_{HEM} directions corresponds to the rutile lattice distortion with respect to the ideal oxygen sublattice of hematite. In prism cross-sections microscopy of pseudomorphs shows a completely different texture; here rutile appears in form of pillars grown in (001)_{HEM} direction through the sample height. TEM investigations further revealed that hematite and rutile form 12 unique interfaces, which trigger three types of special boundaries between rutile domains: (i) 174.4° low angle tilt boundary (the angle enclosed by the rutile c-axes), (ii) 114.4° {101}-type twin boundary, and (iii) 54.4° {301}-type twin boundary. In the vicinity of rutile-lamellas characteristic Ti-rich precipitates were observed at high resolutions, best visible in prism orientations, as remnants of Ti-diffusion paths in surrounding hematite matrix frozen after exsolution of rutile from parent ilmenite. EDS and HAADF-STEM analyses revealed a high content of Ti in hematite in form of irregular patches, implying a diffusion controlled mechanism of transformation within the common oxygen-sublattice. We propose that hematite-rutile intergrowths formed through oxidative leaching of Fe-ions from ilmenite.

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CHARACTERIZATION OF THE HIGLY POROUS NATURAL BUILDING MATERIAL: TUFA FROM PIVNICE NATURAL HERITAGE MONUMENT

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Tufa, a calcium carbonate rock or a variety of limestone, is formed by the precipitation of carbonate minerals from ambient temperature water bodies. In areas with large deposits Tufa was used as building material in the past. Tufa was used as a building material for the graveyard in Pivnice, one of the Serbian National Heritage Monuments of exceptional significance. Today, the erosion is proving to be a great threat for the gravestones; therefore the goal of this project is to determine the best course of action for the conservation of these monuments.

The microstructure and the composition of the samples were investigated by the X-ray powder diffraction (XRD) and SEM analyses. Capillary water sorption was determined on dry samples, with constant moisture level. Subsequently we tested several commercially available synthetic hydrophobic protective coatings on the Tufa samples.

Keywords – calcite, building material, X-ray powder diffraction

PHYSICOCHEMICAL STUDY OF EARLY MODERN AGE PAINTED POTTERY FROM BELGRADE, SERBIA

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Belgrade fortress has been a subject of extensive archaeological investigations for decades. During 2008-2009. excavations at eastern part of Upper Town nearly 1000 ceramic vessels, both cooking and table vessels were found. Due to the welldefined archaeological context of this excavation site they were dated in period from the 17th-18th century. In the present study, group of 50 pottery shards from this site, classified as Austrian-period painted pottery, has been investigated to obtain information about production technology and consequently about workshops. EDXRF spectrometry measurements of investigated pottery have shown that decorations were made of non-transparent glaze based on lead and tin; lead yellow, copper green, cobalt blue pigments were mostly used. Same technique was used for determination of elemental composition of ceramic body. Obtained EDXRF results were further analyzed using pattern recognition techniques to establish compositional groups of the investigated shards. FTIR spectroscopic analysis was performed on ceramic body of investigated samples to obtain information about mineralogical composition. Chemometric tools were also applied on FTIR results, and comparison was made with analog analysis of EDXRF data. The compositional groups were established according to the most discriminate elements in the space of maximal variance. The most discriminate elements were graded as possible carrier of the information about the origin. Discriminant analysis was applied to project linear classifier in order to recognize the shards of unknown origin. Methods applied in this work can be used for effective and simple classification of pottery samples.

THE PROPERTIES OF NANOCRYSTALLINE COMPOSITE MATERIALS OBTAINED BY HIGH PRESSURE SINTERING

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The results of investigations of the microstructure and properties of nanocrystalline composite materials based on nonmetallic and metal like refractory compounds Si₃N₄/SiC, Si₃N₄/TiN and TiN/TiB₂ sintered at high pressures up to 4 GPa and high temperatures from 1000 °C to 1900 °C are presented. Peculiarities of densification and recrystallization processes of different composites during high pressure sintering and pressureless sintering are discussed. The data concerning structure, density, microhardness and fracture toughness are analyzed and compared with literature data.

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SPINEL TWINS AND SPINEL-TAAFFEITE EPITAXIES IN BeO-DOPED MgAl₂O₄ CERAMICS

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Magnesium aluminate MgAl₂O₄ (spinel) has many technological applications due to its outstanding thermal, mechanical and optical properties. In order to obtain the desired physical properties of spinel ceramics one of the main objectives is efficient control of microstructure development during processing. A detailed study of (111) twins in natural MgAl₂O₄ spinel crystals from Mogok (Burma) revealed that twinning is most probably caused by the presence of beryllium during crystal growth, causing the formation of local *hcp* stacking and the formation of twin boundary. Twinning in spinel is related to the formation of intermediate polytypic compounds in the MgAl₂O₄-BeAl₂O₄ system.

In the present work we focused on the synthesis of different polytypic compounds in the MgO-Al₂O₃-BeO ternary system in the presence of melt-forming agent PbF₂. The initial compositions were prepared in stoichiometric ratios for two end-phases MgAl₂O₄ and BeAl₂O₄, and in the ratios for intermediate ternary taaffeites: BeMgAl₄O₈, BeMg₂Al₆O₁₂ and BeMg₃Al₈O₁₆. Sintering experiments were performed in a tube furnace at 1200°C in air. We have successfully synthesized

MgAl₂O₄ spinel and BeAl₂O₄ chrysoberyl, while out of the taaffeites BeMg₃Al₈O₁₆ phase was the only stable intermediate phase at the given sintering conditions. Also we have shown that small amounts of BeO to the MgO-Al₂O₃ system results in abundant twin formation within the spinel grains. Different twin combinations were found in spinel grains, from simple contact twins to complex cyclic ones, similar to those found in natural spinel crystals. The twinning has a dramatic effect on exaggerated growth in MgAl₂O₄-based ceramics. Higher additions of BeO lead to the formation of topotaxial layers of polytypic BeMg₃Al₈O₁₆ on MgAl₂O₄. HRTEM study of spinel-taaffeite intergrowths shows an orientation relationship of [11] 0]·{111}_{sp} || [1120]· {0001}_{taf}. The formation of {111} twins in spinel is therefore not a result of accidental attachment of crystals, but has been shown to be chemically induced. This phenomenon can be used for the production of twinned spinel crystals with complex morphologies.

O - 11

THE EFFETS OF WATER CONTENT AND CHEMICAL COMPOSITION ON THE STRUCTURE AND COMPRESSIVE STRENGTH OF GEOPOLYMERS

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The effects of water content, i.e. $H_2O/(SiO_2 + Al_2O_3)$, SiO_2/Al_2O_3 and K/Al or Na/Al molar ratios on the density, open porosity, microstructure and the thermal and mechanical properties in K and Na activated geopolymers (GPs) were systematically investigated. All GPs samples were prepared from high purity metakaolin using the same mixing and curing conditions. XRD, NMR as well as alcohol immersion technique were used to characterize the structure of processed GPs. It was found that the amount of water used to process GPs is the governing factor affecting their porosity, while SiO_2/Al_2O_3 and K/Al or Na/Al molar ratios play a secondary role. The K- and Na-activated samples have similar amounts of residual water after aging for 21 days at ambient conditions, regardless of the initial water content and SiO_2/Al_2O_3 ratios. Compressive strengths were found to be strongly affected by $H_2O/(SiO_2 + Al_2O_3)$ ratio, only at higher water ratio. At low $H_2O/(SiO_2 + Al_2O_3)$ ratios, SiO_2/Al_2O_3 ratio also plays an important role, i.e. GP compressive strength increases significantly with increasing Si content. Practical implications of results of this study on tailoring thermal and mechanical properties of GPs are discussed in more detail.

FABRICATION OF ACCICULAR MULLITE BY CONTROLED OXIDATION OF MOLYBDENUM DISILICIDE

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Porous acicular mullite was fabricated by using waste $MoSi_2$ heating element and Al_2O_3 . Controlled oxidation of the pulverized heating element led to the formation of a mixture of MoO_3 and amorphous SiO_2 . This mixture was employed as both SiO_2 source and pore former. The effect of fabrication temperature on phase composition, porosity, grain morphology and compressive strength of sintered mullite was examined. Pure mullite with porosity of more than 60% and compressive strength of ~ 20 MPa was obtained at temperature as low as 1300 $^{\circ}$ C. The microstructure consisted of elongated, rectangular, prism-like grains which are known to be effective in filtration of diesel engine exhaust. The increase in sintering temperature caused the change of grain morphology and reduction in the compressive strength.

O - 13

ZINC OXIDE: CONNECTING THEORY AND EXPERIMENT

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Zinc Oxide (ZnO) is a material with a great variety of industrial applications. Those in the ceramic industry are among the most important ones, since ZnO has relatively high heat capacity, thermal conductivity and temperature stability. In particular, ZnO affects the melting point and optical properties of ceramic glazes and enamels. At ambient conditions zinc oxide appears in the wurtzite type of structure ($P6_3mc$, no. 186), while with change of temperature or pressure, it adopts the sphalerite (F-43m, no. 216) or the rock salt type of structure (Fm-3m, no. 225). Therefore, it would be of great importance to find new stable and/or metastable modifications of zinc oxide, and investigate the influence of pressure and/or temperature, and try to connect theoretical results to experimental observations.

In order to reach this goal, we performed several separate research studies, using modern theoretical methods. First, we have performed crystal structure prediction using simulated annealing (SA). The results were in good agreement with previous theoretical and experimental observations, and we have found some additional structure candidates as function of pressure. Next, we have performed calculations using the prescribed path algorithm (PP), where the connectivity among experimental structures on the energy landscape, and in particular transition states, were investigated in detail. With the results of this study we were able to understand more about the influence of temperature in ZnO and to connect our results to the actual synthesis routes. Finally, we performed calculations using the threshold algorithm (TA), where we have investigated the energy landscape of ZnO in more detail (energy barriers, influence of the size, pressure, shear force, etc.). As part of this study, we were able to obtain some new ZnO polytypes, as well as gain new insight on synthesis conditions.

