1st European Conference on Silicon and Silica Based Materials

Miskolc-Lillafüred, Hungary
October 7-11, 2019

BOOK OF ABSTRACTS

Edited by
László A. GÖMZE

ec-siliconf1

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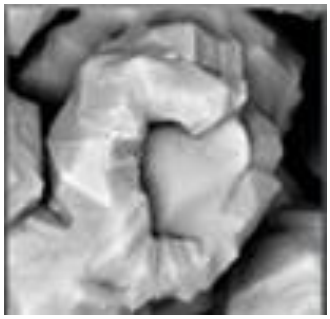

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Miskolc, 2019 August

Prof. Dr. László A. Gömze

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CONTENTS

PLENARY and KEYNOTE LECTURES

PLENARY

On the Synthesis Interconnected Regular Turing Structures with the Topology of Triply Periodic Surfaces of Minimal Energy

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When analyzing the physical properties of a material, especially to assess the prospects for its use in various conditions, the natural desire of technologists and designers is to obtain a regular (periodic) bulk interconnected microstructure that is homogeneous in composition and properties. The first step towards solving this problem is the use of topology in the structural chemistry of matter and materials, including composites, for the realization of triply periodic surfaces of minimal energy (TPMS). This problem was briefly formulated by von Schnering in 1987 - “how nature implements periodic minimal surfaces in chemical structures”. It turned out that in nature there is a mechanism of transition from the atomic interaction, through the nanometric state, to a real structure of a certain type. In 1952, Alan Turing mathematically showed that a two-component reaction-diffusion system with diffusion of reaction components and non-linear conditions leads to the spontaneous formation of spatially periodic structures. Experimental embodiment of the Turing reaction showed that under certain conditions, three-dimensional interconnected structures are observed (gyroid, Turing “fence”, etc.). To realize the conditions of the Turing reaction, it is necessary to select reagents that form the so-called cohenetic (heterogenetic) pairs. The best known pairs are diamond (carbon) and silicon carbide. Composites were synthesized using the “Skeleton” technology developed back in the 1960s. (It should be noted that this term is also used for the structural state of silicon carbide grown from the gas phase.)

For the first time, Turing structures in a diamond (carbon) – silicon composite were observed during the synthesis of chromium carbide too. Thus, the theoretical and experimental possibility of implementing the technology of a new class of materials with a regular (periodic) interconnected microstructure based on triply periodic surfaces of minimal energy is shown.

Keywords: Turing structures, triply periodic surfaces of minimal energy, composite, diamond, silicon, carbide chromium carbide

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KEYNOTE 1.

Optical properties of Si nanocrystals in SiO_2 matrix synthesized by reactive pulsed laser deposition

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After first reports on room temperature visible photoluminescence (PL) in the early 1990s [1], great interest in the optical properties of Si nanocrystals has grown over the last decade because of their potential applications toward Si-based integrated optoelectronic devices. Our group has focused on the formation of silicon nanocrystals and developed the first examples of luminescent Si nanocrystals inside of SiO_2 using ion implantation [2]. Nowadays, it is well known that Si ion implantation into SiO_2 and subsequent high temperature annealing (more than 1000 ^oC) induce the formation of luminescent Si nanocrystals. The PL peaking in the near infrared or visible spectrum (between 1.4 eV and 1.8 eV) is evidently related to implanted Si nanocrystals formed by decomposition of the SiO_x phase and aggregation with high temperature annealing. The PL arising from implanted Si nanocrystals in SiO_2 has been attributed by some investigations to simple quantum confinement, while others have concluded that surface states present in the interfacial layer (including some types of defects) between the Si nanocrystals and the surrounding oxide matrix (localized surface states) play an important role in the emission process.

In this work, we report the optical properties of Si nanocrystals embedded in a SiO_2 synthesized by reactive pulsed laser deposition (PLD) in an oxygen atmosphere. Si sub-oxide (SiO_x, $0<x<2$) films were firstly deposited on Si wafers, by using conventional PLD system with 2nd-harmonic YAG laser (532nm, 10Hz) under controlled low oxygen pressure. After deposition in the oxygen ambient, the produced SiO_x films were annealed using a conventional tube furnace (FA) for several hours at 1050 ^oC in N_2 atmosphere to induce the formation of Si nanocrystals, by decomposition of the SiO_x phase and aggregation. Some of the samples were irradiated with excimer-UV light (172 nm, 7.2 eV, Xe_2^*) for 2 hours with power density of 50 mW/cm^2 in vacuum or rapidly thermal annealed (RTA) at 1050 ^oC in N_2 atmosphere for 5 minutes with a rising rate of 50 ^oC/sec before FA. Room temperature PL spectra were measured at various stages of the processing.

We found that the luminescence intensity is strongly enhanced with UV irradiation and RTA. Based on our experimental results, we discuss the effects of excimer-UV lamp irradiation and RTA process on the formation of Si nanocrystals. In case for PLD produced samples, PL intensity increases with increasing oxygen gas pressure, and then decrease. We also found that the maximum intensity can be obtained with oxygen pressure around 0.6Pa. It is also noted that the peak energies of the PL are affected by ambient oxygen pressure. In some cases, blue-shift, other cases red-shift. The formation process of luminescent Si nanocrystals with UV, RTA and FA treatments can be explained with bond-

breaking (Si-Si and/or Si-O), defect generation, de-nucleation, defect-initiated nucleation and frozen of individual states.

Keywords: Si nanocrystals, Pulsed laser deposition, Excimer-UV, RTA

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KEYNOTE 2.

Silicon-Containing Linkers for Construction of Metal-Organic Frameworks

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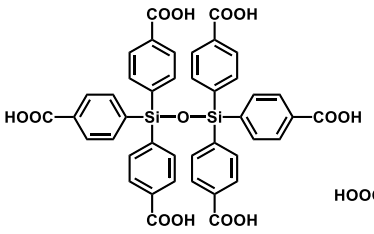
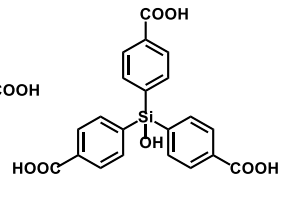
Porous materials containing silicon, often in siloxane (Si-O-Si) linkages, are well known and include zeolites, organosilicas, and POSS hybrids. However, MOFs incorporating Si are relatively uncommon, especially those containing Si-O bonds. This is despite the fact that organosilicon linkers may offer advantages, such as ease of synthesis for complicated polyfunctional linkers, low toxicity, low chemical reactivity and thermal stability. We have prepared a variety of organosilicon linkers and applied them in the construction of coordination polymers and MOFs.

For example, the highly-connected organosilicon polycarboxylic acids (below) have been prepared and applied in the construction of MOFs. **L1**-H_6 itself crystallizes as an unusual interpenetrated 3D hydrogen-bonded framework. Reaction of **L1**-H_6 with Zn(II) gave a MOF with **fsy** topology, the first reported[1] example of a 3D-connected MOF incorporating Si-O-Si functionality. Cleavage of **L1**-H_6 gives a silanol-based triacid **L2**-H_3 which is shown to give a coordination polymer with Zn(II), consisting of 2D layers which assemble by hydrogen-bonding to afford a 3D supramolecular structure with **flu** topology. The tetracarboxylic acid **L3**-H_4 crystallizes through hydrogen-bonding to give a quadruply interpentrated structure comprising 4 identical **mog** nets. Reaction of **L3**-H_4 with Zr(IV) afforded, a 3D MOF built from 8-connected Zr-based nodes cross-linked by **L3** to afford a porous MOF with the rarely encountered **scu**-derived **tty** topology.



