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Prof. Dr. László A. Gömze
chair of conference board
PREFACE

The competitiveness is one of the most important component of our life and it plays key role in efficiency both of organizations and societies. The more scientific supported and prepared organizations develop more competitive materials with better physical, mechanical, chemical and biological properties and the leading companies are applying more competitive equipment and technology processes.

The aims the 4th International Conference on Competitive Materials and Technology Processes (ic-cmp4) and the Symposiums is-ism1, is-icbm2 and is-icm2 are the followings:

- Promote new methods and results of scientific research in the fields of material, biological, environmental and technology sciences;
- Change information between the theoretical and applied sciences as well as technical and technological implantations.
- Promote the communication between the scientist of different nations, countries and continents.

Among the major fields of interest are innovative materials with increased physical, chemical, biological, medical, thermal, mechanical properties and dynamic strength; including their crystalline and nano-structures, phase transformations as well as methods of their technological processes, tests and measurements. Multidisciplinary applications of material science and technological problems encountered in sectors like ceramics, glasses, thin films, aerospace, automotive and marine industry, electronics, energy, construction materials, medicine, biosciences and environmental sciences are of particular interest.

In accordance to the program of the conference ic-cmp4, and its symposiums is-ism1, is-icbm2 and is-icm2 we have received more than 450 inquires and registrations from 46 countries of 5 continents. Finally from them more than 240 abstracts were accepted for presentation, including the 10 PLENARY lectures. Scientists and researchers have arrived to Miskolc-Lillafüred (Hungary) from 41 countries of Asia, Europe, Africa, North and South America and Australia.

Together with co-authors in this book are presented abstracts from more than 700 scientists and researchers.

Prof. Dr. László A. Gömze

chair, ic-cmp4
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PLENARY LECTURES
Mesoscopic Model of Two-phase Composites Damage Under Mechanical and Thermal Loadings

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Metal Matrix or Ceramic Matrix Composites (MMC and CMC) have a non-linear and complex overall response to applied loads due to: different phases, existence of an initial porosity and internal microdefects. All microdefects act as stress concentrators and locally change the state of stress, leading to the development of mesocracks and finally macrocracks. Experimental results show that defects develop mainly intergranularly and cause inhomogeneity and induced anisotropy of the solid. Modelling of such material response is possible by multiscale approach describing different phenomena occurring at different scales:

- the microscopic level is associated with the degradation phenomena developing at the single grain. Micropores inside of grain or at the grain boundaries act as crack initiators. Microcracks spread along grain boundaries,
- the mesoscopic level corresponds to a set of grains, which create Representative Surface Element (RSE). The basic elements of the defect structure are: meso-cracks, which diameters correspond to the single straight facet of the grain boundaries structure, kinked and wing (zig-zag) cracks,
- the macroscopic level corresponds to the dimensions of the tested sample of the material. The composite is treated as a continuum with properties of the polycrystal calculated as averaged values over of RSE.

In this paper we investigate the thermo-mechanical response of 2-phase composites with particular numerical examples concerning WC/Co and Al\textsubscript{2}O\textsubscript{3}/ZrO\textsubscript{2}. The first step is to assess mechanical and thermal properties of these materials with completely random distribution of components with application of Computational Fluid Dynamics (distribution of heat fluxes and temperatures due to heat transfer) and Computational Structural Mechanics (estimation of the elastic thermo-mechanical response of the composite). In the next step stress distribution and places of their concentration were estimated. Concentrations play very important role, because they are sources of defects initiation and further propagation due to temperature variation during heat transfer. Finally the gradual degradation process was considered in both types of analysed composites.

Application of the presented CMC material model was done for modelling of TBC of turbine blades in jet engines. We assumed that the turbine blade has protective covering (TBC) in form of thin ceramic layers. Using sub-modeling technique - the critical stress levels for damage initiation and growth in the turbine blade were assessed.

\textbf{Keywords:} multiscale modelling of MMC and CMC, thermo-mechanical loading, damage initiation and growth, TBC coating of turbine blades
Gas adsorption induced joining for water vapor sealing of flexible device

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Adhesive free molecular joining method (1) was developed and applied for sealing of flexible film devices. Joining has been made by gas adsorption of water and silane to form hydrogen and covalent bonds by low temperature annealing and showed a strong adhesion 10N/25mm. Sealing performance of molecular joining was investigated by using joints of PET films on which high barrier layer was coated. Molecular sealing with 1 mm width was at first carried out by gas adsorption on both surfaces to be joined and then followed by laminating and annealing at low temperature to form bonds by chemical reaction and dehydration at the interface. MOCON measurements showed that water permeation property was better than 5 x 10^{-4} g/m2/day, where thickness of joining for sealing was determined by XPS to be at about 1-2 nm. Practical applications of thus developed molecular joining and sealing method will be discussed from the viewpoints of flexible and wearable film devices such as OLED display, solar cells and sensors.

References
Fifteen years of phase diagrams of materials by the Calphad methodology

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The roots of Calphad methodology takes place in the thermodynamic analysis of systems. We will discuss on the history of CALculation of PHase Diagram (CALPHAD). The history of CALPHAD is achieved in phase equilibria, by a realistic combination of basic thermodynamic principles, mathematical formulations the thermodynamic functions. The CALPHAD approach is born in 1908 after the research of Van Laar, who was the first to apply Gibbs energy concepts to phase equilibria. in that time he didn’t have the necessary numerical input to convert algebraic expressions into phase diagrams. during 1970 to 1980 period the modeling of phase diagram was developed by using computational algorithms and it was the first time when CALPHAD established itself as an accepted tool in the materials modeling. A high level of level of empiricism progressively decreases with time and with the use of new concepts, new softwares and more available experiments. The sentence write on the website calphad.org describe really this approach and lie to understand the development of this approach:

« We believe that substantial progress can be made in a short period of time if we could arrange to work together for one week at one of our facilities to define problems, disband, carry out some individual activities, and meet again for a week at a second facilities to compare results and chart future activities. » Larry Kaufman and Ibrahim Ansara, 1973.

The number of people involved in CALPHAD methods was also increasing and new researchers extends the field of Calphad research. The present state-of-art of CALPHAD is at a high level and it is a very varied and powerful approach to solving problems, associated with thermochemistry and phase equilibria. We will discuss the development during the 15 last years with the introduction of new tools.
Two novel multifunctional optical silicate and borate glasses doped with rare earth ions, molecular clusters, metallic nanoparticles, and semiconductor nanocrystals have been developed for photonic and plasmonic applications. Structure and properties of the materials and their applications for photonics and plasmonics have been demonstrated.

The first multifunctional material presents a silicate photo-thermo-refractive (PTR) glass doped with halogens (fluorine, chlorine or bromine), rare earth and silver ions. The PTR glass changes its refractive index after UV laser irradiation and following thermal treatment. For fluorine PTR glass the refractive index (RI) change is negative and achieves $\Delta n = -1000$ ppm. For chlorine and bromine PTR glasses the RI change is positive and achieves $\Delta n = +1500$ ppm. The PTR glass is successfully used for fabrication of holographic optical elements (HOE) like volume Bragg gratings, spectral filters, wavelength multiplexors, laser beams combiners as well as gradient index (GRIN) optical elements. The HOEs and GRIN-elements in the PTR glass reveal a high chemical stability, thermal, mechanical and optical resistance. The composition of the PTR glass allowed us to use the ion-exchange technology for fabrication of optical waveguides and silver plasmonic waveguides. The rate of etching of the crystalline phase in PTR glass is much higher than one for the glass host by factors of 15. The PTR glass can be successfully used for fabrication of 3D hollow microstructures, microfluidic devices, biosensors, micro-total analysis systems and micro-electro-mechanical systems. It was shown that PTR glass doped with rare earth ions can be very attractive for fabrication of distributed feedback lasers and optical amplifiers with Bragg gratings playing a role of spectral flattening filters.

The second multifunctional material presents a borate glass host with precipitated Cu-molecular clusters, CuCl or MnFe$_2$O$_4$ nanocrystals. The borate glass with CuCl nanocrystals exhibits good non-linear properties and can be used for control optical signals and optical limiters. The borate glass doped with Cu molecular clusters can be used as down-converters for solar cells and luminescent fiber sensors of temperature. And the borate glass with MnFe$_2$O$_4$ nanocrystals exhibits good magneto-optical properties and is very attractive for fabrication of Faraday rotators for optical isolators.

**Keywords:** multifunctional glasses, glassceramics, photo-thermo-refractive glass, silicate glass, borate glass, photonic devices, plasmonic devices.
Formation of carbon matrices by pyrolysis of diphthalocyanines of metals: structure analysis by neutron and X-ray scattering

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By the method of Fourier-analysis of X-Ray data there was decoded the structure of amorphous metal-carbon phase of pyrolysates of diphthalocyanines containing various elements (Y, La, Ce, Nd, Tb). The features of formation of porous carbon structures have been discovered as dependent on annealing temperature, atomic number and mass of basic elements. Low-temperature process (800°C) created rare structures with a small amounts of atoms while above 1000°C it was detected the integration of carbons into globules (size ~ 1 nm, number of atoms \( m \geq 100 \)) with stable size and mass in the range 1000-1600°C. Their density is close to this one for graphite, although at 1800°C the aggregation degree comes down due to losses of carbon and metal. For Yttrium the aggregation numbers showed a moderate growth with temperature up to \( m \sim 30 \), while the transition to heavier elements Lanthanum and Cerium leads to the increase in aggregation numbers up to \( m \sim 140 \). It was established that the growth of aggregates is more intense if a basic element forms carbide. According to neutron scattering data, carbon globules create branched fractal structures at the scale of few tens of nanometer. For instance, for the structures with Yttrium the fractal exponent \( D_f \sim 2.7 \) is higher than that for heavy elements \( (D_f \sim 2.4) \) which are integrated into massive structures inducing intense scattering. Finally it was found a similarity of matrices with multilayered carbon “onions” and volcanic material (shungit) that indicates some universal mechanisms governing self-assembly of carbon atoms at high temperatures.

Keywords: pyrolysate, structure, neutron and X-ray scattering
Novel Approaches of Silicon Carbide and Carbon Allotropes for Energy and Environmental Functions

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Silicon carbide is traditionally grown by Physical Vapor Transport (PVT) and considered for transistors. In recent years we have initiated a number of routes for SiC using modified growth approaches of the PVT method with particular aim for energy applications. Growth of doped hexagonal (6H-SiC) silicon carbide is a recent approach for a new type of white LED in general lighting. The all semiconductor technology has benefits in no need of rare earth metals, pure white light, thermal stability etc. The fluorescent properties are dependent on an interchange between growth and doping stability [1]. In comparison, the cubic SiC (3C-SiC) has a metastable nature that has hindered its progress even though it may be the host of a highly efficient intermediate band solar cell material, or it may generate hydrogen from water splitting or even split carbon dioxide. Recently, we have developed a growth process that allows growth of highly crystalline cubic SiC [2]. A key issue is the control of domains using a step flow growth mode. In addition, SiC is an almost ideal support and source for graphene, in particular with 3C-SiC [3]. However, the planar structure causes some limits in the use of the graphene. Therefore we have initiated studies of attaching carbon allotropes on epitaxial graphene. Carbon allotropes is a platform that consists of 0D, 1D, 2D and 3D structures like in graphene quantum dots, graphene ribbons, graphene flakes, carbon nanotubes, fullerenes, aerographite, nanodiamond, etc. The combined structure of graphene and carbon allotropes would have well defined properties since allotropes are firmly attached to the graphene/SiC. It thereby has additional benefits for example as advanced electrode or functional surface for example in biofuel cells or supercapacitors which could not be obtained by epitaxial graphene or by any carbon allotrope alone. Our new synthesis process of the carbon hybrid material is based on surface reconstruction and vapor species sublimation of SiC. These processes and materials may be further applied to enable new routes for carbon allotrope matrixes combined with Si-C composite films, for example in thermoelectrics.

We describe the new concepts and presents the status of our materials development, as well as brings up the issue of bringing such technology from research to market.

Keywords: energy, carbon allotropes, silicon carbide

Neutron Spectroscopy for Advanced Materials Characterisation and Development: A suite of high end tools for sophisticated analysis.

Anton P.J. Stampfl
Australian Nuclear Science and Technology Organisation

Neutron scattering has an illustrious history since the advent of the nuclear age and in many ways has a similar history of success that is comparable to electromagnetic-based probes, like electrons and photons. Unlike, photons, electrons and other charged particles, however, neutrons gently interact with nearly all materials making them practically the most ideal of probes to use to investigate material problems with.

So called cold, thermal and hot neutrons produced from a reactor or spallation source coupled with their properties of high penetrability (gentle interaction), and high magnetic and isotopic sensitivity, allow for a complete study of most materials, from the atomic to mesoscopic regime at a fundamental level.

Spectroscopy performed using neutrons further enhances studies to the time domain allowing dynamics to explored. Thus neutrons and in particular neutron spectroscopy is used in the characterisation and development of advanced materials from the concept phase through to applications.

At the research reactor, OPAL, in Sydney, a truly impressive variety of spectroscopic studies are being carried out into areas from gas entrapment in pipelines, clay swelling, solar cell development and energy production, immobilisation of radioactive waste, advanced materials characterisation for fourth/fifth generation and fusion reactors, catalysis for the green economy, sequestration, electronic applications (superconductivity, spintronics, quantum computing), and isotopic engineering.

Neutrons are a fantastic tool to use that provide sophisticated high end analysis of the most difficult of problems in advanced materials development and characterisation.
Rheological principles of development hetero-modulus and hetero-viscous complex materials with extreme dynamic strength

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Materials with different crystalline and morphological compositions have different chemical, physical, mechanical and rheological properties, including wear protection, melting temperature, module of elasticity and viscosity [1-4]. Examining the material structures and behaviors of different ceramic bodies and CMCs under high speed collisions in several years the authors have understood the advantages of hetero-modulus and hetero-viscous complex material systems to absorb and dissipate the kinetic energy of objects during high speed collisions. Applying the rheo-mechanical principles the authors successfully developed a new family of hetero-modulus and hetero-viscous alumina matrix composite materials with extreme mechanical properties including dynamic strength. These new corundum-matrix composite materials reinforced with Si$_3$ON$_2$, Si$_3$N$_4$, SiAlON and AlN submicron and nanoparticles have excellent dynamic strength during collisions with high density metallic bodies with speeds about 1000 m/sec or more. At the same time in the alumina matrix composites can be observed a phase transformation of submicron and nanoparticles of alpha and beta silicone-nitride crystals into cubic c-Si$_3$N$_4$ diamond-like particles can be observed, when the high speed collision processes are taken place in vacuum or oxygen-free atmosphere.

Using the rheological principles and the energy engorgement by fractures, heating and melting of components the authors successfully developed several new hetero-modulus, hetero-viscous and hetero-plastic complex materials. These materials generally are based on ceramic matrixes and components having different melting temperatures and modules of elasticity from low values like carbon and light metals (Mg, Al, Ti, Si) up to very high values like boride, nitride and carbide ceramics. 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: Ceramics, composites, diamond-like, elasticity, hetero-modulus, nanostructure, rheology, strength, viscosity

References
Diversity Oriented Synthetic Strategies for the New Molecules

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Synthetic methods that allow rapid access to large number of diverse structural arrays is growing constantly, which served as a new driving force for the advancement of synthetic organic chemistry. In order to synthesize large number of molecules with high level of diversity and complexity, in addition to developing new synthetic techniques and reagents, organic chemists are looking for exploring new methods to design and to evolve new molecules, strategies for new molecules leading to new source of diversity and improving the quality of compound libraries. This diverse new methodologies that will create many structurally diverse compounds efficiently in high yields and with excellent purity and with wide range of functional groups as handles to expand them further. Prof. M.V. Basaveswara Rao and his research group has been engaged in design and development of new efficient methodologies for a wide variety of heterocycles, displaying a range of skeletal and functional group diversity. Our aromatic and heteroaromatic annulation strategies are highly efficient, simple and results in variety of molecules with quantitative yields. The properties exhibited by several heterocyclic molecules make them prime interests of the industry, due to the fact that most of these structural arrays displayed opto-electronic properties. We have initiated work on the synthesis of heterocycles with an aim to understand their properties towards Non Linear Optic materials. We have synthesized indole skeletons initially and utilized them for making other heterocycles, like carbazoles and carbolines and their fused derivatives. All the synthetic methodologies reported by us and their utility will be discussed. Our aromatic and heteroaromatic annulation strategies are highly efficient, simple and results in variety of molecules with quantitative yields.

References
Preparation, Self-assembly & Properties of Hollow Colloidal Spheres

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Hollow micro/nanospheres and two- and three-dimensional ordered architectures have attracted tremendous interest in the past decades due to their well-defined morphology, low density, large surface area, functional characteristics, and wide range of potential applications, such as nanoscale chemical reactors, catalysts, drug delivery, pigments, photonic crystals, supercapacitors, gas sensors, pollutant removal, opto-electronic devices, surface-enhanced Raman scattering devices, solar cells, high-performance display units, capacitors, and transistors. Many physical and chemical methods have been developed to fabricate these hollow spheres and their ordered superstructures. Recently, an oil-water interfacial self-assembly has come to be considered an ideal strategy for the assembly of various low-dimensional nanostructures into nanofilms. The low-dimensional nanostructures are well dispersed in water, and then an oil phase is added to form an oil-water interface. After the addition of an appropriate amount of inducer, the decreased interfacial energy causes the nanostructures self-assembled into closely packed monolayer nanofilms at the interface.

Very recently, we have developed this self-assembly procedure to fabricate monolayer or multilayer nanofilm-based devices, using organic/inorganic core-shell composite spheres, inorganic particles, polymer particles as the building blocks. These ordered hollow or pore nanofilm-based devices display significantly improved properties compared to their nanoparticles and nanowires. In this talk, I will give some examples to demonstrate the recent works in my group.
Scientific basis for creation of construction materials based on titanium and aluminum minerals

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Currently there is a need in affordable and accessible materials with specific physical and chemical properties. Al- and Ti-minerals are excellent test objects for correlation between structure and physical properties of mineral. For example, corundum and ilmenite are related to the same structural type (rhombohedral lattice R3) and possess various physical properties. With the help of modern equipment we studied titanium- and aluminum-containing concentrates of natural raw and also products of various kinds of influences on them, which showed signs of nanostructuring.

The work was done under financial support of project 15-11-5-33.

Keywords: titanium and aluminum minerals, structure and physical properties
Oxides with Anomalous Thermal Properties for Obtaining a Composite Materials

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Materials with negative thermal expansion have received attention of researchers in recent decades. Scientific interest determined the establishment of the causes and explanation of the unique thermal behavior of this group of materials. Materials contracting upon heating can solve the technical problem towards the incompatibility of thermal expansion of the constructional design elements. The combination of materials with positive and negative values of thermal expansion in the required ratio allows obtaining materials with low or zeroing thermal expansion. The field of application of composite materials is widely including such areas as the production of high-precision optical mirrors, the thermal protection of descent module. Using of additive technology can produced ceramic components of complex shape.

It have been studied morphology and properties of ZrW₂O₇(OH)₂·2H₂O-precuror and ZrW₂O₈, obtained under the conditions of hydrothermal synthesis. Using the high-temperature X-ray analysis established the mechanism of formation of zirconium tungstate. The morphology of ZrW₂O₇(OH)₂·2H₂O and ZrW₂O₈ powders was similar and consisted of «whisker – like» particles, what evidenced by isomorphism of crystals.

The effect of temperature on the structure and properties of powders was investigated. The change the size and shape of samples, wetting angle, the contact angle between the sample and the substrate were determined. It was established that the wetting angle remained almost unchanged up to 900 K. A further increase of the temperature up to 1300 ± 23 K led to the decrease and subsequently the increase the θ values, associated with material spreading on the substrate upon heating.

High temperature in situ X-ray studies has shown that zirconium tungstate formed through X-Ray amorphous phase at 625 ± 25 K and remained stable from room temperature to 823 K. Further increase in temperature led to decomposition of zirconium tungstate caused by the change of the lattice structure by restructuring ZrW₂O₈ atoms to form sublattices of WO₃ and ZrO₂.

It have been shown that during sintering Al – ZrW₂O₈ mixtures were observed a decomposition of ZrW₂O₈ and formation of WAi₁₂ и ZrAl₃ on first stage and after 5 hours holding zirconium tungstate formed again whisker-like shape.
Paperboards are recognized to be important raw materials for packaging industry due to their advantages such as high strength-to-weight ratio, good surface smoothness, good printability, recyclability, etc. The paperboards can be converted and used as carton boxes, and they are also used as the raw material for manufacturing corrugated fiberboards (Cfb) which are subsequently converted into corrugated containers. In order to convert a raw paperboard or a Cfb into packaging containers, the boards are firstly printed at printing lines. Then, they are subjected to cutting and creasing processes. The objectivity of these two processes is to convert such printed boards into designed blank forms. After that, the blanks are folded and glued to obtain the containers[1]. Since coated and printed paperboards are kinds of laminated composite materials which are made of multiple plies and composed of numerous fibers, they normally exhibit complicated mechanical properties and forming behaviors. In addition, the successful forming of the paperboards tends to be more difficult than that of other monolithic resin or metallic sheet materials. Regarding the continuous development of advanced packaging materials and the requirement of smart formed products, understanding of sheet’s response behaviors and their forming characteristics are necessary for the converting industry. They strongly contribute to design suitable forming conditions and to develop smart forming technologies.

The author’s group investigated forming characteristics of a white-coated paperboard subjected to a sort of cutting and/or creasing process. In this presentation, a couple of current research works concerned board forming processes are reviewed. One of important forming characteristics is a dynamic stress (or strain) relaxation during a folding/creasing process. Apart from the in-plane uni-axial tensile deformation, the folding of creased part is complicated due to the structure of multiple plies. Recent author’s approach for estimating its dynamic behaviours is discussed[2]. Furthermore, a feasibility study of measuring technology of dynamic cutting characteristics of a coated paperboard is discussed from an aspect of non-destructive monitoring by the use of AE signals which are measured beneath the counter face plate or on the cutting blade[3].

**Keywords:** relaxation, plasticity, dynamic forming resistance, folding, cutting, paperboard

**References**


Multilayer BaTiO$_3$/NiFe$_2$O$_4$ thin films prepared by chemical solution deposition technique

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Development of new materials with advanced and multifunctional properties gives the opportunity to expand the limits of present technologies. Multiferroic materials have found special place in field of microelectronics due to their unique ability to exhibit more than one ferroic property. The aim of our research was to improve electric and magnetic properties of multilayer thin films with optimization of structure by changing the layer thickness, ultimately leading to higher values of magnetoelectric coefficient.

In this research, multilayer thin film structures based on ferroelectric BaTiO$_3$ and ferromagnetic NiFe$_2$O$_4$ have been investigated. The multiferroic thin films were obtained by spin coating of ferroelectric and ferromagnetic layers in alternating order on platinum coated silicon substrates. Sintering was performed at different temperatures up to 1000 °C. All the obtained samples have the thickness below 1 μm (12 deposited layers at most), with defined ferrite and titanate layers, flat and crack-free surface. The presence of perovskite and spinel phases was confirmed by XRD, but a small amount of undesirable secondary phase can be observed in the films sintered at 900 °C. Dielectric, and magnetic properties were investigated to confirm the applicability of the obtained films in microelectronic devices.
Blood combines cells - which have different size, shape, and functionality – that are embedded into a colloidal solution – the blood plasma. The forces between the red blood cells (RBCs) itself, as well as between RBCs and surrounding plasma molecules are relatively low, but can be quantified by means of small amplitude oscillation (SAOS). The shear history influences most measurements, since fresh blood is a suspension of living cells. Therefore blood can respond to external strain on a sometimes-individual basis. For instance, horse blood contains RBCs with pronounced aggregability and generates reproducible results more easily when it is measured after a certain rest following venous withdrawal. Time-dependent effects that overlay stress- or strain-dependent effects further contribute to the complexity of rheological blood measurements. For instance, if RBC aggregation is high in case of inflammatory disease, sedimentation of RBCs with subsequent phase separation in the rheometer gap must be considered at low shear forces. The frequencies that allow measurements in linear viscoelastic mode are close to the physiologic heart rate of the species (1 – 10 rad s\(^{-1}\)). Although human blood has elastic properties at the physiologic hematocrit, we never detected a \(G'\)-\(G''\)-crossover, indicating that native blood is a viscoelastic fluid in principal. However, when the hematocrit increases to pathological values, or when blood is aged, a jellylike texture can occur. RBC concentrates that are stored for transfusion purpose show such properties, although RBCs are morphologically still intact.

Much can be learnt from comparative animal studies. If RBC aggregability is lacking – which is the case in domestic ruminants and many rodents – \(G'\)-values cannot be detected at physiological hematocrit. Due to the low adhesive forces between RBCs and/or between RBCs and plasma molecules, the suspension needs an increment of cells to gain elastic behaviour. The frequency range and the maximum shear stress at which blood is a stable suspension are reduced. In case of severe trauma when RBC count and plasma fibrinogen concentration are low, the same occurs: the frequency range is narrowed and the yield stress of blood is decreased. In contrast, the gain of plasma fibrinogen during chronic inflammatory disease widens the frequency range and allows measurement at higher shear stresses.

We need to consider that SAOS measures blood at quasi-static conditions, if performed in linear mode, a circumstance that rarely occurs in the human circulation. Influences of vascular architecture, hydrodynamic effects, posture, pulsatility of blood flow and vessel wall interrupts the cohesion of bulk blood effectively. To integrate in-vitro rheological data into real clinical circumstances is still a challenging step.
Preparation of calcium carbonate/silicate compositionally graded hollow spherical particle by spray-drying

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In the spray-drying method, an aqueous solution containing dissolved materials is sprayed inside a furnace so that the materials undergo instant drying to form a powder. The powder is formed consists of hollow spherical particles. When the sprayed droplets of the aqueous solution evaporate, the dissolved materials form a particle wall. If two materials with different solubility exist in the solution, the particle wall is formed by depositing into low solubility material first, then, high solubility material deposited in the inner side continuously. I have previously reported the preparation of CaCO$_3$ hollow spherical particles by spray-drying$^2$. However, the mechanical strength of the CaCO$_3$ hollow spherical particles were weak. In the lecture, I will explain about the preparation of the mechanical strength of compositionally graded hollow spherical particles by spray-drying such that the resulting particles consist of CaCO$_3$ on the particle outside and silicate (potassium silicate) on the particle inside that assist mechanical strength.

A spray-drying solution (Ca(HCO$_3$)$_2$ solution) was prepared to blow CO$_2$ gas into a CaCO$_3$ suspension. Then, a potassium silicate solution was added 0–0.3 mass% to a Ca(HCO$_3$)$_2$ solution. The resulting mixture solution was spray-dried at a drying temperature of 100°C, a pressure of 200 kPa. The obtained particles were 2–5 µm in size. The wall thickness of particles without the addition of potassium silicate was approximately 200 nm, and this value was increased to 300 nm by adding potassium silicate. From (Electron Probe Micro Analyser; EPMA) image of the particles, the Ca indicates on CaCO$_3$ was distributed on the particle outer surface, and the Si indicated on potassium silicate was distributed in the inner side of particle wall. Furthermore, these compositions graded, it is not simple double-layered structures. The compressive strength of the obtained hollow particles was estimated by measuring the micro-compressive strength testing equipment of only one particle. The compressive strength of CaCO$_3$/potassium silicate hollow particles was improved about 15% and became 14 MPa.

**Keywords:** spray-drying, hollow spherical particle, calcium carbonate, silicate, mechanical property

**References**
Silicon Carbide, due to its large band gap, thermal stability and high radiation hardness is a unique semiconductor material. Prominent applications are high power devices and radiation detectors. Usually the irradiation (by hadrons, ions, electrons and gamma rays) of condensed matter, semiconductors in particular, results in the creation of point defects following defect reactions ending up in stable defect configurations. These defects, when in the active region, will alter device performance if their electronic defect levels are within the band gap and their density approaches the doping density of the material.

After a brief review on the nature and spectral energy distribution of solar radiation and radiation fields related to sources such as nuclear reactors or the LHC@CERN, the expected defect generation rates in relation to the incident radiation will be revisited. Factors that influence the recombination current in detectors or decrease avalanche voltages in power devices are discussed. Moreover, methods to detect and quantify pertinent radiation induced defects are demonstrated by means of SiC diodes.
Characterization of Materials: Uncertainty Budgets in Measurement and Testing

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The comprehensive knowledge of material properties as engineered and the material behavior in use is of huge importance for the functionality and reliability of processes and products. Hence, the accurate determination of material properties is a prerequisite for the applicability of a given material. Nowadays, macroscopic product features are originated by material properties on the microscopic and nanoscopic scale. This is a general challenge for uncertainty budgets in measurement and testing based on a conformity assessment [1].

Testing labs accreditated according to DIN EN ISO/IEC 17025 have to meet these demands. This paper focuses on the measurement of physical quantities (measurand with value and unit) and on the testing of material properties (qualitative, semi-quantitative or quantitative characteristics) of solid state materials. For both measurement and testing, the expression of uncertainty according to GUM [2] is essential. Regarding the object/sample one has to subdivide in surface and bulk features, from the measurement/testing point of view destructive vs. nondestructive procedures have to be distinguished. Moreover, direct measurement/testing and model-based determination of quantities/properties of interest require different approaches regarding the expression of uncertainty. For surface quantities and properties as discussed here, further considerations have to be made with respect to localized (lateral extension, native or artificial) or stratified (homogeneity and isotropy) material features vs. mapping (integration area) and imaging (lateral resolution and field of analysis) specifications of the measurement/testing procedure.

The concept of uncertainty budgets and the expression of uncertainty is introduced and discussed in more detail for the nondestructive model-based determination of thickness by spectroscopic mapping and imaging ellipsometry, the destructive determination of Brinell and Vickers hardness by means of a hardness tester and centrifuge technology, and the destructive determination of adhesion/cohesion respectively compound (adhesive or bonding) strength by means of the pull-off test using a tensile testing machine and centrifuge technology. It is shown that the expression of uncertainty for these examples and real materials requires different approaches resulting in either quantity values with unit or qualitative/quantititative attributes with calculated/estimated uncertainties in agreement with GUM.

**Keywords:** measurement and testing, uncertainty budgets, expression of uncertainty

**References**
Concentration Fields of the Phase Diagrams for the Materials Genome Decoding

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Materials Genome Initiative was announced on June 24, 2011 [1]. Prof. Hafid Aourag has offered the “Materials Genome Project” 3 years earlier [2].

A similar research was made in Ulan-Ude, Russia (Institute of Physical Materials Science of the Siberian Branch of the Russian Academy of Sciences) by means of 2 projects of the Russian Foundation for Basic Research: # 98-03-32844 “The design of the microstructures of multi-component materials according to the equations of the boundaries of single-phase regions” (1998-2000) and # 01-03-32906 “The genotype of heterogeneous material (boundary of concentration domains with the unique schemes of phase transformations and thermodynamically unstable fragments of phase fields in the multidimensional diagrams of the known and forecasted topological types)” (2001-2003).

These possibilities of phase diagram application for computer-aided design (CAD) of heterogeneous materials were presented in the form of 6 reports in Kyiv (Ukraine) in 2001 at the 6th International School-Conference “Phase Diagrams in Materials Science (PDMS VI)”. In 2004 these papers were published in Germany [3]. Novel interpretation for the term “heterogeneous design” is given in [4-11]. Along with the well-known and wide-spread "molecular design" and "structural design", this new approach in solid state chemistry and physics opens up new opportunities for multiphase materials engineering.

Keywords: materials genome, concentration fields, phase diagram, trajectory of phases

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Structure and Function Tuning of Nanostructured Oxides for Advanced Energy and Environmental Photocatalyst

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Titania (TiO₂) nanotube (TNT) that is categorized as one-dimensional (1D) nanostructured materials can be synthesized by simple solution chemical processing at fairly low temperature without any template [1]. The TNT has significant multifunctions such as photocatalytic, molecular adsorption, photoluminescence, biocompatible properties and so on, due mainly to its unique synergy of semiconductive properties of titanium oxide (TiO₂), low-dimensional and crystalline structures [1,2]. In this research, nanostructures, physical, chemical and photochemical properties of TNTs have been investigated. Special emphasis has been placed to modify the TNT by immobilizing zero-dimensional (0D) metal nanoparticles and doping metal ions to tune and enhance photochemical functions.

Noble metal nanoparticles such as Pt and Pd were loaded to TNTs using ex-situ photoreduction, X-ray or sonochemical methods. These TNT/NPs 1D/0D nanocomposites exhibited excellent hydrogen evolution performance by UV induced water-splitting test, showing their high potential as the energy creating photocatalyst.