O - 14

LAYERED COBALTATES AS THERMOELECTRIC MATERIALS

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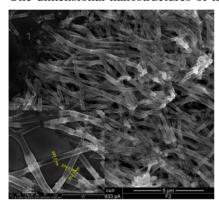
Layered cobaltates are a family of compounds with CdI₂-type layers composed of edge-sharing CoO₆ octahedra as a common structural feature. Most materials of this type contain either alkali metal ions or oxides in rock-salt type arrangement between individual CoO₂ layers. The nature of interlayer species determines average oxidation state of cobalt, which in turn influences electronic correlations and spin degeneracy within the layers giving rise to unique properties of these materials. The discovery that metallic layered cobaltate Na_xCoO₂ exhibits large thermopower spurred the research of oxides as possible thermoelectric materials and triggered numerous studies of physical phenomena within these structures. The chemistry of layered Na_xCoO₂, however, is governed by the mobility of interlayer sodium ions, which renders the crystal structure unstable in contact with atmospheric water and carbon dioxide. On the other hand the misfit cobaltates with rock-salt layers between CoO₂ sheets, while resistant to atmospheric influences, exhibit poorer transport properties. The possibility of engineering new structures based on intergrowth of alkali metal and rock-salt-type interlayers with improved chemical stability and transport properties will be discussed.

SYNTHESIS AND CHARACTERIZATION OF BiFeO₃ NANORODS AND NANOTUBES

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Bismuth ferrite (BiFeO₃) is potentially the only material which is both magnetic and highly ferroelectric at room temperature. Nanostructured BiFeO₃ is promising material for magnetoelectric and spintronic devices and photocatalytic applications. One dimensional nanostructures of multiferroic bismuth ferrite (BiFeO₃) have been



SEM images of BiFeO₃ 1D nanostructure

synthesized using a modified template methodology and characterized by a number of techniques, including XRD, SEM, TEM as well as EDX, DSC, DTA/TG, and Raman and UV-VIS spectroscopy. Factors as starting precursors, dimension of pores, annealing treatments have been considered in obtaining well crystallized 1D structures (100-250 nm in diameters and a few micrometers in length). Magnetic and electric properties were evaluated. The optical band gaps of BiFeO₃ determined nanostructures were favorable band structure allows them to be potential photocatalytic materials.

ON MODELING OF ADSORPTION OF LARGE MOLECULES ON CRYSTALLINE CERAMIC SURFACES

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Adsorption phenomena play an important role in many application areas (gas sensing, decontamination and environmental control, fuel industry, food industry, surface chemistry generally, to mention a few). Adsorption of molecules with dimensions larger than a single adsorption site (from simple cyclic hydrocarbons like benzene to complex macromolecules like DNA) is of particular importance. An often met situation is adsorption of such molecules on nanoporous surfaces of ceramic materials like metal oxides, minerals, etc. This work utilizes an accurate physicochemical model to develop a simplified but still accurate numerical model connecting adsorption kinetics with measurable variables. To this purpose a package of custom routines in MATLAB has been developed and combined with soffware for molecular modeling/editing. A brief review of the state of the art in standalone programs for various platforms (including MS Windows, Linux, iOS, Android) is given. The influence of the molecular size to adsorption on crystalline ceramics has been assessed using the ChemAxon's Marvin software and the PubChem database. Demonstrative examples are given for adsorbates like aromatic hydrocarbons or trimethyl phosphate on crystalline structures of mineral particles like maghemite, hematite and goethite. The obtained results are applicable for work with toxic and hazardous materials, in sensing, etc.

BULK NANOSTRUCTURED ZnO FOR THERMOELECTRIC APPLICATIONS

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Currently, oxide-based thermoelectric materials are investigated for high temperature energy conversion because of their advantages, such as thermally stable, non-toxic and highly resistive to oxidation at high temperature. It has been demonstrated that materials with nanoscale microstructure show an enhancement in the thermoelectric efficiency. For thermoelectric devices dense nanostructured solid bodies are required. The consolidation of nanocrystalline powder into a dense bulk body is very challenging. The high temperatures and long processing time needed for complete densification in conventional sintering inevitably results in grain growth losing the benefits of nanoscaled microstructure. In a pulsed electric current sintering (PECS), full densification can be achieved at low temperatures and short sintering time due to the simultaneous application of the pulsed electric current and uniaxial pressure on the material. In the present work, we studied the thermoelectric properties of Al doped ZnO. ZnO powders were prepared by chemical vapour synthesis. In the light of the obtained data the impact of the microstructure of nanocrystalline material on the thermoelectric properties is discussed.

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SYNTHESIS AND CHARACTERIZATION OF BaTiO₃ SOLS AND THEIR APPLICATION FOR FILMS FABRICATION BY INK-JET PRINTING

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Ink-jet printing is often used as one of the low cost methods for fabrication of ceramic thick films with broad application in microelectronics. By ink-jet printing droplets of a previously prepared sol (ink) can be deposited at predetermined locations on a substrate and after drying and thermal treatment desirable complex structures can be obtained. As it is well known that the sol characteristics determine the performances of the final product, this research is focused on preparation of different BaTiO₃ sols suitable for use in ink-jet printing. Stability of the sols with variation of the processing parameters was investigated. BaTiO₃ sols were prepared with dissolving barium-carbonate in acetic acid and subsequent addition of tetrabutyl-orthotitanate. In addition, formamide was added in the system in several concentrations to improve sol stability. Determination of viscosity, surface tension and particle size distribution, as well as infrared spectroscopy and differential thermal analysis were used for the characterization of the precursor sols. The prepared sols were printed on previously cleaned substrates using a commercial ink printing device. After the drying and heating treatment continuous films were formed. The structure and morphology of the deposited films were evaluated by X-ray diffraction, scanning electron microscopy and atomic force microscopy.

O-19

PREPARATION AND STRUCTURAL CHARACTERIZATION OF NiFe₂O₄/BaTiO₃ MULTILAYERED THIN FILMS

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In recent years multiferroic materials have attracted a lot of attention in a world of material science. Multiferroic composite films with both ferroelectric and ferromagnetic layers are very attractive due to their low losses and easy on-chip integration. In this paper multilayer thin films were prepared with NiFe₂O₄/BaTiO₃ (NFO/BTO) composition, on silicon and alumina substrates by using wet chemical deposition techniques. Two different NiFe₂O₄ sols and one BaTiO₃ sol was used for fabrication of thin films. Optimal processing conditions and deposition technique were investigated. Particle size and viscosity of precursor sols were monitored with time. For structural and phase characterization were used X-ray diffraction (XRD) and high resolution scanning electron microscopy. Fabricated films were crack-free, with thickness of about 500 nm, flat uniform surface texture and with grain size on nanometer scale. Layers of different phases can be clearly distinguished, where each layer was about 50 nm thick. Results from XRD pattern positively confirm existence of spinel and perovskite phase. Also, it was concluded that specific thermal treatment was crucial for successful fabrication.

SYNTHESIS AND CHARACTERIZATION OF SnO₂ FILMS

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Goal of this research was to deposit film of SnO_2 on alumina substrate, which is known to possess good sensing abilities for detection of reducing gases such as H_2S , CO and CO_2 . Tin oxide sols were synthesized at room temperature by hydrolysis reaction of tin chloride dissolved with ethanol as a solvent. Concentration of precursor solution was set in range from 0.5 M to 1 M, and all other processing parameters remained unchanged. Tin oxide thin films were fabricated by chemical solution deposition (dip- and spin- coating) technique on previously cleaned α -alumina substrates. Influence of various processing parameters on film thickness and surface morphology were investigated. Viscosity, surface tension and particle size of precursor sols were monitored with time. Structure of the fabricated thin films was characterized by FTIR spectroscopy, X-ray diffraction, scanning electron microscopy and atomic force microscopy. Dielectric properties of the obtained tin oxide thin films were determined too and correlate with the film microstructure.

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ECOCERAMICS AN ALTERNATIVE FOR INDUSTRIAL SILICATE WASTES

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Numerous silicate based wastes such as: coal ash, slag from steel production, municipal incineration ash, sludge resulting in hydrometallurgical process, different types of sludge from the sewage system and glass cullets or mixtures thereof have been considered for recovery in various ceramic masses. Glassceramic is a polycrystalline material with fine microstructure, which can be obtained by controlled crystallization of glass. The general process involves silicate waste vitrification, or mixtures of wastes, followed by crystallization to form glassceramic. Unlike the glassceramic products made from high purity raw materials for specific applications, those based on industrial wastes are not yet widely available commercially. This paper presents new ways to recycle three industrial silicate wastes: metallurgical slag (LF), oil drilling sludge and ash from power plants through the production of construction materials which would become a viable and successful alternative technique. In this context, the paper seeks to answer new questions about the nature and properties of a geopolymer, if this is a new binder, or new cement for concrete or a new ceramic? The geopolymers are all this and all have a common feature - the silicates. These are new materials for paints and adhesives, new binders for fiber reinforced composites, waste encapsulation, sustainable cement for concrete, which allow the development of green technologies for ceramic production. The variety of geopolymers applications encompass: heat resistant materials, decorative stone, insulation, building materials, tiles, cements and concretes with reduced CO₂ emission, composite for infrastructure fixing, etc. Although there are obvious advantages related to environmental impacts, to be obtained by recycling silicate wastes, it seems that serious efforts will be future-oriented to encourage industrial production and to ensure commercial success. Moreover, especially for toxic residues such as coal ash from incinerators there is a greater pressure from stakeholders for their inertization by an appropriate heat treatment, which will automatically move interest of production of useful products such as glassceramic obtained from vitrified residues in advance. Indeed, it is expected that a wide range of applications and commercial exploitation of ceramic waste products requiring clarification concerns toxicity of fully finished products to ensure their acceptance by the public.

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DOMESTIC RESORSES AND MATERIALS FOR FUTURE OF SERBIA

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Consumption state of refractory materials in Serbia was analyzed by using numerous data related to the amount of both refractories produced by domestic producers and products that world known companies sell in the market of the Republic of Serbia.

In the past, leading producers of refractory materials in Serbia were "Chamotte"-Arandjelovac and "Magnochrom"-Kraljevo. These large factories for production of refractory materials owned well educated stuff, equipment, supporting laboratories, mines, while Magnohrom worked on development new materials and technologies through the researches in the institute.

Today, since the acquisition by foreign companies have been expected for a long time, only mines have remained from all of the above, the equipment is in very poor condition and the number of employees is maintained at minimum. Namely, well-educated stuff mainly left the firm or they have been fired. However, a number of former employees have been turned to development of small enterprises, so nowadays there are about dozen domestic companies that are occupied by preparing the refractory materials in the grain forms, but only five of them are involved in designing and installation with its own construction stuff.