L1-H_6



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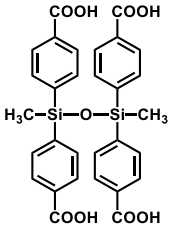
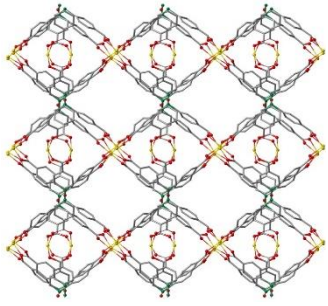


L3-H_6



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KEYNOTE 3.

Thermodynamic Modeling and Calculation of Phase-Chemical Transformations at Ceramic Materials Synthesis and Operation

Alexander Slobodov[1,2], Andrey Syrkov[3], Mikhail Markov[2], Mamoon Sarmini[4], Alexander Melentiev[1], Ricardas Ralys[1], Alexander Sochagin[1,2], Dmitriy Kremnev[1,2], Anna Klepova[1]
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On the basis of minimization principle for characteristic thermodynamic functions the complex of methods and databases (realized in computer program-information system), intended for description, modeling and calculation of phase-chemical transformations occurring under synthesis and operation of functional materials is developed. The complex has no restrictions on componentity, number of potentially possible phases and chemical reactions in a system.

On this basis the calculation of chemical and phase transformations occurring in different ceramic materials at 298 - 2500 K is carried out. It describes and simulates processes occurring in thicker and cells of corresponding refractory with antioxidant on the basis of aluminum and without one. Multiplane picture of temperature and composition influence on phase and chemical transformations in a system is received. These dependencies have allowed to reveal fields of stability for components composing the material. Also, the process of evolution for gas phase composition depending on temperature is investigated, and the efficiency of the antioxidizing addition (suppressing ability of gas phase to oxidize carbon) is quantitatively investigated.

The developed approach has allowed also to investigate and reveal mechanisms of phase-chemical transformations occurring at sialons $Si_xAl_yO_zN_t$ synthesis and operation by means of carbothermy kaolin nitration. The influence of reagents on character and amount of impurities, on areas of thermal stability for sialon phases are investigated, on their thermomechanical and chemical resistance, etc. are studied. The essential influence of temperature on realization of one or another scheme of sialons synthesis is revealed. The conditions of phases recrystallization, mainly gas-phase synthesis process, etc. are determined.

Also, this methodology is used for thermodynamic research of synthesis (from binary compounds) process for another composite - complex carbonitride of titanium, tantalum, tungsten, for micro arc oxidation (MAO) of functional materials, solid state hydride synthesis (SSHS), etc. The correspond complex systems are investigated in wide area of compositions and temperatures (298 - 3000 K). Qualitative and quantitative regularities on influence of temperature and components ratio on character of phase transformations running during synthesis are revealed, on course of homo- and heterogeneous reactions, on formation of intermediate and by- products. The areas and fields of phases stability, their ratio depending on state variables are determined. E.g. the conditions promoting and

preventing formation of "harmful" product – W_2C (forming at input cobalt into alloy, complex carbide $(W,Co)_3C$, that leads to the alloy embrittlement) are defined.

Received theoretically and by calculation way the results for all investigated multicomponent systems not only well agreed with known (truth, by virtue of considered systems complexity, quite often rather fragmentary and limited) experimental data, but also give qualitatively and quantitatively more rich information on mechanisms of running processes.

KEYNOTE 4.

Role of Silicon and Silica in Development of Mechanical and Dynamical Properties of Ceramic Reinforced Armor Shell Composites

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The compounds of silicon are playing important role in mechanical strengths and properties both in traditional [1-2] and technical [3-5] ceramics. The role of silicon nitride and silicon carbide components in ceramic reinforced armor shells are described in several works. During development and testing of a new generation of ceramic based armor shell composite materials the authors have founded that the crystalic structure of a certain parts of α-Si_3N_4 and β-Si_3N_4 components have turned into *diamond-like* cubic structure of c-Si_3N_4 [5-6]. This new *diamond-like* c-Si_3N_4 has density about 5.6 g/cm^3 and considerably increases the mechanical and dynamic strengths of the armor shell composite materials based on ceramic matrix impregnated with light-metals. During their experiments the authors have founded that during the high-energy high-speed collisions the free SiO_2 molecules have turned into stishovite crystals with density of about 5.2 g/cm^3. Both the *diamond-like* c-Si_3N_4 crystals with spinel structures and the stishovite crystals of SiO_2 help to increase the dynamic strengths of the armor shells prepared from ceramic matrix light-metals impregnated composites.

Analytical methods applied in this research were scanning electron microscopy, X-ray diffractions and energy dispersive spectrometry. Digital image analysis was applied to microscopy results to enhance the results of transformations.

Keywords: armor shells, composites, silicon-dioxides, silicon nitrides, stishovite, strengths

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ORAL PRESENTATIONS

Solid-State Reactions in Mixed Micron-Sized Silicon Monoxide and Titanium Monoxide: Nanostructured Composites with Visible Light Absorption

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Silica–titania mixed oxides and composites have been broadly examined for their physical and catalytic properties, but titanium monoxide–silicon monoxide counterparts have yet drawn very little attention. Here we report on structural changes in SiO-TiO mixtures treated by (i) conventional heating, (ii) continuous wave (CW) infrared (IR) laser irradiation and (iii) IR pulsed laser irradiation.

The changes in chemical and phase composition in intimately mixed and treated μm-sized particles of TiO and SiO examined by FTIR, UV–Vis and Raman spectroscopy, X-ray diffraction and electron (SEM and TEM) microscopy. Photocatalytic activity of obtained materials has been tested in term sof methylen blue depletion.

(i) The SiO-TiO mixture conventionally heated at 1000ºC results in depletion of crystalline TiO and formation of titanium suboxides ($Ti_{2.5}O_3$, Ti_2O_3), rutile, elemental silicon, titanium silicide Ti_5Si_3 and amorphous binary SiO_x, TiO_x and ternary $Si_xTi_yO_z$ nano-structures. These constituents of the developed Si/Ti/O composite are explained by SiO disproportionation, reduction of TiO by silicon, oxygen transfer (redox) reactions between TiO_x and SiO_x species and the combination of Ti and Si to obtain titanium silicide. The produced Si/Ti/O composite absorbs visible light and its solar-light photocatalytic activity in decolorization of methylene blue is compared to that of the unheated SiO, TiO and Ti_5Si_3 powders.

(ii) CW IR irradiation of mixed SiO-TiO leads to evolution of nanostructured TiO_2 (rutile and anatase), titanium suboxides ($Ti_{4.5}O_5$, Ti_2O_3), silica and amorphous binary SiO_x, TiO_x and ternary $Si_xTi_yO_z$ nanophases which contain less or more O than SiO and TiO monoxides. These products are ascribed to concurrent silicothermal reduction of TiO and O-transfer between SiO and TiO due to interdiffusion of Si- and Ti-based species. These reactions taking place under transient localized heating are not inhibited by passivation shells around SiO and TiO particles. The laser-produced Ti/Si/O composite shows absorption band at 425 nm tailing up to 1100 nm. Its solarlight photocatalytic activity in decolorization of methylene blue is compared to that of the unheated SiO and TiO powders absorbing only in UV region.