On the other hand, metal ion doping such as Cr and Ru introduced additional optical absorption peaks among the visible light region. These contributed to the visible-light responsibility for the photocatalytic properties. In addition, obtained TNTs exhibited not only photocatalytic molecular degradation performance but also excellent selective molecular adsorption capability for specific organic molecule due to TNTs unique crystalline structure and the surface charge effect introduced by the doping of aliovalent ions to the TNT lattice. Further, we have recently invented simple process to synthesize visible-light responsible TNTs; these exhibit large red-shift of optical bandgap down to 2.8~2.4 eV, which is significantly low compared with that of bulk TiO₂ (3.0~3.2 eV) crystal and TNT (3.4 eV) as well. Resultantly, modified TNT exhibited the both excellent photocatalytic properties.

In this paper, relationships between materials tuning processing, nano/crystalline structures, surface chemistry, environmental and energy functions based on the TNTs physical-photochemical (i.e. photocatalytic) properties will be discussed in detail.

Keywords: titania, nanotubes, doping, metal nanoparticles, photocatalyst, water splitting, multifunction

References:
Studying hot tearing in Al-Zn alloys using in situ XR diffraction during solidification

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Hot tearing or solidification cracking is a detrimental flaw in casting that leads to the rejection of the part. This defect corresponds to the initiation of an intergranular crack during solidification at high solid fractions due to the coexistence of both a poor liquid feeding and the appearance of tensile or shear stresses in the coalesced grain structure. Al-Zn alloys are the base of the aeronautic AA7xxx aluminum alloy series. Depending on their solute content, they can be very prone to hot tearing.

In the present work, castings in dog bone shape molds have been carried out in situ under a high energy XR beam to study by XR diffraction (XRD) the stress/strain build up during solidification. The dog bone shape mold allows the sample to build up internal stress naturally as its contraction is prevented. The cooling on both extremities of the mould induces a hot spot at the middle of the sample which is irradiated by X-ray. Diffraction patterns were recorded every 0.5 s using a detector covering a 426 x 426 mm² area. The change of diffraction angles allowed us to measure the general decrease of the lattice parameter of the fcc aluminium phase. At high solid volume fraction, a succession of strain/stress build up and release is explained by the formation of hot tears. Mechanical coherency temperatures, 556°C – 593°C, and solid volume fractions, ca. 98 %, are shown to depend on solidification time for grain refined Al 6.2 wt% Zn alloys.

Keywords: hot tearing, heat treatable 7xxx aluminium alloys, stresses, XR diffraction.
A non-contact optical measurement technology based on digital image correlation (DIC)

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Non-contact optical measurement technology has made in recent years a major step in terms of speed and accuracy. The measurement system Mercury shows these benefits in practice – it measures general deformations (strains and displacements) and any distance change between two markers set in any axis with an accuracy of up to 0.01 pixels (depending on the frequency of the camera used).

Deformation measurement of samples during material testing by means of Video extensometer Mercury RT allows using multiple virtual probes and advanced image features to be tracked, including the natural pattern of the samples surfaces. The Mercury system enables the user to analyze the measurement either in offline or online modes, thus high-speed cameras can be used. The computation is performed in real-time, where the computed values are transferred to a connected test rig, either via analog or digital outputs.

Typical use is the determination of various material properties (tensile tests, uni/biaxial properties, contractions), measurement of large strain rates, high-speed testing, vibration measurement, crack propagation, dynamic testing, quality control. The Mercury system can be connected to industrial and DSLR cameras. With proper selection of camera lenses the system can be applied to any application from microscopic to macroscopic sizes: metal samples, wires and foils, wooden samples and composites, various fabrics and nanofiber structures, elastomers and tissues, plastic samples and biomaterials, cords and ropes, concrete samples and ceramics materials.

Keywords: material testing, material properties, video extensometer, digital image correlation, vibration, deformation, test rig, non-contact, elongation, contraction, cameras, lenses.
Effect of Annealing on Nanoindentation Hardness and Elastic Modulus of Platinum Implantable Electrodes

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In order to investigate effect of annealing on the cold-rolled platinum used for implantable electrodes 0.03-mm-thick cold-rolled platinum foil stripes with 99.99 wt.% purity were used. Nanoindentation measurements of elastic modulus and hardness were conducted using Agilent Nanoindenter G200 with XP head and Berkovich indenter. Dynamic Continuous Stiffness Measurements (CSM) were performed in order to check the changes of the modulus and stiffness with changes of displacement and standard tests according to ISO 14577 with depth control for different depths. Finally, the microstructure of the stripes was investigated by optical microscopy.

Results of nanoindentation experiments support results of microscopic investigation and allow concluding that recrystallization of platinum starts between 450 and 680°C that is detected by changes in grain size and mechanical properties. While after annealing at 950°C the biggest grains are formed, which result in decrease of elastic modulus and hardness.

Investigation showed that technology, particularly annealing, plays a major role in optimization of platinum electrode properties and functionality. Proper selection of annealing temperature for example can lead to improvement of mechanical properties up to 60%. Nanoindentation proved to be a proper technique in optimization of technology for producing new generation implantable platinum electrodes.

Keywords: nanoindentation, platinum, electrodes, annealing
SESSION 1

Advanced Materials for Bio- and Medical Applications
Osteogenesis by bone marrow cells in a bi-phasic scaffold consisting of circular cylindrical porous hydroxyapatite and acetalized polyvinyl alcohol sponge in vivo

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To regenerate a pulp-dentin complex, bi-phasic scaffold composed of formalin-processed acetalized polyvinyl alcohol sponge (PVF sponge) and cylindrical porous hydroxyapatite (HA) with a hollow center was devised. The scaffold, with rat bone marrow cell (rBMC) seeding in the sponge, was implanted into rat dorsal subcutis. Osteogenesis was evaluated immunochemically and histologically. The purpose of this study was to evaluate the effects of a bi-phasic scaffold for bone formation as a scaffold.

A PVF sponge (Diameter: 5 mm, Height: 10 mm) was inserted into a hollow center (5 mm) of a HA (Diameter: 8 mm, Height: 10 mm). In the sponge, $1.5 \times 10^6$ of rBMCs obtained from femora of 344 male Fischer rats were seeded. In dorsal subcutis of syngeneic rats, bi-phasic scaffold with rBMCs, PVF sponge separated from the scaffold after rBMC seeding, HA without PVF sponge which received rBMCs, or PVF sponge without rBMCs was implanted for 6 weeks and removed ultimately for immunochemical or histological examination.

Osteocalcin (OC) in each material was measured immunochemically. Data are expressed as mean ± standard deviation, and were statistically analyzed with the Tukey-Kramer test ($p<0.01$). For histological examination, 9 µm thick serial sections of each paraffin-embedded material were made.

OC quantity in bi-phasic scaffold with rBMC was $2.86 \pm 0.23 \mu$g/scaffold. In HA removed from bi-phasic scaffold containing rBMCs, OC was $1.70 \pm 0.08 \mu$g/scaffold. OC levels in the PVF sponge, with and without rBMCs, were low.

Histologically, in a bi-phasic scaffold composed of HA and an rBMC-seeded PVF sponge, newly formed bone was observed in both. In the scaffold, it is thought that rBMCs in a sponge would migrate into pores of HA. In many HA pores and in the bi-phasic scaffold sponge with rBMC-seeding before implantation, bone was observed; however, this was absent in sponges removed from the scaffold.

Keywords: osteogenesis, bi-phasic scaffold, hydroxyapatite, polyvinyl-alcohol sponge, bone marrow cells, osteocalcin, in vivo
Pills violate the physico-chemical state of the stomach contents

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We have conducted research of dynamic changes in viscosity (solidity), thermal, acidic and osmotic gastric contents activity in adult healthy volunteers after the ingestion of whole tablets (without grinding) on an empty stomach or portions of ordinary food with thorough mastication in the morning, afternoon and evening under hospital conditions. Studies of changes in similar physical and chemical properties of the contents of 1000 ml translucent colorless plastic containers after the administration of the same “tablets” without grinding or the same food cut and blended in a soft mixture were also carried out under simulated conditions. Shortly after the “tablets” and food natural gastric juice $\mathrm{pH}$ 0.8 – 1.2 was added into the container. Studies under simulated conditions were conducted at a temperature of $+37^\circ\mathrm{C}$.

Initial values of physical and chemical properties of food and “tablets” were explored before their ingestion and addition into containers in vitro at a temperature of $+25^\circ\mathrm{C}$. To realize that prepared portions of food from a typical menu of a hospital were minced and mixed.

The mixture had a soft body like gruel and a very good fluidity. Viscosity values of the mixture ranged from 200 to 500 centipoises; values of acid activity were within the range of $\mathrm{pH}$ 4.5 – 8.0; values of osmotic activity varied from 240 to 340 mOsmol/L $\mathrm{H}_2\mathrm{O}$.

Study outcome of a handful of 20 various tablets showed that all the tablets were dry and solid. The handful itself had a flowing property. The tablets had a disk form of 5.9 – 19.9 mm in diameter, thickness (height) of 2.0 – 6.1 mm, volume of 0.1 – 1.0 sm$^3$ and a specific deformation pressure value ranged from 0.03 to 160 N/mm$^2$.

The examined mixtures had a viscosity value of 100 – 300 centipoises, acid value of $\mathrm{pH}$ 6.0 – 7.1 and osmotic value of 240 – 340 mOsmol/L $\mathrm{H}_2\mathrm{O}$ 30 minutes after having breakfast, lunch and dinner and after adding minced and mixed portions of the same food into the plastic containers. 30 minutes after the administration of tablets in healthy volunteers on an empty stomach or into the plastic containers there was suspension with remains of undecomposed tablets inside the cavities. The fluid had a viscosity value of 0 – 10 centipoises, acid value of $\mathrm{pH}$ 2.0 – 3.3 and osmotic value of 340 – 600 mOsmol/L $\mathrm{H}_2\mathrm{O}$. 
Sodium Hypochlorite Influence on Detoxification System of Man Organism – Practical Application and Thermodynamic Analysis

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Practical applying and thermodynamic analysis, modeling and calculation of chemical transformations occurring as a result of sodium hypochlorite NaClO solution injection in blood plasma are carried out. Thermodynamic research is executed on the basis of methods complex (realized in software-information package) for thermodynamic description, modeling and calculation of phase-chemical transformations in multicomponent systems.

Information on interaction NaClO with components of plasma (form elements - erythrocytes, leukocytes, thrombocytes, etc.), and also on subsequent sodium hypochlorite influence on detoxification functions of organism is necessary for realization of correct and whole calculations.

As is known, under many pathological states in emergency surgery and traumatology one of most complex problems is struggle with endogenous intoxication, which structure include endotoxins accumulation, activation of lipids peroxide oxidation with forming toxic products of radical-free reactions. One of new directions in treatment of endogenous intoxication is using exactly sodium hypochlorite (electrochemistry obtained - by electrolysis of 0,9% NaCl solution).

The results of modeling give a substantiation that the solution NaClO has ability to oxidize not only freely circulating toxins, but also fixed ones on membranes of erythrocytes (0,06% NaClO solution travenous-dropwise injected – for patients with endogenous intoxication of 1 and 2 degrees).

Such parameters of blood, as average molecular mass of substances (AMMS), index of leucocyte intoxication (ILI), index of nuclear shift (INS), index of intoxication (II) and s.c. limulus-test were examined. The last one is referred to specific reactions on presence of toxin into organism.

The checkup was spent at the patients before injection of NaClO and after it through 3, 8, 24 hours. As an analysis result of the laboratory data a decrease for these parameters is revealed: for AMMS - on 37%, ILI - on 49%, INS - on 27%, II - on 30%. On the data of the limulus-test decrease of toxic components concentrations in a blood is marked also.

The recieved data allow to give estimations of thermodynamic activity for main components of blood, of influence upon blood of NaClO solutions and their alkalization, hydroxylation and radical reactions, chloridizing effects of influence on protoplasm cells, and also to check verity of these and other possible mechanisms of considered biological processes.

Thus, the medicine material and thermodynamic physical-chemical methodology finds logical explanation and (that is main conclusion) gives possibilities to study and open too (in number of cases – at presence of minimally necessary thermodynamic information) mechanisms of biological influence NaClO: its application in complex treatment of the patients with endogenous intoxication of 1 and 2 degrees eliminate hypoxia, improves blood circulation, normalizes detoxification functions of organism.

Keywords: medical materials, sodium hypochlorite, blood plasma, injection, composition, oxidation, parameters, thermodynamics, modeling, analysis
Electrospun Biocomposite Scaffolds Based on Polycaprolactone with Silicate-Containing Hydroxyapatite

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INTRODUCTION. The use of bioresorbable synthetic polymer materials in bone tissue engineering has gained a lot of attention. For better calcification and bone formation silicate-containing hydroxyapatite (Si-HA) can be used. This work aimed on the modification of the individual polycaprolactone (PCL) scaffolds with Si-HA particles via electrospinning to enhance its bioactivity. To the best of our knowledge, this is the first research work on use of Si-HA in combination with PCL electrospun fibers in the biomaterials field.

MATERIALS AND METHODS. Preparation of PCL/Si-HA solution: PCL polymer was dissolved in chloroform at a concentration of 9% (w/v). A powder of Si-HA ($\text{Ca}_{10}(\text{PO}_4)_{6-x}(\text{SiO}_4)_x(\text{OH})_{2-x}$, $x = 0.8$) was suspended into the solution at a concentration of 5% (w/w). Electrospinning conditions: 0.55 mm for the needle inner diameter and 10 ml for the volume of a syringe, 2 ml/h for the flow rate, 7 kV for the applied voltage and 50 mm for the distance between tip and collector. Pure PCL fibers were taken as a reference. Mechanical properties: a mechanical testing machine (Instron 3369, USA) was used for determination of tensile parameters. The samples were a size of 0.35mm×0.25 mm×1.3mm. The loading rate was set at 10 mm/min at room temperature. Cytotoxicity study: for in vitro experiments dermal fibroblast (HF) was used for estimation cells viability.

RESULTS AND DISCUSSION. SEM images of pure PCL and PCL/Si-HA composites demonstrate uniform structure of fibers in both types of samples. In case of PCL/Si-HA the difference in fiber diameter is more distinctive and porosity level is higher, compared to pure PCL. The presence of Ca, P and Si was confirmed by EDX analysis. Mechanical testing shows decresing strength of the samples after adding Si-HA particles into the structure. According to in vitro tests all samples show high cell proliferative activity and effect of toxicity was not observed.

CONCLUSION. The modification of PCL fibers leads to increasing porosity parameter. Based on the literature available, it can effect on better cell migration and proliferation. It also was found that with adding Si-HA particles to the structure of PCL fibers mechanical strenght is decreased. The study provides a basis for future optimization and investigation of PCL/SiHA scaffolds for bone tissue engineering.

Keywords: biomaterials, polymer, ceramic, hydroxyapatite, electrospinning
Fibrous Materials on Polyhydroxybutyrate and Ferric Iron (III)-Based Porphyrins Basis: Physical-Chemical and Antibacterial Properties

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Ferric iron (III)-based complexes with porphyrins are the homogenous catalysts of auto-oxidation of several biogenic substances. The most perspective carrier for functional low-molecular substances is the polymer fibers with nano-dimensional parameters. Application of natural polymers, poly-(3-hydroxybutyrate) or polylactic acid for instance, makes possible to develop fiber and matrice systems to solve ecological problem in biomedicine. The aim of the article is to obtain fibrous material on poly-(3-hydroxybutyrate) and ferric iron (III)-based porphyrins basis and to examine its physical-chemical and antibacterial properties. The work is focused on possibility to apply such material to biomedical purposes. Microphotographs of obtained material showed that addition of 1% wt. ferric iron (III)-based porphyrins to PHB led to increased average diameter and disappeared spindly structures in comparison with initial PHB. Biological tests of nonwoven fabrics showed that fibers, containing ferric iron (III)-based tetraphenylporphyrins, were active in relation to bacterial test-cultures. It was found that materials on polymer and metal complexes with porphyrins basis can be applied to production of decontamination equipment in relation to pathogenic and opportunistic microorganisms.
Analysis of structure and dynamics of superfine polyhydroxybutyrate fibers for targeted drug delivery

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Creation of polymer matrix systems for targeted drug delivery into a living organism is a challenging problem of modern treatment of various diseases and injuries. Poly-3-hydroxybutyrate (PHB) is commonly used for development of therapeutic systems. The aim of this article is to examine the changes in structure and morphology of fibers in presence of dipyridamole (DPD) as model drug for controlled release. It was found that addition of dipyridamole led to disappearance of spindle-shaped nodules on fibers of PHB in comparison with pure PHB. The research of thermophysical parameters showed that specific melting enthalpy (and the degree of crystallinity) of PHB fibers increased with the addition of DPD. With the increasing of DPD content in PHB fibers, more perfect and equilibrium crystal structure was formed. According to analysis of intercrystalline regions of PHB fibers, it was found that as the crystallinity of PHB in intergranular regions rose, the corresponding decrease of radical rotation speed was observed. It was concluded that fibers of PHB can be used for creating therapeutic systems for targeted and prolonged drug delivery.
Biodegradable Nanocomposites With Encapsulated Magnetic Nanoparticles For Controlled Drug Release

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Nanocomposites consisting of polymers and encapsulated functional particles represent innovative stimuli-responsive materials, which are currently used in progressive technologies. Among magnetoresponsive nanocomposites (MNCs) the special interest is attracted by matrices with ferrimagnetic nanoparticles (MNPs) that have the direct sensitivity to magnetic fields and also show the local hyperthermal effect used in non-invasive therapy.

This report is devoted to the study of magnetic and diffusion characteristics for the anisotropic and isotropic MNCs. Morphology and kinetics of drug release as well as ferromagnetic resonance (FMR) in the PHB-Cht system were first described. The FMR spectra are sensitive to formation of linear aggregates and allow us to calculate the fraction of particles included in the linear aggregates. The FMR results are also in accordance with the SEM micrographs. MNPs in nanocomposites can control drug diffusion under an external magnetic field. The encapsulation of MNPs in MNCs affects drug release owing to the steric factor, the free volume variation, and magnetic interactions.

Additionally, the properties of MNCs were investigated by Moessbauer spectroscopy and WAXS. It shown that MNPs in MNC exist in two forms: as nanosized superparamagnetic clusters and paramagnetic Fe\textsuperscript{3+} ions. The regulation of drug release transport can be performed by remote magnetic control in novel therapeutic systems.

The work was supported by RFBR No 14-03-01086.

\textbf{Keywords: biodegradable nanocomposites, magnetic nanoparticles, poly(3-hydroxybutyrate), chitosan, controlled drug release, iron oxide, ferromagnetic resonance, Moessbauer spectroscopy}
Free radical oxidation approach to creation of stable protein coatings on magnetic nanoparticles for biomedical applications

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Magnetically targeted nanosystems are considered to be applicable in different areas of biology and medicine such as hyperthermia, magnetic resonance imaging, immunoassay, cell and molecular separation, a smart delivery of drugs to target cells. A novel approach based on protein liability to free radical modification, leading to the formation of intermolecular covalent cross links has been developed for obtaining coatings assembled from protein molecules on the surface of magnetite nanoparticles in dispersions. The properties of the coatings obtained from serum albumin, thrombin, hemoglobin, and immunoglobulin G have been studied with the help of dynamic light scattering (DLS), UV/Vis spectrophotometry, antibody-antigen test and the method of spectral-fluorescent probes. Albumin molecules in MNPs coatings have been shown to retain their capability of binding with a dye and be conformationally stable.

It has been proven that coatings composed of protein macromolecules could be 1) stable, 2) formed around individual nanoparticles and 3) have several nanometers in thickness. The free radical linking of thrombin and immunoglobulin G on the surface of nanoparticles has been shown to almost completely keep native properties of the protein molecules as potential therapeutic products and biovectors.

The free radical linking method also reveals new possibilities for design of single-layer multiprotein polyfunctional coatings on the surfaces of all the nano-, micro- and macroobjects containing metals of variable valence (for example, Fe, Cu, Cr).

The reported study was funded by RFBR and Moscow city Government according to the research project № 15-33-70019 «mol_a_mos», by RFBR, according to the research project No. 16-34-60244 mol_a_dk.
Stable metal nanoparticles study for biomedical and green houses applications

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Metallic nanoparticles (MNP) with diameter ranging from 2 to 100nm have received extensive attention during the past decades due to their high potential applications such as catalysis, biomedical and cancer therapy for noble-metal nanoparticles.1 The main interest in MNP comes from their unique chemical and electronic properties due to their large surface-to-volume ratio.2 The advances in preparing MNPs have improved the MNP samples remarkably giving monodisperse nanoparticles with a good size and shape control and distribution during the synthesis. There is a need for a simple, reliable, surfactant free low cost method for producing homogeneous stable metal nanoparticles and we recently developed one that enables the production of surfactant free stable MNPs under air.3 Their unusual stability in air allows applications previously difficult to implement with metal nanoparticles produced using conventional methods due to their pyrophoric properties. This paper presents the study of ultrastable metal nanoparticles (Ag, Co and Ag_Co nanocomposites) aimed to be used for biomedical applications. Transmission electron microscope micrographs revealed highly crystalline monodisperse nanoparticles and thermogravimetry analyses did not show any weight loss demonstrating the high purity of these MNPs. The biocidal properties of these MNPs were also studied against bacteria and fungi. This study shows that these MNPs do not degrade with time and can be recycled after use. To validate a potential use of these MNPs, their toxicity was also studied using Human Embryonic Kidney Cells (HEK 293) and cancer cells. The properties of these nanoparticles will be discussed in terms of possible biomedical applications. Financial support from through the Estonian Research Council (grant number PUT431), Marie Curie (PERG05-GA-2009-249243) and the Estonian Road Map infrastructure and the NAMUR projects is acknowledged.

References
In the last decades, a wide range of multifunctional systems having different morphologies have been designed for biomedical applications such as magnetic resonance imaging (MRI) contrast agents, hyperthermia, tissue repair, biosensors, immunoassays and drug delivery systems (DDS). In this work, nest-like pore-hierarchical magnetic-sensitive barium hexagonal ferrite (BaO·6Fe₂O₃) microspheres for targeted DDS were engineered by a molecular self-assembly, wet-chemical sol-gel method via spray drying. Structural and morphological characterization was carried out by X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM), respectively. Coalescence of meso-(~12 nm) and macropores (~200 nm) are expected to allow efficient fluid transport and to improve the surface area for the payload on nest-like BaO·6Fe₂O₃ DDS. Moreover, the magnetic behavior allows applying an external magnetic field in order to target the drug at specific sites in the body.

**Keywords:** Biomaterials, Drug delivery, Barium hexagonal ferrite
Development and application of monocrystalline and nanostructural materials for medical X-ray sources

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Results of own works of the authors and works of the Russian, Japanese, Korean and American experts in refractory metals nanocomposite with increased strength characteristics for cathodes and anodes are analyzed in this report. It is shown, that though monocrystals have shown the better characteristics in comparison with traditional polycrystals both as anodes and as cathodes, more radical way of increase of operational characteristics of the x-ray tubes is application refractory nanostructural materials. The results of experiments showed that high-temperature sample annealing resulted in a sharp increase (by a factor of more than 3) in material creep resistance at a temperature of 2200°C. The study of emission characteristics shows that single-crystal tungsten with crystallographic orientation of emitting plane (111) has higher emission characteristics (work function equals 4.43 eV) than polycrystalline tungsten. But best results has nanoscale W. In this work results of research of evaporation of tungsten are presented. It is shown that the monocrystal has essential advantages on this parameter. It is important for materials working at temperatures more 2000°C. An increase in the emission characteristics of a cathode and the strength characteristics of an anode at the same time can be achieved by modifying standard tungsten powders (for cathodes), tungsten and molybdenum powders (for anodes) by nanoscale powders of refractory materials (grain size is 10-100 nm). W, Mo, Nb, Re nanopowders are proposed to be used for producing anodes based on tungsten and molybdenum. It must increase allowable unit loads of an X-ray tube by reducing porosity, increasing strength and plasticity of anode material. Prerequisites for these proposals are some results of work where an increase in density, strength by 10-30%, and plasticity by a factor of 1.5 - 2 was achieved due to modification of steels by ultra-disperse powders of refractory compounds. The authors carried out some experiments to determine the creep of tungsten alloyed by potassium on samples as foils 100 μm thick, used when manufacturing X-ray tube cathodes. The results of these experiments showed that high-temperature sample annealing resulted in a sharp increase (by a factor of more than 3) in material creep resistance at a temperature of 2200°C. The authors think that this effect results from forming nanoscale little bubbles filled with potassium at the grain boundaries. Results of this work can be used when development x-ray tubes with the increased operational characteristics especially of miniature X-ray tubes for oncology.

References
This article deals with the effect of surface treatment on the mechanical properties of biocompatible titanium alloys. In the case of implants made from titanium alloys, it is desirable to ensure good biocompatibility. The biocompatibility is an important property of materials used for construction of medical implants. Generally, the environment in the human body is very aggressive and implants can quickly degrade due to corrosion. The process of corrosion leads to the release of harmful particles into the body. For this reason, for example, titanium alloys with niobe content are preferred before alloys containing harmful vanadium. The corrosion can be reduced through appropriate surface finishes. Another important problem is the ability of the human body to accept an implant. The body is trying to reject foreign objects. A typical example is the failure of dental implants, which are excluded from the gums.

Experience shows that the human body accepts better implants with a rough surface. Vice versa, smooth objects are often expelled from the body (in addition to the most common cases, such as dental implants, other examples can be given such as an extrusion of pedicle-screw from vertebra). From the viewpoint of mechanical properties, the rough surface containing grooves and holes has a negative influence on the fatigue resistance against mechanical loading. Bumps on the surface are often crack initiation sites. One option to make such a rough surface machining. These surfaces are easy to manufacture, but have a significantly negative effect on both corrosion resistance and on the fatigue endurance. The resistance such a surface must be improved by deposition oxides, nitrides or other particles. Currently we are studying the possibility of creating a rough surface by means of irregular and asymmetric deposition of particles (oxides or other elements) on the smooth surface. Particle distribution can be controlled for example by laser or electrical discharges. These particles then form granular surface that is well accepted by the body.

This article analyzes the formation and propagation of fatigue cracks in the material with granular surface. The formation and growth of fatigue crack originated from granular surface is simulated. Also experimental studies were carried out. For experimental study were prepared different types of surfaces. The differences between surfaces were given by size or distribution of the granules and other features. Using results of experimental study and theoretical model were chosen most effective surfaces.

**Keywords:** titanium alloys, surface finishes, surface roughness, granulated surface, fatigue life, biocompatibility, crack growth, crack initiation
Dual-responsive cross-linked prodrug nanogels by a click chemistry for triggered drug delivery system

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A facile drug delivery system based on dual-responsive cross-linked prodrug nanogels was prepared efficiently by alkyne-azide click chemistry. Poly(ethylene oxide)-b-poly(glycidyl methacrylate), PEO-b-PGMA, was firstly synthesized by the reversible addition-fragmentation chain transfer polymerization, and the PGMA parts were subsequently functionalized with azido groups. The cross-linked micelles of the PEO-b-PGMA-N₃ and covalently entrapped Doxorubicin drug were prepared simultaneously using the alkyne-bearing hydrazone-containing drug and disulfide-containing cross-linking agent in the presence of CuSO₄·5H₂O and sodium ascorbate. The cross-linked nanogels showed a much improved drug loading efficiency compared to physically loaded micellar system. The nanogels illustrated the structural stability under physiological condition, while decross-linking through the cleavage of disulfide groups took place rapidly in glutathione circumstance. In addition, the pH-sensitive hydrolysis of the hydrazone groups in the core presented a burst release of the drug at pH 5. The dual-responsive prodrug nanogels enhanced cytotoxicity against human cancer cell in vitro.

**Keywords:** Click chemistry, core cross-linked micelles, redox-responsive, Hydrazone bond
Piezoresistive Carbon-based Hybrid Sensor for Body-Mounted Biomedical Applications

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For body-mounted sensor applications, the evolution of soft condensed matter sensor (SCMS) materials offer conformability, enabling mechanical compliance between the body surface and the sensing mechanism. A piezoresistive hybrid sensor and compliant meta-material sub-structure provided a way to engineer sensor physical designs through modification of the mechanical properties of the compliant design. A piezoresistive fiber sensor was produced by combining a thermoplastic elastomer (TPE) matrix with Carbon Black (CB) particles in 1:1 mass ratio. Feedstock was extruded in monofilament fiber form (diameter of 300 microns), resulting in a highly stretchable sensor (strain sensor range up to 100%) with linear resistance signal response.

The soft sensor was then integrated into a hybrid design including a 3D printed metamaterial structure combined with a soft silicone. An auxetic unit cell was chosen (with negative Poisson’s Ratio) in the hybrid sensor design in order to combine with the soft silicon, which exhibits a high Poisson’s Ratio. The hybrid sensor was subjected to mechanical tensile testing up to 50% strain (with gauge factor calculation for sensor performance), and then utilized for strain-based sensing applications on the body including gesture recognition and vital function monitoring including blood pulse-wave and breath monitoring. A 10 gesture Natural User Interface (NUI) test protocol was utilized to show the effectiveness of a single wrist-mounted sensor to identify discrete gestures including finger and hand motions. These hand motions were chosen specifically for Human Computer Interaction (HCI) applications. The blood pulse-wave signal was monitored with the hand at rest, and in a chest-mounted configuration, different breathing patterns were investigated, including normal breathing and coughing.

Keywords: sensor, polymer, piezoresistive, compliant structure, 3D printing
Surface treatment and corrosion behavior of austenitic stainless steel biomaterial

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Austenitic stainless steels are often used as biomaterials to replace structural components in the human body. They possess more superior tensile strength, fatigue strength, and fracture toughness compared to polymeric and ceramic materials. Such, austenitic biomaterials are used in medical devices such as artificial joints, bone plates, screws, intramedullary nails, spinal fixations, external fixtators, stents, and dental implants. These steels have good biocompatibility, very good mechanical and technological properties combined with good corrosion resistance. Their excellent resistance to uniform corrosion is related to passive oxidic surface film. However, aggressive ions present in environment (halides, namely chlorides and bromides) can cause local breakdown of this protective film, and enable a progress of the local pitting corrosion. This stainless orthopedic implant can corrode in body environment and release iron, chromium, and nickel ions. Resistance of stainless steels to pitting corrosion is affected by a number of internal and external factors. The quality of the surface passive film is one of the most important factors of corrosion behavior of stainless steels. The passive film properties are strongly influenced by mechanical and chemical finishing processes. The biocompatibility of these steels is the requirement of the biomaterial and the surrounding environment coexistence without having any undesirable effect on each other. The surface of the biomaterial is the interface where the material interacts with the surrounding (bone, tissue, blood), so the surface quality is one of the most important factors that determines the acceptance or rejection of a biomaterial in the body. To improve the surface quality, the biocompatibility and corrosion resistance, different surface modifications or surface treatments can be applied on the surface with maintaining the bulk properties of the implant. The one of the basic surface treatment can be electrochemical polishing which results in high quality surface with high surface brightness and decrease in surface roughness but it also improves the corrosion resistance. Another surface modification that improves the corrosion resistance can be oxide coating using atomic layer deposition of aluminum oxide by changing the surface characteristics that affects the interaction between the tissue and biomaterial.

Keywords: Austenitic stainless steel, Biomaterials, Electrochemical polishing, Atomic layer deposition
Biopolymer films or biofoils are formed from natural polymers, of animal or plant origin, such as polysaccharides, lipids and proteins. The biopolymers are neutral and always renewable. Currently, the market for biodegradable plastic products is growing, there is a growing "cult", and even become a fashionable application. In our experiments we were looking for materials for thin film’s production, which are natural and degradable. For preparing our Thin film samples we used many different natural polymer for example: alginate, pektin, guar gum, etc. The biofoil samples were made by casting technique from aqueous solutions. By the analysis of transients relaxation processes were revealed and the relaxation times determined. Depolarization measurements proved as a best resolution power method Setaram TSC II instrument was used in short-circuit mode. As mechanical method dual cantilever bending mode was used with Rheometric Scientific MK III instrument at 1 Hz frequency. This method gives more information about the modulus of elasticity but the resolution power is worse.
SESSION 2.

Advanced Materials for Extreme Applications
Highly Resistant Inorganic Materials – Possibilities and Problems of Thermodynamic Research

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The complex of methods and databases (realized in the computer program-information system), intended for description, modeling and calculation of phase-chemical transformations is developed on the basis of minimization principle for characteristic thermodynamic functions. This 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 system MgO-C-Al-H\textsubscript{2}O-air 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. Multiplan 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 main components composing the composite. 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 methodology has allowed also to investigate and reveal mechanisms of phase-chemical transformations occurring at sialons $\text{Si}_x\text{Al}_y\text{O}_z\text{N}_t$ synthesis by means of carbothermy kaoline 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, and tungsten. The correspond system: TiC-TiN-TaC-TaN-WC is 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. The conditions promoting and preventing formation of "harmful" product – $\text{W}_2\text{C}$ (forming at input cobalt into alloy, complex carbide $(\text{W,Co})_x\text{C}$, that leads to the alloy embrittlement).