Over 600 t of alumina base cement was imported in Serbia during 2011. It means that about 2.500 t of the refractory materials in the grain forms used for production of hydraulically bonded refractories. If imports of 15.000 t of refractory castables, mortars and other mixtures as well as 900 t of other refractory products are added, it is almost 20.000 t of refractory materials in the grain forms imported. Besides that, around 10.000 t refractory bricks were imported too, so the total of 30.000 t of refractory materials were imported and installed in various thermal aggregates. Annual import of refractory materials indicates that domestic production cannot meet the needs of the domestic market. It is disappointing the fact that nowadays Serbia is unable to respond the current need for 30.000 tons, while the former production of over 100.000 t had the purpose of domestic economy usage and exports the same amount.

Burning question is the employment of well-educated people in production, testing laboratories, and consumption, but also in the civil service, as a chamber of commerce, customs and ministries. Unfortunately, well-educated people in this field today are at least presented in the places where they can offer the most; that are production, quality control, supervision of installation, monitoring the process of exploitation and innovation. For example, the most important property of refractory material, refractoriness, cannot be tested in any accredited laboratory in Serbia.

PHOTOCATALYTIC PROPERTIES OF HYDRO-AND SOLVOTHERMALY PREPARED NANOSIZED ZnO

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Two salts of Zn were used as starting materials: $Zn(CH_3COO)_2 \cdot 2H_2O$ for solvothermal and $Zn(NO_3)_2 \cdot 6H_2O$ for hydrothermal treatment. Initially, $Zn(CH_3COO)_2 \cdot 2H_2O$ was dissolved in ethylene-glycol in the presence of PVP and addition of solid NaOH, while solutions of the $Zn(NO_3)_2 \cdot 6H_2O$ and NaOH were mixed to prepare precursor suspensions. The precursors were subjected to solvo- or hydrothermal treatment at $120\,^{\circ}C$ during $18\,h$. The prepared samples are characterized by X-ray diffraction and TG/DSC analysis, while photocatalytic properties were tested according to degradation of Reactive Orange 16.

P-2

NANOPOWDERS OF CEO₂ OBTAINED BY HYDROTHERMAL METHOD FROM THE VARIOUS PRECURSORS

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In this work we have explored different ways for synthesis of nanosized CeO_2 . Four different salts of cerium were used as starting materials for the synthesis as the precursors: two salts of cerium (III): $Ce(NO_3)_3 \cdot 6H_2O$ and $CeCl_3 \cdot 7H_2O$, and two salts of cerium (IV): $Ce(SO_4)_2 \cdot 4H_2O$ and $(NH_4)_2Ce(NO_3)_6$. The precipitated precursors were washed and then subjected to hydrothermal treatment at 200 °C during 18 h. In some cases hydrothermally prepared samples were annealed at higher temperature to obtain phase-pure samples. The phase identification of the samples and analysis were carried out by X-ray diffraction, FTIR spectroscopy, and TG/DSC analysis.

CLAY AS A SOURCE FOR LOW CRYSTALLINITY IRON OXIDE SYNTHESIS

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Clay was chemically treated by aqueous solution of hydrochloric acid (weight ratio of 1:1) following the heat treatment at 600 °C for 2 h. Low crystallinity iron (III) oxide was obtained from the filtrate using ammonium hydroxide as a precipitating agent.

The influence of temperature and type of drying methods on the particle size, chemical composition and crystallinity of the obtained iron (III) oxide powder was investigated. The precipitate was dried in a four different means: at 120 °C, 30 °C, at room temperature in vacuum and at -20 °C under reduced pressure. The study of resulting powders included: crystalline phase identification by X-ray analysis, particle-size determination by DLS (dynamic light scattering) technique, determination of specific surface areas by BET, and microstructural analysis using SEM.

P-4

CHARACTERIZTION OF THE NEW CERAMIC MATERIALS OBTAINED BY THERMALLY INDUCED PHASE TRANSFORMATION OF THE K-LTA ZEOLTE

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The method of the thermal treatment of cation-exchanged zeolites (ZTIT) has proven to be suitable for the synthesis of the alkaline earth and alkaline framework aluminosilicates. In this work, we present the results of the thermally induced phase transformations of K-exchanged LTA zeolites to feldspathoid structure i.e. mineral kalsilite. The phase conversions that occurred in the range from room temperature to 1300 °C were investigated by thermal analysis (DTA/TGA), X-ray powder diffraction (XRD) and SEM/EDAX analyses. Also, we investigated the XRD pattern line broadening and the influence of the potassium cations to the microstructure parameters. The crystal structure and microstructural parameters were refined using the Rietveld method.

Keywords - ceramics, ZTIT synthesis, X-ray powder diffraction, kalsilite.

ANTIMICROBAL ACTIVITY OF SILIVER AND SILICON DOPED CALCIUM-PHOSPHATE SCAFFOLDS

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In this study, spherical particles of silicon substituted hydroxyapatite (HA) doped with different amount of silver were synthesized by hydrothermal method. Thereby silicon addition decreased the phase transformation temperature of HA into silicon-substituted α-tricalcium phosphate (α-Ca₃(PO₄)₂; α-TCP) retaining the spherical morphology of the particles and could have an influence on the biological response of a material and invoke enhanced cell adhesion, differentiation and gene expression additional doping with silver ions was performed in order to improve antimicrobial activity. Synthesized powders were used for scaffold preparation. The scaffolds were prepared by replica foam method. The antimicrobial effects of doped hydroxyapatite powders against pathogen bacterial strains Escherichia coli, Staphylococcus aureus and pathogen yeast Candida albicans were tested on powders preheated at 1150 °C and scaffolds. X-ray diffraction analysis and scanning electron microscopy confirmed complete transformation into α-TCP-HA on all scaffolds and uniform pore distribution. Quantitative test of antimicrobial activity of scaffolds showed that silicon-substituted α -tricalcium phosphate doped with silver had viable cells reduction ability for Escherichia coli, Staphylococcus aureus and Candida albicans up to 30, 28 and 41 %, respectively, related to silicon doped scaffolds, and 69, 98 and 47 %, related to control sample.

P-6

HYDROTHERMAL TREATMENT OF NANOANATASE WITH ALKALI AND ALAKALINE EARTH HYDROXIDES

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Nanoanatase powder was hydrothermally treated with LiOH, NaOH, KOH or $Ca(OH)_2$ at T = 120 °C for 8 h. In all cases, the molar TiO_2 : hydroxide ratio was identical. The obtained samples were washed with distilled water, centrifuged and

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dried at room temperature. Characterization of the samples was done by X-ray powder diffraction, TG/DTA/DSC and SEM analysis. Nanoanatase showed very diverse reactivity toward different alkali and alkaline earth cations. The final products showed different phase composition, crystallinity and microstructure. With ${\rm Li}^+$ and ${\rm Ca}^{2+}$ anatase forms different titanate structures while with ${\rm Na}^+$ and ${\rm K}^+$ it mainly retain original anatase structure.

P-7

SYNTHESIS AND CHARACTERISATION OF Sr-SiO₂ POWDERS WITH ORDERED MESOPORES AND IT POTENTIAL APPLICATIONS IN DRUG DELIVERY

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Mesoporous SiO₂ has a highly ordered structure, large surface area and pore volume – qualities that gives it an excellent drug release profile.

The aim of this study was to incorporate Sr^{2+} into mesoporous SiO_2 in order to develop a bioactive mesoporous material with an improved drug delivery profile.

A series of mesoporous $SrO-SiO_2$ species with different chemical compositions were prepared by a template-induced self-assambling method. As SiO_2 source, tetraethylortosilicate (TEOS) was used in the presence of Pluronic 123 template agent. The chemical and structural characterization of the obtained materials was realized by X-ray diffraction, scanning electron microscopy, transmission electron microscopy, thermal analysis, infra-red spectroscopy, specific surface measurements, pores size distribution and Raman and FTIR spectroscopy.

One described the effect of Sr cations on mesoporous structure and it suitable properties for drug delivery applications.

Keywords: mesoporous materials; SrO-SiO₂; drug delivery

ZnO MESOCRYSTALS FROM SOLVOTHERMAL SYNTHESIS

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Mesocrystals represent a new class of nanostructured materials, made of crystallographically aligned nanoparticles. Due to their unique structural features they have many physicochemical properties, different from nanoparticulate materials and single crystal materials, which can provide better performance in some applications. Zinc oxide mesocrystals have been synthesized by the solvothermal method at 200 °C during 4 hours from slightly basic (pH = 8) precursor (ethanolic zinc acetate solution in the presence of lithium hydroxide). XRD analysis showed that precursor solution consists of zinc acetate and zinc-hydroxy-acetate. Structural and microstructural properties were analyzed using X-ray diffraction, field emission scanning electron microscopy and transmission electron microscopy. ZnO mesocrystals are hexagonal prisms with diameters of 80 - 200 nm and lengths of 100 - 200 nm, but several larger prisms have a hole in the center. Based on characterization results we have discussed the growth mechanism of ZnO mesocrystals. Dipolar nature of ZnO and planar structure of zinc-hydroxy-acetate with free position of the acetate ions between positively charge planes play crucial role in the formation of the ZnO mesocrystals during the solvothermal reaction.

P-9

THE RAMAN SPECTROSCOPY OF TiO₂/WO₃ COATINGS FORMED BY PLASMA ELECTROLYTIC OXIDATION

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The properties of pure TiO_2 and TiO_2/WO_3 coatings formed by plasma electrolytic oxidation (PEO) of titanium in 12-tungstosilicic acid water solution have

been studied by Raman spectroscopy. The Raman spectra of TiO₂/WO₃ coatings obtained after 45, 60, 90 and 180s of PEO process, recorded at room temperature, have been compared to the spectra of pure anatase coatings. The decrease of anatase Raman modes intensities has been registered with the increase of PEO process duration. Also, the increasing intensity of WO₃ Raman modes has been ascribed to enrichment of coatings with WO₃ with the increasing time of PEO process. Moreover, the isolated regions of pure WO₃ have been detected by Raman spectroscopy in coatings obtained by 180s of PEO. The results of Raman spectroscopy have been compared to previously obtained results of by X-ray diffraction (XRD).