(iii) Infrared laser-induced ablation of an equimolar titanium monoxide–silicon monoxide mixture allows deposition of a continuous TiO-doped $Si/SiO_x/SiO_2$ film which undergoes collapse into micron-sized completely amorphous cylindrical-shaped plates composed of TiO nano-objects in prevailing $Si/SiO_x/SiO_2$ phase. Ablatively deposited thin films show a broad red-shifted UV–vis absorption when compared to various TiO_2 or TiO_2-SiO_2 composite materials. In aqueous solutions of Methylene Blue, it enhances the rate of visible light-induced decolorization of this dye. It also reduces methylene blue to leuco-methylene blue in the dark.

Keywords: Silicon monoxide, Titanium monoxide, Solid state redox reactions, nanostructured Ti/Si/O composite, solar-light activity

Effect of H_2 on SiO and SiC Formation

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Based on previous research, hydrogen has an enhancing effect on the kinetics of the Si-O-C system [1]–[3]. In this study silicon carbide (SiC) formation, from a reaction between SiO(g) and carbon, were investigated in a hydrogen atmosphere, an argon atmosphere, and an argon atmosphere containing 10% methane, at temperatures between 1495ºC and 1695ºC. The SiO(g) was generated from pellets comprised of a 2:1 ratio of silica (SiO_2) and SiC. The SiO(g) generation was monitored through analysis of the CO(g) content of the off-gas, the results indicated that the hydrogen atmosphere had an enhancing effect on SiO(g) generation through this reaction. Samples were retrieved from various locations within the set-up: the crucible, the reaction chamber roof, and from the condensation chamber. The reaction products were imaged in SEM using the secondary electron detector.

SiC was found growing throughout the reaction chamber, when using an argon atmosphere, a layer of SiC was found covering the graphite parts, whereas utilising a hydrogen atmosphere, or argon with 10% methane, the SiC would instead grow as whiskers. The hydrogen also had a large effect on the formation of other reaction products. In argon, a brown Si and SiO_2 condensate formed at 1695ºC, while in hydrogen it formed at a lower temperature of 1595ºC. Additionally the hydrogen atmosphere resulted in a large degree of SiC and SiO_2 condensate forming on the graphite. Thermal decomposition of methane is expected to be a challenge in this temperature range [4]. Using a CH_4(g)/Ar gas mixture resulted in carbon deposition within the alumina lance, on top of the raw materials, but most of the deposited carbon ended up within the condensation chamber.

Keywords: silicon, SiC, hydrogen, methane, whiskers

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Evaluation of Microstructural and Thermal Properties of Sol-Gel Derived Silica-Titania Based Porous Glasses

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In recent years, the synthesis of sol-gel derived porous glasses has drawn widespread attention owing to the convenience and versatility of the sol-gel method. The sol-gel synthesis process mainly involves hydrolysis and condensation of precursors followed by drying and stabilization. The characteristics such as pore structure, morphology and compositions of sol-gel derived glasses significantly affect their final properties. In the present study, silica-titania (Si-Ti) based porous glasses with different compositions were synthesized using the sol-gel method. Metal alkoxides such as tetraethoxysilane (TEOS) and titanium isopropoxide (TIP) were used as a source as the source for silica and titania respectively. Nitric acid (HNO_3) was used as catalysts to trigger the hydrolysis reaction and polyethylene glycol (PEG) was used as a polymeric component to induce phase separation. The influence of different processing parameters on the microstructural and thermal properties was investigated. The microstructure of the synthesized Si-Ti based porous glasses was investigated using Scanning electron microscopy (SEM) and the thermal characteristics were evaluated using thermogravimetric analysis (TGA) and thermomechanical analysis (TMA). The main objective of this study is to ascertain the application of sol-gel derived Si-Ti porous glasses as a potential biomaterial for bone tissue regeneration. To understand this facet of Si-Ti porous glasses, the biological performance will be investigated, and their porous architecture will be explored in relation to their interaction with the bioactive nanoparticles.

Keywords: Sol-gel method, Si-Ti glasses, microstructure, thermal properties.

Selective Laser Melting of Silicon Based Ceramics

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During the last decade, silicon nitride has demonstrated a tremendous wave of attention as a bio-structural material and high-temperature component in the automotive and aerospace devices and holds a significant promise in the field of anticorrosive protective coatings. However, the manufacturing of silicon nitride components with netshape is scientifically challenging. Among many approaches, additive manufacturing (AM) via selective laser melting (SLM), i.e. layer-by-layer assembling of components from CAD data, is shown to be powerful and resource-efficient manufacturing method that allows making 3D components of a specified complex geometry. The major feature in processing of ceramics is that it is generally bound by a low absorption of laser beam energy and a poor thermal shock resistance of ceramic materials.

Here we propose a novel strategy of the production of ceramic components with a wide variety of morphologies and characteristics, particularly producing silicon nitride substrate with a tailored design and a specified geometry by sintering silicon precursor with the help of SLM technique.

The direct nitridation of silicon is a complicated process suffering from several disadvantages including a high nitridation temperature (> 1450 °C) and incomplete nitridation due to a very slow diffusion of nitrogen into a molten Si. The introduction of catalysts into the matrix of raw silicon can effectively accelerate silicon nitridation at reduced temperatures and, in addition, facilitate the in-situ formation of Si_3N_4 fibers. The transition metal oxide nanocatalysts were in-situ prepared using alumina whiskers (AW) support and utilized for catalyzed nitridation of a silicon substrate by wet combustion synthesis.

Preliminary, selective laser melting of silicon powder with required shape was accomplished, and nitridation of the as-shaped silicon parts was performed aimed at fabrication of the Si_3N_4 component. Parametric study of the process has been performed for optimization of the sintering parameters, such as laser current, point distance and exposure time. An increase in the point distance negatively affects the density of Si, while increase in laser current from 400 mA up to 1300 mA results in increase in the relative geometric density of the samples from 63 up to 85%. The Vicker's microhardness of the parts increases from 5.5 to 11.8GPa with an increase of the laser current from 500 up to1300 mA. The silicon samples (diameter 10 mm and height 5 mm) consolidated using the laser current of 900, 1100 and 1300 mA and subjected to the compression test exhibit compressive strength of 222, 237 and 432 MPa, respectively.

The nitridation of the sintered Si lead to formation of Si_3N_4 tapes on the surface and inside the pores of the samples. Ni-based catalyst demonstrated the highest conversion degree for the nitridation process.

Keywords: Additive manufacturing; selective laser melting; silicon; silicon nitride

Study on SiC Particulate-Reinforced Aluminum Matrix Composites

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Silicon carbide (SiC) ceramic particle, which has the advantages of low cost, good thermal stability, high hardness and low thermal expansion coefficient, is applied as reinforcement particle in a series of aluminum matrix composites (AMC). Over the past decades, extensive studies have been devoted to developing more efficient preparation method of Al/SiC composites and studying the influence of SiC particle on the microstructure and mechanical properties of AMC. Among the developed fabrication methods, stir casting is the simplest, most cost-effective route for mass production. However, while using this method for fabricating Al/SiC composites, the main challenge is avoiding particle agglomeration and obtaining a uniform distribution of SiC particle.

In this work, 1 wt% micro-sized SiC_P (17 μm) was incorporated into technical pure Al AD0 (1050) by stir–casting. Microstructure analysis revealed that a uniform distribution of SiC particle throughout the matrix was achieved. Compared with the matrix aluminum, grain size of the Al/SiC composites was refined, which is because the introduction of SiC particle promotes heterogeneous nucleation while suppressing the grain growth. Additionally, the hardness and microhardness of the Al/SiC composite was improved. Charpy impact tests showed lower impact energy with incorporation of SiC particle.

Keywords: SiC, aluminum matrix composites, stir casting, hardness, impact bechaviour.