Received theoretically and by calculation way the results for all investigated multicomponent systems not only well will 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.
Topological Insulators: Electronic Band Structure and Spectroscopy

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Topological insulators-TIs (Z₂, Kondo, Dirac, Anderson and Chern) are phases of matter characterized by an order of new kind, that do not follow Landau's classification by spontaneously broken symmetry. TIs that are characterized by metallic surface states inside the bulk band have attracted great attention in recent years [1]. Recently, various searches for new topologically nontrivial phases have been extended to ternary compounds [1,2].

The emergence of surface modes in TIs is a band structure effect which can be understood without invoking interactions. There is great current interest in the possibility of interaction driven topological phenomena. Up until now there are no experimental examples of interaction-driven TIs that preserve time-reversal symmetry. However, several theoretical proposals have been put forward: 2D TIs via spontaneous symmetry breaking in bilayer graphene and optical lattice systems, toplogical Mott insulating phase in Ir-based structure (X₂Ir₂O₇ with X=Nd, Pr). Kondo insulators with the most salient example of SmB₆ and insulating behaviour in filled skutterudites.

In present paper we focus on general principles governing the emergence of chiral metallic states in different TIs and their electronic band structure and optical properties for the different types of TIs. In this process, first principles calculations played a critical role. Up to now, most of TIs were predicted first by first principle calculations, and observed subsequently by experiments.

In all of our calculations that were performed using the ab-initio total-energy and molecular-dynamics program VASP (Vienna ab-initio simulation program) [3] that was developed within the density functional theory (DFT) [4], the exchange-correlation energy function is treated within the GGA (generalized gradient approximation) by the density functional of Perdew et al. [5].

The ground state energies and electronic structures were calculated using the DFT within the generalized-gradient approximation (GGA). The real and imaginary parts of dielectric functions and hence the optical functions such as energy-loss function, the effective number of valance electrons and the effective optical dielectric constant for TIs were also calculated:

1. Band gap values obtained from the band structures of GeTe and SnTe in ferroelectric and paraelectric were calculated as 0.742 eV and 0.359 eV in the ferroelectric phase and 0.376 eV and 0.028 eV in the paraelectric phase, respectively, and the band gap character of these compounds for either of the phases was determined as direct. The change in the energy gap for
the SnTe and GeTe can be understood qualitatively in terms of the difference between the relativistic effect in Ge and Sn (spin-orbital coupling) and the relativistic correction is extremely important in determining the positions of the energy bands. The total and partial densities of states for GeTe and SnTe in ferroelectric phase show that the lowest valence bands occur between about -25 and -22 eV are dominated by d states. Similarly, the valence band that occur between approx. -12 and -5 eV are dominated by s states. The highest occupied valence bands results from the sp-hybridization of Ge and Te bands. The other states also contribute to the valence bands, but the values of densities of these states are so small compared to the sp-hybridization. The lowest unoccupied conduction bands just above the Fermi energy level are dominated by p states.

2. The similar detail calculations were performed for IrX3 (X=Cl, Br, O) and some other examples of Kondo and Z2 TIs.

So, as a result in the present work, we conducted a detailed investigation of the electronic, and frequency-dependent linear optical properties of different types of the new TIs using the DFT

**Keywords:** topological insulator, DFT, band structure, optics

**References**


Electronic Structures, Optical and Magnetic Properties of Some Heusler Compounds for Spintronic Devices: \textit{ab initio} calculations

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Spintronics is a multidisciplinary field and a new research area. New materials must be found for satisfying the different types of requirement. The performance of these devices depend on the spin polarization of the current used for information storage. The most promising candidates for these applications have a semiconductor behaviour with respect to the electrons with a spin orientation, whereas they are metallic in relation to inverse spin orientation, being dominated Heusler compounds. Heusler compounds can be classified into two main groups, namely, half-Heusler ABC alloys and full-Heusler A\textsubscript{2}BC compounds. Here A and B denote the nd-transition elements (n=3, 4, and 5) and C is an s-p element (A=Co, Cu, Ni, Pd, Ir; B = Mn, Ti; C = Si, Ge, Sb, Ga, Al, Sn)

In present work, our research is mainly focused on the electronic structures, optical and magnetic properties of some ABC and A\textsubscript{2}BC Heusler compounds by using \textit{ab initio} calculations within the generalized gradient approximation (GGA). The calculations are performed by using the Vienna ab-initio simulation package (VASP) based on the density functional theory.

The band structure of the Co\textsubscript{2}Mn(Ti)Si(Ge,Ga, Sn) and Cu(Ni)MnSb Heusler alloys for majority spin (spin-up) and minority spin (spin-down) were calculated. It is seen that for these compounds, the majority spin states cross the Fermi level and thus have the metallic character, while the minority spin states open the band gaps around the Fermi level and thus have the narrow-band semiconducting nature. We also find that these Heusler compounds have the indirect band gaps $E_{3}$ in the minority spin channel. For better understanding of the electronic states, the total and partial density of states were calculated, too. The real and imaginary parts of dielectric functions and hence the optical functions such as energy-loss function, the effective number of valance electrons and the effective optical dielectric constant for ABC and A\textsubscript{2}BC compounds were also calculated. In addition, we also show the variations of the total magnetic moment per f.u. and minority spin gap width of these compounds with optimized lattice constants: minority spin gap width decreases with increasing the lattice constant.

\textbf{Keywords:} Heusler compounds, DFT, electronic structure, magnetic properties, optics
Band Structure and Optical Properties of Kesterite Type Compounds: first principal calculations.

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DFT (density functional theory) analyses of band structure dispersion and of different anionic subgroups to the studied electronic structure together with the anisotropy of optical function were performed for the solar cell crystals \( \text{Cu}_2\text{FeSnS}_4 \) (CFTS) and \( \text{Cu}_2\text{FeSnSe}_4 \) (CFTSe) in different structural phases (kesterite and stannite structures). All of the DFT calculations in the present work have been performed with MedeA (WASP) code, where we have applied DFT GGA (general gradient approximation) method for our calculations.

We show that \( \text{Cu}_2\text{FeSnS}_4 \) as well as \( \text{Cu}_2\text{FeSnSe}_4 \) crystals possess a direct energy band gap situated around the center of the BZ. Careful analysis of the total density of states together with the partial contribution of the particular orbital were performed for evaluations of contribution of corresponding bonds to the origin of the chemical bonds. Role of replacing of S by Se is analyzed in the details for the electronic density of states with respect to the nature of chemical bonds. For the selenides and sulfides, the kesterite band gaps are very similar to each other and the corresponding chalcopyrite band gaps.

The principal analysis is performed for the dispersion of the optical constants. The influence of the different chemical bonds into the dispersion of the optical functions is analyzed in order to optimize the optical features.

In summary, the structural, electronic and optical properties of CFTS and CFTSe have been investigated in the present work using DFT GGA method. The different interactions of metal atoms with S(Se) atoms lead to the slight distortion of tetrahedrons (\( \text{CuS(Se)}_4 \), \( \text{FeS(Se)}_4 \) and \( \text{SnS(Se)}_4 \)) resulting in the formation of local internal fields which is especially beneficial to suppress the recombination of photo-excited electron-hole pairs.

**Keywords:** Kesterite compounds, DFT, band structure, optics
The metamaterial based (MA) absorber sensor, integrated with an X-band waveguide, is numerically and experimentally investigated and suggested for important applications including pressure, density sensing and marble type detecting applications based on a rectangular split ring resonator, sensor layer and an absorber layer that measures the changes in the dielectric constant and/or the thickness of the sensor layer. We compared the simulation and experimentally obtained results for the fabricated sample and found a quite good agreement between them.
Modeling of carbon-carbon composites joining using solid-phase synthesis

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Technologies of obtaining and processing materials based on combustion synthesis are quite various nowadays. They are of special importance for joining of carbon-carbon composites. Carbon is known as a material which is not easy to be joined by conventional methods. Since carbon has a very high melting point and cannot be welded, joining using an exothermal chemical reaction would be a suitable technology. Efficiency of combustion joining for forming joints between two cylindrical carbon-carbon composite were proven in [1]. Experimental investigation was carried out with a stack consisted of two disks of C-C composites that were to be joined and a layer of reactive filler between them. The stack was held in place between two electrodes, which were connected to a direct current power supply. In the present work mathematical model of described process is suggested. It contains two thermal conductivity equations and two diffusion equations. The first thermal conductivity equation is for the region occupied by reactive mixture. It includes two additional terms. One of them defines the heat source effect due to chemical reaction and the other one expresses direct current power supply effect. The second one, which corresponds to C-C composite, includes only one additional term characterized heat release due to reaction. Its existence is determined by the fact that reactive mixture penetrates in C-C composite due to diffusion. As for diffusion equations, they contain terms associated with components consumption for new joints formation. The problem is solved numerically. As a result distribution of temperature and concentration at different moments of time with variation of processing characteristics were plotted for both phases.

References
Kinetic-thermodynamic and surface analysis studies on corrosion inhibition of steels by Cetyltrimethylammonium/KI mixture in 5 M HCl medium: The effect of silicon and phosphorus contents in steel

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The effect of silicon and phosphorus content in steel suitable for galvanizing on its corrosion and inhibitor adsorption processes in mild steel/100 ppm of Cetyltrimethylammonium combined with 100 mM of KI (mixture)/hydrochloric acid systems has been studied in relation to the temperature using chemical (weight loss) and electrochemical (Ac impedance and dc polarisation) techniques and scanning electronic microscope (SEM) analysis. All the methods employed are in reasonable agreement. The protection efficiency increases with increasing inhibitors concentration and with increasing temperature. The thermodynamic functions of dissolution and adsorption processes were calculated from experimental polarisation data and the interpretation of the results are given. Adsorption of Mixture was found to follow the Langmuir adsorption isotherm and the ability of the molecule to chemisorb on the steel surface was dependent of the steels composition according to their silicon and phosphorus contents. Surface analyses via scanning electron microscope (SEM) shows a significant improvement on the surface morphology of the mild steel plate.

Keywords: Scanning electron microscopy (SEM), Optical 3D surface characterization, Kinetic-thermodynamic, Steels grade, Acidic media, Electrochemical measurement.
Phosphate sediments of South Eastern Constantine are specified by a rich mineral in P₂O₅ exogangue several sedimentary origin rich in organic matter of humic kind. The origin of the sediment has a direct relation with the chemical composition of each ore. The differentiation of minerals from various mining sites of South-Eastern Constantine region, orient us to a classification based on its chemical composition, which it make it difficult to purification of simply because that each ore class goes through a treatment process appropriate.

**Keywords:** South Eastern Constantine, Phosphate, Chemical composition, Treatment, Purification.
The influences of heat treating processes with hardening sequences on mechanical properties of the martensitic stainless steel AISI 440C were experimentally investigated. The central composite design was used to carry out all experiments by varying process times with specific hardening sequences. Analysis of variance was used to determine whether there were any significant factors of heat treating process affecting hardness and impact toughness. Regression models for hardness and impact toughness were performed to determine an optimal operating condition. The results showed that the quenching time of 11 s had the appropriate values of hardness and impact toughness with the balance of strength and toughness within the range of acceptable hardness of 60.2-60.3 HRC and the impact toughness of 0.36 J.
Discovered that some of blades Damascus steel has an unusual nature of origin of the excess cementite, which different from the redundant phases of secondary cementite, cementite of ledeburite and primary cementite in iron-carbon alloys. It is revealed that the morphological features of separate particles of cementite in Damascus steels lies in the abnormal size of excess carbides having the shape of irregular prisms. Considered three hypotheses for the formation of excess cementite in the form of faceted prismatic of excess carbides. The first hypothesis is based on thermal fission of cementite of a few isolated grains. The second hypothesis is based on the process of fragmentation cementite during deformation to the separate the pieces. The third hypothesis is based on the transformation of metastable cementite in the stable of angular eutectic carbide. It is shown that the angular carbides are formed within the original metastable colony ledeburite, so they are called "eutectic carbide". It is established that high-purity white cast iron is converted into of Damascus steel during isothermal soaking at the annealing. It was revealed that some of blades Damascus steel ledeburite class do not contain in its microstructure of crushed ledeburite. It is shown that the pattern of carbide heterogeneity of Damascus steel consists entirely of angular eutectic carbides. Believe that Damascus steel refers to non-heat-resistant steel of ledeburite class, which have similar structural characteristics with semi-heat-resistant die steel or heat-resistant high speed steel, differing from them only in the nature of excess carbide phase.

Matrix (dark background) - sorbite (about 0.2 µm). Excess phase (bright background) - eutectic carbide (about 10.0 µm).

**Keywords:** Bulat; Damascus steel; Wootz; Indiansteel.
Fish scale-cellular composite structure for impact protection: numerical and experimental proof-of-concepts

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Fish scale structures are known to have excellent penetration resistance while cellular materials are lightweight and have excellent energy absorption capacity under compressive loading. In this study, a novel composite structure which combines fish scale structure and cellular material is explored for protection against low-velocity impact loads. The two-layer composite structure is made by using a fish scale structure as an outer layer – which is formed by an assembly of overlapping plates representing “scales” – underlain by an inner layer made of a cellular material.

Numerical simulations and experimental proof-of-concepts are performed to assess the feasibility of the composite structure for impact protection. The peak stress transferred by the composite structure when subject to impact is used as a measure of impact performance. The impact performance of specimens with different shapes of the overlapping scales is compared with that of a conventional sandwich design with the same volume of materials and subject to the same impact energy.

The simulation results show that the composite structure performs optimally when the scales dissipate most of the impact energy while the underlying cellular layer acts as a cushion to limit the magnitude of stress transferred. The numerical simulations and experimental tests also show that the composite structure can perform better than a conventional sandwich design with the same volume of materials provided the scale are curved. Due to additional hoop resistance from their curved shape, the scales are able to dissipate a larger proportion of impact energy before they are flattened and start compressing on the underlying layer. Nonetheless, improved impact performance can only be achieved if the curved scales have the right geometrical configuration, i.e. having aspect ratio, curvature, degree of overlapping, and size that fall within certain bounds.

Keywords: biomimicry, finite element simulation, drop weight impact, composite
SESSION 3

Advanced Nanomaterials with Predesigned Properties
An efficiency strategy for controlled growth of 1D nanomaterials under high gas pressure

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1D Nanomaterials, including nanowires, nanobelts and nanotubes, have drawn many interests during the past decades. In most cases, inorganic 1D nanomaterials are prepared via high temperature processing under low atmospheric pressure. However, the yield of nanomaterials is usually very low so that it is hard to be used for application. We have focussed on the high yield preparation of 1D nanomaterials with high gas pressure processing for more than 10 years.

In this presentation, we will introduce a new efficiency strategy: the high gas pressure processing from principle to application. Kinds of 1D nanostructures[1-3], such as SiC, Si₃N₄, BN, Sialon, etc. are all prepared for large-scales by this strategy. The relationship between the gas pressure parameters and the morphology and microstructures of nanomaterials are investigated. The nanomaterials are well characterized by XRD, XPS, SEM, TEM, HRTEM and so on. Results show that the high gas pressure processing is an efficiency way to prepare high qualified 1D nanomaterials.

**Keywords:** 1D nanomaterials, high gas pressure processing, nanostructure, controlled growth, high yield

References

The corrosion resistance of zinc coatings in the presence of boron-doped detonation nanodiamonds (DND).

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The use of zinc as a chemical protection due to a combination of resistance to the environmental influences and low cost of inorganic zinc compounds.

Nanodiamonds are a new generation of composite additives. They are easily regenerated, evaluated and are environmentally friendly.

Boron-modified in the explosion nanoparticles of detonation nanodiamonds (DND-boron), that included in the coating, are barriers of microcracks, defects and coating’s dislocations that means hardening of the material and corrosion resistance increasing. The most common electrolytes are alkaline galvanizing electrolytes (zincate electrolytes), that requires the presence of surface-active agents (surfactants), in the absence of which the spongy zinc precipitation obtained. In this Work we used the surfactant A-1DM (production of «Chemeta» company). This additive increases the cathodic polarization, leads to polarization curves shifting towards negative potentials. This is because of the additive A-1DM adsorption at the cathode and the sharp drag effect of zinc discharge. This improves the structure and performance of the zinc coating.

For investigation of the DND-boron influence on the galvanizing process an electrolyte of the following composition and mode used:

\[ \text{ZnO} - 10-12 \text{ g/L}; \text{NaOH} - 100-120 \text{ g/L}; \text{PAВ A-1ДМ} - 2-10 \text{ mg/L}. \]
\[ \text{t} - 18 - 25^\circ\text{C}; i = 2 - 3 \text{ A/dm}^2. \]

Previously, we studied the additive DND-TAN (DND, modified with ammonia at high temperature and pressure), which has little effect on the zinc cathodic and anodic polarization, without changing the zinc discharge mechanism [1]. Addition of DND-boron adsorbed on the cathode, together with the addition of A-1DM leads to a significant shift in the polarization direction of negative potential (more than 100 mV) and obtaining fine crystalline precipitates. Addition of DNA-boron additive (2g / l) significantly improves the electrolyte scattering - 4 times as compared with the electrolyte additive A-1DM. Progress hydrogen curves testifies to the fact that the coating includes DND-boron additives together with A-1DM.

The corrosion resistance of the coatings was determined by two methods: fast - evaluation of corrosion currents in the 3% NaCl and evaluation of corrosion resistance of the coating in salt spray (visual and gravimetric).

Corrosion current in the coating obtained in the presence of DND-boron (7 g / l) is 2.6 times lower than in the coating without the additive.

Tests carried out in the salt fog chamber, confirmed that the best results were obtained on samples deposited in the presence of DND-boron additive at concentration of 5 and 7 g / l, which agrees well with corrosion currents. The microhardness increased by 10-15% in the presence of DND-boron additive, indicating the effect of this additive on the coating structure.

**Keywords:** detonation nanodiamonds, DND, zinc-diamond coatings, corrosion resistance, zinc, composite additives, explosion

**References**
Multilayer Structures of Nano Ferrocene/Schiff Base Compound Embedded in Inorganic Matrix for Optical Limiting Devices

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Metal organic nonlinear optical (NLO) materials with large second-order nonlinearity and high-speed response are recently investigated materials due to their important applications in total optical communications systems. Nano Ferrocene / Schiff base organic compounds take advantage of the ferocenes’s excellent thermal and photochemical stability in electron donor-acceptor organic complexes which exhibit intramolecular charge transfer under external stimulus. We report on some new materials prepared by the sol-gel synthesis consisting of SiO\(_2\)-P\(_2\)O\(_5\) multilayer structures doped with ferrocene-derivative. Fourier transform infrared spectroscopy (FTIR), atomic force microscopy (AFM), scanning electron microscopy (SEM) and UV-VIS spectroscopy were used for structural and optical properties investigations. The nonlinear optical efficiencies were measured using a femtosecond Ti:sapphire laser. The non linear efficiencies of the films are correlated with the number of layers and thermal treatment temperature.

**Keywords**: ferrocene-derivative; Sol gel; NLO-active; Chromophores; Silicophosphate matrix
The size and shape of particles and magnetic properties of composite films

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Metal and alloys $Me$ (CoFeZr, CoFeB, CoTaNb, Co) and dielectrics $De$ (Al2O3, SiO2, Zr2O3, ZrO) were used for the production of films. The films were prepared by ion-beam sputtering on a mylar substrate in argon atmosphere. The thicknesses of the films were 0.1÷1.0 μm. Areas with micro- and nano- sizes of surfaces of the films at various metal concentrations $x$ were observed using atomic force microscopy (AFM). The silicon probe coated with a layer of cobalt was used. Magnetic particles sizes of 10÷100 nm and domain structure in some series of the films were found. Phase transition as a result of analysis the sizes of the magnetic and metal areas of the composite films was detected. The spectra of the permeability and tangent loss of the composite films were obtained. Spectra permeability explained based on these of magnetic force microscopy (MFM) images of magnetic phase contrast on surface films with different $x$ of metal or alloys. Changing the behavior of the spectra was observed in area of the percolation threshold of the films. Away from the transition permeability depends weakly on the frequency. The metal granules are not arranged in groups ($x<0.3$) and the relaxation frequency of the magnetization vector of each granule have large values. For some composite films were obtained giant permeability value (about 1000) up to 250 MHz. This fact shows that the coherent rotation of the magnetization vector and minor losses in the films. This property of the composite films can be used to creation of electronic devices operating in the HF region. The tangent loss is growth with increasing of frequency and the stronger the higher $x$.

Keywords: micro- and nano- sizes, composite films, AFM, MFM, magnetic permeability, HF spectra,
The paper presents results of measurements of basic thermophysical properties of ethylene glycol based yttrium aluminum garnet (Y3Al5O12–EG) nanofluids. Nanofluids used in presented experiments were prepared with two-step method based on commercial nanoparticles. Basic rheological properties were investigated on HAAKE MARS 2 rheometer (Thermo Electron Corporation, Karlsruhe, Germany). Dynamic viscosity curves in the range of shear rates from 10 to 1000 s\(^{-1}\) at a constant temperature of 298.15 K were determined. Additionally, the temperature dependence of the viscosity in the range from 273.15 K to 333.15 K was measured. To determine thermal conductivity of nanofluids a KD2 Pro Thermal Properties Analyzer (Decagon Devices Inc., Pullman, Washington, USA) was used. The dependence of thermal conductivity of Y3Al5O12–EG nanofluids on the concentration of nanoparticles was measured at constant temperature of 298.15 K.

References
Inorganic, organic and hybrid aerogels: the synthesis and properties.

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Aerogels (AGs) are unique mesoporous materials possessing ultra-low density, high porosity and a high specific surface area. AGs are widely used as very effective thermal and acoustical insulators,1 sorbents,2,3 catalyst supports,4 optical materials,1,5 etc. The preparation of aerogels is a multistage process, typically involving gel synthesis by a sol-gel technique, aging and washing with a desired solvent, followed by supercritical drying (SCD).

The present work is focused on the development of different types of aerogels – inorganic, organic and hybrid. Different methods of AGs’ surface modification are discussed – SCD solvent type, surface modification by fluorine substituents, incorporation of inorganic and phosphororganic fragments into aerogels’ matrix. It is shown that the incorporation of long fluorine-containing chains increases the AGs’ hydrophobicity but decreases their transparency. A special attention is paid to the influence of SCD solvent on textural properties of aerogels. It is shown that the change of the supercritical solvent can dramatically change their specific surface area, porosity, etc.

It is shown that the incorporation of chiral lactate moiety into aerogel matrix leads to a formation of chiral aerogels.

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Keywords: aerogels, supercritical drying, silica, modified aerogels, hybrid materials, hydrophobicity.

References
Titania-Ceria Surfacant Assisted Sol-Gel Synthesis and Characterisation.

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The nanoparticles of titanium dioxide are important in a wide range of applications such as catalysis, environmental remediation and solar energy conversion [1]. While cerium oxide is rare-earth oxide material used in the fields of photoluminescence, photosensitive material and anti-UV radiation [2]. Furthermore, research on the synthesis of nanoparticles of titanium dioxide has given rise to different methods such as sol-gel, in solid states, hydrothermal processes, among others. The process of solid state synthesis is most often used for the technical production of ceramic materials, while the sol-gel has gained much popularity in recent decades [3-5]. Between these methods, microemulsion of reverse micelles technique, is one of the most versatile allowing control of particle properties such as size distribution, morphology and surface area [6]. In this work, titania-ceria nanoparticles in anatase phase were synthesized by sol-gel method assisted by microemulsion reverse micelle using butoxide de titanium and cerium nitrate hexahydrate as the precursors. The sample was heat treated at 600 °C at a rate of 3 °C min⁻¹. The residence time of the sample at this temperature was 2 hr. IR, RAMAN, TEM and XRD analysis were used to characterize the samples obtained. According to the results obtained, the obtained nanoparticles present spherical morphology and have a size distribution of 5 nm for CeO₂, 9.5 for TiO₂ and 14, 17 and 20 nm for the doped TiO₂ with 5, 10 and 15 % CeO₂, respectively. The results indicate that the CeO₂ was incorporated into the network of titania.

**Keywords:** Nanoparticles, Titania, Ceria, Microemulsion, Sol-gel.

**References**


45S5 Bioglass porous scaffolds functionalized with Vitamin D and Collagen.

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The development of smart biomaterials has become a promising area in regenerative medicine, even though some materials have proved their bioviability, there is a need to decrease the bioassimilation time and improve the regenerated tissue mechanical and biological characteristics. This work involves the development of several bioscaffolds that mimics the cancellous bone tissue structure using nanostructured silver-doped sol-gel bioactive glass, the bioscaffold surface was bioactivated with bone development molecules such as cholecalciferol and collagen. The osteogenic and antimicrobial properties were analyzed by in vivo and in vitro systems, were we induce the different reactions which take place inside the human body. The bioactivity was examined in vitro and in vivo with respect to the ability of hydroxyapatite (HA) layer to form on the scaffold’s surface as a result of contact with simulated body fluid (SBF) and the CAM of the hen’s egg, after 2, 4, 6 & 8 days. XRD, FTIR, EDS and SEM studies were conducted to analyze the evolution of the HA layer. Antimicrobial assays were developed by using Escherichia coli cells, determining in 1.75% the bioglass minimal Ag composition to inhibit the bacterial growth.
At present, carbon nanostructures are being actively developed and finding more practical applications. The interest in these materials has been caused due to their high operational properties such as great tensile strength and elasticity, and some other important parameters. However, when applying nanomodifying additives, it is rather difficult to distribute them uniformly in the construction material matrix, since they cannot be evenly dispersed due to their high ability to form agglomerates. Thus, to overcome these forces, multistage and labor-consuming processes are required. In this regard, the successful solution of this task can allow to open new opportunities for creating nanostructured construction composites with improved qualitative and functional characteristics.

Considering the aforementioned, the present work describes the technology of fabrication of a nanomodified construction binder appointment, in which the uniform distribution of carbon nanoparticles was achieved due to the directed joint total synthesis of nanofibers in the binder structure. The synthesis of carbon nanomaterials was performed via chemical vapor deposition (CVD) of hydrocarbons onto catalysts. A propane-butane mixture was used as carbon source. Cement particles were used without any additional pretreatment. The distinctive feature of the above-mentioned technology is that cement or the other construction binders are sprayed into a synthesis chamber together with metal-oxide catalyst particles. Process takes place in the chamber at a temperature of hydrocarbons pyrolysis.

The proposed technology is directed to the production of construction materials and modification of various types of binders. The use of the nanomodifying additive results in improving the physicomехanical characteristics of the construction composite. Experiments aimed at studying the effect of the synthesized additive on the construction composite features were carried out with fine-grained concrete samples. The obtained results demonstrate a stable increase in the physicomехanical and operational properties of the material.

**Keywords:** synthesis, carbon nanomaterials, catalytic pyrolysis, construction materials, concrete, nanomodification, physicomехanical properties.
Comparative Studies on Mechanical Properties and Microstructure of Boroncarbide Based Nanolaminate Coatings

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Recently, in modern material research intensive efforts being exerted to develop novel nanolaminate coatings with unique physical, mechanical and chemical properties. Materials with ³BC structure belong to nanolaminates with similar structure as so called MAX phases [1,2] (M= transition metal, A is a group A element and X is either boron or carbon). These materials exhibit high hardness and elastic modulus together with high fracture resistance, which make them industrially very attractive, for example, as protective coatings.

The aim of the present work was to study the microstructure and the mechanical properties of M-B-C (M=Mo, Ta, W) nanolaminate coatings which show a favorable combination of high stiffness, hardness and elastic modulus together with moderate ductility [1,2]. The coatings were deposited on high speed steel and hard metal (WC-Co) substrates using magnetron sputtering of three targets: B₄C, C and M. The thickness of the prepared coatings ranged from 1 to 2 µm. As it was shown in our previous study [3], the mechanical properties of these coatings depend on the nature of their microstructure. In this study, the X-ray diffraction, performed on Rigaku Smartlab X-ray diffractometer with a fixed angle of incidence, and selected area diffraction (SAD) patterns were used in order to determine whether the material is crystalline, nanocrystalline or amorphous. Special focus was given on the analysis of the mechanical and tribological properties of the coatings. The mechanical properties were studied using nanoindentation tests under quasistatic as well as dynamic conditions. Microindentation tests were performed using indentation loads around 1N in order to study the differential hardness and the fracture resistance of the coating/substrate system. The adhesion of the coatings to the substrates was studied using scratch tests. The details of microstructure in the vicinity of residual indentation prints were studied by transmission electron microscope (TEM). Thin lamellar cross sections for TEM observations were prepared using a focused ion beam (FIB) in SEM.

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Keywords: keyword, nanolaminates, nanocomposites, mechanical properties, fracture toughness, protective coatings, magnetron sputtering

References
Modified SiO$_2$-based aerogels: the effect of the fluorinated substituent on the properties of materials

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Aerogels are mesoporous solid materials possessing unique properties including very low bulk density, large specific surface area, high porosity, etc [1]. Due to unique properties aerogels can be used as effective thermal insulators, catalysts and catalyst support, sorbents, etc. [2-4]. Aerogels are prepared by sol-gel method, followed by supercritical drying (SCD).

The presented work is devoted to the modification of aerogels – synthesis of the pre-constructed monomers containing polyfluorinated fragments with subsequent gelation, as well as studying the effect of solvents on the properties of aerogels.

Different polyfluorinated acids (R = CF$_3$, C$_2$F$_5$, C$_2$F$_5$OCH$_3$, C$_6$F$_{13}$, C$_8$F$_{17}$) were used for the aerogels’ preparation. It was shown that all obtained aerogels possess high hydrophobicity. The influence of the substituent on the hydrophobicity, opacity, and specific surface area of aerogels was investigated.

The effect of the solvent used for SCD on the aerogels’ properties was studied. We have found that the specific surface area of aerogels obtained by SCD in isopropanol and CO$_2$ was higher than that for samples prepared in methyl tert-butyl ether and hexafluoroisopropanol. It was also shown that the type of supercritical fluid could affect the aerogels’ hydrophobicity.

Keywords: aerogels, supercritical drying, silica, modified aerogels, hydrophobicity.

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References
Characterization of Ta-B-C nanostructured hard coatings

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Hard coatings are commonly deposited on cutting tools to protect the tool surfaces from mechanical and chemical damage and hence to improve lifetime and performance. Protective coating materials used for cutting require high stiffness and hardness to lower wear rates. On the other hand, since crack initiation and growth have to be avoided, the protective coating materials should also possess moderate ductility. In the last decade, new hard coating materials of X2BC type fulfilling the mentioned criteria have been proposed based on correlative experimental and theoretical research [1]. Due to their orthorhombic crystal structure with high aspect ratio these materials (e.g. Mo2BC) have been described as a nanolaminated materials. They exhibit a high stiffness in combination with moderate ductility [2] and resistance to brittle fracture [3]. In this paper we report on microstructure and mechanical properties of Ta-B-C nanocrystalline layers prepared by magnetron sputtering. DC magnetron sputtering was used to prepare 1-2 micrometre thin layers on rotated substrates. Various deposition parameters were tested. Microstructure of layers was studied using a Tescan LYRA 3XMU SEM×FIB scanning electron microscope (SEM), a Philips CM12 STEM transmission electron microscope (TEM) and a JEOL 2100F high resolution TEM. Thin lamellar cross sections for TEM observations were prepared using a focussed ion beam (FIB) in SEM. Both undisturbed layers and the volume under relatively large indentation prints (load of 1 N) were observed. The microstructure observations were correlated with mechanical properties characterized by means of nanoindentation experiments in both the static and the dynamic loading regime using a Hysitron dual head TI950 triboindenter with Berkovich tip. Elastic modulus, indentation hardness and fracture resistance of prepared nanostructured coatings were evaluated and discussed. The research has been supported by the Czech Science Foundation (Project 15-17875S).

References
How Mesoscopic Physics Explains the Super small Quantities Action of Metal/Carbon Nanocomposites in Different Liquid Media

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Present investigations include the discussion of theoretical and experimental studies of the Metal/Carbon Nanocomposites super small quantities influence on the different liquid media. A number of hypothesis and investigation results are explained by means of mesoscopic physics principles, and also by the fractal theory equations. Self organization in system and self similarity (image) are the ground notions in mesoscopic physics as well as in the fractal theory and nanotechnology. For example, to explain the improvement of polymeric materials properties by super small quantities of nanostructures it is necessary to use the notion about the electron transport. Nanocomposite, having the great dipole and magnetic moments, vibrates with ultrasonic frequency in the field of medium molecules. Besides the every molecule has own electrical and magnetic fields. In this notion Metal/Carbon Nanocomposite is considered as vibrator, which radiates electromagnetic waves. These processes lead to the medium molecule self-organization and to the formation of nanostructured fragments (fractals) in the media, and also in the material composition. These changes can be accompanied by the processes of active particles appearance with the change in IR spectra of medium. After the addition of nanocomposite in the active dispersed media the lines intensity increasing in their IR spectra is observed. According to IR spectra, the intensity of some lines is increased more than two times. The changes of media electron structure are possible under the nanocomposites super small quantities influence that it is confirmed by the X-ray photoelectron spectroscopic investigations. The change of X-ray photoelectron spectrum of PVA, containing 10-3% of Cu/C nanocomposite, is concluded in the widening and in the appearance of satellites sp\textsuperscript{2} and sp\textsuperscript{3} lines.