P-10

SYNTHESIS AND CHARACTERIZATION OF NANOMETRIC TUNGSTEN CARBIDE

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Nano-sized tungsten carbide (WC) powder was prepared by thermical treatment of mixture of tungsten powder and activated carbon cloth. The effect of different temperature, time of heat treatment and C/W ratio on obtained powder properties was studied. The reaction products were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Specific surface area was measured by Brunauer–Emmet–Teller (BET) method. It was possible to obtain pure WC after eight-hour heat treatment at temperature of 1000°C with C/W ratio 3.

STRUCTURAL AND MICROSTRUCTURAL CHARACTERIZATION OF BST CERAMICS OBTAINED BY HYDROTHERMALLY ASSISTED COMPLEX POLYMERIZATION METHOD

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Barium strontium titanate powder $Ba_{0.8}Sr_{0.2}TiO_3$ (BST) has been obtained by hydrothermal treatment of precursor solution containing titanium citrate, previously prepared by complex polymerization method, and barium and strontium acetates. The powders were calcined at $700^{\circ}C$, pressed into pellets and further sintered at $1280^{\circ}C$ using different times (from 1 to 32 h). The phase compositions of sintered samples were followed using X-ray diffractometry and EDS analysis. Microstructural properties were investigated using scanning electron microscopy. It was found that BST sintered samples contained a two-phase structure. Sintered samples underwent an abnormal grain growth, whereby some grains grow faster than the other due to the presence of two-phase structure.

P-12

FUNCTIONALIZATION OF THE TITANATE NANOTUBES WITH A SILANE COUPLING AGENT

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In order to develop new nanosized filler compatible with the thermoplastic polymer Nylon-11, trititanate nanotubes (TTNTs) were synthesized by standard alkaline hydrothermal treatment of a TiO₂ anatase powder in 10 M NaOH at 120 °C for 24 h. After the synthesis, the as-obtained nanopowder was washed differently (either with water or HCl), in order to prepare TTNTs with high and low sodium contents. Chemical functionalization of TTNTs was performed with 3-aminopropyltriethoxisilane (APTES) coupling agent using two different reaction

media (water and an ethanol/water mixture) with the aim to improve the bonding between inorganic hydrophilic fillers and hydrophobic polymer matrix. Fourier transform infrared spectroscopy (FTIR), thermogravimetry analysis (TGA), transmission electron microscopy (TEM), zeta potential and CHN elemental analyses were used to elucidate the grafting mechanism of APTES at TTNTs surface. The obtained results shown that: APTES coupling agent is bounded covalently to the TTNTs surface (Ti-O-Si bond was identified after deconvolution of the IR bands at 800-1000 cm⁻¹); grafted amount of APTES is almost independent of the reaction media; protonation of –NH₂ groups shift the isoelectric point from pH 2.4 to pH 6.6; TTNTs kept their original size and shape after silanization.

P-13

APPLICATION OF THE LAYERED TITANATES IN WATER PURIFICATION

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Titanate nanostructures (e.g. nanotube and nanosheets) possess a unique combination of physical and chemical properties due to their specific structure (high surface area, large ion-exchange capabilities and electroconductivity) that can provide a wide range of possible applications, such as those in photocatalysis, lithium batteries, sensor applications, hydrogen production and storage, water purification etc. As one kind of main organic pollutants in water, dyes have been widely used in industry and our daily life. The layered structure has advantages in making effective exchange with most cations in water, while the large surface area provides active surface for adsorption. In this work, layered titanates were synthesized by simple hydrothermal procedure in highly alkaline conditions, starting from commercial titania powder (Degussa P25). To examine the adsorption and the photocatalytic activity of the synthesized layered titanates, methylene blue (MB) was employed as a target compound in response to visible light at ambient temperature. The morphology and structures of as-prepared samples were investigated by transmission electron microscopy, X-ray diffraction, Raman spectroscopy and N₂ adsorption/desorption. The concentration change of MB due to adsorption and photocatalysis was monitored by visible spectrophotometer at the maximum absorption wavelength of MB ($\lambda = 664$ nm). Prepared layered titanates have high values of the specific surface areas (higher than 300 m²/g) which make them good candidates for different types of applications, especially for water purification, since these materials showed remarkable adsorption capacity for MB removal.

PROPERTIES OF MULTIDOPED CeO₂ NANOPOWDERS SYNTHESISED BY GNP AND MGNP METHODS

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Multidoped $Ce_{0.8}Nd_{0.01}Sm_{0.04}Gd_{0.04}Dy_{0.04}Y_{0.07}O_{2-\delta}$ nanopowder, with constant composition, was synthesized by glycine nitrate procedure (GNP method) and modified glycine nitrate procedure (MGNP method) by varying the ratio of glycine (as fuel) to nitrate (as an oxidant) in the interval of 0.6 - 1.67. The influence of different relations glycine/nitrate on properties of nanopowders was investigated by XDR method, Raman spectroscopy, BET and SEM methods. Parameters such as crystallite size, microstrain, the degree of agglomeration, surface area, and SEM analysis show that the modified glycine nitrate procedure is the better technique than GNP method for the manufacturing nanopowders with the good performance.

P-15

SOLID-STATE SYTHESIS AND CRYSTAL GROWTH OF PURE VALENTINITE ANTIMONY(III) OXIDE

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Antimony(III) oxide is inorganic substance of great importance in industry. Its two polymorphs, senarmontite and valentinite, have different properties and therefore different area of application. Obtaining a single-phase Sb₂O₃ with

thoroughly determined morphology could be essential for further processing. The reaction was performed in an agate mortar by grinding a mixture of SbCl₃ and NaOH as solid-state precursors. After 3 hours standing at ambient conditions, the white slurry was rinsed several times with water and ethanol. The product was further dried at 60 °C for 1 hour. Obtained powder was characterized by XRD and by microstructural analysis using SEM, FESEM and TEM. X-ray analysis and SEM followed by HRTEM and SAED confirmed well-crystallized singe-phase valentinite Sb₂O₃. Morphological examination showed submicronic prismatic forms of Sb₂O₃ crystals with hexagonal, lozenge and deformed octagonal basis that are typical for this mineral. Crystal shapes were analyzed through the appearance of visible crystal faces and their comparison to the available mineral data.

P-16

ENCAPSULATION OF MAGNETITE NANOPARTICLES IN HUMAN SERUM ALBUMIN

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At present day biocompatible materials arouse great interest among scientist. They find uses at every branch of medicine – as implants, drug carriers or imaging contrasts. As of late, the accent is put on multifunctionality due to the need to minimize the dose of agent to which the patient is subjected. We present here a study of magnetite nanoparticles embedded in albumin microspheres. The albumin microspheres show great promise for drug targeting, and magnetite nanoparticles doped with rare earth metals can be used as imaging contrast and as heating medium in hyperthermia therapy. The microspheres are synthesized in a two-step process. First, the ferrofluids based on doped magnetite nanoparticles are synthesized, characterized and their heating potential (SPA) is measured. Then, the albumin microspheres, which entrap nanoparticles, are created. Dual Beam microscopy showed that microspheres have very large effective surface and that the nanoparticles form clusters inside the microspheres.

CESIUM ADSORPTION AND PHASE TRANSFORMATION OF CLINOPTILOLITE

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This work was focused to provide promising solid-phase materials that combine relatively inexpensive and high removal capacity of some radionuclides from low-level radioactive liquid waste. Cesium adsorption from aqueous solutions onto clinoptilolite and their thermal transformation in this work was investigated. All samples were characterized by scanning electron microscopy and X-ray diffraction analysis. The elemental composition of the zeolitic material clinoptilolite was also determined with XRF. The content of Al and Si was determined using ICP-OES. The cesium adsorption by zeolites was carried on in a batch system where a contact time was 24 h. The cesium was detected by atomic absorption spectrometer. The results for Cs adsorption efficiency of clinoptilolite are very satisfactory, especially for concentration of 10 mg/l, but this material didn't change into pollucite stable phase.

P-18

EXAMINATION OF NANOSTRUCTURED Ca_{1-x}Gd_xMnO₃ (x=0.05; 0.1; 0.15; 0.2) OBTAINED BY MODIFIED GLYCINE NITRATE PROCEDURE

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Starting $Ca_{1-x}Gd_xMnO_3$ powders (x=0.05, 0.10, 0.15, 0.20) were prepared by combustion of solutions containing mixture of glycine with metal nitrates in their appropriate stoichiometric ratios. The so-obtained powders were annealed at the temperature of 850 $^{\circ}$ C to 950 $^{\circ}$ C for 10 minutes to produce final nanostructured

samples with the average nanoparticle size of about 20 nm. Properties such as phase evolution, lattice parameters, chemical composition and magnetic properties were monitored by DTA, X-ray diffraction, SEM/EDS and magnetic measurements. The possibility of incorporation of Gd ions in the positions A of the perovskite structure was investigated by X-ray methods. Influence of Gd on unit cell volume of the perovskite compounds, occupation numbers and distances between atoms were analyzed by Rietveld refinement. Microstructure size-strain analysis was performed, as well. The results revealed that Gd entered positions A in the structure. It revealed that synthesized material is single phase of the orthorhombic-perovskite structure described by Pnma space group. Differential thermal analysis (DTA) revealed phase transition at ≈918°C. Magnetic measurements show that electron doping by Gd³⁺ ions substantially changes CaMnO₃ antiferromagnetic (AFM) behaviour. After introduction of Gd³⁺ ions, significant ferromagnetic (FM) component appears due to an emergence of double exchange interaction between Mn³⁺-Mn⁴⁺ ions. This resulted in appearance of a low temperature plateau in field cooled (FC) magnetization as well as in emergence of hysteresis loop with the relatively high coercivity up to 2300 Oe.