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Preparation and Investigation of Alumina-Zeolite- Composite Materials

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Great attention and huge work have been recently done to synthesize ceramic reinforced ceramic matrix composites (CMCs) with promising characteristics like high strength, good hardness, superior refractory properties and fracture toughness. Normally the ceramic composite materials are made in such a way that it enhances the strength and reduce the brittle behaviour of the ceramic material which restricts their application [1]. Several research works have been demonstrated regarding the preparation of ceramic reinforced CMCs [2-3]. In this research work, the authors were successfully prepared Alumina-Zeolite ceramic composite materials using natural zeolite from Tokaj region (Hungary) and MOTIM Al_2O_3 (98%) powder through mechanical activation and reactive sintering techniques. The structure, topography, morphology and different properties of the complex composite samples were examined using various characterization methods such as, scanning electron microscopy (SEM), X-ray diffraction (XRD) and thermo-analytical analyzer (DTA), The XRD analysis of the natural zeolite from Tokaj region has revealed the existence of many minerals with different contents for instance montmorillonite, quartz, cristobalite, clinoptilolite, and calcite. By controlling the mechanical activation, compaction and the reaction conditions such as sintering temperature and sintering time, new Alumina-Zeolite samples with different compositions and properties were achieved. These materials could be a candidate for many practical applications.

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Hierarchically Porous Aluminosilicate Substrates Based on Geopolymer Materials

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Recently, wastewater treatment has become a critical issue due to particular concerns for sustainable environment, climate change and industrial growth. There has been a need for readily available and inexpensive solutions between research and industry. Porous aluminosilicate materials based on geopolymer systems have proven their effectiveness as adsorbents, ion-exchangers, membranes, anti-microbial filters, pH buffers and stabilizers for water treatment residues [1]. Furthermore, catalysts and/or catalyst supports for pollutant degradation in liquid phase reactions have been studied [2]. Geopolymer materials exhibit synergies in the cleaning and depollution process and can be regarded as cost effective and novel in the context of removing organic pollution from water and air. Geopolymer catalytic performance is closely linked to the composition, microstructure and preparation methods [3]. High permeability, surface area, chemical resistance and mechanical strength are essential for these applications.

Here we present a facile synthesis routes based on replica technique and sol-gel pipetting of highly porous substrates with open cells and water-floatable spherical beads with closed porosity. Characterization of prepared structures was performed with respect to their porous architecture, surface properties and mechanical integrity. Scanning electron microscopy (SEM), digital microscopy (DM) and micro-computed tomography (micro CT) revealed the relationship between the inner/outer structure and the open/closed porosity ratio. Thermal behavior was studied by thermogravimetric (TGA) and differential thermal analysis (DTA) up to 1000 °C and 1300 °C, respectively. In addition, mechanical stability was determined and a procedure for coating nanoparticles with respect to their photocatalytic activities was proposed.

Keywords: Aluminosilicates, Porosity, Morphology, Microstructure, Thermal properties

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Synthesis of Graphene Oxide (GO) – Silica (SiO_2) Composited with Titania (TiO_2) for Textile Wastewater Treatment

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In this study, rice husk (RH), an agricultural solid waste from rice production, is used as carbon and silicon sources for synthesizing graphene oxide (GO)-silica (SiO_2) nanocomposites applied for textile dye treatment by an adsorption process. GO-SiO_2 nanocomposites were synthesized via a modified Hummers' method by oxidizing throughout $KMnO_4$ in concentrated H_2SO_4 and H_3PO_4. To enhance the dye treatment, a combination of absorption and photocatalysis processes was studied by incorporation of titanium dioxide (TiO_2) with the synthesized GO-SiO_2 nanocomposites. TiO_2 nanoparticles were synthesized via a sol-gel method using titanium (IV) butoxide (TBUT) as the titanium (Ti) precursor. Gels dried were calcined at 500^{o}C for 3h prior use. The GO-SiO_2 nanocomposites were incorporated with the synthesized TiO_2 using an impregnation method with ratios of GO-SiO_2 to TiO_2 of 25%, 50%, and 75% w/w to obtain GO-SiO_2/TiO_2 nanocomposites. Structural characteristics and chemical properties of the nanocomposites were investigated by the characterization techniques of X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, Field emission scanning electron microscopy (FE-SEM), and X-ray photoelectron spectroscopy (XPS). According to the experimental results, TiO_2 nanoparticles consisting of anatase and rutile phases were successfully introduced between the surfaces of GO-SiO_2 layers. Photocatalytic activities of the GO-SiO_2/TiO_2 nanocomposites tested under UV irradiation conditions were evaluated by photodegradation of methylene blue (MB) in aqueous solutions used as a dye model of textile wastewater. MB concentrations remained were detected by a calibration technique of the UV-Vis spectrophotometer. The results show the achievement of the MB degradation by the synthesized GO-SiO_2/TiO_2 nanocomposites. Kinetic studies of the MB degradation were finally discussed. This present study reveals that the GO derived from RH is beneficial for further utilization.

Keywords: GO-SiO_2/TiO_2 nanocomposites, Rice husk (RH), Textile dyes, Wastewater treatment, Adsorption process, Photocatalysis process

Effect of Activation and Exfoliation on the Formation of Cabon Nanosheets Derived from Natural Materials

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Carbon is a widely used material, which can be found from nature or synthesized through heat and chemical processes. It can be applied in electronics, thermal, adsorption or can be manufactured as composite materials for use in a variety of industries, such as carbide materials. Chemical activation has been shown as a very efficient method to obtain carbons with high surface area and narrow micropore distribution. Among all the chemical activation agents, alkaline hydroxides as potassium hydroxide (KOH) or sodium hydroxide (NaOH) are reported to be highly interesting from the performance point of view. Nettle and peanut are natural materials were used to prepare carbon in this paper. Natural structures of nettle and peanut shell consisting of cellulose, which can be an important precursor in the preparation of highly ordered carbon nanosheets. Potassium or sodium hydroxide aqueous were used for activation, sulphuric (H2SO4) acid was used for exfoliation in the experiments. The developed process is easy and yields high percentage of carbon element in nanostructured form. Carbon nanosheets were characterized by their microstructure, chemical composition, specific surface area, micropore volume and pore diameter. X-ray Diffraction measurements allow to demonstrate that the activation and exfoliation process is partly controlled by the structural organization of the precursor. These results will help to better understand the different activation and exfoliation behaviours observed with natural carbon nanosheets of different ranks.

Keywords: Activation, Exfoliation, Carbon nanosheets

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Characterization of Glass Foams produced from Waste CRT Glass and aluminium dross

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Currently, the problem of increasing the energy efficiency of buildings has become very relevant. One of the directions being investigated to solve this problem, is to produce products that efficiently meet structural and engineering characteristics, thus decreasing the effect on the ecosystem and general population. This also includes the economical use of financial resources, to meet the increasing demand for sustainable structures. This will be implemented by employing eco-friendly materials such as glass foam. Glass foaming is a complex process that depends on the mode of foaming and the initial composition of the mixture [1]. This study deals with the investigation of glass foam properties based on recycled bottle glass material, CRT glass and aluminium dross. Cathode-ray tubes (CRTs) come from computer monitors and TV sets. They are considered electronic waste and over the century this kind of waste has increased exponentially [2]. However, there is a few CRT recycling facility in Europe and the rest of the world. Managing this problem is critical from the viewpoint of creating functional WEEE treatment systems [3]. The other material being investigated is aluminium dross, which is a by-product of melting aluminium scrap, via the application of NaCl-KCl-CaF_2 based salt flux, where the composition depends on the type of scrap.