Keywords: metal/carbon nanocomposite, mesoscopic physics, fractal theory, Avrami–Kolmogorov equation, IR spectroscopy, X-ray photoelectron spectroscopy, super small quantities.
Ytterbium-Erbium Doped Lead-Fluoride Nano-Glassceramics for Temperature Sensors

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Currently, transparent glassceramics are of great interest for the modern element base of photonics. Because they stay at intermediate state between crystalline materials and glasses, these ceramics combine the best properties of crystals and glasses. If the dopants (erbium, neodymium, etc.) enter the crystalline phase, the spectral, luminescence and laser characteristics of glassceramics become close to those of laser crystal analogues. Erbium doped fluorescent materials have often been used as temperature sensors because its absorption and emission properties are temperature dependent. Thus, transparent fluorine-containing nano-glassceramics doped with erbium ions are promising media for up-conversion temperature sensors.

In the present work we have prepared nano-glassceramics 30SiO₂-18PbF₂-7.5Al₂O₃-5ZnF₂-29CdF₂-3YF₃ -5YbF₃ wt % doped with different Er³⁺ concentration (0.05 -0.5 wt %). In these systems, the crystalline phase is precipitated upon heat treatment. Rare-earth ions play a role of nucleation centers. An X-ray diffraction analysis of the samples after the secondary heat treatment showed that the crystalline phase has a composition of PbYOF₃.

Up-conversion luminescence from two thermally coupled excited states \(^{2}H_{11/2}\) and \(^{4}S_{3/2}\) of Er³⁺ centered at 522 and 547 nm in the nano-glassceramics were recorded in the temperature range from 300 to 750 K for all erbium concentration and different secondary heat treatment time. Fig 1 shows dependence of up-conversion luminescence intensity from temperature.

![Fig 1. Up-conversion luminescence intensity dependence from temperature.](image)

As you can see, relationship between luminescence peak at 522 and 547 nm depend on temperature (fig 1, a). Luminescence peak dependence on the temperature is close to linear (fig. 1 b) which allows as measuring temperature. We have evaluated this material as a potential candidate for use as temperature sensors.

**Keywords:** nano-glassceramics doped with erbium, up-conversion, luminescence temperature sensors
A hybrid surface modification method of copper mesh for enhancing thermal performance of ultra-thin heat pipes

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As high performance and down sizing being the prerequisite in the consumer electronics market, heat pipes are attracting more and more attention with its outstanding performance in thermal management system. In this study, we introduce a hybrid surface modification method for the copper mesh being inserted into ultra-thin heat pipes.

The hybrid method is the combination of a solution-etching based chemical method and a sol-gel method with SiO2 nanoparticals with 40 nm in diameter being dip-coated onto the copper mesh at a slightly higher pulling rate, which is about 2 centimeters per minute. According to the SEM result of the modified copper mesh, it is observed that the method results in a hybrid micro structure, which further shows the white-flake-shaped CuO in its fine structure roughly wrapped by the SiO2 nanoparticles.

In order to enhance thermal performance, copper mesh is used as wick structure attached on the inside of pipe body to drive the condensed water back to the evaporator side. A well-modified copper mesh surface should be able to carry as much working fluid, which is liquid water in our case, as possible. This hybrid structure enhance the porosity by the flake-shaped CuO and as well as the hydrophilicity by the SiO2 nanoparticles. These two main reason would essentially improve the thermal performance of heat pipes and we are looking forward to bringing this method into a practical use for industry.

**Keywords:** heat pipes, surface modification, copper mesh, hybrid method
Pulsed Nd:YAG Deposition of Nanostructured FeS_{1-x} Containing Metastable Phases

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Iron sulfide thin films attract attention because of its potential application for solar cell materials and due to interesting electrical semiconducting and magnetic properties of this material. Its advantage in comparison with more effective cadmium, lead, indium or selenium represents non-toxicity, abundance, and cheap price. The iron sulfide (Fe-S) system possesses a complex phase diagram including seven phases where just pyrite (FeS_2) and pyrrhotite (Fe_{1-x}) are stable. Pulsed near-IR laser irradiation of ferrous sulfide (FeS) in a vacuum allows a noncongruent ablation and deposition of nanostructured FeS_{1-x} thin films. Deposition has been performed on Al, Ta and Cu substrate and analysed by scanning (SEM) and high resolution transmission electron (HRTEM) microscopy and electron diffraction. Morphologically, the similar homogeneous, dark, metallic and adhesive appearances has been revealed for all the coats deposited on various substrates (by SEM). However, using HRTEM in agreement with electron diffraction, different phase composition on various substrates has been detected. Cubic pyrite phase (FeS_2) has been detected on Ta substrate. Cubic pyrite (FeS_2) and metastable rhomboedric smythite Fe_9S_{11} have been found in case of Al substrate. Cubic pyrite (FeS_2), metastable rhomboedric smythite Fe_9S_{11} and metastable orthorhombic marcasite (FeS_{2m}) revealed HRTEM analysis of the film on Cu substrate. In case of all deposits the detected crystalline nanograins were surrounded by amorphous matrix.

These appearances represent the first example of the ablative deposition of metastable phases of ferrous sulfide which may evoke more interest in phase modification of FeS and other materials by this simple method.

Keywords: ferrous sulfide, laser noncongruent ablation, metastable phases, amorphization
Control of the location and size of quantum dots nucleation using of a functional substrate inducing a lateral self-organization by a buried dislocation network

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In the last decade, nanotechnology and nanostructures are the subject of increasing interest because of for their industrial applications in varied fields, from electronics to medicine through the aerospace, defense and the environment. Control over the location, distribution, and size of Quantum dots (QDs) is essential for the engineering of next-generation semiconductor devices employing these remarkable nanostructures.

One way to control the location of quantum dot nucleation and their size is the use of a functional substrate inducing a lateral self-organization by a buried dislocation network. The organization driving force in this case is the strain field induced on the surface. The network periodicity is controlled by the orientation angle between the substrate and the bonded film.

In this work, the anisotropic elasticity theory is used to determine the elastic fields (strain, stress fields and the energy density) generated by a square network of screw dislocations, at the free surface of a finite layer bonded onto a semi infinite substrate. The effect of the thickness of the layer and the periodicity of dislocations are also shown. The Si/Si(110) system is chosen in this study. Finally computational results and discussions are given and then compared to those obtained in the isotropic elasticity. The results obtained show that:

- For a fixed period, maximal strain increases when the layer thickness decreases. This evolution is exponential; therefore, it seems so natural to thin a maximum the layer to benefit almost in surface of the effects of the elastic field.
- For a fixed thickness, maximal strain increases with the periodicity of the network, until reach a limit value.
- The maximal energy value calculated at the free surface and extremes are located above the medium of the dislocation lines.

Finally, these results show that by using a strain-selective etching, we can manage to define a nanopatterned surface in a well-controlled way.

Keywords: Self-organization- Nanostructures- Dislocations- Quantum dots- Nanotechnology- Misfit- Self-assembly-Anisotropy.

References
SESSION 4

Biomaterials Derived Ceramics and Composites
Porous Composite Materials ZrO$_2$(MgO)-MgO for Osteoimplantology

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Bone integrity regeneration in terms of postoperative recovery and aesthetic due to the cancer resection or injuries is one of very important issues. Applications of ceramics for using as osteoreplacement material had a special interest now. The most actively developed studies in this area is investigations of zirconia ceramic (ZrO$_2$) included in ISO register as a material for bone replacement. Ceramics based on zirconia stabilized with magnesium oxide (MgO) besides with absence of chemically interaction with body tissues and resistant to most ways of sterilization. Moreover, magnesium is involved in protein synthesis and stabilization of DNA molecules, which can improve regeneration processes.

In this work were studied pore structure and phase composition of ceramic composite material ZrO$_2$(Mg)-MgO at different sintering temperatures. The main mechanical characteristics of the material were determined and it was shown, that they are close to the characteristics of natural bone tissues. It was found, that ceramic strength directly depends on microstresses and at high microstresses ceramic has a low strength.

During sintering of porous ceramic were formed bimodal porosity structure with mean size 26-30 and 94-110 µm which had a positive effect on the pre-osteoblast cells proliferation. In-vitro studies of pre-osteoblast cells, cultivation on material surface has shown a good cell adhesion, proliferation and differentiation of MMSC by osteogenic type.
Bioactive glass ceramics from natural calcium phosphates

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Meat processing facilities produce significant amount of raw animal bone waste representing a serious biohazard. For this reason the safe deposition of these bones is of utmost importance. The main mineral constituent of these bones is calcium phosphate, therefore they can be used as a precursor for production of bioactive ceramics. A process was developed to convert animal bones into a valuable secondary raw material of calcium phosphate based ceramics for bone replacement, tissue repair, and augmentation. Bioactive glass-ceramics containing apatite/\textbeta-\textit{whitlockite and wollastonite were manufactured from chemically and heat treated, protein-free bovine bone as well as a base glass prepared by melt quenching. Body fluids (similar to the blood plasma) enhance apatite formation on the surface of calcium phosphate based bioceramic implants, which in turn improve the bone-binding strength of such devices. A systematic investigation of hydroxyapatite formation on the surface of bioactive glass ceramics immersed into simulated body fluids (SBF) was therefore conducted on glass ceramic samples produced from pre-treated animal bones. Significant changes of phase composition, microstructure, as well as microhardness of surface prior to and after SBF-treatment were observed. Open porosity correlates with other results. Dissolved constituents (Ca and P) of bioactive glass ceramics were quantified by X-ray fluorescence analysis of SBF. Optimum conditions were determined by using microhardness, porosity, and dissolved Ca as independent variables.

\textit{Keywords:} apatite, glass ceramics, implants, bioactive
SESSION 5

Glasses, Coatings and Related Materials
High Performance Ceramic Coatings as Friction Surfaces of Brake Rotors

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Thermal spraying is an industrially widespread coating technology for manufacturing of cost-effective and performant protective and functional coatings for a variety of challenging applications. The coating materials range from metals and metal alloys to advanced ceramic and cermet systems. In the automotive industry, thermal spray coatings are successfully used in series production: as thermal shock protection of lambda sensors, wear protective coatings on synchronizer rings and as cylinder liner coatings, where the friction properties are improved or even tailored and the crankcase weight is significantly reduced by these coating technologies. In the present work the lightweight design potential is used for an engineering concept, where a ceramic layer is applied on light metal alloy brake rotors by atmospheric plasma spraying. Thereby, the layer composite component obtains a wear and corrosion resistant surface with optimum braking characteristics. The substrate material enables significant weight savings compared to the reference steel rotor, subsequently improving the handling characteristics due to lower rotating and unsprung masses. The paper describes the development of such coated lightweight brake rotors with the focus on the coating process technology. The studies include brake dynamometer test with subsequent evaluation. The brake rotors were successfully used in a Formula Student car in international racing competitions; therefore these were designed with particular features, such as a temperature-independent and constant friction coefficient to meet the special competition specifications.
Bromide photo-thermo-refractive glasses

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Present work is focused on the development and characterisation of novel bromide photo-thermo-refractive (PTR) glasses with positive refractive index change (RIC) after UV irradiation and subsequent heat treatment (HT). Classicaly, UV irradiation and following HT of fluoride PTR glasses lead to precipitation of silver nanoparticles and growth of NaF nanosize crystalline phase on them in the glass host [1]. This effect results in a negative refractive index change between exposed and unexposed areas and is used for highly efficient volume phase holograms recording [2,3].

In this work we synthesized and studied the PTR glasses of Na$_2$O-ZnO-Al$_2$O$_3$-SiO$_2$-NaF-NaBr system with different bromine concentration, and doped with Ag$_2$O, Sb$_2$O$_3$ and CeO$_2$. The influence of UV irradiation dose, heat treatment duration and bromine concentration on silver bromide crystals growth have been examined in present work. Optical properties studying and XRD-analysis of silver bromide phase have been conducted. Variation of glass composition, namely, bromine, antimony and silver oxides concentrations as well as dose of UV radiation, and TT time and temperature result in growth of silver bromide nanocrystals on silver nanoparticles with different long-wavelength shift of silver nanoparticles plasmon resonance peak up to 60 nm. Apparently the precipitation of silver bromide also leads RIC up to 800ppm. The possibility of Bragg gratings and planar waveguide structures recording on bromide PTR glass via photo-thermo-induced crystallization have been shown. The possible Mechanisms of phase separation and some photonic applications have been discussed.

Keywords: Photo-thermo-refractive glass, glass-ceramic, holographic material, planar waveguide

References:
Effect of colouring pigments on the microstructure of crystal glazes

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In this research work, different types of crystal glazes were prepared to investigate the effect of the colouring pigments on the microstructure. Thermal analysis (including DTA and HSM measurements) were carried out to determine the processes during melting and crystallization. SEM and XRD test were done to identify the crystallized phases. Composition, size and morphology of the crystal phases were also compared. Scratch, wear and hardness tests were executed to get information about the mechanical properties of the glazes.
Glasses of the system: (90-x) TeO2 + 10Nb2O5 + xTiO2, where x = 2.5, 5, 7.5, 10 and 12.5 mole% were prepared by melt quench technique. Dependencies of their glass transition temperatures (T_g), density, molar volume, glass stability, fraction force constant and infrared (IR) absorption spectra on composition were investigated. It is found that the gradual replacement of oxides, TiO2 by TeO2, increases the glass transition temperature and decreases the fragility of the glasses. Also, IR spectra revealed broad weak and strong absorption bands in the investigated range of wave numbers from 4000 to 400 Cm^{-1}. These bands were assigned to their corresponding bond modes of vibration with relation to the glass structure. The compositional dependences of the above properties are discussed and correlated to the structure of tested glasses.

**Keywords:** Glass transition, Infrared properties, Tellurites, Titanates.
An introduction of CO\textsubscript{2} absorbent of [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] into the coating material of polypropylene itaconate (PPIA) eventually opens the gate of the further investigation of the functional coating related to the air or gas adjustment in the pipe, chamber and rooms. In this paper, fabrication of functional coating materials based on PPIA modified with [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] will be presented. Thermal stability, surface morphology, and the CO\textsubscript{2} absorption ability of the obtained materials have been investigated. The color of the surface coating turned from the yellowish transparent into blue opaque surface after incorporation of [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}]. For the best application of the coating material of PPIA, incorporation of [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] into PPIA shall not more than 20% (w/w) as the coating turned from smooth surface to coarser surface and rigid form. The thermal stability of the PPIA coating decreased as the [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] content in the PPIA increased. The content of 1 until 5% [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] affected on the visible CO\textsubscript{2} adsorption of the coating compared to the without incorporation with slightly increasing value. Above 10%, the CO\textsubscript{2} adsorption of the coating was more than 50% of the adsorption value of [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] MOF only. With the incorporation of 20%, the CO\textsubscript{2} absorption reached about 84% of the absorption value of Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2} MOF. This study proved that the PPIA coating incorporated [Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2}] MOF could afford a coating material with a function to adsorb CO\textsubscript{2} gas.

\textbf{Keywords:} functional coating, Cu\textsubscript{3}(BTC\textsubscript{2})\textsubscript{2} MOF, PPIA, CO\textsubscript{2} adsorption, incorporation
The influence of post-deposition annealing on the microstructure and properties of TiBx/TiSiyCz multilayer deposited by PLD method

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Amorphous multilayer coatings of TiBx /TiSiyCz type were deposited on M2 steel by pulsed laser deposition method using TiB2 and TiXSiYCZ targets. Microstructure, mechanical and friction-wear properties of coated samples were studied in as-deposited state and after annealing at 500 Â°C for 10 minutes. Mechanical properties were determined in nanoindentation test using diamond indenter of Berkovich type geometry loaded at 1, 5 and 10 mN in series of 5 individual indents and in continuous measurement of stiffness by indentation depth mode under load increased up to 50 mN. Hardness and elastic modulus of coated samples were calculated by Oliver & Pharr method. Friction-wear performance were studied in a scratch-test with diamond pin of Berkovich geometry as a counterpart. Microstructure of multilayers was studied after annealing and after nanoindentation test by transmission electron microscopy, high-resolution transmission electron microscopy and scanning - transmission electron microscopy, accompanied by chemical composition analysis by electron dispersive spectroscopy and compared to as deposited state. For microstructure observations, thin lamellas were prepared by focused ion beam method. Samples were cut perpendicularly to the surface of produced coatings. Post deposition annealing of produced TiBx /TiSiyCz multilayers was beneficial for hardness, adhesion to steel substrate and resistance to wear. Hardness of annealed sample was two times values calculated for the same load for coated steel in as-deposited state and four times those for uncoated ones. In scratch tests, lower critical load (Lc1) for annealed samples was tree times higher the value determined for sample in as-deposited state. The values of coefficient of friction for as-deposited and annealed samples were in the range 0.11-0.23, e.a. four time lower than for uncoated steel (0.8). Increased hardness, adhesion to steel substrates and resistance to wear of annealed samples were associated with crystallization of 5-10 nm in size particles of TiB and TiB2 phases in amorphous TiBx matrix. Post deposition annealing did not influence the microstructure of TiSiyCz layers nor interlayer diffusion of elements in the coating. KEYWORDS TiB2, coating, PLD, microstructure, hardness, friction coefficient, wear

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References
SESSION 6

Hetero-Modulus and Hybrid Materials
Self-Healing of SiC/Y$_2$SiO$_5$ Hybrid Composites via Thermal Oxidation

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The use of silicon-based ceramic composites as structural hot-section components for jet engine systems would allow significantly higher operating temperatures than using conventional nickel-based superalloys. Silicon-based ceramic composites improve thermal efficiency, which leads to lower specific fuel consumption, and decreased weight. However, the factor that currently precludes the use of silicon-based ceramic composites is poor in environmental durability of these materials in combustion environments. Water vapor, a combustion reaction product, reacts with the protective silica scale, forming gaseous reaction products. External ceramic coatings are the most common method for protecting these materials from the degradation by water vapor.

In candidate materials of environmental barrier coatings (EBCs) for silicon-based ceramics, Y$_2$SiO$_5$ ceramic is a promising oxide because of a variety of desirable thermal properties such as low thermal conductivity and low oxygen permeability over a wide temperature range. Its thermal expansion coefficient matches well with silicon-based ceramics. But low fracture toughness is a disadvantage on this material.

Besides, the crack healing phenomena on some oxide ceramic composites dispersed with SiC particles were reported, for examples, SiC/Al$_2$O$_3$ and SiC/mullite. Crack healing in the SiC dispersed oxide ceramic composites is attributed to thermal oxidation of SiC particles. Oxidation products including SiO$_2$ and its multiple compounds are filled up in the surface cracks and results in decreasing the stress intension at the crack tips. The reduction in stress concentration at crack tips leads to recovery of mechanical strength. In the present study, capability of crack healing as well as recovery of mechanical strength was investigated on SiC particles dispersed Y$_2$SiO$_5$ composites via high temperature oxidation.

**Keywords:** Y$_2$SiO$_5$, self-healing, high temperature oxidation, strength recovery, ceramic matrix composites.
Inorganic composites with two phosphors in glass for higher lightening properties for white LEDs

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The emergence and development of LED technology can significantly improve the energy efficiency of light sources in combination with good color rendering index. For some applications, such as domestic lighting and greenhouses lighting, the usage of radiation sources with higher color rendering index (Ra> 90) or with the red shifted spectrum is necessary. In white LEDs production the standard yellow-green phosphor (YAG: Ce³⁺) for getting wide integral spectrum consisting from blue (from InGaN diode) and yellow-green (Ce³⁺ luminescence) emission is used. That WLEDs type have a limit on the color rendering index (Ra < 75) and color temperature due to a lack of cerium luminescence intensity in red spectrum region. To increase the color rendering index, for example, the red phosphor based on oxynitrides (often SiAlON:Eu²⁺) is added to yellow-green. The summary radiation given by these sources (diode and two phosphors) corresponds to the warm white light and has a high color rendering index Ra> 90.

For fixing the phosphors powder (as the binder) on the surface of emitting diode element the organic media - silicone is normally used. During exploitation, the LED is heating to a significant temperature (about 150 °C) and as the result of the organic component in phosphor structure gets color centers because of overheating. That lead to reduction in its strength, and therefore the value of the radiation luminous efficiency falls that consequently reduce the real life time of such light sources. To get rid of those problems, the replacement of silicone to inorganic optical component has been proposed.

Such inorganic materials, like glasses and ceramics, are more stable as polymer binders. The perspective material for LEDs phosphor is phosphor in-glass (PiG). The main advantage is that PiG allows to obtain samples with two phosphors in any proportions. PiG is a simple mixture of typical commercial phosphors (YAG: Ce³⁺; SiAlON:Eu²⁺) and glass powders (or frits). After a heat treatment, glass powders can be formed into a stable matrix for the phosphors through the viscous sintering process. The sintering temperature can be considerably lower (~600 °C) than those for phosphor ceramic sintering processes (which are more 1000 °C). In present work, silicate glass systems SiO₂-PbO-PbF₂-AlF₃ as frit materials was taken. Sample with different glass to YAG:Ce³⁺ and SiAlON:Eu²⁺ ratios were prepared. Powders were pressed into disks and heat-treated. The spectral and luminescent properties of these PiG samples have been investigated to define the relationships between light conversion efficiency, composition and structures. Color temperatures and color rendering indexes of samples with varying composition were measured. The dependence of these parameters from the SiAlON:Eu²⁺ content has been received. The possibility to increase the color reproduction by adding SiAlON:Eu²⁺ was shown. In conclusion, the new phosphor is very attractive medium for fabrication the phosphor-converted white LEDs.

Keywords: LED, color rendering index, color temperature, Phosphor in glass, YAG:Ce³⁺, SiAlON:Eu²⁺, glass
This paper focuses on the influence of TiC content on material properties of (Al₂O₃-ZrO₂)-TiC composites with 5-30wt.% of TiC. Bending strength, fracture toughness, hardness and structure of materials have been studied. We have used commercial Al₂O₃ powder with average particle size 4.7 µm; TiC powder with particle size 5 µm and ZrO₂ powders with nominal particle size 30 nm.

Composites were obtained by hot pressing in an argon atmosphere at a sintering temperature of 1400, 1500, 1600°C, with a pressure of 50 MPa. The holding time was 5, 10, 15, 20 minutes. The heating rate up to hot pressing was 50, 75, 150 and 300 °C/min.

It has been shown that changing temperature and heating rate one can obtained composites with a fine structure, very low porosity and uniform distribution of the components in a matrix of alumina: measuring of the average grain size of the individual components had shown that grain size of alumina was 1.5 microns; zirconia - 0.8 microns; titanium carbide - 2.5 microns. X-ray analysis showed that the phase content does not changes and same as in initial mixtures.

It has been shown that hardness are increased linearly with TiC content while fracture toughness had maximum at 20wt.% of TiC which corresponds its optimal content.

**Keywords:** alumina, composite materials, titanium carbide, hardness, toughness, structures
Interesting behaviour of polymers contained multiwall carbon nanotubes

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Mixing is a very important polymer process. Nanocomposites were made by a new type of shear mixer, IDMX. The nanocomposites contained different amount of multiwall carbon nanotubes. Test pieces were prepared by injection moulding method. Thermal, flowing and mechanical properties were measured.
SESSION 7

Light-Weight Metals and Alloys
One-step consolidation and precipitation hardening of nanostructured AA7020 powder by Spark Plasma Sintering

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Lightweight materials are very attractive in the global industry, and more specifically in the automotive and the aeronautic fields. The design of the mechanical parts is already optimized to minimize the mass of the structure while some aluminium alloys exhibit enhanced strength. In order to go further, it is possible to carefully adapt the process of a light material to increase its mechanical properties by combining different strengthening mechanisms such as the Hall-Petch effect and precipitation hardening. In this case, the challenge is how to prevent the grain growth while performing a heat treatment.

The powder metallurgy route was considered. The heat treatable AA7020 aluminium alloy well-known for its high mechanical properties / behaviour was chosen for this study. Commercial powder of this alloy was high energy ball milled and then consolidated through the spark plasma sintering (SPS) technique, whose parameters were optimized to preserve the ultrafine microstructure while producing hardening precipitation. The proposed one-step consolidation process was proven successful and there was no need for further time-costing heat treatment usually performed to improve the mechanical behaviour of such alloys.

The developed material was mechanically tested in quasi-static and dynamic conditions, and compared to an un-milled material and to a commercial heat-treated AA7020-T651 alloy. The strengthening precipitation of the $\eta$-phase (MgZn2) was highlighted by microstructural characterisations such as SEM, TEM and XRD. The Hall-Petch, Orowan and solid solution strengthening were quantified to dissociate the different contribution in order to prove the interest of such a process.

**Keywords:** Strengthening mechanism, Al alloy, Ultrafine grained materials, Spark Plasma Sintering, mechanical properties, high energy ball milling
Development and optimisation by Spark Plasma Sintering (SPS) of the AZ91 magnesium alloy from a metastable as-atomised powder

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The reduction of the energy cost combined with the environmental conservation has contributed to raise the scientists’ interests in structure lightweighting within the sector of transports. In this context, magnesium alloys are of interest in order to substitute the main structural metals in use – aluminium alloys and steel – thanks to their lower density and similar specific strength. Among these alloys, AZ91 is part of the favourite ones because of its moderate cost, enhanced oxidation resistance and improved mechanical strength through a precipitation hardening by essentially $\text{Al}_{12}\text{Mg}_{17}$ precipitates.

As casting generates coarse precipitates and grains, powder metallurgy appears to be an effective route to reduce segregation and preserve a finer microstructure. From all the available powder consolidation processes, Spark Plasma Sintering (SPS) stands out through its ability to establish short heat cycles, restricting the grain and precipitate growth.

The present work aims at developing the AZ91 alloy through this powder metallurgy route to gain improved mechanical properties by an adjustment of the SPS processing parameters. The grain sizes, volume fractions and sizes of the $\text{Al}_{12}\text{Mg}_{17}$ precipitates were experimentally determined as a function of the sintering conditions and related to the mechanical properties of the sintered alloy. While keeping a slightly superior ductility (+12%), higher hardness (+16%) and improved compressive strength (YCS +59%, UCS +32%) were also obtained for the optimised SPS sintering condition in comparison with a conventional cast AZ91 alloy aged under the T6 condition.

**Keywords:** Magnesium alloys, Spark Plasma Sintering (SPS), Mechanical and Microstructure characterisation.
Composites of aluminium alloy and magnesium alloy with graphite showing low thermal expansion and high specific thermal conductivity

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High thermal conductivity, low thermal expansion and low density are three important features in novel materials for high performance electronics, mobile applications and aerospace. Spark plasma sintering was used to produce light metal-graphite composites with an excellent combination of these three properties. By addition of up to 50% in volume of macroscopic graphite flakes, the thermal expansion coefficient of magnesium and aluminium alloys was tuned down to zero or negative values, while the specific thermal conductivity was over four times higher than in copper. No degradation of the samples was observed after thermal stress tests and thermal cycling. Tensile strength and hardness measurements proved sufficient mechanical stability for most thermal management applications. For the production of the alloys, both prealloyed powders and elementar mixtures were used; the addition of trace elements to couple with the oxidation of the powders was studied.
SESSION 8

Materials with Extreme Dynamic Strength
Numerical Simulation of Mechanical Behaviour and Prediction of Effective Properties of Metal Matrix Composites with Consideration for Structural Evolution under Shock Wave Loading

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Mechanical behavior on mesoscopic scale level and effective mechanical properties of stochastic metal matrix composite materials under shock wave loading were investigated by means of computer simulation methods. Shock waves propagation in the mesoscopic volume of metal matrix composite (MMC), deformation processes, nucleation and growth of damages, and evolution of the structure of composite materials consisted of an aluminium matrix and randomly distributed ceramic (B₄C and SiC) inclusions were numerically simulated.

The results of the numerical simulation were used for numerical evaluation of effective elastic and strength properties (elastic moduli and elastic limits) of MMC with different values of volume concentration of ceramic inclusions. The numerical values of the effective mechanical characteristics of investigated materials were obtained, and the character of the dependence of the effective elastic and strength properties on the structure parameters of composites was determined. It was shown that the dependence of the numerical values of effective elastic moduli on the volume concentration of ceramic inclusions is nonlinear and monotonically increasing. The effective values of the elastic limits increase with increasing concentration of the inclusions, however, for the considered composites, this dependence is not monotonic.

Keywords: composite materials, mechanical behavior, effective properties, structure, shock-wave loading, numerical simulation
SESSION 9

Membranes and Catalysts
Designing a New Mixed ZrO2-Al2O3 Oxide Calalysts for Low Temperature Dehydration of Bio-ethanol

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Receiving ethylene and ether from alcohols on oxide supported catalysts is of particular interest because bio-ethanol, obtained by the fermentation of biomass, has advantages from the environmental point of view. For increasing activity oxide catalysts for this reactions are usually promoted by active metals. However, the expensive cost of noble metals still limited the widespread application of these catalysts. In this regard, mixed oxides are considered promising catalysts in dehydration and dehydrogenation of alcohols. Metal oxides and their combinations, such as ZrO₂, Al₂O₃ and CeO₂, were found to show high catalytic activity in acid-base and oxidation–reduction reactions, comparing to supported catalysts.

In the present study, we investigated the effect of preparation method, phase composition and calcination temperature of the (Ce-TZP) – Al₂O₃ mixed oxides on their structural features and catalytic performance in alcohol conversion. Sample powders with different (Ce+Zn)/Al atomic ratios were prepared by sol-gel method from the precursor solutions ZrOCl₂, Al(NO₃)₃, Ce(NO₃). Precipitation was carried out with aqueous ammonia at 250°C for 120 minute. Oxides samples were obtained at 180°C and 500°C. At T=180°C powder contains a substantially amorphous phase, with appearing of tetragonal ZrO₂. At T= 500°C phase composition of the powder is represented by a crystalline phase of the solid solution based on t-ZrO₂ and amorphous Al₂O₃. The catalysts were characterized by XRD, ASAP, REM, N₂-adsorption and UV spectroscopy (pyridine adsorption from solutions). Ethanol conversion was carried out at 200°C-400°C with chromatography analysis of gas mixture (FID, helium as a carrier gas).

The main reaction was dehydration (as well as intramolecular dehydration) with selectivity for olefin and ether 60%. Alcohol dehydrogenation proceeds in parallel (less than 10% conversion). The selectivity is correlated with the catalyst composition and depends on calcination temperature. Calcined at 500°C samples showed the highest catalytic activity due to recognized strong acid sites on the surface of catalyst.

Keywords: mixed oxide, alcohol conversion, acid-base catalyst, zirconia oxide, aluminum oxide, ether, ethylen, bio-ethanol
Design of nanocomposite active components and structured catalysts for steam/autothermal reforming of biofuels

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Design of efficient, inexpensive and stable to coking catalysts for transformation of biofuels into syngas and hydrogen is an urgent problem of sustainable and renewable energy field including hydrogen production, synfuels synthesis, internal reforming in solid oxide fuel cells, etc. In this work, nanocomposite active components comprised of nanoparticles of Ni(Ru) or their alloys and CaTiO₃ perovskite have been synthesized by different methods and studied as bulk and supported on γ-Al₂O₃ promoted by Mg. The structural and redox properties of the catalysts were characterized by XRD, BET, TEM with EDX, UV-vis, XPS, TPR-H₂, FTIRS of CO test molecule. Their performance as fractions and layers on structured ceramic, Ni-Al alloy and carbon-Ni-Al ceramometallic substrates has been studied in steam reforming of ethanol and glycerin. The influence of preparation method and catalyst composition on the structural and redox properties of the catalysts have been elucidated. High activity and coking stability of the catalysts are provided by a high dispersion of metallic Ni and Ni-Ru alloy due to strong interaction with CaTiO₃/MgO/γ-Al₂O₃ support and suppressing support acidity by Mg cations. The optimal catalyst loaded on the foam supports with a good thermal conductivity has shown more effective performance in reforming reactions as compared with ceramic ones. The catalyst on a carbon-Ni-Al ceramometallic substrate shows highest selectivity for hydrogen, while the catalyst on Ni-Al foam provides a stable performance in a realistic reaction mixture during 50 hours.

Keywords: perovskite, CaTiO₃, Ni-Ru alloy, structured catalyst, steam reforming, biofuels
High Capacity MnOxZrO\textsubscript{2} sorbent for elementary mercury capture: preparation, characterisation and comparison to other sorbents

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Elemental mercury removal from gas streams needs oxidative transformation of mercury prior or parallel to load it to the sorbent if almost complete mercury removal is required [1-2]. The oxidative capture of mercury is well documented in aqueous solution for analytical purposes. Potassium permanganate in acidic solution is generally used for trapping mercury. Janssen et al.[3] used MnO\textsubscript{2} for collecting mercury vapour from air. Granite et al. [4] compared a number of different sorbents, including MnO\textsubscript{2} supported on alumina, which, together with other metal oxides, e.g. Cr\textsubscript{2}O\textsubscript{3}, and metal sulfides, e.g. MoS\textsubscript{2}, were found to exhibit moderate capacities for Hg capture among the candidates considered. The use of manganese oxides for Hg removal was the subject of a patent by Pahlman et al. [5], but the surface areas reported were quite low (1-10 m\textsuperscript{2}g\textsuperscript{-1}), which resulted low removal efficiency. Thus, to make manganese oxides more effective, there is a need to improve surface area and this can be achieved by co-precipitation with a support. Since the oxidative nature of the sorbent also seems to be important the support also crucial.