P-19

PREPARATION OF YMnO₃ POWDER FROM POLYMERIC PRECURSORS

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Multiferroics are the vast group of materials which exhibit both ferromagnetic and ferroelectric properties in the same phase. This specific feature makes them an interesting subject of scientific research. The aim of this work was to prepare yttrium manganite (YMnO₃), a well-known multiferroic material by chemical synthesis. Polymerizable complex method, a modification of Pechini method, was used for the synthesis of YMnO₃. Starting materials were Mn(CH₃COO)₂ and Y(NO₃)₃, citric acid and ethylene glycol. The precursor solution, which contained polymer network with Mn(III) and Y(III) ions coordinated by citric acid, was slowly evaporated for five days. Afterwards, the obtained resin was thermally treated at 900°C for 10 h. The results of TEM and FE-SEM analysis indicate that particle size is above 100 nm. According to results of XRD analysis, hexagonal YMnO₃ was

obtained. No obvious reflections that would indicate the existence of orthorhombic YMnO₃ or other phases were detected. However, the TEM analysis indicates that small amount of o-YMO is present in the powder. Magnetic measurements show that T_N is 42 K, which suggests the presence of mixed valence manganese state in the obtained samples. The minor magnetic hystereses and very low coercive fields indicate that the samples are basically antiferromagnetic with weak ferromagnetism.

P-20

THE EFFICIENCY OF PURE AND La-DOPED ANATASE NANOPOWDERS SYNTHESIZED BY SOL-GEL AND HYDROTHERMAL METHOD IN PHOTOCATALYTIC DEGRADATION OF ALPRAZOLAM

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Nanopowders of pure and La³⁺-doped anatase were synthesized by sol-gel and hydrothermal methods. In both methods the hydrogel was synthesized by hydrolysis of TiCl₄ at 0 °C. The doping of TiO₂ was performed by using of LaCl₃·7H₂O with 0.5 and 1 mol %. The sol-gel route involves the hydrogel conversion into its ethanolgel, drying of obtained alcogel at 280°C and calcination at temperature of 550°C for 4 h. In hydrothermal method the hydrogel was placed in autoclave at 200°C for 24 h.

Structural properties of nanopowders were analyzed by X-ray diffraction (XRD) spectroscopy. Dependence of structural and morphological characteristics of nanopowders on synthesis methods and La³⁺ content is also investigated by the Raman spectroscopy. Very intensive modes observed in the Raman spectra of all nanopowder samples are assigned to anatase phase of TiO₂.

Synthesized nanopowders (1 mg cm⁻³) were tested for their photocatalytic activity in the degradation of alprazolam (0.03 mmol dm⁻³), a benzodiazepine derived from 1,4-benzodiazepine of new generation mainly used to treat anxiety disorders. Nanopowders synthesized by hydrothermal method showed a higher photocatalytic activity and a little dependence on the content of La³⁺, in contrast to nanopowders synthesized by sol-gel method.

COMPARATION OF NATURAL RADIOACTIVITY AND PHYSICO-CHEMICAL PROPERTIES OF CLINIOPTIOLITE AND SYNTHETIC ZEOLITE

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The content of naturally occurring radionuclides ²³⁸U, ²³²Th and ⁴⁰K in two natural clinoptilolite (originated from sites in Serbia and Bosnia and Herzegovina) and synthetic zeolite A4 were measured. The obtained results and the effect of structural changes caused by physicochemical properties of zeolite are presented in this paper. The structure has been characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and X-ray fluorescence. The specific activity of ²³⁸U, ²³²Th and ⁴⁰K of different samples was determined by gamma spectrometry using the HPGe semiconductor detector and obtained values ranged from 28 to 44 Bqkg⁻¹ for ²³⁸U, from 59.4 to 71.4 Bqkg⁻¹ for ²³²Th and from 335 to 517 Bqkg⁻¹ for ⁴⁰K.

P-22

SYNTHESIS AND CHARACTERIZATION OF Fe-DOPED MULLITE

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Amorphous powders which composition coresponds to Fe-doped 3:2 mullite (3Al₂O₃·2SiO₂) powders were prepared by sol-gel combustion process using ethanol-water solutions of TEOS, Al(III) nitrate, Fe(III) nitrate and urea. The gel was heated on a hot plate in order to evaporate solvents and initiate combustion

process. The obtained powders were heat treated at 800 °C for 4 h to remove retained organic substances. Afterwards, the powders were uniaxially pressed into pellets and sintered at 1550 °C for 4 h to produce mullite solid solutions. The obtained compositions with up to 15 wt.% of Fe₂O₃ were investigated. XRD analysis confirmed that the powders were amorphous while sintered samples depicted single mullite phase. Also, the lattice parameters of mullite increase with increasing Fe content due to replacement of Al^{3+} - by larger Fe³⁺ -ions in crystal structure. TGA/DSC analysis showed a decrease of crystallization temperature of Fe-doped mullite. Density of sintered samples have increased with enhenced Fe content. Microstructure and composition of powder particles as well as sintered pellets were examined by SEM and EDAX. SEM images indicate that powder particles are highly agglomerated while the grains of sintered pellets have a rod-like shape.

P-23

NANOSTRUCTURED Fe₂O₃/TiO₂ THICK FILMS

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Thick films of nanostructured pure TiO_2 , α -Fe₂O₃, Fe₂O₃/TiO₂ (ratio 2:3 and 3:2) and a hetero-junction in the form of a TiO_2 layer over a Fe₂O₃ layer have been fabricated by screen printing technology on a glass substrate. The pastes used for film preparation were obtained by adding an organic vehicle to the oxide powders together with a small percentage of binding glass frit. Samples were dried up to 100° C and sintered at 650° C/60 minutes. Structural, morphological and optical studies have been carried out using XRD, SEM, EDS analysis and UV/Vis spectroscopy. Fe₂O₃/TiO₂ thick films had a homogenous nanostructure and no new compounds were formed. Indirect band gaps were determined from the measured transmission spectra.

SYNTHESIS OF POROUS COMPOSITE CERAMICS BASED ON Al₂O₃/YAG

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Alumina/YAG composite was obtained by using Alumina powder compact and wather solution of aluminium nitrate nonahydrate, Al(NO₃)₃·9H₂O and yttrium trinitrate hexahydrate, Y(NO₃)₃.6H₂O. Alumina powder compact was pressed with three different pressures: 5, 10 and 15MPa and sintered at five different temperatures: 1000, 1200, 1300, 1400 and 1550°C. Dribbling solution in vacuum, drying at 120 °C and calcination at 700 °C was repeated six times to increase content of yttria. Later, samples were thermaly shocked at temperature 50 °C lower than inicially. The yttria-alumina system contain four phases Al₂O₃, YAG, YAP, YAM in different ratio, depending on initial pressure and temperature of sintering. The phase composition and morphology of the samples were observed by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

P-25

THE SORPTION BEHAVIOR OF INTERSTRATIFIED MONTMORILLONITE/KAOLINITE CLAY AND CLAY/ TiO₂ COMPOSITE

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The sorption behavior of 19 h milled interstratified montmorillonite/kaolinite clay and its composite with 20% TiO₂ was investigated. The microstructure changes of samples have been investigated by XRD, PSD and SEM and correlated with

sorption behavior. The results showed that composite has a higher removal efficiency in range of pH<4 in comparison with raw clay milled for 19h. The best improvement in removal efficiency is expressed for Pb (II), afterwards for Cu (II) and Zn (II) and the least for Cd (II).

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STRUCTURE AND MORPHOLOGY OF SILVER IN Ag/C HYBRID OBTAINED BY HYDROTHERMAL ROUTE

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Ag/C hybrids were produced by the redox of fructose and silver nitrate (AgNO3) in the presence of various acids and base which acts as ionic liquids, under hydrothermal condition. Carbon microspheres encapsulated Ag nanoparticles which structure and morphology depends on the ionic liquid. The products were characterized by XRD, and SEM and energy dispersive X-ray spectroscopy (EDX). Morphology of these silver nanostructures could be varied by changing the reaction conditions.

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PARTICLE EROSION OF CERAMIC COATING ON ALUMINIUM FOAM

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Aluminium foam is, principally, a composite material consisting of aluminium or aluminium alloy matrix and of pores filled up with a gas distributed throughout the matrix. This unique structure possesses an unusual combination of properties, such as low thermal conductivity, high impact energy absorption capacity, very high specific toughness and good acoustic properties. One of the disadvantages of

aluminium foam is low wear resistance. Particle erosion is wear loss from surface of solid body because of relative motion of fluid with solid particles. In this paper, samples of aluminium foams with different porosity were thermal sprayed with two different ceramic coating (on the basis of Al_2O_3 and ZrO_2) and eroded with SiO_2 particles. Test results were presented in loss of mass after the testing and compared with erosion resistance of thermal sprayed coating on the basis of molybdenum.

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INFLUENCE OF MICROALLOYING ELEMENTS ON THE SURFACE ACIDIC-BASE AND STRUCTURAL CHARACTERISTICS OF CERAMICS OBTAINED BY SINTERING OF ALUMINOSILICATE BASED COMPOSITE PARTICLES

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Composite particles (coated particles) are composed of solid phase covered with thinner or thicker layer of another material. By application of these coatings - layers, the surface characteristics of the initial solid phase are modified and sintering conditions could be better controlled. Composite particles can be obtained in several ways and in this study the procedure of Fe³⁺ precipitation onto dispersed particles of bentonite was applied. Subsequently, material coated with hydrated iron oxides was microalloyed by two aliovalent metal cations (Ca²⁺ and Sn⁴⁺) and then sintered in order to obtain two types of ceramics. Gran plot method and XRD technique were employed to *characterize ceramics* acidic-base and structural properties. In either case, a microalloying and sintering of composite particles cause crystal grain surface layer amorphization and the creation of clusters and non-stoichiometric phases. Ceramic microalloyed with Sn⁴⁺ possesses significantly higher concentration of surface acid-base sites than its counterpart microalloyed with Ca²⁺. Acid-base activity of ceramics manifests itself in contact with the water by changing its pH during interaction.

ECAP AS CONSOLIDATION METHOD FOR PRODUCTION OF ALUMINIUM POWDER COMPOSITES WITH CERAMIC REINFORCEMENT

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Aluminium based metal matrix composites are rapidly developing group of materials due to their unique combination of properties that include low weight, elevated strength, improved wear and corrosion resistance and relatively good ductility. This combination of properties is a result of mixing two groups of materials with rather different properties with aluminium as ductile matrix and different oxides and carbides added as reinforcement with Al₂O₃, SiC and ZrO₂ being most popular choice of reinforcement material. One of the most common methods for producing this type of metal matrix composites is powder metallurgy since it has many variations and also is relatively low-cost method. Many different techniques of compacting aluminium and ceramic powders have been previously investigated. Among those techniques equal channel angular pressing (ECAP) stands out due to its beneficial influence on the main problem that arises during powder compaction and that is a non-uniform distribution of reinforcement particles. This paper gives an overview on ECAP method principles, advantages and produced powder composite properties.