In this study, CRT glass with particles sizes of D_{90}=63 µm and D_{90}=32µm were added in quantities of 5 to 10 wt%. Aluminium dross was leached with water and added in quantities of 10 wt%. The foaming agent (SiC) was added in quantities of 2 wt%. Weighted mixtures were homogenized in a laboratory mixer for 10 minutes at 200 rpm and poured in a cylindrical mold and pressed under 11 MPa.

Experiments were carried out to optimize the process. The thermal behavior was characterized by using heating microscopy for the mixtures and derivatograph for the aluminium dross, then the optimal foaming temperature was determined. The viscosity was calculated using characteristic temperatures from HSM curves. The density of the samples was also calculated. Thermal conductivity measurement was also conducted. Microstructure and pore size distribution were analyzed using optical microscopy. Phase composition was examined using X-Ray powder diffraction. Finally, the compressive strength was measured as well. Effect of the CRT glass and the aluminium dross on the properties of the foams was evaluated in this paper.

Keywords: waste management, CRT, Aluminium dross, Glass foam, Viscosity

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The Effect of Cristobalite Formation on Disintegration of Quartz

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Quartz is used as a raw material for the production of silicon and ferrosilicon. Together with various carbon sources and energy, quartz is added to a submerged arc furnace, where the temperature reaches above 2000 °C. When the quartz is heated it will disintegrate [1] [2] [3]. This is not favorable to the furnace operation because of problems related to clogging. This study investigates if there is any relation between the amount of fines produced upon shock heating for one quartz type and the ability to transform into the high temperature silica phase, cristobalite. The amount of amorphous intermediate material is also investigated. Based on earlier studies [4] [5] it is indicated that the amount of cristobalite will increase the amount of fines produced during shock heating. One reason for this is the volume expansion from quartz to cristobalite of 17 %. In this study four different quartz types used in the Si and FeSi industry has been shock heated to 1500 °C for 10 minutes. Afterwards, the amount of phases has been measured using X-ray diffraction. The quartz types used has different geological origin and appearance. Additional samples have been heated in another furnace to 1600 °C and 1700 °C to obtain a more thoroughly picture of the phase transformations from quartz to cristobalite and the different reaction rates in the quartz types. The ability to transform from quartz is very different in the various quartz types. After heating to 1600 °C, the amount of quartz varies from 5-95 % between two quartz types. No correlation is found between the amount of fines produced during shock heating for one quartz type, and the ability to transform the other silica polymorphs cristobalite and amorphous silica. In fact, the least disintegrated quartz type showed the largest amount of cristobalite in the shock heated sample.

Keywords: cristobalite, quartz, disintegration, silicon, ferrosilicon

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Water and Hydroxyls Groups in High Purity Quartz Sand

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High purity quartz (HPQ) is used for applications in semiconductor production, lamp tubing, microelectronics, telecommunication and photovoltaics.[1]. The latter has recently the highest demand in volume due to rapidly increasing market of silicon photovoltaic panels for green energy production. Single crystal silicon for these applications is produced in the Czochralski (CZ) process, in which HPQ sand is used as a material for crucibles. To meet the rigorous process requirements, extremely low level of impurities is required. [2]

For many years, engineers were mostly focused on removal of metal impurities and with little focus on the role of water and hydroxyl groups in high temperature treated quartz sand. It was often assumed that water could be easily removed during a calcination process. Only recently effect of the liquid inclusions on properties of quartz and quartz glass in high temperatures was investigated [3]. The results showed that the presence of water and hydroxyls causes breakage of Si-O bonds and lowers the viscosity of quartz at high temperatures. Nevertheless, fundamental understanding of water and hydroxyl groups behaviour at high temperature is still limited.

In this work various spectroscopy methods were applied to characterize water and hydroxyls behaviour in quartz sand treated at high temperatures

Our results show that a temperature higher than 600 ^{o}C is needed to remove water from inclusions, however only at as high temperature as 900 ^{o}C most of bridged Si-OH groups are removed, and after this treatment only non - bonded silanols are present.…

We can conclude, therefore, that the removal of the water from inclusion is at large extend because of the formation of hydroxyl groups and subsequent condensation of these groups.

Keywords: High purity quartz, IR spectroscopy, liquid inclusions, hydroxyls

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The Effects of Magnetic Silica Nanoparticle Addition on Dynamic Spin Susceptibility of Topological Kondo Insulator

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A topological insulator is a material with topologically protected metallic boundaries and an insulating bulk. The strongly-correlated Kondo variety, such as SmB_6, has recently been widely investigated owing to its promise of being the first realized topological Kondo insulator (TKI). We study here a generic TKI system by performing a mean-field theoretic (MFT) calculation within the frame-work of slave-boson protocol. We assume infinite Hubbard-type interaction among the quasi-localized electrons (QLE). We also investigate the consequences of break-down of the time-reversal symmetry (TRS) due to bulk substitutional magnetic impurities (MIs), such as magnetic silica nanoparticles (MSN). These impurities couple with itinerant electrons of the system. One of the note-worthy outcomes is the impurities change the single-particle excitation spectrum in a fundamental way. We obtain the grand canonical potential of the system and self-consistent equations for MFT parameters minimizing the potential with relative to these parameters. The parameters enforce constraints on the pseudo-particles due to the infinite Coulomb repulsion and the need of the formation of singlet states between an itinerant electron and a localized fermion at each lattice site in order to have a Kondo insulator.

A fundamental quantity describing the magnetic response of a system is the dynamic spin susceptibility. The Kondo screening mechanisms are mostly characterized by this quantity. We have calculated the bulk spin susceptibility of the generic TKI system in the metallic and insulating phases of the system at a fixed chemical potential and found them behaving differently with relative to change in the MSN impurity field (M). The difference between the metallic and insulating phase is in the sign of nearest neighbor hopping of QLE: It is positive for the metallic and negative for the insulating phase. The spin susceptibility is found to be negative in the latter and positive and decreasing function of M in the former. Furthermore, when the expectation value of the slave-boson field tends towards zero, the spin susceptibility diverges signaling a transition from the Kondo insulator phase to a normal insulator phase. There are many challenges in the processing of these exotic materials to use the metallic/insulating states in functional devices, and they present great opportunities for the chemistry and materials science research communities.

The Use of Silicon-Boron Alloys in Latent Heat Thermal Energy Storage System

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Si and B are considered as phase change materials (PCMs) in the latent heat thermal energy storage (LHTES) system due to their high latent heats and melting points. Proper Si-B alloy composition and ceramic refractory materials that can survive long time corrosion at high temperatures should be determined before successful implementing them into reality. Here, we investigate serials of Si-B alloys with boron range of 2-11 mass % in graphite crucibles at temperatures between 1450 ^{o}C and 1750 ^{o}C. Furthermore, the wetting behaviour of Si-3.25B eutectic alloy in contact with hexagonal boron nitride (h-BN) was examined at temperature up to 1450 ^{o}C with 3 times melting/solidification process. This work shows that the interaction between Si-B alloy and graphite goes into two stages. A SiC is formed at the interface when the boron content is 2 mass %. Otherwise, SiC and B_4C are formed at the interface when the boron content is higher than 5 mass %. Moreover, the wetting test demonstrates that the contact angle of 120^{o}, 90^{o}, and 50^{o} is maintained at the first, second, and third cycle in the Si-3.25B/h-BN system, respectively. Additionally, the results of structural characterization are supported by FactSage thermodynamic calculations. These results provide reliable data in the use of Si-B alloys as PCMs in the LHTES system.

Keywords: Si-B alloys, hexagonal boron nitride, graphite, wettability, phase change material.