Therefore manganese oxide-zirconia type (MnO\textsubscript{x}:ZrO\textsubscript{2}) sorbents were prepared based on the sol gel technique. The heat treatment below 500 °C resulted a high surface area amorphous solid structure consist of (Mn\textsubscript{2}O\textsubscript{3}Bixbayit) with and amorphous ZrO\textsubscript{2} phases.

The Hg(0) removal capacity by the MnO\textsubscript{x}ZrO\textsubscript{2} sorbents were tested at different Mn/Zr atomic ratio and by the MnO\textsubscript{x} or ZrO\textsubscript{2} both have been formed the same process as the MnO\textsubscript{x}ZrO\textsubscript{2} sorbents. The zirconia alone (used as support) displays negligible Hg uptake. The MnO\textsubscript{x}ZrO\textsubscript{2} sorbent prepared with 0.87 Mn:Zr atomic ratio achieved an exceedingly high breakthrough capacity larger than 15 m/m % . The breakthrough capacity of 15 m/m % is over 200 times higher than that for activated Norit-Darco FGD carbon, which has been widely used for in direct injection trials for Hg capture from flue gas [5] The precipitated MnO\textsubscript{2} prepared in the same manner as the co-precipitated MnO\textsubscript{x}ZrO\textsubscript{2} sorbent was found have a much lower breakthrough capacity, as anticipated from the lower surface area and the lack of oxidation promoter support.. However, this was still considerably higher than for bulk manganese oxide represented by Fischer chemicals.

The preliminary investigation into the effect of temperature has proved that the sorption capacity does not fall until temperatures above 150°C. However, even at 300 °C, the breakthrough capacity still remains higher than 5% w/w. It can state that the sorbent be a candidate not just ambient temperature but moderately high temperature till 450 C applications.

References
SESSION 10

Minerals for Environmental and Medical Application
Effect of mechanical treatment on properties of natural zeolites

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Zeolites have been extensively used in various applications due to their unique properties for catalys, ion exchangers, adsorbents. Zeolites are crystalline aluminosilicate materials having microporous (zeolite pore) structure. It is well known that specific surface area is a dominant parameter for zeolites and mechanical ball milling activation will change the structure, specific surface area, phase content etc. and may improve their properties.

The goal of this work was to study changes induced by mechanical activation on the structure and properties of natural zeolite Tokaj Mountain deposite.

Mechanical activation of powder was performed in a ball mill for 600 minutes. Structure, phase composition, morphology, specific surface area were studied by XRD, SEM, BET methods.

The SEM data showed that particles of zeolite powders has irregular shapes with an average particle size of 27 µm. After activation of 20 minutes particle mean size was 5.5 µm and after 600 minutes of milling time the particle size became 28 µm with spherical shape.

Initial zeolite powder has a specific surface area of 19 m²/g. During the first 60 minutes, the specific surface area of zeolite powder increases, reaching 33 m²/g. After 120 minutes, the specific surface slightly decreases and after 600 minutes of activation, it was 20 m²/g. The changes of the specific surface area of the zeolites correspond to the theoretical curve of the developed specific surface as function of the activation time – on first stage one can observed Rittinger-zonewhen increasing of the specific surface is proportional to the activation time; on second stage we have aggregation zone when specific surface almost does not change and last stage - agglomeration-zone decreasing specific surface with the activation time. It have been shown that after 180 min of mechanical activation on material was change its mineral composition.

Keywords: zeolite, mechanical activation, specific surface area
Removal mechanism of arsenic by hydration of calcium aluminoferrite

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Our demand for water is increasing due to the continuous development of industry and population growth. Therefore, the depletion of water resources is becoming a problem, and it is necessary to develop new water resources. Arsenic polluted groundwater is one potential resource that can resolve this problem. We have focused on the use of calcium aluminoferrite (6CaO\cdot2Al_{2}O_{3}\cdotFe_{2}O_{3}: Ca_{6}A_{2}F), which can remove arsenic by a simple process and low cost, and we have previously reported that it is effective at removing arsenic \cite{1}. However, the removal mechanism of arsenic by Ca_{6}A_{2}F is not clear. Therefore, the purpose of this study is to elucidate the removal mechanism by hydration of Ca_{6}A_{2}F.

A 0.5 g quantity of Ca_{6}A_{2}F was added to 45 mL solutions of As(III) and As(V) at concentration of 5-50 mg\cdot L^{-1} and the mixtures were stirred for 60 min at room temperature. Finally, the suspensions were separated into liquid and solid phases.

After 60 min, over 90\% of As(III) and As(V) was removed from the solutions regardless of arsenic valence by hydration of Ca_{6}A_{2}F. XRD patterns showed Hydrogarnet (HG) and a small amount of an AFm phase appeared in any case. A gel was formed on the HG crystals. EDX analysis showed that it contained Fe and As elements. Therefore, we conclude that As was removed by adsorption to Fe gel on the surface of the HG.

\textbf{Keywords:} removal arsenic, calcium aluminoferrite, Ca_{6}A_{2}F, AFm, Hydrogarnet

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Characterization and Synthesis of Silica Nanoparticles-Crosslinked Hydrogels with Decorations of Aminopropyl Silane

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Biopolymers are materials of great interest within the scientific community thanks to their biocompatibility, toxicity, solubility, accessibility and affinity with the environment. Biopolymers have a wide variety of applications in biotechnology, biomedicine and in the pharmaceutical industry. Carboxymethyl cellulose is a polysaccharide with biopolymer properties, and it allows the formation of hydrogels for the controlled release of drugs. However, its degradation rate demands a cross-linking process that would improve its properties and the self-healing in corrosive media. Cross-linking of biopolymers by nanoparticle inclusion generates an interaction with the biopolymer matrix, thus allowing the optimization of mechanical, thermal and oxidative properties. Silica nanoparticles were synthesized by the Stöber method using an alcohol-based tetraethyl orthosilicate precursor and ammonium hydroxide as catalyst. The nanoparticles were decorated by suspension in dimethyl sulfoxide in order to add aminopropyl triethoxysilane and functionalize with amine groups. The hydrogels were obtained from carboxymethyl cellulose. The carboxylic groups from this polymer were activated by carbodiimide chemistry with the purpose of forming covalent bonds with the amino groups of the nanoparticles. The presence of functional groups and nanoparticles within the hydrogels was determined by FTIR and SEM. Their distribution and sizes were also studied using these techniques. The degradation of the modified hydrogel was analysed by TGA and swelling tests, which were used to evaluate its absorption capacity.

References
Synthesis and crystallization of titanium dioxide in supercritical carbon dioxide

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In this work, a versatile, low-temperature route to synthesize brookite phase titanium dioxide (TiO$_2$) particles with supercritical carbon dioxide is presented. The specimens were synthesized using tetra-$n$-butyl orthotitanate as precursor of TiO$_2$ in the supercritical chamber. The pressure and temperature of the chamber were 150 bar and 50 °C, respectively. After reaching treatment parameters deionized water was introduced into chamber with a co-solvent pump to enable hydrolysis reaction of TiO$_2$ precursor. A mixer was employed inside the chamber to ensure proper mixing of water and precursor. Reaction times of 10, 60 and 300 minutes were used to determine an effect of synthesis time to characteristics of the specimen. Characterization was made by X-ray diffraction, differential scanning calorimetry, transmission electron microscopy and nitrogen adsorption testing. The results showed that the particles synthesized with reaction times of 10 and 60 min contained mainly amorphous and brookite phases whereas longer reaction time (300 min) produced the mixture of amorphous and crystalline anatase phase. The specific surface areas obtained with reaction times of 10, 60 and 300 min were 567 m$^2$/g, 320 m$^2$/g and 274 m$^2$/g, respectively.

*Keywords:* Titanium dioxide, Brookite, Supercritical carbon dioxide
Nanosrtuctured Organic Alkali-Soluble Silicate for Innovative Industrial and Construction Applications

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In this paper we analyzed the properties of the water-soluble high-modulus silicate systems, and their technology for producing. We have shown how these systems are transformed, from lower to higher oligomers, through the formation of the silica sol and the implementation of the sol-gel process for these oligomers. We have conducted advanced research of various aspects of the use of these materials as the binder. Modifiers have been proposed for making of hybrid nanostructured composite materials by a sol-gel process. Have been shown of structuring phenomena some aspects, synthesis and application of hybrid materials based on silica with grafted polymers. Production of polymer concrete, which was nanostructuring of silicon dioxide, serves as an example of the application of silicate systems. It has been shown, the possibility of modifying compositions using the nanostructuring agents such as tetrafurfuryloxysilane, and an aqueous dispersion of chlorosulfonated polyethylene and other polymers. In the present work are also described methods of synthesis products for modifying a sol-gel process using polyurethanes. They include applications of sols for producing of hybrid nanocomposites, monolithic blocks and fire-resistant materials and technology for the production of new nanocomposite materials and acid-resistant coating for protection aggressive environments.

It should be emphasized that even small changes in process parameters in the manufacture of nanocomposite materials can have a significant impact on the final product. On the one hand, this increases the complexity of the system and, on the other hand, offers an excellent opportunity to develop their own, individual solutions of practical problems, which are often associated with minimal changes in the composition and production technology.

\textbf{Keywords:} nanostructured materials, organic alkali-soluble silicates, hybrid nanocomposites, nanocomposite materials, fire-resistant materials, sol-gel process.
Matrix-Isolated Smart Nanocomposite Materials – Alumina Silicon Flocculants Coagulants

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Matrix-isolated smart nanocomposite materials aluminum-silicon flocculant-coagulant ASFC is one of the few binary compositions, composed of only inorganic components: a coagulant - aluminum sulfate and an anionic flocculant - active silicic acid. Action ASFC based on the reaction of the primary components ASFC - coagulant aluminum compound and flocculant active silicic acid, adapting to the state of the aquatic environment to be cleaned. Complex compounds are formed with higher flocculating ability. There is a synergistic effect - an increase the efficiency of the impact, as a result of integration of individual processes into a single system.

However, existing methods, allowed receiving such materials only in the form of solutions. Their lifetime is not more than 2-3 weeks. This factor is holding back the practical use of ASFC, in industrial practice for wastewater treatment.

The task was solved with a processing of aluminum-silicon raw material with sulfuric acid, separating the liquid phase from the solid and liquid phase dehydration. Dehydration of the resulting solution to obtain a dry product, carried out at a temperature below the boiling point of water by evaporation under vacuum, or by dispersing in a high-temperature high-speed gas stream of coolant.

These processing methods have allowed “freezing” and isolating the solid phase matrix components produced flocculant-coagulant, which are in a nanodispersed state. Quick transfer of active ingredients in a solid state can dramatically reduce the rate of diffusion processes and, thus, preserve the activity of the material.

Experiments have shown that the material thus obtained can be stored for a long time. For some samples were observed to preserve 90% of the activity for over 2 years. For effective water treatment the reagent is required in much smaller quantities. An important feature is the use of powdered ASFC in water treatment from oil contaminations.

**Keywords:** composite flocculants-coagulants; water treatment agents; aluminum-silicon and flocculants-coagulants; ferrum-silicon flocculants-coagulants; matrix isolation.
SESSION 12

Novel Synthesis and Processing Technology
Recovery of phosphoric acid from steel slag by ion-exchange resin (H-R) dissolution method

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It is said that phosphate rock will be exhausted in about 50 years. Many researchers have been studying about the recovery process from unused inorganic wastes, including phosphorous. In particular, phosphate was removed during the steel making process which was condensed into emitted steel slags. Therefore, phosphorus recovery method from steel slags have been focused. However, steel slag includes harmful heavy metals and these metals was difficult to remove. The authors paid attention to ion-exchange resin dissolution method (H-R dissolution method). This method is able to dissolve low solubility salt by ion-exchange resin easily, and remove heavy metal ions by ion-exchange reaction. In the present study, we investigated dissolved properties of steel slag by using H-R dissolution method.

Steel slag within an ion-exchange capacity was suspended into deionized water. Then, 5 g of strong acid cation-exchange resin was added. The mixture was stirred up to 360 minutes at room temperature or at 80°C. Samples were taken at various intervals. The sample solutions were analyzed by ion chromatography and ICP-OES.

We investigated dissolution behavior of steel slag by H-R dissolution. Concentration of phosphoric acid was increased with incensing immersing time to dissolve steel slag. On the other hand, metal cation were removed by ion-exchange resins. The eluted concentration of these ions were slight. H-R dissolution method was able to dissolve steel slag and it was easy to obtain less cation solution. Therefore, this novel method is promising as a way of recovery phosphoric acid recovery from steel slag.

Keywords: Steel slag, Phosphate recovery, Ion-exchange resin
Preparation of silica microsphere particles by spray-drying method

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Silica microsphere particles are used in various applications such as in sealing materials and precision polishing agents. These particles can be prepared using well-known methods such as the Stober method and the emulsion method. However, these methods result in the formation of sub-micron or nanometer sized particles. Preparation methods for obtaining micron-sized particles have hardly been reported. Furthermore, most of the reported methods use expensive tetraethoxysilane (TEOS) as the silica source. We have focused on a spray-drying method for preparing micron-sized spherical particles. In this present study, we investigate the preparation conditions for obtaining silica microsphere particles using spray-drying.

We used potassium silicate solutions of different concentrations as the spray-drying solution. The solutions were spray-dried at a temperature of 100°C and an atomizing pressure of 100 kPa, to produce potassium silicate microsphere particles. These particles were washed with 3M HCl solution in order to obtain spherical silica particles by removing potassium ions.

Spherical particles having diameters of 1-10 µm and smooth surfaces were obtained depending on the experimental conditions used. Moreover, filled particles were obtained from dilute solutions of potassium silicate, but infinite and hollow particles were obtained with concentrated solutions of potassium silicate. Potassium could not be detected using energy dispersive X-ray (EDX) analysis of the obtained particles after HCl treatment, revealing that the products were composed of pure SiO₂. A compressive strength of approximately 670 MPa was obtained on examining one particle of silica microsphere.

Keywords: Silica particle, microsphere, spray-drying method, compressive strength
Preparation of pierced hollow calcium carbonate particles by addition of seed crystals

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CaCO₃ can be synthesized by blowing CO₂ gas into a Ca(OH)₂ suspension. Basic calcium carbonate (2CaCO₃·Ca(OH)₂·1.5H₂O, BCC), an intermediate product, is generated under specific conditions and a Basic drop (B-drop) in the electric conductivity curve is attributed to the generation of BCC. In contrast, Yamada et al. reported that the addition of a seed crystals under BCC generation conditions affected the control of CaCO₃ particle size and shape [1]. In the present study, we investigated the morphological control of CaCO₃ by adding a seed crystals under the BCC generation condition.

We prepared a 5 mass% Ca(OH)₂ suspension. CO₂ gas was then blown into the suspension with stirring in 100 rpm at a flow rate of 0.34 dm³·min⁻¹ at 5°C for 140 min. The electric conductivity was measured to investigate the reaction process. Reagent grade CaCO₃ was added as seed crystals at several addition time.

The X-ray diffraction study indicated that the products were calcite CaCO₃. Generation of BCC and a B-drop in the electric conductivity curve were observed at 5°C without seed crystals. The obtained crystals were about 0.1 μm in size. When the seed crystals was added several addition time, rhombohedral crystals of about 0.2 μm size were obtained. Moreover, we only obtained pierced hollow CaCO₃ particles to add seed crystals at reaction starting immediately.

Keywords: calcium carbonate, calcium hydroxide, seed crystal, morphological control

References
The composite reinforcement production in digital manufacturing: experimental validation of the heat transfer and cure modeling results

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The experimental validation of the heat transfer and cure modeling results for 8-mm fiber-reinforced thermosetting composite reinforcement is reported in this article. The temperature and degree of cure of composite reinforcement are predicted using a two-dimensional heat transfer and curing model. The model uses the infrared radiant heating theory and takes into account the heat transfer between the composite rod and the surrounding air. The implicit finite difference method was used to solve the system of governing equations. The results obtained using mathematical model were compared to experimental data: the temperature field inside the composite reinforcement was measured by means of naked thermocouple; Differential Scanning Calorimetry (DSC) was used to measure the degree of cure of the final product. Calculated and measured temperature and degree of cure fields were in good agreement.

Keywords: Fiber reinforced plastic (FRP) composite rods, IR heating, nidltrusion, heating chamber
Influence of Processing Time and Concentration in Phosphoric Acid Treatment of Zinc Oxide

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Zinc oxide is often used as a white pigment for cosmetics; however, it shows photocatalytic activity that causes decomposition of sebum on the skin when exposed to the ultraviolet radiation in sunlight. In this work, zinc oxide was reacted with various concentrations of phosphoric acid for several hours to synthesize a novel white pigment for cosmetics. The chemical composition, powder properties, photocatalytic activities, colors, and smoothness of these pigments were studied.

The obtained materials exhibited X-Ray diffraction peaks relating to zinc oxide and phosphate after phosphoric acid treatment. The ratio of zinc phosphate to zinc oxide was estimated from inductively coupled plasma results. Sample treated for 6 hours had high P/Zn ratio, therefore this sample contained larger amount of zinc phosphate. The concentration of phosphoric acid had less influence on P/Zn ratio than the processing time. Particle size of samples became larger by phosphoric acid treatment. The photocatalytic activity of zinc oxide was inhibited by phosphoric acid treatment. The obtained samples had enough high reflectance at the visible light region.

**Keyword:** zinc oxide; phosphoric acid treatment; photocatalytic activity; particle size
Comparative Analysis of Inorganic Polymers Obtained by Raw Materials with Different Properties

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Aim of present research is the development of new binders which could – partially – replace the widely used cementitious materials. Studies has shown that industrial wastes can also be used as raw material for the manufacture of these new kind of binders. Aim of present research was to carry out a comparative test in which a new types of binder (geopolymer) with the most favourable properties can be produced, with regards to its strength. We investigated the influence of various chemical compositions and particle size distribution of the starting materials (fly ash and slag) on strength properties.

During experiments fly ash and slag powder with three - three different specific surface area was used to produce geopolymers. As a first step change in strength properties was examined by applying various initial parameters (surface area, Si / Al ratio, the concentration of NaOH). In case of slag-based geopolymers test samples the maximum compressive strength exceed the highest strength class (52.5 MPa) of cement standard (MSZ EN 197-1). Furthermore the relationship between strength and structure of these geopolymers was investigated, using FTIR for examining the polymer structure, XRD for phase composition and SEM for morphological studies.

Based on results geopolymer with most favourable strength properties can be achieved by the following parameters: surface area of slag with 3578 cm²/g, Si / Al ratio = 7.0 and 12 mol/dm³ concentration of alkaline solution. The available strength is comparable to the values of classical cement standard and the evolution of strength can be well monitored.

**Keywords:** Geopolymer, Fly ash, Furnace slag, Specific surface, Compressive strength, X-ray diffraction, FTIR spectroscopy, Scanning electron microscopy
The one of perspective directions of new materials creation is shock-wave synthesis and compaction, using powder mixtures. The intensive researches of compressibility of mixtures are conducted in this direction to create materials with desired properties, in particular, heat-resistant and high-strength ceramics.

The results of numerical experiments on modeling of shock wave loading of mixtures with allowance for phase transition components in their composition are presented. The significant change in volume in the region of phase transition components included in the mixtures allows us to expand the range of variation of thermodynamic parameters of the mixtures under shock wave loading. This fact allows us to purposefully create the necessary conditions for the synthesis of new materials. The calculation model is based on the assumption that all components of mixture under shock-wave loading are in thermodynamic equilibrium (model TEC). Equations of state of Mie–Grüneisen type are used to describe thermodynamic parameters of condensed phases, taking into account the dependence of Grüneisen coefficient only from temperature [1]. The model TEC allows us to describe, in particular, and the region of the polymorphic phase transition, considering the material in the region of phase transition as a mixture of low-pressure phase and high-pressure phase.

A good agreement of these model calculations with the data of different authors defined on the basis of experiments is obtained. Thermodynamic parameters of the nitrides mixture, solid and porous mixtures with quartz as component were reliably described [2, 3]. It is shown that conditions for the start of the phase transition of a pure substance can also be used in the simulation of mixtures consisting of one or more components experiencing a phase transition under shock-wave loading. This model is useful for determining the compositions and volume fractions of the components of the mixture to obtain the specified parameters of solid and porous materials under shock-wave loading.


Keywords: equation of state, shock adiabata, powder mixture, thermodynamic equilibrium, phase transition.
Influence of binding composition on the structure and properties of steel work-pieces obtained by injection molding

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Manufacturing of work-pieces using the method of injection molding is a prospective technology. Raw material for this technology is powders (metal and ceramic) and a binding substance. In MIM-technologies multi-component bindings are used, one of which is a low-molecular compound and another – a high-molecular one. It provides sufficient hardness to a work-piece after removal of a low-molecular component of a binding substance and good gas permeability for its subsequent removal. Practically, two-component bindings with SAS-additives have found wide application. The ratio of binding components may be varied, and the content of a high-molecular component may stay in the range from 80 to 20% volume ratio [1].

The mode of unbinding plays an important role [2]. Changing heating rate and holding time, it is possible to influence the porosity of the work-piece.

The paper presents the results of the research analyzing the influence of binding composition on the structure and properties of the stainless steel obtained by injection molding. We have determined the tailored composition of binding, which provides sufficient feedstock viscosity, low porosity of work-pieces, etc. Three binding compositions have been studied: 1:6, 1:2, 2:1 polypropylene and paraffin wax, respectively. Stearinic acid has been used as a SAS (surface active substance). The work defines wetting angles of steel substrates with binding, rheological properties of feedstocks, porosity, and properties of MIM-products.

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Keywords: Powder/binders Feedstocks, Injection Molding, Sintering

References
Investigation of austenitic stainless steel properties obtained with the use of PIM-technologies

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Injection molding technology is a prospective direction of powder metallurgy, as far as it allows obtaining sintered work-pieces of an irregular shape with high density and balanced shrink. The properties of sintered work-pieces are highly affected by a grain-size composition, initial powder shapes, their preprocessing, etc. Therefore, to obtain the work-pieces with high mechanical characteristics it is necessary to study the properties of the initial “feedstock”.

The properties of the initial “feedstock” and sintered material are investigated in the paper. Feedstock appears to be granules of a cylindrical shape with a diameter of 3-3.5 mm and a length to 4 mm. The basic feedstock material is the mixture of nickel-chromium and steel powders, the major feedstock phase is ferrite. The distribution of powder particles according to the size is polydisperse, about 85% of particles in the powder mixture have a spherical shape. While sintering a phase transformation occurs, and after sintering the ferrite phase of feedstock transforms into the austenitic phase. Chemically, the sintered material corresponds to the class of austenitic stainless steels. The material density after sintering is more than 98% of full density. The distribution of pores outside diameter is polydisperse, the basic amount of pores (> 50 %) is relatively fine pores with a diameter of 3-5 µm. Microhardness of the sintered material is 1.6 hPa, its modulus of elasticity – 115 hPa.

Keywords: powder injection molding, metal injection molding, stainless steel, feedstock, properties of steel
Certification of specifications for PIM-product powder compositions (after mechanical activation)

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Powder production techniques influence their physical, chemical and technological properties, which, in their turn, have a significant impact on manufacturing and properties of MIM-products. To manufacture products using the method of injection molding it is necessary to have the information about surface morphology, pour density, mean-squared deviation of a chemical content in the powder composition from the nominal one, the percent of ball-shaped particles, and an average size of particles.

The paper presents the results of certification for SS-powder composition after various times of mechanical activation – 0, 1, 5, 10, and 20 min, respectively.

It is shown that at mechanical activation powder surface morphology changes, conglomerates are formed from particles and particles obtain a longish, flattened shape. With the time increase of mechanical activation specific surface of powder composition decreases, consequently, linear sizes of particles increase from 8 µm without mechanical activation to 250 µm after 20 minutes of activation. The use of mechanical activation leads to the increase of pour density by 15%. However, density of sintered samples decreases by 20%. Meanwhile, hardness of sintered samples is the highest one at 5 minutes of mechanical activation.

The work is performed in accordance with the plan of Research and Development SB RAS, project 23.2.3, and, partially, with funding from Ministry of Education and Science of the Russian Federation, project № 14.578.21.0035-RFMEFI57814X0035.

Keywords: mechanical activation, powder, properties, injection molding
Influence of mechanical activation in powder composition 03X17H12B on product properties

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The method of shaped work-pieces molding with the use of PIM-technologies (Powder injection molding) includes MIM (Metal Injection Molding) and CIM (Ceramic Injection Molding). Powders (metal or ceramic) and binding substances serve as raw materials for this technology. The properties of initial powders (their shape and size) and their mechanical activation play a crucial role in the properties of the products sintered on their basis[1, 2].

The paper presents the investigation analyzing the influence of activation time in 03X17H12B powder composition on the properties of sintered samples obtained by injection molding.

The 03X17H12B composition was prepared with the use of powder metallurgy technologies via mixing initial powders in necessary weight concentrations. The composition was mechanically activated in a planetary mill. Rotation rate of mill shells was constant, and it was of 50 Hz what provided acceleration to 30 g. The time of activation was 1 min, 5 min, 10 min, and 20 min. Density, an average size of pores, hardness, an average grain diameter, and the sizes of coherent scattering regions of main phases in sintered PIM-products are determined in the paper.

The work has been completed within the framework of Federal Target Program project № 3.1386.2014.

Keywords: mechanical activation, powder, properties, injection molding

References
Effect of laser power and laser fluence on the size of TiO$_2$ nanoparticles synthesized by pulsed laser ablation in deionized water

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Pulsed laser ablation in liquids (PLAL) is a novel and green technique to synthesize nanoparticles directly into liquids in the form of a suspension. There has been extensive research on synthesis of nanoparticles of metals, metal oxides, semi-conductors, nano-diamonds, and carbon nanoparticles by PLAL$^1$. Researchers have reported PLAL as a simple yet effective method for synthesis of nanoparticles with the possibility to control their properties using process parameters$^2$. In order to effectively produce nanoparticles for a particular application, it is extremely important to know how the nanoparticle size changes with the process parameters.

Despite the importance, there has been little research on employing the process parameters to determine how they affect the nanoparticle size. In this study, we discuss how the process parameters of PLAL technique, such as laser power and laser fluence, affect the size of nanoparticles synthesized. This makes it extremely important and interesting lecture for the scientific as well as industrial community. In this study, a nanosecond fiber laser 1062 nm was used to cause ablation at the surface of titanium target dipped in deionised water and synthesize TiO$_2$ nanoparticles in a green and energy efficient way with varying laser fluence and laser power. Small angle x-ray scattering (SAXS) technique, TEM, and XRD were used to characterise the nanoparticles.

**Keywords**: nanoparticles, pulsed laser ablation in liquids, SAXS, TEM, laser parameters, nanoparticle size, laser fluence, laser power, nanosecond pulsed laser

**References**


A computational homogenization method to reducing the time required to design, mesh and calculate the effective elastic response of three-phase composites

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The development of multiphase composites knows considerable effort recent years. The control of the technical development and study of the elastic mechanical properties requires very complex methods of calculation taking into account the three-dimensional character of geometry and multiphase material. In this work, a computational homogenization method is used to reduce the time required to design, mesh and calculate the effective elastic response of three-phase composites. The influence of interphase parameters like stiffness and volume fraction of interphase on effective material properties of transversely randomly distributed uni-directional fiber composites and randomly distributed spherical particle composites is studied. The numerical results are compared with the finite element method presented in KARI et al (2008). Comparing the results gives a very good agreement, with a maximum relative error not exceeding 0.014%.

**Keywords:** homogenization, multiphase composites, effective material properties, elastic response.

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![Variation of effective material properties with increase of interphase stiffness for transversely randomly distributed fiber composites](image1.png)

(a): Effective shear modulus of fiber composites.

(b): Effective Young modulus of randomly distributed spherical particle composites.

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Fig. 1. Variation of effective material properties with increase of interphase stiffness for transversely randomly distributed fiber composites (compared with the finite element method presented in KARI et al (2008));

(a): Effective shear modulus of fiber composites.

(b): Effective Young modulus of randomly distributed spherical particle composites.
Research of Products of High Temperature Synthesis and Their Use for Initiation of Reaction with the High Power

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The method of production of materials by out-furnace process of self-propagating high temperature synthesis (SHS), flowing in the conditions of action of centrifugal force, is developed presently. The primary purpose of working is achievement high level of generating of energy and use of it for forming of steady meta-stable crystalline phases with an uncommon set of physical and chemical properties. The high-temperature centrifuge which provides special conditions for carrying out SHS without external supply of heat is for this purpose created.

The features of process consist in forming into the revolved reactor of adiabatic wave of combustion in the time of aluminothermic recovery of metals from their oxides. The recovered metal forms clusters which move along of a reactor axis with acceleration. Velocity of particles of metal in a stream reaches tens of meters per second. Total kinetic and thermal energy of clusters is sufficient for a rupture of chemical bonds in molecules and for initiation of chemical reactions in mixes of reagents with a high barrier of activation. Temperature in the front of a wave of burning reaches 4 000 K. Duration of process there are 0,4 – 1,5 pages.

Features of process consist in formation in the rotating reactor of an adiabatic wave of burning at aluminothermic restoration of metals from their oxides. Products of the high-temperature synthesis proceeding in extreme conditions significantly differ in structure and properties from SHS products in the stationary mode. Methods of the quantitative X-ray phase analysis, electronic microscopy, an electronic paramagnetic resonance, the combined dispersion of light were applied to their research.

The phases possessing semiconductor properties (CuAlO2), phases with a free valency (Al80B4O36, Mg3B2O6), the conductive oxide (CuBO2) are received.

**Keywords:** combustion, centrifugal acceleration, high-temperature synthesis, adiabatic wave, the free valence
SESSION 13

Phase Diagram as a Tool of Materials Science
Design of efficient thermoelectric materials via first principles calculations

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Adaptation of thermoelectric materials to industrial applications demands to design relatively complex materials. The search and optimization of these materials requires not only to have an in depth knowledge of their thermoelectric properties but also of other physical properties such as their mechanical and thermodynamical stability often linked to the presence of structural defects. Experimentally the study of these properties can often not be performed exhaustively and is sometimes difficult to tackle especially concerning the defects.

This is where *ab initio* calculations can be of a precious help especially in permitting to select better materials in silico before going to the synthesis.

In this presentation we will illustrate the input of first principles calculations on three aspects and on three different thermoelectric materials:

- the influence of defects/dopants on the thermoelectric properties of ZnSb [1]
- the phase stability of High Manganese Silicides (HMS) [2]
- the mechanical and thermal properties of Ni-Ti-Sn half-Heusler and Heusler compounds [3]

References

Predicting a composite material for thermoelectricity. About the phase separation in the multicomponent system, Hf-Ni-Sn-Ti-Zr

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The phase diagram of the pseudoternary (Hf,TiZr)NiSn system is of interest for the selection of the compositions and for the prediction of the stability of the final material prior to material synthesis in the scope of thermoelectric properties. A review of the experimental data in this system shows that it is difficult to obtain stable and single phased alloys by mixing component in different proportions. To overcome this difficulty we have performed thermodynamic stabilities by means of ab-initio calculations in order to predict the possible phase separations in the half Heusler phase. Our calculations are consistent with the available experimental data and we obtain the evolution of the solid-solution region for different temperatures around the optimal operating temperature of these thermoelectric materials. The limits of the solubilities of the phase separation are also plotted at different temperatures. The limit of solubilities of the phases are plotted at different temperatures from 500 to 1300K; its show the shape of the phase separation through the phase boundaries.
The effect of rejuvenation treatment on microstructure and mechanic properties of Ni-superalloy in gas turbine blade failures.

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Nickel-based superalloys on gas turbine blades are normally present in mechanical components requiring resistance to high temperatures and extreme mechanical stresses. The large amount of γ'-precipitates coherent with the γ-matrix in these superalloys ensures excellent properties at high operating temperatures. Nevertheless, after long service periods, the γ'-precipitates grow and lose coherence with the matrix, which leads a blades failure. Intermediate thermal treatments are a potential solution to recover the microstructure, extending the component lifetime. Thermal cycles for γ'-dissolution and re-precipitation define the efficiency of such thermal treatments. The aim of this work was to study the effects of solution and re-precipitation treatment temperatures on evolution microstructural and mechanical properties of Ni-based superalloy Inconel 738, after a long service (50000 hours). Which allows making a better choice of treatment for reduce the cost on blades failures repairs. The resulting microstructures were analyzed using scanning electron microscopy, optical microscopy, X-ray diffraction and Vickers hardness measurements. The results obtained show that the performance of our superalloys is due to size, shape, and distribution of the hardening phase γ' [Ni3 (Al,Ti)], then them carbides. It was found, that increasing the hardness of the Inconel 738 is related to increase of the rate of γ' secondary precipitates and the decrease of their size.