PROPERTIES OF ANODE-ELECTROLYTE BI-LAYERS OBTAINED BY SLURRY SPIN-COATING TECHNIQUE

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In this study microstructural and electrochemical properties of anode-electrolyte bi-layers NiO-BaCeZrY||BaCeZrY| were investigated. Electrolyte powders were obtained by citric-nitric auto-combustion method and cermet anode powders were synthesized by mixing of oxides. Electrolyte films BaCeZrY were deposited on the cermet anode substrates NiO-BaCeZrY using slurry spin-coating method. SEM and XRD analysis were used for characterization of samples after co-sintering. Obtained electrolyte films were dense, single phase systems, with thicknesses around 10 μm . Microstructures of anodes showed homogeneous distribution of constitutive phases and porosity. Sintered samples were reduced and their microstructural and electrical properties before and after reduction were compared and analyzed using XRD and SEM analysis. Electrochemical impedance spectroscopy measurements in wet hydrogen confirmed their applicability as of anode substrates, low anode-electrolyte interfacial resistance, high density of electrolyte films and their consequent lack of permeability to gas.

SYNTHESIS AND CHARACTERIZATION OF NANO BIO-CALCIUM PHOSPHATES

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Calcium phosphates such as octacalcium phosphate (OCP, $Ca_8H_2(PO_4)_6 \cdot 5H_2O$), hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(HO)_2$), brushite (CaHPO₄·2H₂O) and monetite (CaHPO₄) are of great scientific interest in the field of biomaterials. HAP is the main constituent of bone, dental calculi and enamel. OCP is structurally similar to HAP, and is a precursor for formation of HAP in bone, also it has very good biodegradabile and osteoconductive characteristics. Brushite is a biological mineral that occurs in bones and teeth and as a transitional phase in the crystallization of HAP. It is also used as a CaP cement, and as an abrasive in toothpaste. When bushite loses its structural water it transforms to monetite.

The preliminary objective of this study is synthesis and characterization of these CaP phases. Materials were obtained by titration of the solution (CH₃COO)₂Ca·H₂O with the solution NaH₂PO₄·H₂O in different molar ratios, under constant stirring, and temperature around 60 °C. Brushite forms at pH around 5, and at pH around 7 OCP and HAP form. This was confirmed by XRD and Raman spectroscopy.

Pure brushite nanomaterial was obtained, and the crystallite size was modified by grinding in the vibrating mill. The sample was ground five times for 2.5 minutes and analyzed by XRD between grinding. Particle sizes were determined from the XRD patterns using Scherer equation. After about 5min of grinding an increase in temperature causes a phase transformation from brushite to monetite.

BET method indicates that synthesized brushite is micro porous. After 5 minutes of milling brushite is mezzo porous, these results were confirmed by SEM images.

Rietveld refinement showed that we didn't get pure OCP. Synthesized OCP was examined also by SEM method.

STRUCTURAL PROPERTIES OF HYDROCHAR OBTAINED BY HYDROTHERMAL TREATMENT OF FRUCTOSE

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A carbon-rich solid product, here denoted as hydrochar, has been synthesized by hydrothermal treatment from fructose with HNO $_3$ at temperature of 140°C and the same time of reaction with four different mass of sample. pH value for every sample was the same, 0.99. The formation of the carbon-rich solid through the hydrothermal carbonization of fructose is the consequence of dehydratation reactions. This material is made up of spherical micrometer-sized particles that have diameter in the 4-10 μ m range, which can be modulated by modifying the synthesis conditions, which in this case is concentration of the fructose in solution. The best results are given with smaller concentrations of fructose. Spherical particles have more regular shape and they are less agglomerated.

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HYDROTHEMAL CARBONIZATION OF PLANE TREE SEED

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A carbon-rich solid product has been synthesized by the hydrothermal carbonization of plane tree seed. This material can be modulated by modifying the synthesis conditions such as the concentration of the aqueous solution or by adding different acid or bases, the temperature of the hydrothermal treatment, the reaction time, etc. This carbon materialss were characterized by, X ray diffraction, scanning electron microscopy and/or nitrogen adsorption/desorption isotherms measurements. It was noticed that all mentioned process parameters affected structure and characteristics of obtained carbon material.

INFLUENCE OF DOPANTS ON BARIUM BISMUTH TITANATE ELECTRICAL PROPERTIES

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The Aurivillius structure has capability to host ions of different size, so a large number of different dopants can be accommodated in the BaBi₄Ti₄O₁₅ (BBT) lattice. It was detected that various substitutions of Bi³⁺ and Ti⁴⁺ ions can affect the change of microstructure and electrical properties of barium bismuth titanate ceramics. Doping of BBT ceramics is very important due to possibility to obtain materials with required characteristics.

In this work, pure and niobium and lanthanum doped barium bismuth titanate powders were prepared by conventional solid state method, according to formulas $BaBi_{4\text{-}x}La_xTi_4O_{15}$ and $BaBi_4Ti_{4\text{-}5/4x}Nb_xO_{15}$ (x=0.05). Obtained powders were uniaxially pressed and sintered at different temperature depending on the composition.

The influence of dopant type on structure change, grain size reduction and microstructure development was analyzed. XRD measurements showed formation of orthorhombic BBT crystal structure without presence of secondary phase in doped samples. Dopants had influence on shifting of temperature phase transition peaks to the lower temperatures, broadening of ε - T curves and increasing relaxor behavior of phase transition. Temperature dependence of the electrical conductivity pointed out that niobium as a donor dopant decrease conductivity and lanthanum as a isovalent dopant increase conductivity of BBT ceramics. Obtained results were analyzed in the frame of the influence of the grain and grain boundaries contribution to the dielectric behavior through impedance spectroscopy.

NANOSTRUCTURE AND PHASE ANALYSIS OF SPARK PLASMA SINTERED COMPOSITE POWDER OF ZrC AND β-SiC WITH LIYO₂

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The aim of this research is to evaluate the suitability of spark plasma sintering (SPS) in sintering of ZrC/β -SiC composite powder in the presence of $LiYO_2$ as sintering additive. The composite powder was obtained by carbothermal reduction of natural mineral zircon, $ZrSiO_4$. The composite material fabricated by SPS process was characterized by the X-ray diffraction (XRD) and Raman analysis. Microstructure/chemical analysis was conducted by using scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS). An insight into the phase composition change of material surface was obtained from AFM phase images by generating phase distribution. The material was homogeneously distributed and some grains were 10 to 15 nm in diameter. Fracture toughness and Vickers hardness of the composite material were found to be 5.07 MPam $^{1/2}$ and 20.7 GPa respectively. Based on the obtained results, it can be concluded that ZrC/β -SiC composite is promising material for structural application.

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THE INFLUENCE OF BORIC ACID ON SYNTHESIS OF POROUS SILICA CERAMIC

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Porous ceramics based on silicon dioxide was prepared using inexpensive method and starting raw material, clay (surface coal mine Kolubara, Serbia). Boric acid was used as binding and sintering aid. Content of boric acid was varied up to 1

wt%. Clay was purified using heat treatment (600 °C, 2 h) and aqueous solution of HCl (weight ratio of 1:1). The powder was compacted using low different pressures of 20, 30 and 40 MPa. Pressed samples were sintered for 4 h at 850, 1000, 1150 and 1300 °C.

X-ray diffraction (XRD) and scanning electron microscopy (SEM) measurements were employed to characterize the phases and microstructure of obtained ceramics. Also a measurement of densities and porosities by immersion technique according to Archimedes principle was used.

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BIOMIMETIC SYNTESIS AND PROPERTIES OF CELLULAR SIC

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Mechanical properties and microstructure of SiC ceramics derived from wood were investigated. Biomorphic silicon carbide were prepared by infiltration of tetraethyl-ortosilicate into a carbon template derived from five kinds of wood (alder, walnut, wild cherry, oak and ash) at room temperature. Infiltration was followed by carbothermal reduction at three temperatures in N_2 . The carbon template and bioSiC samples were characterized by X-ray diffraction and scanning electron microscopy. Mechanical properties of biomorphic SiC were investigated by three-point bending, compression and hardness testing. Bending and compressive strength increased with increasing silicon content. The mechanical properties showed correlation with initial density of the wood.

THE KINETICS SINTERING OF FINAL FLOTATION WASTE FROM RTB BOR

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In the process of flotation enrichment and metallurgical processing of smelter slag from RTB Bor, in addition to copper concentrate, which is transported to purification, the resulting FFW (final flotation waste) appears, which is transported to compaction, and after that, it is deposited to the tailings. These dumps are large areas of degraded land, and they are permanent polluters of soil, water and air. To discuss the application of FFW in the manufacturing of new materials from the glass-ceramic group, besides elementary addition, phase and mineral composition were examined as well as thermal properties (the temperature interval of sintering, plastic deformation and melting) whose knowledge is necessary for usage in the production of glass ceramic. The paper presents the examination of kinetics of sintering FFW by measuring the change of sample contraction over time, at constant temperature. The obtained results indicate that the kinetics of liquid-phase development and the sintering kinetics FFW depend on granulometric distribution. The theoretical model fits quite well the experimental data.

EFFECT OF HIGH PRESSURE SINTERING ON THE MICROSTRUCTURE AND PHYSICAL-MECHANICAL PROPERTIES OF BORON CARBIDE

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Sintering under high pressure, in contrast to hot pressing, accelerates the process of consolidation of powder and allows fabrication of a pore-free, fine-grained ceramics with high performances without any sintering additives. The influence of temperature and time of sintering under high pressure of 4 GPa on the microstructure and physical-mechanical properties of boron carbide was studied. The starting powders of different dispersion were used. Nonmonotonic dependence of the microhardness of B_4C samples on sintering time was found. Ceramic composite material with density close to the theoretical value and high physical-mechanical properties was obtained.

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DENSIFICATION OF MICRO- AND NANOCRYSTALLINE SIC WITHOUT ADDITIVES UNDER HIGH PRESSURE

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Nanocrystalline materials based on silicon carbide are interesting in the context of the expectation of their good physical-mechanical characteristics and thermal stability. Densification of α - SiC and β -SiC powders with different particle size

varying from 25 nm to 5-7 μ m at pressure of 4 GPa and temperatures of 1500-2000°C was studied. Highly dense ceramics (99%) was obtained. It was found that microhardness of ceramics increases with increasing dispersion of starting powder. The maximum value of microhardness was 37 GPa for β -SiC ceramics.