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The Role of Purity Level in Foundry Silica Sand on its Thermal Properties

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For foundry molding and core making applications, silica sand is the most commonly used mineral due to its availability, thermal and chemical attributes. However, there are many additional requirements foundry sands need to meet regarding their sizing, chemical purity, physical durability and thermal properties. Consistent particle size distribution, for instance, is crucial for achieving maximum performance and efficient operations. Its role is well known; the grain size distribution of the sand determines its binder demand, the compactibility and permeability of the molds, etc. [1] The chemical composition is also one of the most important attributes that are considered when selecting from different silica sand products since it impacts the performance of the sand and also the behavior of the molds and cores. The relationship between the grain size distribution and thermal properties like thermal expansion is a commonly studied topic in foundry research. [2] However, the correlation is explained purely by the effect of the physical size difference of the fractions and neglects the state of composition.

This research studies the thermal properties of a foundry silica sand comprehensively. After separating the sand batch into numerous grain size ranges, the chemical composition and thermophysical properties of the fractions were investigated, respectively. By means of this approach, the chemical properties and thermal behavior can be directly linked. The silicon dioxide content shows a strong correlation with the thermal expansion properties of the various fractions. The results give us a better understanding of the high-temperature behavior of foundry silica sands and clarify the role of affecting factors on the thermophysical properties.

Keywords: chemical composition, foundry sand, molding material, silica sand, thermal expansion

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SHORT ORALS
(and Posters)

SO-1 (P21)

Synthesis by Laser Pyrolysis of Si Quantum Dots as Active Materials for LED

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Silicon quantum dots have recently attracted great interest due to their excellent optical properties, nontoxicity, and ease of surface modification. The size of Silicon nanocrystals (Si-nc) and chemical characteristics of their surface have a great influence on their optical properties. The most challenging task in preparing Si-nc is to succeed in controlling the particle size and morphology as well as the surface properties. To this respect, the technique of laser pyrolysis of silane gas (SiH_4) appears as a very flexible tool for the production of Si-nc in developmental quantities.

Using a CW CO_2 laser, we have synthesized Si-nc of high purity, selected dimensions from 5 to 10 nm, different surface characteristics and productivity up to 1g/h. In this synthesis technique, the condensable products result from laser induced chemical reactions at the crossing point of the laser beam with the molecular flow of gas precursors. The physical and chemical properties of the nanoparticles depend directly on the process parameters, such as flame temperature and residence time, that are determined by the nature and the flow rate of reagents and sensitizers, the reactor pressure and the laser power.

The produced Si-nc show weak luminescence. In order to enhance and wavelength-tune the luminescence emission, Si-nc were dispersed in polymers, deposited as films and processed by an innovative methodology that employs laser to induce modification of surface nanoparticles and to obtain luminescent materials.

Chemical and optical characterization of the nanoparticles were performed by conventional and advanced techniques such as FTIR, BET, UV-VIS, XPS.

Keywords: silicon nanocrystals, CO_2 laser pyrolysis, luminescence, laser writing

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SO-2 (P22)

Preparation and Synthesis of Hydroxyapatite Bio-Ceramic from Bovine bone by Thermal Heat Treatment

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Calcium phosphate, particularly hydroxyapatite (HAp), is important materials in biomedical engineering applications. The development of HAp is due to the continued rising similarity and biomimetic requirements to the hard tissue of human body such as bone and dental. The purpose of our work was to produce and describe HAp bioceramic powder from environmental and cheap source (Bovine bone) by thermal process at various calcination temperatures. The analysis of FTIR verified the formation of HAp because of the existence peaks related to phosphate and hydroxyl groups. The analysis of Raman confirmed findings of the FTIR the formation of HAp due to the appearance of peaks at 960 and 920 cm−1 related to a phosphate group. A result of EDS also referred to Ca/P atomic ratio at 1000 ºC was 1.6 that has been near stoichiometric hydroxyapatite (1.67) in human body.

SO-3 (P23)

Examination of Mullite Ceramic Specimens Made by Conventional Casting Method from Kaolin and Sawdust

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By using relatively low-cost materials (conventional kaolin and sawdust powders) and technology the authors have developed new ceramic composite materials which can meet successfully different industrial requirements. The kaolin and mullite are important materials in the industry therefore are many research about the thermal decomposition of kaolin minerals [1-3].

In this research casting masses (slurries) were made by milling different compositions of the powders and by adding distilled water. The test specimens were made by conventional slip casting method and after the drying process the specimens were sintered in an electric kiln under oxidation and reduction atmosphere at 1250°C max sintering temperature. The prepared and sintered specimens were tested on geometrical sizes, microstructures and morphologies by scanning electron microscopy. In this work the authors present some parts of the results of their research and investigation.

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SO-4 (P24)

Silicon Containing Cu-C Mesoscopic Composite and the Application for the Improving of Epoxy High Filled Compound

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The mechanic chemical synthesis silicon containing Cu-C mesoscopic composite is considered. The chemical mesoscopic ideas [1] are used at the discussion of the flowing processes. At the beginning the characteristics of Copper Carbon mesoscopic composite are given. The structure of Copper Carbon mesoscopic composite with active carbon shell is defined by means of the complex of methods including x-ray photoelectron spectroscopy, transition electron microscopy with high permission, electron microdiffraction and also EPR spectocopy.

According to the transition electron spectroscopy (TEM) with high permission the carbon shell structure for cluster of copper is presented as Carbon fibers. This fact is confirmed by electron micro diffraction results. The Carbon fibers formation is caused by realization of reduction oxidation (Red Ox) process with the appearance of reduced Copper and the carbonization of polymeric hydrocarbon chains [2].

The composition of metal containing phase in this mesoscopic particle corresponds to $CuO\text{-}Cu_2O\text{-}Cu$ (1.17%-5.19%-93.64%). In accordance with C1s spectra the carbon fibers contain the carbine and poly acetylene fragments.

According to the EPR spectrum of Cu-C mesoscopic composite carbon shell the coordination processes lead to the charges of metal electron structure with unpaired electrons formation. This process is accompanied by the metal aromic magnetic moment growth, as well as by the appearance of unpaired electrons on the carbon shell surface: g-factor is equaled to 2.0036, number of unpaired electrons is $1.2\cdot10^{14}$ spin/g, atomic magnetic moments are equaled to 1.3 μ_B.

The presence of active double bounds and delocalized electrons in carbon shell of Cu-C mesoscopic composite gives possibility for its modification by means of Red Ox and addition processes [3].

Therefore the interaction reaction of Silicon containing substance with the aforesaid mesoscopic composite is possible. The fact of this reaction is confirmed by Si2p x-ray photoelectron spectra. According to Si2p spectrum the relation of peak intensives shows the reduction oxidation process development on 51.4%. At the same time C1s intensity peak for C-H bond in containing Cu-C mesoscopic composite less on 65% in composition with the intensity peak of initial mesoscopic composite. In this case simultaneously the Copper atomic magnetic moment growth to 3 μ_B and the increasing of the unpaired electrons quantity to $3.4\cdot10^{19}$ spin/g is observed.

At the application of the Silicon containing Cu-C mesoscopic composite for the improving of properties of epoxy high filled compound leads to the increasing specigic impact elasticity more then to 2 times and shear strength on 20%.

Keywords: Cu-C mesoscopic composite, composite modified be Silicon, epoxy high-filled compaund

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SO-5 (P25)

Production of Lightweight Geopolymer Concrete

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Lightweight concrete has been known all around the world for decades. As a masonry material there is an advantage: it has sufficient strength despite of its light weight. Mainly it is used for the production of large wall elements and thermal insulation boards.