Keywords: Ni-Superalloy, aging treatment, solution treatment, rejuvenation treatment, precipitation, γ' phase, TCP phase.
First-principles study of solubility and diffusion of interstitial elements in tetragonal ferrite

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There are accurate observations which indicate the reluctance of excess carbon within bainite or martensite to partition into adjacent austenite in spite of prolonged heat treatment at temperatures where carbon atom can move. Whereas the equilibrium between ferrite and austenite in iron-carbon system is well established, that of austenite with tetragonal martensite has not been studied. To explain this, the first principle calculations have been conducted for the solubility of carbon and nitrogen in tetragonal ferrite that is in equilibrium with austenite and activation energies for diffusion in tetragonal ferrite. A combination of ab initio method and phase stability calculations proposes that the solubility is dramatically increased relative to the cubic form of ferrite. In addition, a consideration of tetragonality might form a better basis for a variety of kinetic theories on industrially important processes such as the quench and partitioning process.

Keywords: first principle calculations, tetragonality, carbon, nitrogen, steel
SESSION 14

Polymer Derived Ceramics and Composites
Radioactive waste pollution entails consequences dangerous to human health and the environment. Increasing pollution scale requires fast and effective ways to protect it, the use of advanced technological processes.

Borosilicates polymeric compounds are complex boron and silicon, generated during the exchange reaction, dehydration and polycondensation. The peculiar properties of borosilicate glass explains the change in the structural state of boron. Synthesized 5 kinds of X-ray amorphous borosilicates: $\text{Na}_2\text{B}_6\text{SiO}_{12}$, $\text{Na}_3\text{B}_5\text{O}_9\cdot3\text{SiO}_2$, $\text{Na}_6\text{B}_4\text{O}_{11}\cdot3\text{SiO}_2$, $\text{Na}_4\text{B}_6\text{O}_{11}\cdot2\text{SiO}_2$, $\text{Na}_2\text{B}_8\text{O}_{13}\cdot\text{SiO}_2$ at 1200 °C. Analysis borosilicate $\text{Na}_x\text{B}_y\text{O}_z\cdot n\text{SiO}_2$ with TSL spectra showed that it has long afterglow in comparison with the other glass and has the shape of a plateau in the temperature range of 180-230 K.

Basis on the sodium borosilicate obtained composition with sulfur polymer. Sulfur polymer is the product of the sulfur processing of sludge and waste oil refineries in Kazakhstan. Use properties of polymer sulfur to form strong composites with borosilicate. The process takes place at low temperatures (130-180°C) than getting glasses (1200-1500°C).

The compositions adhere well to the metal surface and exhibit high chemical activity, do not react with the corrosive environment, including seawater.

Obtained borosilicate compositions based on natural materials: glauconite, borate, lead dust, schungite, barite, ferrophosphorus and other waste chemical and metallurgical industries. The degree of absorption of gamma radiation such composite materials is 34% and can be offered as a container for storing solid and liquid wastes of medium and high radioactivity.
In this study the influence of different types of three-dimensional reinforcement on the mechanical properties of basalt fiber reinforced SiOC-composites was investigated. Basalt fiber reinforced, polymer derived SiOC-composites are able to bridge the gap between low temperature polymer matrix composites (PMC) and high temperature ceramic matrix composites (CMC). They combine the benefits of cheap raw materials and adapted state of the art polymer manufacturing technologies with an increased thermal and tribological stability compared to PMC. Limiting factors are the weak interlaminar strength values attributable to the pyrolysis step. In order to increase these values, a variety of 3-dimensional reinforcements were evaluated with standardized test samples to qualify the influence. Sample plates composed of basalt fabric were manufactured. In the preforming step 6 different types of thru thickness reinforcement were applied to the preform and infiltrated via RTM. After pyrolysis and subsequent PIP steps, the samples were evaluated regarding bending strength, interlaminar shear strength and orthogonal adhesion strength. The obtainable values compared to a non-reinforced composite are, in some cases, significantly higher. Additionally the influence on shrinkage, porosity and their qualification for a series production were evaluated with respect to potential and already proven applications. Keywords: interlaminar strength, ILSS, 3D-reinforcement, basalt fiber, thru thickness, SiOC composites
SESSION 15

Processing, Properties and Applications of Ceramics
Transport Properties of Aliovalent Substitution Solid Solutions of the System (1-x)PbF$_2$-xYF$_3$-SnF$_2$

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In the system (1-x) PbF$_2$ – xYF$_3$ – SnF$_2$, aliovalent substitution solid solutions of tetragonal system, isostructural $\beta$-PbSnF$_4$ compounds (space group P4/nmm), are formed in the concentration range 0 < x ≤ 0.17. The fluorine anions in the synthesized compounds, as in $\beta$-PbSnF$_4$, occupy three structurally nonequivalent positions. The mobile fluorine anions are localized mainly in interstitial positions (F$_3$). Their concentration at 300 K is over 32% and increases with rising temperature, reaching limiting values of 78 – 83% at 523 K. In the case of the aliovalent substitution of Y$^{3+}$ for Pb$^{2+}$, the number of additional anionic vacancies at 300 K is practically independent of the YF$_3$ content, and at temperatures above 430 K, it increases with YF$_3$ concentration. Fluorine ion exchange between nonequivalent subsystems increases with increasing temperature and degree of substitution, which tells on plots of electrical conductivity against temperature. For each of the compositions of the synthesized samples, a kink is observed in the temperature range 435–475 K; it is characteristic of most solid electrolytes with fluorite and antifluorite structure and may be caused by an increase in the mobility of fluorine anions on temperature rise.

At the low concentration of aliovalent cations-substituents Y$^{3+}$ (x ≤ 0.07), the conductivity of solid solutions is much lower than that of starting $\beta$-PbSnF$_4$. At larger x values, an increase in the amount of mobile fluorine ions (passage of a part of locally mobile and immobile ions to the subsystem of highly mobile ones) is observed. The sample of the composition Pb$_{0.91}$Y$_{0.09}$SnF$_{4.09}$ has much the same conductivity values as the phase of stoichiometric composition, $\beta$ – PbSnF$_4$, in the high-temperature region. The samples containing 13.0 mol% YF$_3$ have the highest anion mobility.

At temperatures below 300 K, the conductivity of the synthesized compounds is practically independent of the concentration of the aliovalent substituent. Only at temperatures of over 400 K in samples containing over 7.0 – 9.0 mol% yttrium trifluoride, the electrical conductivity increases with aliovalent substituent concentration. No contribution of the surface conductivity of the crystallites of the synthesized compounds to their total bulk conductivity has been detected.

The transport numbers of fluorine anions in the entire concentration range of the synthesized compounds are close to unity and are practically independent of the YF$_3$ concentration. The electronic component of conductivity is two orders of magnitude lower than the ionic one and is not over 2.6 · 10$^{-4}$ S/cm at 453 K.

The results of impedance and $^{19}$F NMR spectroscopy suggest that the high conductivity of samples (∼ 10$^{-4}$ S/cm at 300 K) is due to the translational diffusion of fluorine ions in their anion sublattice.

Keywords: solid solutions, lead, tin, yttrium fluorides, aliovalent substitution, conductivity, ion mobility, transport numbers.
Synthesis for Pr\textsuperscript{3+} doped CaTiO\textsubscript{3} Spherical Particle using Water-Soluble Ti Complex

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Titanium reagents commonly used for liquid phase synthesis are susceptible to hydrolysis. Recently, selective preparation for polymorphism of TiO\textsubscript{2} by hydrothermal method using water-soluble Ti complex were reported. The author concluded that the structural similarity between the dissolved titanium complex molecules and the nuclei of TiO\textsubscript{2} phase is important. These interesting findings motivated us to investigate the structural effects of Ti complex to Pr\textsuperscript{3+} doped CaTiO\textsubscript{3} prepared using the water-soluble Ti complex via hydrothermal reaction. In this paper, we prepared Pr\textsuperscript{3+} doped CaTiO\textsubscript{3} from precursor solution consisted of water-soluble titanium complex, CaCl\textsubscript{2}, and Pr(NO\textsubscript{3})\textsubscript{3}·6H\textsubscript{2}O under hydrothermal condition and investigated effects of ligands to chemical composition, morphology, and emission behavior of the powder obtained.

The precipitate obtained by hydrothermal reaction at 423K for 24h were calcined at 923K for 1h in air after drying at 358K for 18h. Glycolic, citric, and lactic acids were used for ligand of water-soluble titanium complex. X-ray diffraction (XRD), scanning electron microscopy (SEM) and photoluminescence spectroscopy (PL) were employed to investigate crystal structure, surface morphology and fluorescence intensity of samples.

On the case of citric acid ligand, perovskite phase was observed in XRD pattern. However, the phase was invisible in the powder obtained from glycolic and lactic acid precursor solutions without increasing pH value using NH\textsubscript{3}. Therefore, we focused on citric acid precursor solution. PL intensities of samples increased with fraction of Pr\textsuperscript{3+} doped perovskite phase. SEM analysis revealed that spherical particles with a diameter of 2\textmu m were produced with progress of hydrothermal reaction. EDS analysis revealed that chemical compositions of these particles were varied with citric acid concentration in the precursor solution. These findings indicated that the structure of Ti complex have an effect on the emission behavior of our samples.

\textit{Keywords:} water-soluble Ti complex, hydrothermal reaction, perovskite, red phosphor, spherical particle
Optical Properties of Polycrystalline Alumina and Ruby
Fabricated by Pulsed Electric Current Sintering with Pre-Powder Treatments

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Alumina (Al₂O₃) is widely used in industries because of its excellent combination of properties. It has high hardness, high thermal conductivity, good abrasive resistance, chemical inertness and optical transparency. Single crystal alumina, commonly called sapphire, has been used in many optical applications. With the additive of Cr³⁺, ruby can be formed with beautifully red color. Single crystal ruby has been used as a solid-state laser host material, especially in holographers.

Recently, transparent polycrystalline alumina and ruby have been successfully fabricated by various sintering techniques¹(1)-(5). However, because of the influences of grain boundaries as well as residual pores, the transparency of polycrystalline alumina and ruby is still lower than single crystal ones. Improving the transparency of them is important for many applications.

By two-step pulsed electric current sintering, highly transparent polycrystalline alumina and ruby were obtained with nearly 100% of relative density⁴(4),(5). Even though, microscopic pores were observed as black dots inside transparent alumina by optical microscope and scanning electron microscope. Those pores derived from hard agglomerates in starting powder. By various chemical treatments to break agglomerates inside powders, the transparency of polycrystalline alumina was improved⁶(6).

In this study, the starting Al₂O₃ and ruby powder were treated with polyethylene glycol in order to deagglomeration in these powders before sintering. The influences of various powder treatments on the density of black dots within transparent polycrystalline alumina, the optical transmittance of alumina and ruby, and the photo-luminescence of polycrystalline ruby were evaluated.

**Keywords:** transparent polycrystalline alumina, polycrystalline ruby, agglomeration, pulsed electric current sintering

References

Evolution of A.C. conductivity of WET illitic clay during drying

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The evolution of the electrical conductivity during drying as well as the relationship between sample volume and moisture of a green sample were investigated. The samples were prepared from illitic clay (80 wt.% illite, 4 wt.% montmorillonite, 12 wt.% quartz and 4 wt.% of K-feldspar) and distilled water with initial moisture content 35% and were freely dried on air. Conductivity was measured by the volt-ampere method with AC power supply 5 V in the frequency range of 50 Hz to 10 kHz. The samples exhibited good conductivity and their volume decreased when the moisture was higher than 8 wt.%. Below this value, the electrical resistivity steeply increases and the sample’s dimensions do not significantly change. That means, that the evaporation of the free water molecules from pores is over. The dependence of the moisture on relative volume change is presented in a form of the Bigot’s curve.

Keywords: Illite, Conductivity, Bigot’s curve, Drying
Important high value adding applications of modern structural ceramics are in the field of tools and dies in manufacturing engineering. Processing of highly abrasive materials in powder injection molding or extrusion requires mold materials with high wear resistance to increase the durability of the tools and to sustain a high quality and precision of the manufactured products. High performance ceramics which exhibit high hardness, bending strength and toughness feature the perfect combination of properties for these applications. Their drawback is, that they cannot be economically customized in complex shapes and small lot sizes, as they are required in tool and mold design. Recent development of electrically conductive oxide ceramics enabled the use of EDM, the most efficient process for machining of hard materials, as alternative to conventional ceramic manufacturing technologies. Combining the shaping and final machining of ceramics by EDM in one process step, complex shaped assemblies with fine structures, small tolerances and the benefits of ceramic material properties can produced. The focus is on ZTA based ceramics with addition of titanium carbide that can be machined by wire EDM and die sinking. Mechanical and electrical properties of the materials as well as the characteristics of the machining process and its influence on the workpiece material are analyzed. Additionally the feasibility of the ceramic material for tool inserts is shown by real wear tests in extrusion dies.

**Keywords:** EDM, electrically conductive ceramics, wear resistance
Influence of the granulation processing on the compaction behavior and properties of alumina granules

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The manufacturing of technical ceramic materials often requires the use of very fine powders whose particle size distribution is less than one micrometer. This low particle size is necessary to enhance the surface reactivity of particles during the sintering. Indeed, a fine microstructure (small grains and reduced pore volume fraction) is required to favor high performances for structural applications. Concerning the shaping of ceramics, one of the most widespread methods is uni-axial pressing because of its simplicity to carry out. However, a suitable rheological behavior of powders is required to facilitate the filling of the die. That is why a step of granulation of the powder with addition of a small amount of organic additives is necessary.

In this work, different aqueous suspensions of alumina have been prepared in order to form granules by two different ways: (i) with a classical heat flow granulator for which the humidity is removed by a hot airflow and (ii) with the freeze-granulator processing in which the water goes out of the granules by sublimation. The characterization of granules and powder compacts (density, pore volume fraction by mercury intrusion porosimetry, green mechanical strength determined by indirect tensile test (Brazilian test) and compaction behavior) has been investigated for different pressures of compaction.

Results have shown that the microstructure of individual granules is very sensitive to the way of granulation. Moreover, compaction ability and mechanical strength of powder compacts are significantly different.

A realistic numerical simulation of the compaction behavior of granules taking into account elasto-plastic law of behavior has also been carried out.

**Keywords:** granulation, powder compaction, porosity, numerical simulation
Contribution to the understanding of the tribochemical damage process of Si₃N₄/graphene composites worn in different ball-on-disc tribosystems

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Silicon nitride ceramics are excellent structural ceramics used widely in wear resistant applications. The improvement of frictional properties is of great interest to increase the lifetime and to decrease costs due to wear initiated failures. The low friction behaviour of the engineering ceramics is well known, in general. The contribution from adhesion and deformation is characteristically low furthermore tribofilm formation in presence of water for Si₃N₄ and SiC ceramics is also known. The tribofilm can provide lubrication lowering the friction and decreasing the wear rate even by orders of magnitude. The resultant friction behaviour and degree of the wear damage is highly dependent on the kinetics of the competing processes, i.e. the rate of the film formation and detachment of tribofilm that is influenced by the critical film thickness. The understanding of the real and complex tribological processes is indispensable for appropriate modelling the wear damage, necessary for reliable life assessment of these types of ceramics. The existing models range from the initial phenomenological descriptions up to the latest nano-scale friction models giving an atomic interpretation of the tribochemistry of the processes responsible for the tribochemical degradation.

Recently a systematic comparative ball on disc wear test series has been accomplished on multilayer graphene (MLG) reinforced silicon nitride composites produced by hot isostatic pressing and spark plasma sintering to investigate the effect of the MLG addition, processing method and counterpart material (Si₃N₄ and SiC) on the tribo-corrosion process of these silicon nitride ceramics. It was established that SPS can improve the wear resistance of Si₃N₄ ceramics, but the achievable improvement was strongly affected by the MLG content and the applied counterpart material [1].

The paper aims at providing further analysis of the formerly published results and contributing to the understanding of the physical background of the experienced differences for the investigated tribosystems. Since the best combination of the tested systems represented by those pairing the SPS sintered Si₃N₄ disc with MLG addition sliding against SiC counterpart (see k=1.39E-05 in Fig.) the focus of the analysis has been put on the discussion of the different nature of triboemission of the silicon nitride and silicon carbide ceramics, furthermore on the analysis of the different physicochemical behaviour of these ceramics. The obtained results and the physical explanation of the damage process based on a microscopic model of the wear mechanism can serve as useful data for optimization of the wear behaviour of silicon nitride friction couples. Beside the advantage of a significant decrease of the wear rate provided by the best tribosystems, the emphasis is also put on the benefits providing by the cheaper and environmentally friendly alternative sintering technology, i.e. spark plasma sintering.

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The effect of the addition of ground olive stones on the physical and mechanical properties of clay bricks

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This study deals with the effect of ground olive stones (GOS) on the performance of fired clay bricks. Seven different clay-GOS mixes with 0, 1, 2, 3, 4, 5 and 10 wt % of GOS respectively were used for making fired brick samples. All samples were fired at 900 °C. The technological properties of the resultant material were then determined, including shrinkage, apparent density, pore size distribution, thermal conductivity, water absorption, and compressive and flexural strength. The addition of GOS to the mixture reduced the compressive strength of fired clay bricks. All clay brick pieces exhibited low firing shrinkage. It was apparent that as the percentage of GOS increased in the body, there was a noticeable increase in porosity. The water absorption coefficient decreased with increasing additions. The results indicated that thermal conductivity decreases with decrease in density and increase in porosity in fired clay bricks.

Keywords: Brick; Organic material; Temperature; Compressive strength; Thermal Analysis
Low loss and Large Electrostrain in CuO-added (1-x)KNbO₃-xBaZrO₃ Lead-free Piezoelectric Ceramics

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The CuO-added KNbO₃ (CKN) ceramics exhibited the double polarization versus electric field (P-E) hysteresis loop and the sprout-shaped strain versus electric field (S-E) curve. These CKN ceramics showed a large coercive field (E_c) of 3.5kV/mm, P-E, and S-E curves, indicating that it is difficult to use these ceramics for actuator devices. The (1-x)CKN-xBaZrO₃ [(1-x)CKN-xBZ] ceramics with 0.00 ≤ x ≤ 0.07 sintered at 960°C also showed the sprout shaped S-E curve and the double P-E hysteresis loops without aging process. The formations of these S-E and P-E loops were explained by the presence of defect polarization formed between the Cu²⁺ ion in Nb⁵⁺ site and the oxygen vacancy. These ceramics showed a large strain of 0.11% - 0.12% at 6.0 kV/mm and they exhibited a small S-E, P-E curves, and E_c of 1.5 kV, indicating that these ceramics have a low electric and electromechanical loss. Moreover, these ceramics maintained the high strain of 0.12% after 10⁶ cycles with the applied electric field of 3kV/mm and thus these ceramics also have a good fatigue properties. In this work, the detailed explanations on the low loss and small S-E and P-E curves of (1-x)CKN-xBZ will be discussed.

Keywords: Lead-free, Piezoelectric ceramics, Actuators
Synthesis of [001]-Oriented PbTiO$_3$ Platelets by Topochemical Microcrystal Conversion Method using PbBi$_4$Ti$_4$O$_{15}$ Precursor

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In this study, PbTiO$_3$ (PT) platelets were successfully synthesized through molten salt and topochemical microcrystal conversion (TMC) methods. First, PbBi$_4$Ti$_4$O$_{15}$ (PBT) platelets having Aurivillius structure were prepared by the molten salt method at 1080°C for the precursor of PT platelet. The average size of PBT platelet was 3 ~ 5 μm with the thickness of 0.5 μm. PT platelets were synthesized at 1010°C by replacing the Bi ions in PBT precursor with the Pb ions through the TMC reaction. The [001]-oriented PT platelets with the average size of 3 ~ 5 μm and the thickness of 0.2 μm were obtained. However, the PT platelet has a rough surface with irregular shape. It is considered that the surface roughness and morphology of the PT platelets were determined during the exfoliation of [Bi$_2$O$_2$]$^{2+}$ layers from the PBT platelet. Therefore, it is important to control the exfoliation process to synthesize the PT platelets with a smooth surface and a high aspect ratio. In this work, exfoliation process of the bismuth oxide sublayers was systematically investigated by controlling the amount of PbO. Moreover, the synthesis processes of Pb(Zr,Ti)O$_3$ platelets will be also presented. The PT and Pb(Zr,Ti)O$_3$ platelets can be used for the reactive templated grain growth of the PZT-related ceramics.

**Keywords:** platelet, topochemical microcrystal conversion, molten salt, reactive templated grain growth, aurivillius structure, perovskite structure.
Synthesis and Ferroelectric Properties of KNbO$_3$ Thin Film Grown on a Pt/Ti/SiO$_2$/Si Substrate using the RF Magnetron Sputtering Method

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A homogeneous KNbO$_3$ (KN) thin film was grown on a Pt/Ti/SiO$_2$/Si substrate using the RF sputtering method. A K$_{2.88}$Nb$_5$O$_{15}$ secondary phase was formed in the KN film when the deposition and the annealing temperatures were greater than room temperature (RT) and 800°C, respectively, owing to the evaporation of K$_2$O. On the other hand, KNb$_3$O$_8$ and K$_3$Nb$_{5.45}$O$_{15}$ secondary phases were formed in the KN films when the annealing temperature was less than 800°C or the annealing time at 800°C was shorter than 90 min. A homogeneous KN thin film was formed when it was deposited at RT and subsequently annealed at 800°C for 90 min under the K$_2$O atmosphere. This KN film exhibits a relative permittivity of 884 with a dissipation factor of 6.71% at 100 kHz. The leakage current density of 1.06 x 10$^{-6}$ A/cm$^2$ at 0.1 MV/cm and a breakdown field of 1.5 MV/cm were observed from this film. This film showed a saturation polarization of 21.9 µC/cm$^2$, with a remnant polarization of 8.3 µC/cm$^2$, and a piezoelectric strain constant of 125 pm/V.

Keywords: Ferroelectric, Lead-Free, Piezoelectric, Sputtering, Thin Film
The \( \text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3\)-\( \text{Pb}[(\text{Zn}_{1-y}\text{Ni}_y)_{1/3}\text{Nb}_{2/3}]\text{O}_3 \) (PZT-PZNN) ceramics have been textured using [100]-oriented \( \text{BaTiO}_3 \) (BT) platelets, which were synthesized by topochemical microcrystal conversion method. The sintering temperature of the PZT-PZNN ceramics was approximately 1100°C but when the BT platelets were used as the seed for the templated grain growth (TGG), the PZT-PZNN ceramics were well-sintered even at 900°C. The lotgering factors (LF) of the BT-added PZT-PZNN ceramic sintered at 1100°C was approximately 66%, indicating that this ceramic was not well textured. On the other hand, the BT-added PZT-PZNN ceramic sintered 950°C showed a large LF of 95%, indicating that this ceramic was well textured. Therefore, the sintering temperature of the PZT-PZNN ceramics should be decreased for the TGG. The PZT-PZNN ceramic without the TGG showed the strain (0.176 %) at 3.0 kV/mm. However, the textured PZT-PZNN ceramic exhibited the increased strain of 0.276 % at 3.0 kV/mm. In this work, the TGG processes and the strain enhancement will be systematically investigated.

**Keywords:** Ceramics, Piezoelectric, Texturing, Single-Crystal, Actuator
Cu-doped nanostructured ZnO coatings: Effect of Cu-doping on band gap energy and photocatalytic activity

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ZnO is widely studied semiconductor material with interesting properties such as photocatalytic activity and wide range of applications, for example in the field of opto-electronics and self-cleaning and antimicrobial applications. Doping of photocatalytic semiconductor materials has been shown to introduce variation in the band gap energy of the material and thus it could improve photocatalytic activity in visible light.

In this work, Cu-doped ZnO nanorods were grown on a stainless steel substrates using hydrothermal method. Zinc nitrate and hexamethylenetetramine were used as precursor materials and the growth was conducted at 80 °C for 2 h in order to achieve a well-aligned evenly distributed nanorod structure. Copper was introduced as copper nitrate that was added in the precursor solution in the beginning of the growth. The as-prepared films were then heat-treated at 350 °C and band gap measurements were performed for the undoped and doped films. It was found that increase in the copper concentration in the precursor solution decreased the band gap of the ZnO coating. Methylene blue discoloration tests were then performed in order to study the effect of the Cu-doping on photocatalytic activity of the nanostructured ZnO films.

Keywords: Zinc oxide, copper doping, hydrothermal synthesis, band gap, photoactivity
Ceramic-like open-celled geopolymer foam as a porous substrate for water treatment catalyst

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This paper presents results from experimental study on microstructural and mechanical properties of geopolymer-based foam filters. The process for making porous ceramic-like geopolymer body was experimentally established, consists of (a) geopolymer paste synthesis, (b) ceramic filler incorporation, (c) coating of open-celled polyurethane foam with geopolymer mixture, (d) rapid setting procedure, (e) thermal treatment. Geopolymer paste was based on potassium silicate solution $n(\text{SiO}_2)/n(\text{K}_2\text{O})=1.6$ and powder mixture of blast-furnace slag and calcined kaolin. Various types of ceramic granular filler (alumina, calcined schistous clay and cordierite) were tested in relation to aggregate gradation design and particle size distribution. The small amplitude oscillatory rheometry in strain controlled regime 0.01% with radial velocity 1 rad/s was applied for determination of rheology behavior of prepared mixtures. Thermal treatment conditions were applied in the temperature range 1100 – 1300 °C.

The developed porous ceramic-like foam effectively served as a substrate for highly active nanoparticles of selected Fe$^{+2}$ spinels. Such new-type of nanocomposite was tested as a heterogenous catalyst for technological process of advanced oxidative degradation of resistive antibiotics occurring in waste waters.

Keywords: geopolymer foam, open-celled, catalyst

Macro/microstructural characterization of open-celled geopolymer foam.
Properties of Oxide-Hydroxide Sintered Ceramics

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The development of material based on oxides is a fundamental challenge to modern materials which used for manufacturing of filters, catalyst supports, membranes, etc. Porous oxide ceramics are also of interest for usage in medicine as its structure is close to the natural bone. The traditional method for obtain a highly porous ceramics based on “burn-pore-forming” additives; however using this route a carbon impurities formed on the pore internal surfaces. To form a multi-level pore structure without addition of pore-former one can use the method using decomposition of hydroxides to oxides during sintering which accompanied by the release of gas and, as a consequence, the formation of porosity.

The aim of this work is to study the formation of porous structures in ceramics synthesized from aluminum hydroxide during in the sintering process decomposition of hydroxide to the oxide. Aluminum hydroxide with gibbsite modification Al2O3*4H2O obtained by the decomposition of the aluminates solution was used as a starting component. Cylindrical form samples were pressed at 20 kN load, sintered in air at 1,300, 1,400 and 1,500 °C, with isothermal one hour holding. X-ray diffraction studies were carried out on X-ray diffractometer with filtered CuKα radiation. Mechanical properties of the samples were obtained by compression tests.

The method of forming pores, the pore-forming burnout comparisons decomposition during sintering, allows obtaining the same porosity as in the “burn-pore-forming” additives about 15-65%. Porosity decreases with increase of the sintering temperature. The pore size decreases from 1.9 μm to 0.9 μm and size dispersion decreases from 3.1 μm to 0.6 μm. It has been shown that the compressive strength increases with increasing sintering temperature, minimum compression strength is 20 MPa for sintering temperature 1300°C and the maximum 800 MPa for 1500°C besides with decreasing pore volume from 65 to 15%.

Based on these results, one can conclude that the aluminum hydroxide can be used as pore formation material for sintering ceramics with controlled porosity. The obtained alumina structure is very close to inorganic “burn-pore-forming” additives and is promising material for bone implants.
Recent experiments on polycrystalline materials show that microcrystalline materials have a strong dependency to grain size. In this study, mechanical and electrical properties of polycrystalline materials in micro level were studied by using averaging theorems. To completely understand the size-dependency of polycrystalline materials, an integral non-local approach was presented that can predict the experimentally observed stress-strain relations for these materials. In microcrystalline materials, crystalline and grain-boundary were considered as two separate phases. The mechanical properties of the crystalline phase were modeled using crystalline brittle material and is composed of randomly distributed and orientated single crystal anisotropic elastic grains. For microcrystalline materials, the surface-to-volume ratio of the grain boundaries is low enough to ignore its contribution to the elastic deformation. Therefore, the grain boundary phase was not considered in microcrystalline materials and the mechanical properties of the crystalline phase were modeled using an appropriate an integral non-local approach. Finally, the constitutive equations for polycrystalline materials were implemented into a boundary integral equation and the results obtained from the proposed constitutive equations were compared with the experimental data for piezoelectric ceramic.

**Keywords:** boundary element method, crack, crack propagation, multi-scale modelling

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Study of Zn$_{1-x}$Mg$_x$O materials for UV diode laser application

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Our work aims to investigate the insulator conductor oxide (ITO) compounds Zn$_{1-x}$Mg$_x$O which includes two structures ZnO and MgO. Where x representing the concentration of the element Mg substituted for the element Zn in the ZnO matrix. The Zn$_{1-x}$Mg$_x$O compounds have a gap covering a wide spectral range from 3.4 to 6.6 eV. They are cheap compared to GaN compound for use in optoelectronics as lasers diodes. The doping n-type and p-type for ZnO are now possible. Moreover Zn$_{1-x}$Mg$_x$O has a refractive index that varies according to the proportion x of Mg substituted for Zn. Consequently a good optical confinement is achieved for the Zn$_{1-x}$Mg$_x$O / ZnO diode laser.

In this work we highlight the interesting properties of the junction ZnO (+) / ZnO (-) on the substrate Zn$_{1-x}$Mg$_x$O depending on x. The functionality of laser diode emission will depend on these properties. Using the rate equations we simulate the diode laser ZnO Zn$_{1-x}$Mg$_x$O for different proportion of Mg into ZnO matrix.

**Keywords:** conductor oxides, element Mg, emission, laser diode, rate equations, refractive index, ZnO matrix
Synthesis of kaolinite–methanol intercalation complexes using cost-efficient homogenization and solvothermal methods

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From the point of view of the potential industrial use of kaolinite intercalation complexes, cost-efficiency, low reaction time and reagent consumption are important factors. In order to minimize costs, reaction time and reagent consumption, we have performed systematic experiments to analyze the effect of reaction parameters (the temperature and time, the amount of reagent) on the intercalation using homogenization and solvothermal methods. The influence of reaction parameters was characterized by X-ray diffraction. Cost-efficient homogenization method was applied to synthesize the kaolinite–urea pre-intercalation complex of the kaolinite–methanol one. The tested homogenization method required an order of magnitude lower amount of reagents than the generally applied solution method. To synthesize kaolinite–methanol complexes solvothermal methods were applied, which required less time and liquid methanol than the generally used multiple washing-and-centrifugation treatment at room temperature. Our optimized solvothermal method proved simple, effective and cost-efficient (an order of magnitude lower amount of methanol was required) due to lack of shortcomings of the traditional multiple treatment. Furthermore, our optimized method resulted in a practically complete replacement of pre-intercalate reagent (urea) by methanol forming the 1.12-nm kaolinite-methanol complex.
Manufacture of Industrial Waste Based Alkali Activated Cement Using Mechanochemical Activation

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Alkali activated cements play a unique role as a successful substitute of ordinary portland cements due to their superior durability and eco-friendliness. Industrial wastes as granulated blast furnace slag and fly ash proved to be valuable source material for manufacturing alkali activated cements, but especially slag is an equally important component for cement industry. In present study a new industrial waste source (crystallized slag) was investigated with very similar composition to granulated blast furnace slag.

Research focused on the activation of crystallized slag for the purpose of producing a raw material suitable for binder manufacture. Activation was realized by mechanochemical activation, which means short and intensive dry grinding by planetary ball mill. The grinding parameters (grinding time, rotation speed, and sample to grinding body mass ratio) were altered during the experiment. The amount of amorphous phase, which may have a direct effect on reactivity, of differently treated samples was determined using X-ray diffraction. The compressive strength of binder mortars derived from activated crystallized slag was measured at 7 days of age and compared to alkali activated binders derived from granulated slag.

Results indicate that mechanochemical activation of crystallized slag can be a new and valuable method to manufacture alkali activated cements.

Keywords: Alkali activated cement, X-ray diffraction, Compressive strength, Mechanochemical activation
Development high-tech ceramic composites based on $\text{Al}_2\text{O}_3$, $\text{SiO}_2$ and IG-017 additive

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Based on high purity alumina and quartz powders and IG-017 biooriginal additives of Igrex Ltd. the authors have developed new ceramic composite materials for different industrial purposes. The main goal was to fine a material and morphological structures of high performance ceramic composites as frames for development complex materials for extrem consumptions. For this the mixt powders of $\text{Al}_2\text{O}_3$, $\text{SiO}_2$ and IG-017 were uniaxially pressd at different compaction pressures into disc sapes and were sintered in electric kiln under the following 3 conditions:

- oxidation atmosphere,
- reduction atmosphere,
- reactive atmosphere.