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SINTERING OF NANOCRYSTALLINE CeO₂ UNDER HIGH PRESSURE

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 ${\rm CeO_2}$ nanopowders with different particle size were sintered under high pressures up to 4 GPa at temperatures from 1100 °C to 1500 °C. Starting nanopowders were prepared by self propagating room temperature synthesis (SPRT) and by method of precipitation in urea and subsequent calcination at temperature of 1200 °C. The particle size was 3-5 nm and 70 nm respectively. XRD analysis and SEM were used for the investigation of microstructure of sintered samples. The density and electrical resistance of these samples were also studied.

CAVITATION EROSION OF SILICON CARBIDE-CORDIERITE CERAMICS

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Covalent bond in SiC ceramic request high temperature for sintering. In order to decrease sintering temperature of SiC ceramics we used reactive sintering method. SiC particles were joined with cordierite phase between them. We have created insitu SiC/ cordierite composite at 1300°C. Cordierite precursor was made from commercially available spinel, alumina and quartz. Composite material was made from 70wt% of SiC and 30 wt% of cordierite.

The fluid dynamic system of the experimental methodology was used to produce ultrasonic erosive wear. Mass loss and level of degradation were measured before and during the experiment. Level of degradation of the samples, average erosion ring diameter and average erosion area were monitored using Image Pro Plus program for image analysis. Obtained results pointed out that after 150 minutes sample exhibited excellent erosion resistance compared to metallic and ceramic samples. Level of degradation did not overcome 7.5 % of destruction measured from original surface.

Keywords: ceramics composite, surface erosion, image analysis, erosion ring area and diameter

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NATURAL RADIOACTIVITY IN IMPORTED CERAMIC TILES

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Ceramic tiles are one of the commonly used decorative building materials. Body of ceramic tiles is a mixture of different raw materials including clays, quartz materials and feldspar. The body may be glazed or left unglazed. Due to the presence of zircon in the glaze, ceramic tiles can show natural radioactivity concentration significantly higher than the average values for building materials. This study contains a summary of results obtained by a survey on imported ceramic tiles which were analyzed in radiation and environmental protection department, Institute Vinča. The survey consisted of measurements of concentrations of natural radionuclides using gamma spectrometer. Based on the obtained concentrations, gamma index, radium equivalent activity, the indoor absorbed dose rate and the corresponding annual effective dose were evaluated to assess the potential radiological hazard associated with these building materials.

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THE RELATIONSHIP BETWEEN FLY ASH-BASED GEOPOLYMER STRENGTH AND MAJOR STRUCTURAL ELEMENTS

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Geopolymers are relatively new type of inorganic binder materials, which can partly substitute Portland cement. Application of geopolymer binders is very favorable from ecological point of view for two reasons: because of considerably lower amount of carbon dioxide formed during production of geopolymer binders (compared to Portland-cement production) and because waste materials, such as fly

ash, can be used as raw material for geopolymers production. One of the most limiting factors for wider use of geopolymers is the fact that production of fly ash-based geopolymers often requires curing at elevated temperature (up to 100°C). This article is focused on investigation of the strength development and changes in the microstructure of fly ash-based geopolymers cured at room temperature up to 180 days. The differences in microstructure between geopolymer samples at different ages were characterized by scanning electron microscopy (SEM/EDS) and correlated to the mechanical properties. It was established that the most significant geopolymer strength gain as well as the greatest microstructural changes occurred within the first 28 days of reaction. After this initial period, less significant changes of fly ash-based geopolymer strength and microstructure were observed.

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SYNTHESIS PROCEDURE AND PROPERTIES OF NiFe₂O₄ – BaTiO₃ COMPOSITES

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NiFe₂O₄ (NF) powder was prepared by auto combustion method starting from nickel and iron nitrates. After the process of self-ignition, fine precursor powder was thermally treated and forming the nickel ferrite powder. XRD analysis proved the formation of well crystallized nickel-ferrite cubic spinel structure.

Cubic barium titanate (BT) powder was prepared by soft chemical method (modified Pechini process).

Composites (NF-BT) with the general formula x NiFe₂O₄ – (1-x) BaTiO₃ (x = 0.2, 0.3, 0.5) powders were prepared by mixing previously obtained powders of nickel ferrite and barium titanate in planetary ball mill. As a milling medium were used tungsten carbide balls and iso-propanol. Powder was pressed and sintered at 1170 °C for 4 h and from X-ray measurements the presence of NF and BT phases was detected. No secondary phases were found. Magnetic measurements of composite materials were carried out. Saturation magnetization moment of composite materials decrease with barium titanate amount and the fields at which saturation occur increase with BT content. The coercivity H_C (Oe) increases with barium titanate concentration in obtained multiferroic material.

CHEMICAL STABILITY ENHANCEMENT OF DOPED BaCe_{0.9}Y_{0.1}O_{3-δ} AS AN ELECTROLYTE FOR PROTON CONDUCTING SOFCs

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 $BaCe_{0.9-x}Nb_xY_{0.1}O_{3-\delta}$ and $BaCe_{0.9-x}Ta_xY_{0.1}O_{3-\delta}$ (where $x=0.01,\ 0.03$ and 0.05) powders were synthesized by solid-state reaction method to investigate the influence of dopant and concentration on chemical stability and electrical properties of the sintered samples. The dense electrolyte pellets were formed from the powders after being uniaxially pressed and sintered at 1550°C for 5h. The electrical conductivities determined by impedance measurements in temperature range of 550-750 °C in wet hydrogen atmosphere showed a decreasing trend with increase of Nb and Ta content. On the other hand, stability of the sintered samples treated in 100% CO₂ at 700 °C for 5h determined by X-ray analysis was enhanced with increased concentrations of Nb and Ta. It was found that BaCe_{0.87}Nb_{0.3}Y_{0.1}O_{3-δ} is the optimal composition that satisfies the opposite demands for electrical conductivity and chemical stability, reaching 8.010⁻³ Sm cm⁻¹ in wet hydrogen at 650 °C compared to 1.0·10⁻² Sm·cm⁻¹ for undoped electrolyte. Similar results obtained by doping with Nb and Ta can be explained by almost equal size and same valence of Nb and Ta cations, as well as their similar electronegativities. The electrolyte characteristics are strongly dependent on these properties, whereas doping with Nb showed slightly higher conductivities for each dopant concentration.

PREPARATION, CHARACTERIZATION AND INVESTIGATIONS OF STRUCTURAL, MAGNETIC AND ELECTRICAL PROPERTIES OF NANO SIZE ZINC FERRITE

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Zinc ferrite, $ZnFe_2O_4$ was prepared by a soft mechanochemical route from a mixture of (a) $Zn(OH)_2$ and α -Fe₂O₃ and (b) $Zn(OH)_2$ and Fe(OH)₃ powders in a planetary ball mill for 18 h. The powder samples were sintered at 1100 °C for 2 h and were characterized by X-ray diffraction (XRD), Raman spectroscopy, magnetic measurements and scanning electron microscopy (SEM). The sample obtained from mixture (a) has the cation inversion degree 0.3482 and the sample obtained from mixture (b) 0.4. Magnetization measurements were confirmed that the degrees of the inversion were well estimated. Comparison with published data shows that used method of synthesis gives samples of $ZnFe_2O_4$ with extremely high values of saturation magnetizations: sample (a) 78.3 emu/g and sample (b) 91.5 emu/g at T = 4.5 K. Impedance spectroscopy techniques were used to study the effect of grain and grain boundary on the electrical properties of the prepared samples. A difference in dielectric constant (ϵ) and dielectric loss tangent ($\tan \delta$) of $ZnFe_2O_4$ samples obtained by the same methods but starting from different initial components was observed.

NONSPECIFIC REVERSIBLE ADSORPTION PROCESSES AND THEIR FLUCTUATIONS IN MEMS BIOSENSORS BASED ON PIEZOELECTRIC CERAMIC MATERIALS

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MEMS (microelectromechanical systems) devices based on piezoelectric ceramic materials offer a promising technology platform for highly sensitive biological sensors. One class of these devices is film bulk acoustic wave resonators (FBAR). For biosensing applications one FBAR's surface is coated with functionalizing molecules, serving as specific binding sites for molecules to be detected. Adsorption of target molecules changes the resonant frequency of a resonator which is related to the adsorbed mass of target molecules on its surface. Binding of biomolecules is often reversible, causing fluctuations of the resonant frequency, known as the adsorption-desorption fluctuations, which affect a sensor minimum detectable signal. However, molecules from a sample also bind nonspecifically to the FBAR's surface out of functionalizing sites, thus contributing to the total adsorbed mass and the sensor's output signal, as well as to its fluctuations. In this paper we present the analysis of the influence of nonspecific adsorption on the sensor's signal. We also analyze the contribution of the nonspecifically adsorbed molecules to the total adsorption-desorption fluctuations of the FBAR's resonant frequency. Results show that this contribution can be significant and thus necessary to be taken into account in order to estimate and improve the sensor's limiting performance.

INVESTIGATION OF POSSIBLE TILT SYSTEMS IN CaMnO₃ PEROVSKITE USING AN ab initio APPROACH

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The CaMnO₃ based ceramics are highly interesting materials for the industrial application, especially because of their good electrical conductivity and their magnetic properties. At ambient conditions CaMnO₃ adopts orthorhombic Pnma space group and $a^{\dagger}b^{\dagger}a^{\dagger}$ tilt system. Adoption of the specific tilt system in the perovskite structure type is dependent on composition, temperature and pressure. Using SPuDS (Structure Prediction Diagnostic Software) code we have obtained ten more possible tilt systems for the CaMnO₃ compound. They are all members of the group – subgroup relation between *Pnma* and the cubic aristotype, *Pm3m*. Initial data for the calculation model were experimental structures taken from the Inorganic Structure Database (ICSD). In cases where has been no experimental model in the group – subgroup relation graph, than the predicted unit cell and atomic coordinates given in the SPuDS were used. For each of these systems, a local optimization on the ab initio level using Hartree-Fock, density functional theory (LDA), and hybrid (B3LYP) functional was performed. Our results are in a good agreement with previous experimental and theoretical results, and hybrid (B3LYP) functional shows the best fit to the experimental observations. Finally, a group - subgroup relation graph was constructed, showing tilt systems of CaMnO₃ perovskite phases which could exist on ab initio level. In addition, we have found a new post perovskite phase in our calculations, which was observed for the first time in the CaMnO₃ compound.