This research focuses on the production of lightweight geopolymer concrete samples. Aggregates, glass foam and alkaline solution (NaOH solution and water glass) were used as raw materials, lightweight aggregates and alkaline activator. During the examinations compressive strength, thermal conductivity and density of the geopolimer concrete samples were determined. Material structural and morphological tests were obtained by scanning electron microscopy (SEM) to charaterize the geopolymer concrete samples.

Keywords: lightweight, geopolymer, concrete, glass foam

POSTER PRESENTATIONS

P26

The Effect of Additives Materials on Phases and Properties of Lightweight Expanded Clay Aggregates

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This work is focused on studying the effect of additive materials such as tuffs and perlite on physicial, mechanical, expansion properties and mineral phases of expanded clay minerals. Samples were collected from Mályi quarry Miskolc, Hungary. They were characterized by XRF, XRD, SEM microscopy and heating microscope. The physico-mechanical properties of specimens were measured according to relating standards. Results showed that, perlite can be enhanced the compressive strength and expansion properties of the aggregates, in addition, these materials can be decreased the bulk density of the lightweight aggregates. In addition to, the sintering temperature approximately was decreased by 25˚C lower than the temperature used by the LWA manufacturing sector.

P27

Case Study of Failure Investigation Procedure of CMOS Imager Modules in Automotive Electronics Unit

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In this paper a case study will be introduced describing a failure analysis (FA) approach to CMOS imager modules of automotive electronic control unit (ECU). The units were investigated after life cycle tests. Functional deviation was observed during electrical parameter testing. To define the physical failure mode of this phenomena different kind of FA technics were compared in order to determine which gives detailed results in the most effective way. The applied FA technics were the following:

- scanning electron microscopy (SEM) after metallographic sample preparation: embedding in epoxy resin, perpendicular mechanical grinding, ion-polishing
- scanning acoustic microscopy (SAM): without sample preparation, but the sample were placed in ion-exchanged water (coupling fluid) during the measurement
- infra-red (IR) microscopy after simplified sample preparation: embedding in epoxy resin, parallel mechanical grinding to reach the bulk side of the CMOS sensor, polishing

Physical analysis revealed that the active area of the CMOS sensor module was delaminated from the bulk silicon in some areas.

Destructive and partly destructive analysis methods were performed to localize the failure and determine the failure mode. Delamination between the bulk and the active layers of the CMOS structure was detected by metallographic cross-sectioning and SEM. In order to eliminate the possibility of artifacts (caused by grinding and polishing) other failed CMOS modules were inspected parallel by SAM. The delaminated area could be detected, but higher resolution was required in order to accurately identify the concerned areas of the CMOS circuit. To reach that, IR microscopic investigation was performed on the same CMOS modules which were inspected by SAM. Imaging through silicon with near-IR light enables partly nondestructive analysis (sample preparation is necessary, but it does not affect the delaminated layers) which ensures that no additional external load applied by the grinding process. The investigation showed that the concerned area is always the same two side of the CMOS sensor where the bond pads are located. With IR imaging it was possible to determine the exact location and shape of the delamination in each unit.

This case study revealed that, among the used technics the IR microscopy seems to be the most effective FA technique to demonstrate the most detailed result.

Keywords: silicon chip, CMOS, IR microscopy, SEM, SAM, physical root cause analysis

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P28

Controlled Release Formulation of Spherical Silica Hybrid Liposome Particles for Cosmetic Applications

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This work investigated the various factors influencing the formation of silica-encapsulated liposome particles (SLPs) including the concentration of silica precursor and lecithin, and reaction temperature and solvent used, and then demonstrated a controlled release cosmetic application with a formulation of natural hemp-seed extracts as an anti-oxidant material. SLPs were prepared by sol-gel encapsulation reaction with tetraethyl orthosilicate (TEOS) as a silica precursor. The reaction on the hydrophilic region of lecithin was evaluated as a function of reaction time, concentration of silica precursor, and amounts of lecithin used, respectively. The SLPs obtained from various conditions were characterized by SEM, particle size analyzer, and FT-IR spectrophotometer to confirm their morphology and particle size. Then, a cosmetic application of the SLPs was demonstrated using the controlled release of natural hemp-seed extracts. The SLPs release profiles showed a sustainable release pattern with a maximum hemp-seed extract release as high as 90 $mgmL^{-1}/g$ per gram.

P29

Elimination of Plasticized Poly(vinyl Chloride) Degradability by Using Nanocement: A novel study

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There is no supernatural material in the universe, but there is a material can be having more than one characteristic that makes it unconventional and this which was discovered in this study. Based on the PVC heat stability measurements which obtained by dehydrochlorination test, it has been shown that nanocement is not only a material used to improve the properties of concrete; but also a material that has shown a significant indications in the stabilization process of plasticized poly(vinyl chloride). Where the rate of degradation of polyvinyl chloride was effectively decreased after adding nanocement, which means the nanocement act as an effective stabilizer for plasticized poly(vinyl chloride).

Keywords: Plasticized poly(vinyl chloride), Nanocement, Dehydrochlorination.

P30

Tunable Photoluminescence Controlled by Interface Defects Between Boron-Doped Nanocrystal Silicon and Silicon Oxide

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Boron (B) -doped silicon rich silicon oxide (SRSO) films with color-tunable photoluminescence (PL) are achieved by RF co-sputtering technique followed by post-annealing treatment. It is found that the interface defects in the interface between silicon nanocrystals (Si-NCs) and silicon dioxide matrix would change with the dopant increase which results in the variety of PL. In the B-doped SRSO films, the PL from the Si-NCs is quenched, while interface defects acting as luminescent centers dominate the PL. The interface defects including the weak oxygen bond, neutral oxygen vacancy and Eδ' centers are attributed to the origin of the luminescence. It is found that the ratio of these interface defects depends on the doping concentrations of B atoms. Hence, we can harvest color-tunable PL through the variety of interface defects controlled by the B doping concentrations, which may have a promising application in fluorescent powder or other Si-based optoelectronic devices.

P31

Usage of the Amorphous Silica for the Mullite Ceramic Preparation

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The scientific-practical attention of the research work focuses on the high-temperature heat-insulating mullite ceramics, which allows reducing heat loss, withstands rapid changes in temperature and simultaneously serves as a load-bearing construction material. Ceramic materials and its properties are formed as puzzles from several components and conditions. Each stage of the ceramics production, such as the choice of raw materials and ratio, preparation method, drying and firing of the ceramics, is important both individually and in common. During the formation of the ceramic material, it is necessary to balance all stages. Development of the modern technologies requires the production of materials with perfect and sometimes universal properties. The aim of this work is to investigate the influence of different ratio of kaolin, silica and alumina with different grain size of raw materials on the mullite formation and properties of the mullite ceramic.

The porous mullite materials were prepared by slip casting of suspension of raw materials where the aluminium paste (0.18 wt.%) was used as a pore former. Pore formation occurs due to the hydrogen formation in a chemical reaction between aluminium paste and raw materials, when water suspension is with pH>7. This method is more ecological than the method of additive of combustible matter [1, 2].

Keywords: mullite ceramics, SiO_2, kaolin, porous ceramics.

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P32

The Influence of Moulding Wastes on the Quality of Autoclaved Sand-Lime Materials

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The paper presents the results of investigations on the applicability of selected types of moulding wastes i.e. moulding and core waste casting masses and dusts from regeneration processes as alternative or supplementary materials in relation to quartz sand of natural origin used in manufacturing of sand-lime bricks. In the production technology of lime-sand products, quartz sand is used as aggregate. A rational factor in favour of such a technological solution is the high content of crystalline silica in the waste moulding or core compounds, in which the mineral matrix is composed of good quality quartz sands of natural origin.