The grain size distribution of the raw materials were determined by laser granulometry, the mineralogical composition by X-ray diffractometry. There thermo-physical properties were also determined by derivatography.

The prepared and sintered specimens were tested on geometrical sizes, material composition (X-ray diffraction), microstructure and morphology by scanning electron microscopy, porosity and mechanical strength. In this work the authors present the results of their research and investigation.

**Keywords:** additives, alumina, biooriginal, ceramics, composite, materials, morphology, quartz, thermoanalysis, X-ray diffraction
SESSION 16

Testing and Characterization of Materials - Methods, Equipment and Errors
A Study On The Development And Evaluation Of Ritonavir - Albumin Nanospheres

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The objective of the present study was to prepare and characterize the ritonavir-albumin nanospheres. The nanospheres were made by desolvation technique. In this method proportion of drug and carrier ratios with varied by altering pH medium. Then they were cross-linked with glutaraldehyde. The FTIR spectra and DSC thermal studies indicated that there was no chemical interaction between drug and the BSA. The nanocarriers were in spherical in shape with an average diameter of 365.5 nm. The entrapment efficiency of the nanocarriers increases on increasing the concentration of the BSA up to a particular concentration. The in vitro release data of all the NC’s were fitted with various mathematical models to establish the drug release mechanism from the NC’s and was found to be non-Fickian diffusion with n >0.5. The nanocarriers were stable and appreciable difference was not observed during 90 days stored at 25°C.

**Keywords:** Ritonavir, bovine serum albumin, modified desolvation, nano carriers, mechanism.
A Study On The Development And Characterization of Antiviral Nanoparticles With Albumin

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The objective of the work took up was to characterize the nanoparticles made with an antiviral, lamivudine and the carrier albumin. The FTIR studies revealed no drug-carrier incompatibility. The yield of the formulation ranged from 59.6-77.3 and loading capacity was 19.8-32.7. The ratio of drug to carrier played significant impact on the release of the drug from the nanoparticles. The in vitro release data of all the NP’s were fitted with various mathematical models to establish the drug release followed first order kinetics. The nanoparticles obtained were spherical in shape and gave zeta potential -21.8mv with average particle size of 283.5nm and average PDI of 0.245. The nanoparticles were stable and appreciable difference was not observed during 90 days stored at 25°C.

Keywords: Lamivudine, bovine serum albumin, modified desolvation, drug release, stability studies.
Investigation of thermal properties of raw materials of asphalt mixtures

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Asphalt mixtures are composite materials, which are made from different grades of mineral aggregates and bitumen. During the mixing process mineral materials blended with bitumen at relatively high temperature (~200 °C). As the binding process come off in these higher temperature range, therefore thermal properties of asphaltic materials are important. The aim of this project is to reveal the thermal properties of raw materials. During our research two types of mineral aggregates were tested (limestone and dolomite) by different methods. Differential thermal analysis, thermal expansion and thermal conductivity were investigated on technologically important temperatures. The results showed that the structure of mineral materials was not changed at elevated temperatures, expansion of samples was negligible, while thermal conductivity was changed by temperature.

Keywords: asphalt, mineral, thermal analysis, thermal conductivity, thermal expansion
Efficiency Of Surface Treatments Between Dies And Forging Steels By Measuring Coefficient Of Friction

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The seizure sometime occurs between dies and forging steels in hot forging process. The seizure is a serious problem to decrease productivity. Many studies on die-materials and lubrications to prevent the seizure are reported. However, the seizure mechanism in die and forging steels was not understood well. Surface treatments are often applied to die to prevent seizure and to increase the life time. These are utilized for prolong die life because of modifying surface condition e.g. hardening, changing surface layer to lubricant one, etc. It is necessary to consider the influence of surface treatments on the seizure mechanism.

The objective of this study is to apply friction test as evaluation methods of frictional behavior using the surface-treated die metals by measuring coefficient of friction. Friction tests were conducted by a pin-on-disc test equipment. From these experiments, coefficients of friction between surface-treated (Heat treated, Nitrided and Sulphonitrided) alloy tool steel (chemical composition C:0.37 Si:0.93 Mn:0.46 P:0.020 S:0.020 Cr:5.22 Mo:1.21 V:0.80 Fe:Bal (in mass%) (SKD61 in JIS) and carbon steel (C:0.55 Si:0.35 Mn:0.75 P:0.020 S:0.020 Fe:Bal) (S55C in JIS) were measured at room temperature.

From these experiments, coefficients of friction gradually decreased in order to heat treated, nitride, sulphonitrided surface treatments. These results corresponded with present common sense of surface treatment.

This research is partly supported by SIP(Cross-miniserial Strategic Innovation Promotion Program).

Keywords: Heat treated, Nitrided, Nitriding Sulphonitrided, Sulfonitriding, Die, Forging, Seizure, Alloy tool steel, Carbon steel, Friction coefficient, Pin-on-disc test
Plastic deformation effect of the corrosion resistance in case of austenitic stainless steel

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The corrosion forms are different in case of the austenitic steel than in case of carbon steels. Corrosion is very dangerous process, because that corrosion form is the intercrystallin corrosion. The austenitic stainless steel shows high corrosion resistance level. It knows that the plastic deformation and the heat treating decrease it's resistance. The corrosion form in case of this steel is very special and the corrosion tests are difficult.

We tested the selected steel about its corrosion behaviour after high rate deformation. We wanted to find a relationship between the corrosion resistance decreasing and the rate of the plastic deformation.

We wanted to show this behaviour from mechanical and electrical properties changing.
Determination of pressure dependency of shear viscosity of polyethylene terephthalate melt

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Pressure sensitivity of polymer melt is a very important parameter in polymer processing. There are several simulation software using this values. Polyethylene terephthalate (PET) is a widely used polymeric material in packaging. Reuse and reprocessing of the collected material is in the focus of researchers. Our investigation is aimed on the determination of the pressure on the viscosity of PET.

Original and regrind PET were used. Rheological behaviour of the melts were determined at different pressures (50 bar to 1000 bar) and temperatures (260 oC - 300 oC). Capillary rheometer was used to determine the flow curves of the materials.
Method development and validation for the quantitative application of mid-range infrared spectroscopy in GLP environment

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The main analytical role of the mid-range infrared spectroscopy was the structural investigation of organic compounds for decades until the cheap, versatile and easily interpretable mass spectroscopic methods slowly occupied the area. Nowadays new perspectives are open for the MIR users due to the multiplied computing capacity of the personal computers. It does not only mean stronger hardware but the development of chemometric data evaluation methods and softwares. Nevertheless thanks to the new sample introduction techniques that provide good repeatability with minor or no sample pretreatment. The significantly quicker method development, the shorter analysis time, the smaller cost of chemicals and labor-intensive work phases compared to the conventional chromatographic methods are the other reasons of this “renaissance”.

The laboratories operating in GLP/GMP environment must fulfill rigorous requirements. The analytical performance must be demonstrated for any methods applied in quantitative determinations. Depending on the application field different validation procedures exist (such as FDA or ICH1 guidelines) but they are almost exclusively set up for univariate - mainly chromatographic - methods. As probably the only exemption, there exist methodology for the validation of near infrared systems2, but due to the different nature of NIR and MIR not all the parameters correspond to each other.

In our presentation the development and the validation of a Fourier-transform mid-range infrared method will be demonstrated with special focus on the assay specificity parameter.
On specifics of rheological measurements for heavy highly-viscous oil

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Various heavy and high-viscous oils are neither fluid nor solid and their rheological behavior strongly depends on external conditions. Their inner structure determines pertinent physical properties and can be affected by measurements in shear stress regime common for oil industry. Hence, the results mayn't be accurate, can vary even within one's lab and depend strongly on sample preparation and measurement protocol. All these factors complicate comparison of rheological results for such oils if experimental protocol isn't properly documented. To compare results, we simultaneously performed shear-stress and dynamic measurements for crude heavy oil (0.965 g/cm3; concentration of parafin, resins, asphaltenes = 1.1, 31.1, 9.9 mas%, respectively). Experiments were performed at 10-70, with cone/plate configuration of rheometer (shear-stress regime with 3.0 and 300.0 1/s of shear rate. Yield point, indicating an inner structure, decreases from 1905.0 (at 10 C)to 753.5 Pa (30 C) and disappears at higher temperatures. Viscosity values at high shear rate show untypical pattern, being lower at low temperatures, the maximum occurs at 40 C and even at 70 C viscosity is higher rhan at 10 C.

The phenomenon is presumed to be due to phase separation under high mechanical stresses, since it completely disappears at low low shear rate and in oscillatory-mode experiments. Besides, viscosity values at 3.0 and 300.0 1/s below 40 C differ greatly, up to 3-4 orders, and become comparable only at 60-70 C. Meanwhile, viscosity data from oscillation experiments are quite similar to those at low shear rate: e.g., 115.08 and 159.95 Pa.s at 10 C, respectively (i.e., differ by a factor of 1.4); 0.652 and 0.681 Pa.s at 70 C (the difference within measurement error). The results illustrate dependence of rheological data on experimental approach.

We believe that dynamic measurements are preferable for studying heavy highly-viscous oils, though may require more efforts for getting and interpreting the data.
Effects of post-discharge nitriding on structural and corrosion properties of 4140 low-alloy steel

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The nitriding effects of 4140 low-alloy steel were investigated by exposing it to a post-discharge nitriding process at a temperature of 810 K and holding times of 20, 40 and 60 minutes. The effects of holding time on the microstructure was investigated employing scanning electron microscopy, high resolution transmission electron microscopy, X-ray diffraction, corrosion test and hardness measurements. The results showed that as the nitriding time is increased, the microstructure of the layer changes from a dual phase (Fe₂₃N and Fe₄N) to a single phase (Fe₄N). Experimental results of the nitrided samples surface composed of Fe₄N single phase shown improved corrosion resistance.

Keywords: Post-discharge nitriding; Microhardness, Potentiodynamic Test; Corrosion Resistance
SESSION 17

Mechanical Properties and Processing Technology of Advanced Materials
The designed three dimensional complex shapes could've be machined on the insulating materials with electrical discharge machining method about twenty years ago. This machining method was proposed by authors research group. It was named as the assisting electrode method by authors group. In this method, the electrical conductive material was attached on the surface of workpiece before machining. The discharge started from the attached material, and continued to the workpiece contact surface. As the discharge occurred in the working oil, the dissolute carbon was generated on the workpiece surface, the electrical conductive layer was attached on the workpiece surface which was composed by the carbonized material. On the suitable discharge conditions, the later could've be attached after the machining of attached surface material which I was act as role of earth. Many insulating ceramics materials such as Al2O3,ZrO2, Si3N4, SiC, AlN, Sapphire,and diamond could' be machined. The machining priorities of machining speed,surface roughness etc. was observed. The machining speed was not enough to use as the actual method. In this paper, to obtain the better machining properties, the new compound machining system was designed with the ultra sonic machining method. The following experimental factors were examined, (1)vibration frequency,(2)amplitude,(3)way of vibration. The effects of added vibration were confirmed on the machining speed and surface conditions. The action of discharged debris and the oil was examined with the designed system. The exhausting phenomena of discharge debris on the machining atmosphere was observed.
Metallic foil stacked on adhesive tape [1] is used in the housing of the electronic equipment for electromagnetic cut-off shield and static electricity protection purpose. A wedge indentation processing is widely used for cutting off a sheet material into a complicated formed pattern such as carton boxes, labels, insulation films and similar metal thin sheets [2]. The wedge cutting has a moderate accuracy and productivity for stripping various complicated patterns. When the wedge cutting is applied to the copper foil stacked on adhesive tape, trouble sometimes occurs due to the air inflow in detached layer, peeling of the cut end-surface and the glue flow out. The characteristics of trouble phenomena have not been clarified sufficiently [3], while actual troubles are empirically taken by the hand operation of experts.

In order to predict the cutting profile of copper foil stacked on glue layer and control the corresponding mechanical condition of cutting device, the effect of cutting wedge condition on dynamic deformation of laminated structure should be clarified.

In this study, therefore, the effect of indentation speed and the apex angle of cutting blade on the deformation characteristics of copper foil and load response, during the wedge indentation to a copper foil of 0.035mm thickness stacked on acrylic based glue of 0.050mm thickness and paper mat of 0.115 mm thickness, were experimentally analyzed. Regarding various combinations of cutting speed and wedge (apex) angle, the cutting test was investigated, and after cutting, the sheared profile of foil was observed by a laser microscope. As the result, it was confirmed that the apex angle of wedge and the indentation speed affected the cutting characteristics of the copper foil stacked on glued paper mat: the warpage of copper foil in the neighborhood of wedged zone, the macro scale bent-up angle, the correlation between the maximum force (as the foil broken point) and its timing position.

**Keywords**: pressure sensitive adhesives, cutting, shear, warpage

**References:**
A study on the improvement of the strength of weld part by core pin drive injection molding method

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A study of the improvement in the strength of the weld line using a core pin drive injection molding method. The weld line is a typical molding defect in plastic injection molding. It is known to be a cause of poor strength and appearance. A poor appearance can be hidden by painting the product. High-strength structural members and mechanical parts have been made using resin. However, weld lines in thick plastic parts with complex shapes are problematic. Development and disseminating weld line strength improvement technology are in demand.

A number of research reports have been published concerning the weld line. For example, “molding conditions affect the weld line”, “weld line loss due to molding conditions,” and Umemura and colleagues conducted a study reporting that additives affect the weld line strength. Tomari and colleagues presented a research report concerning the weld line strength in multi-cavity short fiber-reinforced resins. The research report from Tomari and colleagues is a reference for the fracture behavior of the weld line. This report is a good example of deficient weld line. The authors have proposed a weld line strength improvement technology. The method, after filling the resin into the mold, is a mechanism that forces flow through the core layer before solidification. The tensile load, etc. can be improved using this method.

The core pin driving method is arranged at an arbitrary position in the mold with any amount of movement and arbitrary timing followed by forcing flow into the core layer. The core pin drive method is a weld line strength improvement technology. This study is a report on the relevance of the following: the tensile load and the forced flow conditions, the forced flow amount and the tensile fracture mode, and the forced flow volume and the fiber orientation. There were three tensile failure modes. In the case of a small amount of forced flow, the failure mode destroys the weld line. A moderate amount of the weld line is destroyed by a forced flow band. A maximum amount of the weld line is destroyed from the weld line remaining on the surface.

As a result, the tensile strength was improved to a strength of approximately 80% of a test piece with no weld lines. However, not reach the strength of the test piece with no weld line. For this reason, the weld line remaining on the surface of the test piece prevents strength improvement. However, this technique may be considered as a practical weld line strength improvement technology. This study was analyzed using flow analysis (Moldex3D). The analysis items were the gate position, the shape of the mold and other similar structures. CAE analysis is an effective tool.

Keywords: strength of weld, core pin drive, injection molding, forced flow

References
Consolidation by Spark Plasma Sintering of Polyether-Ether-Ketone

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For decades, high-performance thermoplastics such as thermostables polymers have aroused considerable interest. In this class of polymers, polyetheretherketone (PEEK) has been extensively investigated thanks to unique combination of temperature stability, high wear resistance, stiffness, chemical resistance and high mechanical properties [1-3]. For these reasons, it is already well-known to be of great interest for engineering applications, particularly in the automotive, aerospace and biomedical industries [2, 3].

However, conventional processing like injection or extrusion molding applied to this category of polymers present several limitations. Indeed, the high viscosity in the molten state makes the shaping difficult and limits the use of fillers. In addition, the high temperature processing leads to some degradation and shrinkage problems during crystallisation due to the difference in density between amorphous and crystalline phases [4].

A solution to these problems is the use of uncommon method for the development of such materials. In this way, the Powder Metallurgy approach was considered and more precisely a consolidation process called “Spark Plasma Sintering” (SPS). Contrary to conventional techniques, its main interest is the processability at low temperature under the melting point. This way confers the capacity to keep a degree of crystallinity close to the native powder’s one and hence the potential to increase the mechanical properties.

First, the effects of SPS parameters such as temperature, pressure, and dwell time on density, mechanical properties and structure of PEEK were investigated. Finally, the mechanisms of polymer’s sintering were studied and a particular attention on the role of pressure’s application on the chains mobility was considered.

Key words: Polymer processing, Spark Plasma Sintering, Polyetheretherketone

References
Calculation of the effective properties of composites with a transition layer

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Liquid-phase methods of preparation of composite materials allow producing of complex shape products with minimal further processing. The process of composite forming by casting with crystallization under pressure is used to increase density of the casting and improve uniformity of particle distribution in the metal matrix.

Of particular interest is represented the processes at the phase boundary, defining the physical and mechanical properties and performance characteristics of the product.

In order to improve the interfacial wettability, both particle surface modification using coating and matrix alloying with are employed. The inclusions are coated previously to improve the interphase bonds. During the crystallization process the transition layer is formed at the interface between the particle and matrix, changing the thermal properties of composites. Similar composites are widely used and analyzed in the literature [1].

In this work we calculated the effective properties of matrix composites. In this problem we discuss the effective thermal expansion coefficient for a spherical inhomogeneity with transition layer. The transition layer is modeled using differential method of replacing an inhomogeneous inclusion by an equivalent homogeneous one. The effect of this layer and its thickness on properties of composite is discussed. The effective properties are calculated using Maxwell’s homogenization scheme in terms of thermal expansion contribution tensor of the inhomogeneity, considered in the work [2].

**Keywords:** effective thermal expansion coefficient, transition layer, Maxwell’s homogenization scheme, thermal expansion contribution tensor, spherical inhomogeneity.

**References**
Development of Dynamic Spallation Test for Oxide Scale with Fast Hard Ball Collision

-Influences of Collision Material for Spallation of Oxide Scale-

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Oxide scales on the metallic materials are formed in high-temperature applications and materials processing of alloys at high temperatures. Spallation of the oxide scale leads various problems such as acceleration of oxidation rate and degradation in surface performance of materials. Spallation test methods are important to understand spallation of oxide scale. A spallation test of oxide scale with high velocity collision of hard ball to oxidized alloys is proposed in the present study. In order to apply the spallation test to a variety of oxide scales, influences of test conditions should be discussed. The purpose of this study is to investigate the spallation behavior of the oxide scale with different density of hard ball to be collided.

Samples (Φ20 ×10) of carbon steels (0.55 mass%C) and Cr-Mo steels (1 mass%Cr-0.1 mass%Mo) were oxidized at 800°C in air. Spallation of oxide scale on the oxidized samples was tested with collision of a hard ball (20 ~ 100 m/s) at room temperature. In this study, three type of hard balls were used; bearing steel balls (7.8 g/cm³), silicon nitride balls (3.3 g/cm³) and cemented carbide balls (14.6 g/cm³). All the hard ball are 2.5 mm in diameter.

The spallation of the oxide scale of carbon steels occurred between steel substrate and oxide scale. On the other hand, the spallation of Cr-Mo steel occurred in the inside of oxide scales. Figure 1 shows spallation area as a function of impulse of ball shot. With increasing density, the spallation area is increased at the same impulse. Faster velocity at the same impulse results in larger spallation for the scales on the carbon steels and Cr-Mo steels.

Fig. 1 Spallation area as a function of impulse of ball shot on various hard ball type

**Keywords:** Oxide scale, Collision test, Spallation, Hard ball
Establishment of identification method of the oxide scale spallation on collision test using a hard steel shot

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Oxide layer of metals has a role of thermal and corrosion resistant in high temperature corrosive environment. Spallation of oxide layer is often related to severe damage such as a breakage of turbine blade. Spallation behavior has been investigated by several material tests and has also been proposed several new methods to evaluate the spallation properties. A collision test is one of the evaluation methods in actual situation, and is establish to identify the properties of spallation behaviors. The purpose of this study is to develop the evaluation method for spallation behaviors of oxide scales on several materials by collision test using a hard steel shot and to identify the properties and the characteristics of spallation behavior for various materials. Collision tests were conducted for carbon steel, Cr-Mo steel, stainless steel, Ni and some other materials with non-oxidation and oxidation by shooting a shot. The shot was used a steel ball with a diameter of 2.5mm accelerated up to 60 m/s approximately by compressed air with 0.25 MPa using a prototype collision test equipment. The specimens were observed each indent on the surface and measured the surface profile in each indent. Indent area and spallation area were compared with non-oxidation and oxidation and were discovered in relation to the collision rate in all materials. Spallation of the oxide scale was able to distinguish by the occurrence region where were inside of oxide scale or boundary between the material and the oxidation scale. As the several points of view were summarized, the characteristics of the oxide scale spallation were discovered and categorized in accordance with the mode of spallation on collision test.
The designed three dimensional complex shapes could've be machined on the insulating materials with electrical discharge machining method about twenty years ago. This machining method was proposed by authors research group. It was named as the assisting electrode method by authors group. In this method, the electrical conductive material was attached on the surface of workpiece before machining. The discharge started from the attached material, and continued to the workpiece contact surface. As the discharge occurred in the working oil, the dissolute carbon was generated on the workpiece surface, the electrical conductive layer was attached on the workpiece surface which was composed by the carbonized material. On the suitable discharge conditions, the later could've be attached after the machining of attached surface material which I was act as role of earth. Many insulating ceramics materials such as Al2O3,ZrO2,Si3N4, SiC,AlN,Sapphire,and diamond could' be machined. The machining priorities of machining speed,surface roughness etc. was observed. The machining speed was not enough to use as the actual method. In this paper, to obtain the better machining properties, the new compound machining system was designed with the ultra sonic machining method. The following experimental factors were examined, (1)vibration frequency,(2)amplitude,(3)way of vibration. The effects of added vibration were confirmed on the machining speed and surface conditions. The action of discharged debris and the oil was examined with the designed system. The exhausting phenomena of discharge debris on the machining atmosphere was observed.
Study of Mechanical Properties of Polymer-Ceramic-Metal Composite Systems

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An objective of today’s development engineers’ is to reduce the mass of the designed machines, and in this way, saving considerable energy. This energy-saving, among other things, can be obtained by the reduction of the mass of the machine parts. For this reason, there is an increasing tendency for the employment of special materials used in industry. More and more metal parts are substituted by metal foams, by several fiber-reinforced polymer composites, which besides their reduced mass-possess better mechanical properties, and in this way, they are able to completely perform the expected function. [1]

Metal foams are low density materials, which have unique mechanical, electric, thermal and acoustic properties. Owing to their great specific strength and good soundproofing ability, they are widely used in building industry. Their low thermal conductivity and excellent thermal diffusion are advantageous for electronic industry, while their good energy-absorbing ability revolutionized wrapping technique, military industry (for example, padding of bulletproof vests), vehicle industry, where sandwich panels are made from these materials. [2]

The advantages of polymer composites are their planned mechanical properties, low density, electric and magnetic insulation ability, great strength and corrosion resistance. [3]

The aim of our article is the study of the mechanical properties of fiber-reinforced polymer and metal foam composite systems in order to develop a system which has better properties than the metal foam and the fiber-reinforced separately.

Finally, we make proposals for the application of the examined advantageous properties in industry.

Keywords: metal foams, fiber-reinforced polymer, mechanical properties

References:
Laser Cutting of a Thick Glass Plate

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Laser cutting method of a glass plate in the present study is that crack propagates by local thermal stress generated by laser irradiation without melting and vaporization of the material. In this method, occurrence of micro-cracks can be suppressed and there is no material loss compared to conventional mechanical cutting method. According to the previous research works, the laser cutting method has been mainly applied to a glass plate with thickness about 1-2mm. In this study, the laser cutting method was applied to a glass plate with thickness of 10mm. According to the experimental results, it was not able to cut a glass plate completely in thickness direction by the laser cutting carried out in air. Unstable crack propagation behavior such as crack deflection and branching was observed when higher heat input was applied. In order to change temperature distribution and history, the laser cutting experiment was carried out in water. A glass plate with thickness of 10mm was successfully cut by the laser cutting method. Unstable crack propagation behavior was suppressed in the laser cutting experiment conducted inside water. Crack was propagated from the both sides of the specimen surfaces according to observation in the cross sectional surface cut. It was considered that in case of laser cutting of a thick glass plate, thermal stress was not enough generated to grow a crack in the thickness direction, however, unstable crack propagation easily occurred when higher heat input was applied. Distribution of thermal stress was very localized and rapid heating / cooling history was induced during laser cutting process conducted inside water. Material properties may strongly affect the laser cutting.

Keywords: Laser cutting, Glass plate, Thermal stress
The secondary salinization of grounds is one of the main problems of agriculture that has been taken place in Mirza-chul region in the process of developing the huge tracts of lands for irrigation. The soils of this oasis are subjected to irrigative salinization; therefore, it is necessary here to carry out anti-salinized measures, without which one cannot again use irrigated lands valuably.

In Sir-Darya province the general area of lands makes up 286.9 000 of ha: out of them there are 2514.4 thousands of ha in agricultural recycle, 14332.45   ha being difficult –reclamation ones. As a result of non-cultivating agricultural arable lands, there 1845.1 ha of grounds turned into uselessness ones, the ones of poor-reclamation conditions makes up 1432.45 ha.

The development of new cultivation technologies for different land-reclamation grounds with using agro-reclamation and agro-technical measures, structure-formers and cultivating the culture-developers will allow raising fertility of the soils and efficiency of their use. This does define actuality of the problem. It studies changes of the main properties in soils under the influence of bio-reclamation cultures. Agricultural manufacturers lose on harvest up to 50% because of soil toxicity excess in the upper layers of soils. The used methods in reclamation of grounds are connected with intense washing up the grounds and considerable volume of collector-drainage flow taken to the rivers and they, in general, are the main reason of deterioration of water quality in their bottom currents.

Purpose of the research was to evaluate the influence of licorice cultivation on physical and technological properties of law-fertile soils in Sir-Darya province. This research was conducted in Galaba economy, Bayaut region, Sir-Darya province. The Sir-Darya economy is located on the second over flood-meadow terrace of Sir-Darya River structured with alluvial sediments in sierozem –meadow soils.

For this purpose, there were established 6 key sites with different duration of licorice up to growing of one, four, six and ten years of licorice bushes and cotton field. On each site there were established basic cuts up to the surface of ground waters at their close bedding up to the depth of 3 meters lower than surface of earth at their deep bedding.

In investigated soils the size of volume masses is changing in wide limits from 1.20 up to 1.67 g/sm\(^3\), the least indicators being in arable horizon of six years in licorice cultivation. Within the limits of top horizons an under-arable horizon differs by the largest soil compaction. To the bottom the soil decomposition takes place, the density of the soil structure has an optimal estimations (1.26-1.39 g/sm\(^3\)). Therefore, under licorice bushes of four, six and ten years, the soil has more soft consistence not only in the top part of the soil profile, but in the soil-forming rock as well. It should be noted, that the study of spreading licorice bushes in soil profile on the field with bushes of ten years in licorice showed, that the licorice roots penetrating deeply into the soil promote soil decomposition which result in conditions for developing micro and macro-aggregates.

For granular metric composition in sierozem-meadow soils of Galaba economy it is characteristically the richness of them by large-dust fractions (particles of 0.05-0.01mm), their content ranges from 24 up to 50% in upper meter thickness. The content of agronomical valuable macro aggregates ranges from 50-59% on ploughed field profile in sierozem-meadow, heavy-loamy, strongly salinized soils. The content of water-stable aggregates (3-2 and 5-3 mm) were changing from 2.6 and from 4.3 up to 4.8 % in sierozem – meadow soils with close bedding of the level in strongly-mineralized ground waters of arable horizon.

Analysis by the wet sieving method on wetting and capillary humidifying showed that the content of particles by the size of> 2mm were reducing with increase of the remoteness in licorice...
In arable and under-arable soil layers the minimum of particle content makes up 34.5 and 32.5%.

Of six years in licorice cultivation.

Mechanical stable aggregates of one, four and ten years of licorice cultivation make up from 354-367 up to 450-492 kg/sm² in meter layer, the most ones being under licorice of six years of cultivation i.e. 369-725 kg/sm².

In pasture soil firmness makes up within 19-20 kg/sm² of one and 4 years in licorice cultivation in plowed layer, but the most size i.e. 40-45 kg/sm² are noted of 6 years. In deep moisture and low-density horizons the soil compactness ranges within 5 kg/sm².

Keywords: salinization, irrigation, agricultural, micro-aggregate, ploughed, ground, bedding
Synthesis and characterization of Zirconia-Ytria nanoparticles in t' phase by sol-gel and spray drying.

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The synthesis of Zirconia-Ytria nanoparticles in phase t', non transformable tetragonal phase of the zirconia, is important for the reinforcement of different ceramic matrixes with nanometric and submicronic structures, in order to enhance the mechanical resistance of the composite obtaining a better and homogeneous stress distribution. The objective of this research is to obtain the phase t' by sol-gel synthesis and spray drying of the gel suspension. The precursors used in this study were: zirconium oxychloride octa-hydrate and yttrium oxide which was dissolved in hydrochloric acid and water, after salts hydrolysis, the suspension subsequently undergo to spray drying and the obtained spherical nanostructured aggregates are calcined at 650 °C. Non transformable tetragonal composition employed was 7.5YSZ (7.5% mole YO₁.₅), according to the equilibrium diagram of ZrO₂-YO₁.₅ system. The products obtained were characterized by XRD and SEM, verifying obtaining the phase t’ and analyzing the microstructure of the obtained particles. From XRD results, it was determined that calcination temperature was enough for the complete obtention of the phase t'.
The Exchanges of Metal/Carbon Nanocomposites Electron Structures and Properties due to the p – Elements Intercalation

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The functionalized nanoproducts with introduced p-elements (Si, P, S) are produced by the mechanical – chemical interactions of Copper or Nickel/Carbon nanocomposites with Silicon-, Phosphorus- and Sulphur containing compounds.

The possibilities of these processes are determined by quantum chemical modeling. The probability of RedOx processes which lead to the oxidation states changes for correspondent p-elements is established. In these cases there is the formation of two states for p elements in reduced and oxidized states. The possibility of metal coordination with p-elements groups as well as the metal charge changes is also defined.

Experimental investigations confirm the quantum chemical modeling results. The characteristics of initial reagents and the conditions of the synthesis are following;

According to PEM studies, Metal/Carbon Nanocomposites are metal containing clusters, associated with Carbon Fibers, which have Carbene Chains. In other ways, Si, P or S containing compounds introduce p elements with oxidation states +4(Si), +5(P), +4(S) accordingly. Mechanical Chemical Synthesis of functionalized nanocomposites is carried out with the pounder application.

After thermal stage the nanoproducts obtained are investigated by means of IR spectroscopy and X-ray photoelectron spectroscopy. In IR spectra the lines, correspondent to P–C and Si–C bonds, are found. According to the results of X-ray photoelectron spectroscopy the P2p lines correspondent to P0 (EP2p = 130 eV), and Si2p – to Si0 (ESi2p = 99 eV) are appeared, and also the metal atomic magnetic moments growth takes place. For example, the atomic magnetic moment for Si containing Copper/Carbon Nanocomposite is increased to 2.5 µB.

**Keywords:** metal/carbon nanocomposites, intercalation, p-elements, RedOx synthesis, atomic magnetic moments, oxidation states
Mechanical properties of nonwoven geopolymer composites

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This experimental research focuses on mechanical properties of nonwoven glass fabric composites binded by geopolymeric matrix. This study investigates the effect of different matrix composition and amount of granular filler on the mechanical properties of final composites. Matrix was selected as a metakaolin based geopolymer hardened by different amount of potassium silicate activator and modified by slag addition for optimal mechanical properties and time of hardening. Optionally the ceramic granular filler was added into the matrix for investigation of its impact on mechanical properties and workability. Prepared pastes were incorporated into the nonwoven fabrics by hand roller and final composites were stacked layer by layer to final thickness. The early age hardening of prepared pastes were monitored by small amplitude dynamic rheology approach and after 28 days of hardening the flexural strength and modules of elasticity in bending and torsion were examined. The optical and scanning electron microscopy were used for complex description of final composites. The imaging methods revealed good wetability of glass fibers by geopolymeric matrix and results of mechanical properties indicate usability of these materials for constructional applications with elimination of risks originating from corrosion of steel reinforcement constituents.
It is well known that ZrW₂O₈ is an excellent isotropic negative thermal expansion (NTE) material over a wide temperature range (-273-777 °C) and has been widely used to create composites with controlled thermal expansion. However, after synthesizing zirconium tungstate remains metastable and decomposes into constituent oxides upon heating. Therefore, one can expect some specific behavior of ZrW₂O₈ during the interaction with metals near the melting point. One of the simplest ways to observe this effect is using aluminum due to its low melting point. In addition, aluminum-based materials are lightweight, demonstrate excellent mechanical properties, thermal and electrical conductivity, and therefore are promising candidates for developing new generations of composites with invar effect.

Al – ZrW₂O₈ composites sintered as powder mixtures in argon atmosphere at 600 °C for 1 and 5 hours have been studied. Structure and mechanical properties and interaction between ZrW₂O₈ and aluminum during intense mechanical activation and sintering near the melting point of aluminum in these composites were investigated.