HYDROTHERMAL SYNTHESIS AND MAGNETIC STUDIES OF MULTIFERROIC BiFeO₃

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Pure-phase BiFeO₃ powders were prepared by applying low-hydrothermal synthesis route. Bi(NO₃)₃·5H₂O and Fe(NO₃)₃·9H₂O were used as starting materials and 8 M KOH was utilized as mineralizer. The phase composition of obtained samples was determined by X-ray diffraction (XRD) analysis. It revealed that synthesized material crystallize in space group R3c with cell parameters a = b = 5.5780(10) Å and c = 13,863(3) Å. Morphology of synthesized BiFeO₃ powders were analyzed by using scanning electron microscopy (SEM) while the particle size and distribution was determined by small – angle X-ray scattering (SAXS). Obtained powders were also characterized by SQUID techniques, which showed that synthesized material is magnetic.

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THERMAL SENSOR FOR WATER WITH A RANGE CONSTANT VOLTAGE SUPPLY

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A thermal sensor for water was formed using two thick film segmented thermistors. The first thermistor was used to measure the incoming water temperature. The second thermistor was self-heated at a constant voltage and measured the water volume flow. The range constant voltage (RCV) power source changes the power supply in steps of 2-3 V for a change in 5° of input water temperature. Thick film segmented thermistors were produced by screen printing of thermistor paste composed of $Cu_{0.2}Ni_{0.5}Zn_{1.0}Mn_{1.3}O_4$ powder obtained by a combination of mechanical activation and thermal treatment, an organic vehicle and glass frit. The sensor system response to changes in the water volume flow rate were measured and analyzed in a static regime and also for different volume flow rates and temperatures of incoming water from the water supply mains.

TiO₂ FILMS PREPARED FROM NANO-TiO₂ PASTES AND THEIR PHOTOVOLTAIC PROPERTIES

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We have prepared several pastes with different contents of commercially available nano-TiO₂ (surface area ~ 326 m²/g) and organic functional additives: acetylacetone, PEG 6000 and Triton X-100. The pastes were deposited onto fluorine-doped tin oxide (FTO) substrates (12-14 Ω /sq) using tape-casting film applicator, and as deposited films were calcined at temperatures up to 550°C. The effects of paste composition and processing parameters on the morphology, porosity, thickness and adhesion of prepared films were investigated. The thermal treatment conditions of films were chosen in accordance to the results of viscometric and TG/DTA analysis, and the microstructure of fabricated films was analyzed by SEM, FESEM and AFM. The films with most favorable microstructural properties were used to fabricate dye-sensitized solar cells (DSCs), and their photovoltaic performances were investigated by measuring current density-voltage characteristic.

MAGNETIC PROPERTIES OF MECHANOCHEMICALLY SYNTHESIZED YTTRIUM MANGANITE

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Yttrium manganite (YMnO₃) is a multiferroic material, which means that it exhibits both ferromagnetic and ferroelectric properties, making it interesting in spintronics. In this work, single-phase YMnO₃ powders were prepared for the first time by mechanochemical synthesis in a planetary ball mill. The YMnO₃ was formed directly from the highly activated constituent oxides, Y_2O_3 and Mn_2O_3 , after 60 min of milling time. However, the X-ray diffraction analysis and the Rietveld refinement indicated that already after 240 min of milling time a pure orthorhombic perovskite structure could be obtained. Particle size analysis along with SEM revealed the agglomeration of powders with prolonged milling time. The magnetic properties of the obtained YMnO₃ powders were found to change as a function of the milling time in a manner consistent with the variation in the nanocomposite microstructure. In addition, small magnetic hysteresis at low temperature and a discrepancy between the ZFC and FC curves with negative paramagnetic Curie-Weiss temperature indicate that the obtained samples are basically antiferromagnetic with weak ferromagnetism.

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MECHANOCHEMICALLY ASSISTED SOLID-STATE SYNTHESIS OF Cu SUBSTITUTED THERMOELECTRIC SODIUM COBALTITE

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Polycrystalline samples of Cu substituted NaCo_{2-x}Cu_xO₄ (x=0, 0.01, 0.03, 0.05, 0.1) were prepared using mechanochemically assisted solid-state reaction method

starting from the Na_2CO_3 , Co_3O_4 and CuO powders. Each powder mixture was mechanically activated by grinding for 3 h in a planetary ball mill with ball to powder mixture ratio 20:1, at the basic disc rotation speed of 360 rpm. The asprepared powders were pressed into disc-shaped pellets and subsequently subjected to a heat treatment at 880 °C for 24 h in inert argon atmosphere. Changes in structural characteristics of the samples and particle morphology, caused by Co substitution by Cu, were characterized using X-ray diffraction and scanning electron microscopy, respectively. It should be emphasized that milling process reduced the time necessary for obtaining pure sodium cobaltite. From the results obtained in this study, observed changes in microstructure were correlated with changes in the lattice parameters, indicating the influence of Cu ion incorporation in $NaCo_2O_4$ crystal lattice.

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NANOINDENTATION OF NICKEL MANGANITE CERAMICS OBTAINED BY COMPLEX POLIMERIZATION METHOD

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Nickel manganite is very interesting NTC material due to its outstanding properties and wide field of applications mainly in microelectronics and optoelectronics and lately in the mobile phone industry. In this work, chemical synthesis of this material was performed by complex polymerization method (CPM). The presence of pure nickel manganite phase was confirmed by X-ray analysis. The obtained fine nanoscaled powders were uniaxially pressed and sintered at different temperatures: 1000 °C -1200°C for 2h. Microstructure development during sintering was observed by scanning electron microscope (SEM). Indentation experiments were carried out using a three sided pyramidal (Berkovich) diamond tip. Young's modulus of elasticity at various indentation depths and hardness of NTC ceramics were calculated. It was found that the highest hardness and elastic modulus exhibit the ceramics sintered at highest temperature.

MAGNETIC PROPERTIES OF HYDROTHERMALLY SYNTHESIZED YMnO₃ POWDERS

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The hexagonal YMnO $_3$ is multiferroic material exhibiting ferroelectricity and antiferromagnetic properties with a strong coupling between them, with ferrielectric order up to high temperatures $>600^{\circ}\text{C}$ and simultaneous ferrielectric and antiferromagnetic order below 75 K. In this work single phase hexagonal YMnO $_3$ powder was prepared starting from Y(CH $_3$ COO) $_2$ ·xH $_2$ O, Mn(CH $_3$ COO) $_2$ ·4H $_2$ O, KMnO $_4$, and KOH using a method of microwave assisted hydrothermal synthesis (200°C for 2 h) followed by calcination at 1200 °C for 2 h. According to FESEM analysis calcined powder consisted of submicronic, partially sintered YMnO $_3$ particles uniform in shape and size. Magnetic measurements indicated ferrimagnetic properties which were explained by a nonstoichiometry of the obtained compound and an excess of manganese confirmed by ICP analysis.

RECONSTRUCTION OF PRODUCTION PROCESS OF NEOLITHIC POTTERY FROM PLOCNIK NEAR PROKUPLJE

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Plocnik near Prokuplje is the location of the excavations of the interesting Neolithic settling having very developed pottery production for a period of about 800 years. Pottery artifacts are carrying information about the technological skills of Neolithic people and are giving the insight about their ability to control fire and firing process. This ability is performed in producing different color of their vessel and having bowls having very fine structure. In order to reconstruct the pottery production technique in the scope of the experimental archaeology two approaches were used. The classical geology approach was used to identify the origin of the clay used in the production. Petrography gives insight into the structural characteristics of the pottery. In order to reveal the technique of production of bowls and other utility pottery objects the multi slice scanner was used. The pottery has the density that is comparable to that one of the bones and the imaging techniques used for bone structure identification are used in this study.

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- Magnezitne vatrostalne mase
- Smolom vezane temperovane magnezitkarbonske opeke i mase

Osim proizvodnje vatrostalnog materijala Bomex se bavi i veleprodajom betonskog čelika iz proizvodnog programa fabrike Arcerol Mittal, Zenica, na tržištu Srbije, Crne Gore, Makedonije i Hrvatske, uz realizaciju od oko 100.000 tona.

Veoma velika povezanost sa crnom metalurgijom, poslednjih godina uvela je Bomex u dve relativno nove delatnosti: građevinarstvo i brodogradnju. Stambeno poslovna gradnja fokusirana je najvećim delom na tržište Beograda.

Bomex je 2007. godine investirao u novo brodogradilište, koje je do sada izgradilo 28 brodova. Brodogradilište je izgrađeno u severoistočnoj Srbiji, na reci Begej, 50 km od Beograda. Pozicija brodogradilišta je veoma dobra, s obzirom da je ono rekom Tisom povezano sa Dunavskim koridorom a preko njega Crnim, Kaspiskim, Sredozemnim morem i Atlanskim okeanom, što mu daje logističke mogućnosti pristupa veoma velikom tržištu.

Brodogradilište je osposobljeno da gradi različite tipove brodova za tehničke flote, sve vrste brodova za unutrašnju plovidbu, teretne brodove za rasute terete, kontejnerske brodove, tankere, hemikal-tankere, dužine do 135 metara i širine do 11,50 metara, morske teretne brodove dužine do 135 metara, širine do 30m i visine (od vodene linije) do 12m do 20 metara, platforme i barže za gas dužine do 90 metara i širine do 30 metara, rečne i morske remorkere za rad u rejonu velikih luka, brodove za prevoz žive ribe, brodove za odmuljivanje rečnog dna i slično. Brodogradilište je građeno sa nizom tehničkih novina koje mu omogućavaju ne samo visok kvalitet, već i postizanje komparativnih prednosti u odnosu na konkurente iz Zapadne Evrope.

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*TERS - Tip-Enhanced Raman Scattering **SNOM - Scanning Near-field Optical Microscopy

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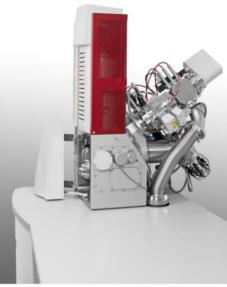
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