The research concept covers production of a series of samples of autoclaved silicate materials formed by pressing, from raw material mixtures involving the discussed waste materials. In the laboratory tests conducted for the preparation of autoclaved materials of the sand-lime brick type, apart from traditional raw materials in the form of natural quartz sand and quick lime, different waste moulding and/or core sand materials and post-regenerative dusts were used for composing raw material mixtures. Mentioned foundry industry wastes were introduced into the basic raw material mix based on the gradually increasing substitution of quartz sand, in the amount within the range of 0-100% (% by weight).

The assessment of the possibility of using the discussed waste materials in the indicated direction was based on the result of a comparative analysis, covering the basic operational characteristics of two types of materials, i.e. reference material, obtained on the basis of a basic raw material mix, containing no such waste and several series of experimental materials, produced with various quantitative and qualitative contributions. The characteristics of the obtained autoclaved materials in the above range are also supplemented by the results of the leaching tests for heavy metals elements and the analysis of selected aspects of the microstructure carried out by the SEM + EDS method.

The results of the research show that it is possible to use processed waste casting compounds in the production of autoclaved lime-sand products. This processing of wastes is two-stage operation. The purpose of the first stage is to restore the original grading of quartz sand used to obtain foundry moulds and cores, while the second stage is to remove the organic binder residues that appear on the quartz sand grains in the form of thin layers hindering the reaction with quick lime used as a binder.

P33

Effect of Bismuth on the Structural and Optical Properties of the Sol Gel Processed Barium Titanate.

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In the family of ferroelectrics, barium titanate ($BaTiO_3$) is the most frequently utilized ferroelectric material and even after seventy five years of its disclosure, it remained as a most essential material which have excellent dielectric, optical, piezoelectric and ferroelectric properties [1]. Also, it is one of the most extensively studied leadfree ferroelectric materials due to its broad range of device applications viz. capacitors, transducers, non-volatile memories, positive temperature coefficient (PTC) thermistors, dynamic random access memory (DRAM) and many more [2]. $BaTiO_3$ is generally not used in pure form, but it is doped with special additives to modulate its properties for the purpose of very specific applications.

This work, tackles the synthesis by the sol gel process and characterization of pure and Bismuth doped Barium titanate ceramics. Powders were characterized by X-ray spectroscopy (XRD), Raman Spectroscopy, Infrared spectroscopy and optical measurements. The structural characterization of BBiT by X-ray diffraction (XRD) shows that our samples are crystallized in pure perovskite phase. Both Bi doping decrease lattice parameters and the tetragonality leading the development of pseudo-cubic symmetry, Raman spectroscopy spectra of pure and doped samples are in agreement with these XRD results. Our results show that BTO is a tetragonal-phase ferroelectric material with a band gap Eg≈3.21 eV. Moreover the Uv-visible spectroscopy measurements performed on the samples showed a decrease in the band gap with increasing Bismuth concentration.

Keywords: Ceramics, BaTiO3, synthesis, Sol-Gel, XRD, Raman, band gap.

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P34

The Content of the Adsorption Centers of High-Silica Porous Glasses

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High-silica porous glasses (PS) obtained by leaching two-phase alkaline borosilicate (ASC) glasses are ideal matrices for creating materials for selective filtration, because they have a unique set of properties, such as through porosity with nanoscale pores, chemical and thermal properties, as well as biological stability. PSs have a well-branched surface formed by the liquation channels of a chemical unstable phase, extracted by acid treatment of the original glass, and colloidal particles of the "secondary" silica inside the pore space [1,2]. Estimated long-term operation of PS as a membrane filter has led to the study of the dependence of the structural and surface characteristics of PS on the storage time in water. PS NFF of the composition (mol.%) $0.17Na_2O \cdot 5.96B_2O_3 \cdot 93.75\ SiO_2 \cdot 0.07P_2O_5 \cdot 0.05F$ was obtained by acid etching in the HNO_3 solution of the original two-phase alkaline borosilicate glasses of the composition (mol.%) $6.8Na_2O \cdot 22.1B_2O_3 \cdot 70.4SiO_2 \cdot 0.19P_2O_5$ 0.52F (thermal treatment 550 ° C/40 hrs) [3], kept in water for a long time, after which measurements were taken of the specific surface and examined for the presence of active centers on the surface formed by siloxane Lewis base centers (LBC) and silanol groups (Bronsted acid centers (BAC)). The specific surface was measured by the method of thermal desorption of nitrogen on a specific surface analyzer "Sorbtometer-M" with subsequent calculation by the BET method. The study of surface-active centers of various acidic forces was carried out using an indicator method [4] based on the selective adsorption of acid-base indicators from aqueous solutions on the surface of solids. The method of adsorption of acid-base indicators on the surface of the PS samples under study determined the content of LBC with pKa -0.3 (indicator - o-nitroaniline), corresponding to oxygen atoms in the siloxane bridging groups, and BAC with pKa 2.5 (indicator - m-nitroaniline), corresponding to hydroxyl groups (acid groups ≡Si – OH). It was found that with an increase in the thickness of the PS NFF plates, the number of siloxane groups of LBCs increased and the number of silanol groups of BACs a decreased. In addition, by the example of PS with a thickness of 2 mm, it was shown that a significant decrease in the specific surface was accompanied by a decrease in the content of the LBC and BAC groups, which can be explained by the recondensation of colloidal silica inside the pore space. The work was done as a part of the state assignment of the IChS RAS (theme No. 0097-2019-0015).

Keywords: high silica porous glass, Lewis and Bronsted centers, indicator method

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P35

Laser Ablation of Silicon Monoxide and Titanium Monoxide in Liquids: Formation of Mixed Catalytic Colloidal Dispersion with Catalytic Activity

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Silica–titania mixed oxides and composites have been extensively studied, whereas the titanium monoxide (TiO)–silicon monoxide (SiO) counterparts still remain to be explored. Laser ablation of silicon and titanium monoxides in liquids is in according with literature completely unexplored. Here we report on Nd:YAG pulse laser ablation of SiO and TiO in water and ethanol which allows generation of SiO and TiO nano/microparticles. Size distribution, process of coagulation and values of zeta potential of individual monoxides colloids and their mixture have been measured by DMS zetasizer. Morphology and chemical composition of ablatively nanoparticles and their agglomerates were analysed by SEM and EDX after evaporation of the solvents on Ta substrate. Catalytic activity of individual silicon and titanium monoxides and of their mixture has been tested in terms of methylene blue (MB) degradation under the daylight. Synergic effect of mixed monoxides has been studied.

Keywords: silicon monoxide, titanium monoxide, laser ablation in liquid, mixed colloid, catalytic activity

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P36

Role of micro silica on rheological properties of fresh concrete

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In this work the influences of micro silica on physico mechanical and rheological properties of cement pulps were studied by the authors. For these three types (A, B, C) of micro silica were used in 5 different composition (30, 40, 45, 50 and 55 m%) at 5 different (60°C, 65°C, 70°C, 75°C and 80°C) temperature conditions at continuesly increasing shear rates. During the experiments 5 different plastizesers were used. First the authors have prepared the mixtures of cement pulp and fresh concretes then the rheological tests were made in 1, 2, 3, 4, 5 and 6 hours later. The realized experiments have shown a very strong dependence of rheological properties on volumes of micro silica components, times and shear rates. During their experiments the authors have founded that the mass ratio of micro silica components has influenced on the rheological (forming) properties of fresh cement concretes and on the final (28 days) mechanical strengths of the prepared specimens. During the experiments Hake RS80 Rheotester and scanning electron microscopy were used by the authors.