The data obtained using scanning electron microscopy and XRD analysis demonstrate that formation of the composite goes on through decomposition of zirconium tungstate, synthesis of WAl₁₂ and ZrAl₃ intermetallic compounds after 1 hour of sintering and subsequent formation of ZrW₂O₈ microfibers after 5 hours of sintering. Moreover, ZrW₂O₈ is not distributed uniformly in the matrix and one can observe dense areas with high concentration of ZrW₂O₈ with Vickers hardness of such areas is several times higher than hardness of matrix and increases with increase of sintering time.
The influence of metakaolin substitution by blast-furnace slag in alkali-activated inorganic binders

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In this study the effect of metakaolin replacement by milled blast-furnace slag in alkali-activated geopolymeric binder was investigated in accordance to their rheological and mechanical properties. It was demonstrated that slag addition into the metakaolin binder can improve mechanical properties of final products. Our investigation was focused on broad interval of metakaolin substitution in the range from 100 to 40 volume per cents of metakaolin so that the volume content of solids in final binder was maintained constant. Prepared binders were activated by alkaline solution of potassium silicate with silicate module of 1.61. The particle size analyses were performed for determination of particle size effect on rheological properties. The rheological properties were determined in accordance to flow properties by measurements on Ford viscosity cup and by oscillatory measurements of hardening process. For the investigation of hardening process, the strain controlled small amplitude oscillatory rheometry was used in plane-plate geometry. For determination of applied mechanical properties were binders filled by ceramic grog in the granularity range 0-1 mm. The filling was maintained constant at 275 volume per cents in accordance to ratio of solids in dry binder. The mechanical properties were investigated after 1, 7 and 28 days and microstructure was documented by optical and scanning electron microscopy. The results indicate that slag addition have beneficial effect not only on mechanical properties of hardened binder but also on flow properties of fresh geopolymer paste and subsequent hardening kinetics of alkali-activated binders.

Keywords: Geopolymer, metakaolin, slag, rheometry, mechanical properties
The superplasticity of a fine-grained Sn-1Bi alloy, processed by Multi Directional Forging (MDF), was studied by shear punch testing (SPT) at room temperature. The grain size decreased from 4.2 to 2.6 μm, after 2 passes of MDF and Continue the process had little effect on it, so that eventually reached 2 μm after 8 passes, however, the microstructural homogeneity increased greatly with increasing the number of MDF passes. The results indicate the strain rate sensitivity (SRS) of the shear yield stress increase from 0.11 to 0.53, as the number of passes increased from 2 to 8. The high SRS index of 0.53 is indicative of a superplastic deformation behavior dominated by grain boundary sliding.
Ferrite-austenite duplex steel with 1.5 GPa strength and 50% ductility by transformation-induced plasticity of non-recrystallized austenite

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As a promising method to achieve ultra-high yield and tensile strengths of 1.4~1.5 GPa together with excellent ductility above 50 %, a simple annealing treatment of a cold-rolled Fe-Mn-Al-C-based steel was suggested in this study. The ultra-high yield strength of 1.4 GPa was achieved mainly by the non-recrystallization of austenite, while it was also beneficially affected by the precipitation hardening of V and V+Mo carbide, solid solution hardening of Si, and ultra-fine ferrite grain with high dislocation density. The persistent elongation up to 50 % was attributed to TRansformation-Induced Plasticity (TRIP) in the non-recrystallization of austenite which could easily reach the critical strain for martensitic transformation. These results were unexpected ones because the deterioration of elongation in the non-crystallized region was generally accepted in commercial steels. However, the 650°C-annealed steel showed a steady stress flow behavior because of inhomogeneous stage-by-stage-type deformation, which could achieve ultra-high yield and tensile strengths and excellent ductility. Since the present duplex steels have superb properties of strength and ductility, easy manufacturing process, and reduced density and alloying costs, they give a promise for various automotive applications requiring excellent properties.

Keywords: Ferrite-austenite duplex steel, Recrystallization, TRansformation-Induced Plasticity, Non-recrystallized austenite
Electron beam technologies of modification coating are complex and varied. In them is observed a whole complex of physical and chemical processes and phenomena which determine the formation of the phase and the chemical structure of the surface layers and their mechanical properties.

In this paper, the mathematical model of sintering the Ti powder due to electron beam treatment with regard to explicit porosity evolution of powder layer has been suggested and investigated. Due to porosity reduction the shrinkage occurs, thermal – physical properties (the heat capacity, thermal conductivity coefficient) change. For the numerical realization of the model has been developed special numerical algorithm and computer program. It has been shown that additional mechanism of convective heat transfer connects with powder layer thickness evolution. It was detected that model obeys some prognostic properties and allows to reveal the area of technological parameters of sintered layer form.

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*Keywords:* Electron beam processing, Mathematical simulation
Analysis and prevention of liquid metal embrittlement occurring in resistance-spot-welded TWIP steel sheets

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Hot-dip-galvanized TWinning Induced Plasticity (TWIP) steel sheets were fabricated, and liquid metal embrittlement (LME) occurring during resistance spot welding was clarified in relation with microstructural parameters including various constituent phases and influences of welding heat effect. The detailed microstructural observation of initiation and propagation of Zn infiltration or crack occurring during very-short-time welding was explained by tracing procedures of liquid Zn infiltration and formation of $\alpha$-Fe(Zn) and $\gamma$-iron phases due to welding heat effect. $\alpha$-Fe(Zn) particles formed on the steel surface provided paths for liquid Zn infiltration, while they played a role in accelerating the LME as brittle phases themselves. Particularly in the case of increased content of Al, the LME was more activated rather than in the case of increased thermal or tensile stresses due to the increased welding current. Thus, the appropriate combination of Al content and welding current was essentially needed because the further reduction in Al content might promote the LME, while the reduction in current deteriorated the weldability and productivity.

Keywords: Liquid metal embrittlement, Resistance spot welding, Galvanized TWinning-Induced Plasticity (TWIP) steel, Liquid Zn infiltration, Al content
Polymer Melt Viscosity Measuring by an Injection Machine

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In our work we present an investigation of polycarbonate melt shear viscosity in wide range shear rate. In the course of our research we aggregated more testing method results and mathematically determined viscosity equation of the tested material.
The 1st International Symposium on Intelligent and Smart Materials
Researches of mechanical behavior of the bone microvolumes and porous ceramics under uniaxial compression

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The long-term experience of the artificial materials' use to repair the bone elements points out that the biochemical compatibility of the implant with the bone is an essential, although insufficient, condition for functioning of the implant - bone system as a whole. Their mechanical compatibility is an important condition too.

The research results of the mechanical behavior are presented and the effective mechanical characteristics under uniaxial compression of the model bone micro volumes are defined subject to the direction of the collagen-mineral fibers, porosity and mineral content. The experimental studies of the mechanical behavior are performed and the effective mechanical characteristics of the produced porous zirconium oxide ceramics are defined. The recommendations are developed on the selection of the ceramic samples designed to replace the fragment of the compact bone of a definite structure and mineral content.

\textbf{Keywords:} osteoimplants, bone microvolumes, porous ceramics, mechanical compatibility
Self-Recovering Section of RPV Steel Radiation Embrittlement Kinetics as Indication of Material Smart Behavior

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Dependence of the materials properties on neutron irradiation intensity (flux) is a key problem while usage data of the accelerated materials irradiation in test reactors to control and forecast their capacity for work in realistic (practical) circumstances of operation. Investigations of the reactor pressure vessel steel radiation degradation dependence on fast neutron fluence (embrittlement kinetics) at low neutron flux reveal the instability in the form of the scatter of the experimental data and wave-like sections of embrittlement kinetics appearance. Disclosure of the steel degradation oscillating is a sign of the steel structure cyclic self-recovery transformation as it take place in self-organization processes. This assumption has received support through the discovery of the similar “anomalous” data in scientific publications and by means of own additional experiments.

Data obtained stimulate looking-for ways to management of the structural steel radiation stability (for example, by means of nano - structure modification for radiation defects annihilation intensification) for creation of the intelligent self-recovering material.

Expected results:
- radiation degradation theory and mechanisms development,
- more adequate models of the radiation embrittlement elaboration,
- surveillance specimen programs improvement,
- methods and facility development for usage data of the accelerated materials irradiation for forecasting of their capacity for work in realistic (practical) circumstances of operation,
- search of the ways for creating of the stable under neutron irradiation self-recovery smart materials.

Biography
Krasikov E. is Head of Laboratory, Department of Reactor Materials and Technologies at the National Research Centre “Kurchatov Institute” (Moscow, Russia), with key qualification: responsible executor in Radiation Damage Physics of Solids. Master’s Degree in Material Science – 1970, Ph.D. – 1974, D.Sc. -2005. He has published more than 100 peer-reviewed papers.
Synthesis of Fluozirconate Glass ZBNL with Partial or Complete Substitution of Samarium, Europium and Ytterbium Difluorides for Barium (Lanthanum) Fluorides

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The formation conditions of glass and glass-crystalline phases as well as phase transitions under the partial or complete substitution of difluorides of rare-earth elements (REEs (Ln): samarium, europium, ytterbium) for lanthanum (barium) fluorides in fluorozirconate compositions ZBNL (mol%): (53.5) ZrF₄ – (20.0) NaF – (20.0) BaF₂ – (6.5 - x) LaF₃ – (x) LnF₂ (0 ≤ x ≤ 6.5 ) (composition I) and (53.5) ZrF₄ – (20.0) NaF – (20.0 - x) BaF₂ – (6.5) LaF₃ – (x) LnF₂ (0 ≤ x ≤20.0 ) (composition II) have been determined by differential-thermal, X-ray phase analysis and infrared spectroscopy. The obtained glassy samarium- and europium-containing samples are colorless, and the ytterbium-containing samples are green. At higher rare-earth fluoride (II) content of the original mixtures, the formation of glass-crystalline phases is observed (for over 6.5 mol% samarium and europium and over 15 mol% ytterbium). For all samples of the investigated systems, the vitrification temperature \( t_g = 200 \text{ – } 250 ^\circ C \). Exothermic crystallization effects are observed in the following temperature ranges: \( t_{x1} = 320 – 340 ^\circ C \), \( t_{x2} = 380 – 460 ^\circ C \), \( t_{x3} = 415 – 490 ^\circ C \), and an endothermic melting effect is observed in the range \( t_{\text{melt}} = 460 – 490 ^\circ C \). When the LnF₂ content is increased (x ≥4.0 mol%), endothermic effects, which relate to the melting of samples based on lanthanide difluoride as a modifier, are observed in the range 545-690 °C. The Hruby criterion, which characterizes the crystallization resistance of glass, has the maximum value of 0.65 (1.0 mol% SmF₂), 0.86 (2.0 mol% EuF₂), 0.67 (3.0 mol% YbF₂) for composition I and 0.78 (5.0 mol% SmF₂), 0.63 (3 mol% EuF₂), 0.64 (1.0 mol% YbF₂) for composition II.

The results of IR spectroscopy suggest that glass in the investigated compositions is composed of fluozirconate skeletons, which consist of ZrF₆ polyhedra linked by bridge bonds. The simultaneous change of the ratios Zr/Ba and Zr/Ln (composition II) gives rise to absorption bands typical of Ln-F bonds. The absence of Ba-F absorption bands indicates that the barium cations are in fluozirconate skeleton voids and form ionic bonds to fluorine anions. When REEs are largely substituted for barium, extra absorption bands appear, which indicate localization of REE cations in different positions depending on their oxidation state. The fact that the synthesized glass samples contain REE cations in different oxidation states is evidenced by the results of diffuse reflectance spectroscopy; their spectra exhibit bands at 200-400 nm and 980-2200 nm, which are typical of 4f-5d electron transitions in Ln²⁺ ions and 4f-4f transitions in Ln³⁺ ions respectively.

Key words: fluozirconate glass, synthesis, samarium, europium, ytterbium difluorides, partial or complete substitution
Repeatability of Crack Healing on 5 vol% Ni/ Al₂O₃ Nanocomposites

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Engineering ceramics have high chemical stability, high-temperature resistance and good abrasion resistance. However, fracture strength of engineering ceramics is sensitive for cracks or voids. Ni/ Al₂O₃ nanocomposites have a crack healing function [1], which is caused by high-temperature oxidations of dispersed Ni particles. In this composite system, surface cracks were filled with the oxidation products within Al₂O₃ matrix [2]. Oxidation of Ni dispersion also results in formation and growth of oxidized zone, which consists of NiAl₂O₄ grains in Al₂O₃ matrix. After healing, Ni dispersion was consumed. It is not known that healing ability will appear or not again at the position surface cracks were healed before. Evaluation for repeatability of crack healing is necessary to investigate for estimating the life time of the present material system.

Samples of Ni/Al₂O₃ nanocomposites were prepared with pulsed electric current sintering. Surface cracks were introduced with the Vickers indentation at 49 N for 10 s. The samples were heat-treated at 1200°C for 6 h in air to heal the surface cracks. A surface crack was re-introduced at the same position of the surface. The samples were heat-treated again for crack-healing.

Fig. 1 shows surface SEM image of Ni/Al₂O₃ on repetition of crack-healing and crack introduction. Surface cracks included the first indentation were disappeared completely. Surface cracks prepared by the second indentation on the healed sample were also disappeared completely again.

Fig. 1 Repetition of cracks and indentation on a sample surface.

References:
Importance of surface charge on solid-liquid interfaces in nano/microfluidic channels for membrane, sensor and energy conversion applications

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Micro/nano fluidic systems are widely used in physical, chemical and biomedical fields. In many technical applications, the physico-chemical processes on the solid-liquid phase play an important, often even decisive role. The surface charges of the nano/microfluidic channels (membranes, sensor/actuators, energy harvesting devices, technical nano/microchannels) result in a number of phenomena that arise on the solid-liquid interface.

Thus, for example, the surface charge of a nano- and ultrafiltration membrane determine the separation and fouling properties of filtration processes. In nanofiltration, charged substances can be separated depending on the polarity and value on the basis of the membrane charge.

In nano/microfluidic channels electrokinetic phenomena arise, such as electroosmosis and streaming potential, based on the charges of the inner capillary surface. E.g. using an electroosmosis pump a fluid in a lab on chip channel can be moved. Electroosmosis can be used as an actuator principle, too.

Electrokinetic sensors based on the reversal effect, the streaming potential, can detect the adsorption of chemicals or drugs, or biological markers. The charge modification of the surface of a coated metal oxide semiconductor can be also evaluated. In this way in our group pH sensors were prepared and characterized on the basis of the field-effect occurring in the semiconductor.

The electrokinetic phenomenon, the streaming potential, plays a key role both as a characterization method of surface charges as well as sensor signals. The effect can also be understood as an energy conversion, and was investigated in energy harvesting applications as electrokinetic power generator or battery.

By external modulation of the surface charge by applying of an electric field, the charge-based surface effects are specifically reinforced. Separation efficiency of nanomembranes, sensitivity of sensors and efficiency of the energy harvesting applications are controlled and improved.

Keywords: micro/nanofluidic channels, solid-liquid interfaces, surface charge, electrokinetic, streaming potential, nanofiltration, field effect sensors, energy harvesting
Role of Zn$^{2+}$ ion on the formation of reversible thermochromic polydiacetylene/zinc oxide nanocomposites

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Polydiacetylene(PDA)/zinc oxide(ZnO) nanocomposite is a class of materials that exhibit reversible thermochromism with controllable color-transition temperature. The nanocomposites also change color upon exposure to various types of acids and bases. In our continuation efforts to develop this material, we obtain new evidences that reveal true nature of the nanocomposite formation. X-ray diffraction (XRD) technique detects significant increase of the interlamellar distance within the nanocomposite compared to that of the pure constituent PDA. The investigation of nanocomposite systems prepared from 10,12-tricosadiynoic acid and 10,12-pentacosadiynoic acid provides consistent results. This indicates an intercalation of the Zn$^{2+}$ ions, leaking out of the ZnO nanoparticles, between the PDA layers. An atomic absorption spectroscopy confirms the existence of Zn$^{2+}$ ions in aqueous solution. The XRD result also shows that the use of only Zn$^{2+}$ ions to prepare nanocomposite can provide the intercalated PDA/Zn$^{2+}$ structure. However, the enhanced inter- and intrachain interactions within this system is not sufficient to provide reversible thermochromism. An infrared spectroscopy detects the variation of vibrational frequencies of carboxylate head groups within the systems of PDA/ZnO nanocomposite and PDA/Zn$^{2+}$ assemblies, corresponding to different types of interactions. Therefore, strong interfacial interaction between PDA head groups and ZnO nanoparticle is essential for obtaining the reversible thermochromism. We also attempt to prepare the nanocomposites by using a combination of Zn$^{2+}$ ions and various types of nanosubstrates including TiO₂, SiO₂ and Al₂O₃ nanoparticles.

**Keywords:** color transition; polydiacetylene; reversible thermochromism; self-assembling, temperature sensor
Fractal nature structure method in designing micro/nano-structure properties

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Nanotechnology, the ability to use and control matter, including fractals, at the nano-scale, is resulting and will continue to result in unique material, structure, and device properties which can be used to address a myriad of challenges confronting society including energy, the top priority. Material models and methods that a priori accurately predict material parameters as a function of temperature are needed and can be used to reduce the time and cost of discovering and developing affordable materials with properties that outperform materials in current technologies as well as that provide novel properties upon which new technologies can evolve. The fractal algorithms being applied on perovskites, silicate, refractory and other ceramics, are now extrapolated towards nano-sizes with the aim to retrieve the real microstructures. Based on the grains and pores perimeters fractal analysis, their reconstruction is made using different algorithms. It gives a more realistic picture than that is obtained in the Euclidean geometry frame, which replaces the role of modelling, because it gives real micrographs shapes, on one hand, and from the obtained passive micrograph, through the shape reconstruction, leads to its prognoses possibility with designed microstructure properties. Applying known technology processes phases, obtained samples are the investigated objects, leading toward a more ceramics electronic materials properties exact calculations. Thus, the new possibilities, in microstructure characterization applications, are directly introduced, with which the hardware is reduced only to present engineering system, providing new solutions related to software support. Such solution opens industrial production and application possibilities, and that is a base for the “fractal electronics” development.

Keywords: Perovskite ceramics, fractals, micro/nano-structure
Control over the photophysical properties of conjugated polymer nanoparticles: The effects of solvent and molecular weight

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In this study, we investigate the effects of initial solvent, media solvent and molecular weight (MW) on photophysical properties of poly(2-methoxy-5-(2’-ethylhexyloxy)-1,4-phenylvinylene) (MEH-PPV) and regioregular poly(3-octylthiophene) (rr-P3OT) nanoparticles. The variation of initial solvent, media solvent and MW of MEH-PPV and rr-P3OT significantly affects morphologies and photophysical properties of conjugated polymer nanoparticles (CPN). These properties are important for their applications such as optoelectronics, photonics, bio-imaging, bio-sensing and nanomedicine. The preparation process involves the injection of small amount of polymer solutions in tetrahydrofuran (THF) or dichloromethane (DCM) into non-solvent, alcohols and water. We demonstrate that water solubility of the initial solvent is a major factor dictating mechanism of the CPN formation in water. When the DCM and THF are used as initial solvents, the resultant CPNs exhibit quite different size and photophysical properties. The use of DCM and THF as initial solvents provides the MEH-PPV nanoparticles with green ($\lambda_{\text{max}} = 535$ nm) and red ($\lambda_{\text{max}} = 590$ nm) photoemission, respectively. The variation of media polarity also affects photophysical properties of the CPNs. The use of methanol as a medium provides MEH-PPV nanoparticles with dual light emission at two regions. The PL spectrum exhibits two peaks at ~470 nm and ~585 nm, indicating the co-existence of collapsed chains and aggregates within the nanoparticles. Interestingly, the ratio of these two species varies with polarity of the alcohols. The MW of MEH-PPV and rr-P3OT also affects the photophysical properties of the nanoparticles. We observe that the decrease of MW promotes the PL emission of collapsed coil.

**Keywords:** conjugated polymer nanoparticle, solvent effects, photophysics, self assembling
Investigating the anisotropies of magnetic nanoparticles is crucial for further development of magnetic data storage media, MRAM (magnetoresistive random access memory), magnetic logical circuits, or magnetic quantum cellular automata. Former theoretical and experimental examinations have revealed the possibility to gain highly symmetric nanoparticles with increased numbers of magnetic states per storage element.

In a recent project, we have investigated low-symmetry T-shaped 2D and 3D particles from iron using the micromagnetic simulation software MAGPAR which is based on solving the Landau-Lifshitz-Gilbert (LLG) equation of motion for a mesh built from tetrahedral finite elements. To examine the influence of the reduced symmetry, simulations were performed on the 3D double-T particle with the field applied in different directions in the x-y base plane, ranging from 0 to 180° in 5° steps. Additionally, the external magnetic field was rotated laterally under different angles with respect to the x-y plane, i.e. 5°, 22.5°, and 45°. Similar simulations were executed for the 2D single-T particle.

Our results show the strong impact of the shape anisotropy and the respective possibility to tailor magnetic anisotropies according to the desired behaviour by modifying the nanoparticles’ form.

**Keywords:** magnetic nanoparticle, magnetic anisotropy, micromagnetic simulation, MAGPAR
Development and characterization of textile batteries

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During the past years, smart textiles have gained more and more attention. Products cover a broad range of possible applications, from fashion items such as LED garments to sensory shirts detecting vital signs to clothes with included electrical stimulation of muscles. For all electrical or electronic features included in garments, a power supply is needed – which is usually the bottleneck in the development of smart textiles, since common power supplies are not flexible and often not lightweight, prohibiting their unobtrusive integration in electronic textiles.

In a recent project, textile-based batteries are being developed. For this, metallized woven fabrics in different material combinations (e.g. copper and zinc, silver and zinc) are used as well as combinations of metallized woven fabrics and carbon fabrics. The article gives an overview of our recent advances in optimizing power storage capacity and durability of the textile batteries by tailoring the gel-electrolyte.

The gel-electrolyte is modified with respect to thickness and electrolyte concentration; additionally, the influence of additives on the long-time stability of the batteries is examined.

Keywords: textile battery, electrolyte, textile electrodes, layer thickness, electrolyte concentration

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The 2nd International Symposium on Innovative Construction Materials
One of the 3 largest ceramic roof tiles plants can be found at Lenti in Hungary thanking to the high quality and large quantity of conventional brick clay deposit on the place. To support the productivity of the technological lines and maintain the consistently high quality, adequate material and specific energy consumption as well as competitive prices regular tests and investigation of mined clays are required. To supply the technological lines with clay raw materials of permanent quality the following material tests and investigations were made by the authors:

- Determination of mineral composition (XRD tests),
- Determination of specific surface (BET and Langmoir)
- Structural and morphological tests (SEM, EDAX)
- Thermo-analytical tests (DTA, TG, DTG)
- Dilatometry tests (specific dimensional change, thermal expansion coefficient)

For tests and examination of conventional brick clay “Lenti” the samples were taken from 4 different mine layers (I, II, III and IV) at 2 different location (Kelet=East and Nyugat=West) and 3 different depths. The results of these tests and investigation are present by the authors in this work.

Keywords: ceramics, clay minerals, dilatometry, mechanochemistry, mineralogy, morphology, roof-tile, specific surface, thermoanalysis, x-ray diffraction
The elaboration of computer models for T-x-y diagrams TiO$_2$-SiO$_2$-Al$_2$O$_3$, ZrO$_2$-SiO$_2$-Al$_2$O$_3$ [1], TiO$_2$-ZrO$_2$-Al$_2$O$_3$ and TiO$_2$-ZrO$_2$-SiO$_2$, based on the schemes of mono- and of invariant states, data about the structure of binary systems and surfaces of primary crystallization, permits to create complete geometric construction of T-x-y diagrams [2], that can be used for the prediction of structure of phase diagram for quaternary system TiO$_2$-ZrO$_2$-SiO$_2$-Al$_2$O$_3$.

Models of T-x-y diagrams are constructed taking into account the immiscibility of liquid phases in binary systems TiO$_2$-SiO$_2$ and ZrO$_2$-SiO$_2$ and the presence of five binary compounds: two incongruently melting compounds ZrO$_2$·TiO$_2$, ZrO$_2$·SiO$_2$; two congruently melting ones 3Al$_2$O$_3$·2SiO$_2$, Al$_2$O$_3$·TiO$_2$ and the compound ZrO$_2$·SiO$_2$ decomposed without the melt appearance.

The simplest structure has the system TiO$_2$-ZrO$_2$-SiO$_2$. It is characterized by two invariant transformations and the immiscibility region. Systems TiO$_2$-ZrO$_2$-Al$_2$O$_3$ and TiO$_2$-SiO$_2$-Al$_2$O$_3$ has a similar structure and contain three invariant point, but the system TiO$_2$-SiO$_2$-Al$_2$O$_3$ is complicated by the immiscibility region. The most complex structure has a system ZrO$_2$-SiO$_2$-Al$_2$O$_3$ with four invariant transformations and immiscibility regions. The peculiarity of phase diagrams is the presence of internal liquidus surface corresponding to the compound ZrO$_2$·SiO$_2$ decomposing without the melt appearance in the binary system.

The obtained computer models allow to compare different variants of diagrams with the ambiguity of experimental data and to design the microstructure of ceramics.

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**Keywords:** phase diagram, computer model, oxide systems, ceramics, titanium, zirconium.

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Concrete is the most commonly used building material and its significance to basic infrastructure is immeasurable. Today, many concrete structures are designed with service lives of over 80 years. At the same time, concrete infrastructure from bridges to nuclear containment buildings, are undergoing ageing degradation and at the same time, agencies and owners have limited funds for repair and replacement. In order to ensure this, designers must evaluate the concrete’s resistance to the ingress of moisture and ions in order to control age related degradation. Concrete’s ability to resist damage due to outdoor exposure conditions such as freeze-thaw cycles, de-icer salts, sulfates, and reactive aggregates is typically evaluated by accelerated laboratory tests specified in standards such as ASTM, CSA, RILEM etc.. These tests are valuable experiments which can discriminate between concrete mix designs which perform well or poorly. However, challenges associated with interpretation of the material performance are: (i) the evaluated durability does not necessarily correlate to the material damage that occurs in the field with age, (ii) the test specifications do not typically account for more than one degradation mechanism acting simultaneously, and (iii) conservative models of field performance of concrete need to account for implications of coupled degradation mechanisms. These challenges warrant advancement in research to bridge the gap between laboratory testing and models of field performance through establishing the linkage between micro-scale and macro-scale material characterization of concrete exposed to the natural environment. This paper is focused on current research related to coupled degradation mechanisms which are of relevance and on the forefront of aging concrete infrastructure in Canada. Outcomes of this research indicate that service life estimates vary markedly when consequences of high early temperature exposure are considered with respect to implications on the concrete microstructure, particularly the air void system.
Evaluation of the shrinkage and creep of medium strength self compacting concrete

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The difference between self compacting concrete (SCC) and conventional concrete (CC) is in fresh state, is the high fluidity at first and the need for vibration at second, but in hardened state, both concretes must comply with the resistance specified, in addition to securing the safety and functionality for which it was designed. This article describes the tests and results for shrinkage and creep at some medium strength Self Compacting Concrete with added sand (SCC-MSs) and two types of cement. The research was conducted at the Laboratorio de Tecnología de Estructuras (LTE) of the

Universitat Politècnica de Catalunya (UPC), in dosages of 200 liters; with the idea of evaluating the effectiveness of implementation of these new concretes at elements designed with conventional concrete (CCs).

References

Development of spraying agent for reducing drying shrinkage of mortar

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Mortar used to repair is sometimes exposed to drying state in the early ages after construction and a few days later water is sprayed frequently on the surface of the mortar in order to prevent cracks.

This research studied on shrinkage characteristic of mortar subjected to drying conditions like this. The result showed that the water spraying on the mortar after initial drying did not have any effect to prevent shrinkage, but increased. And it also showed when various chemical agents are mixed and used in water spraying, it had the prevention effect on shrinkage. This report was to understand this kind of phenomenon and clarify the mechanism. In addition, based on the results, the new spraying agent was developed to reduce drying shrinkage.

Keywords: mortar, drying shrinkage, water spraying, new spraying chemical agent,
As the traffic grows, the traffic requirements such as axle loads and traffic volume on the existing infrastructure are rising still. This is especially problematic for old steel bridges. Higher load coupled with many load cycles generally leads to fatigue damage to the structures. This causes an issue for the infrastructure owners as the existing methods of repair are difficult, time consuming and expensive. So there is a need to find some easier alternatives, such as the use of carbon fiber reinforced polymers (CFRP). They are being successfully used for repairs and strengthening of concrete structures however their application on steel is still relatively new.

The purpose of this work is to analyze the behavior of deteriorated steel members reinforced with CFRP under fatigue load not only in the laboratory environment, but also exposed to the real weather on the bridges.

First, the numerical study was performed to establish the suitable dimensions, loads and methods. Based on that, the extensive series of experiments was designed. There are two sets of mild steel specimens. Each set has different level of surface deterioration (corrosion pits or corrosion holes). Some specimens are reinforced using hand laid wet layup composites, some are left unreinforced. They are subjected to fatigue loading and based on the preliminary study, it is expected, that the reinforcement will prolong the life expectancy by half. The results on the reference samples are compared with the samples, some of which exposed to the freezing cycles, chlorides and wet environment.

**Keywords:** CFRP, strengthening, fatigue, mild steel
Ultra Lightweight Cement Composites for energy efficient buildings

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Energy efficient building is defined as achieving satisfactory internal environment and service with minimum energy consumption. One of the most important parameters that affect the heat transfer through the building envelope is thermal conductivity. The thermal conductivity of lightweight concrete is generally lower than that of normal-weight concrete due to the lower thermal conductivity of air. Although introducing voids in concrete reduces its thermal conductivity and increases its insulation capacity, the mechanical properties are generally compromised.

This presentation provides a summary of the development of ultra lightweight cement composites (ULCCs) for energy efficient buildings. The lightweight was achieved by incorporating hollow cenospheres, a by-product of coal combustion at thermal power plants, in ULCCs as micro aggregate. The ULCCs had 1-day densities ranged from about 1150 to 1450 kg/m$^3$ and 28-day compressive strengths ranged from about 30 to 70 MPa. In general the compressive strength, flexural tensile strength, and elastic modulus of the ULCCs were reduced with the decrease in density. Compared with conventional normal weight concrete of similar 28-day strength, the ULCCs have lower elastic modulus. The thermal conductivity of the ULCC was about 80% lower than that of the normal weight concrete of similar 28-day strength. Effect of thermal conductivity on the heat transfer through panel specimens exposed to a sun simulator under controlled solar irradiance, air temperature, and wind velocity was experimentally determined and evaluated.

**Keywords:** lightweight, cement composite, cenosphere, density, mechanical properties, thermal conductivity
Multi-functional building coating materials: An emerging approach to mitigate air pollution and reduce carbon footprint

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Enabling building envelope to mitigate airborne pollutants by employing photocatalytic coating materials is an emerging approach to enhance living environment of large cities. The capability of such photocatalytic coating materials is demonstrated by removing selected gaseous pollutants, while more evidence is needed to show how efficiently such building coating materials can remove airborne particulate pollutants, which impose increasing concerns on public health. This study investigated the removal efficiency of photocatalytic building coating materials by focusing on two types of major particulate components, organic aerosols and black carbon, BC (or soot). The former has been reported as one of the major components of airborne fine particulates. The latter, typically emitting from incomplete combustion processes, is stubborn against being removed from the ambient environment, and is reported as one of the most significant global warming components.

Results showed that mortar specimens coated with silicate containing titanium dioxide (TiO₂) removed a substantial amount of BC depositing on the surface with a degradation rate constant of \(~0.02\ \text{hr}^{-1}\). This indicates that a “BC-resistant” building surface is capable to preserve the surface color and reduce unnecessary absorption of ambient solar irradiation, a heat load on building envelope. Our laboratory results are supported by field measurements demonstrating that the slab surface coated with TiO₂-containing silicate satisfactorily preserved the surface color after an exposure to complicated industrial and transboundary air pollutants in a warm humid tropical city for more than 3 years.

**Keywords:** black carbon, soot, particulate, aerosol, silicate, TiO₂, coating, building envelope,
Admixture Compatibility of Alternative Supplementary Cementitious Materials

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This paper aimed to study the compatibility between some natural pozzolans including perlite (PL), pumice (PM) and zeolite (ZL) with one type of superplasticizer by means of rheology and heat of hydration analysis. The plain cement paste and cement paste containing flysh (FA) were considered as the reference mixes. The results showed that the inclusion of pumice and zeolite led to an increase in yield stress and plastic viscosity with time, compared to the mixtures with perlite or fly ash, or the plain cement mixture, regardless of mixture temperature. The results of the calorimetry analysis revealed that the inclusion of zeolite accelerated the hydration reaction significantly and released almost the same cumulative heat compared to the plain cement paste, while pumice accelerated the reaction slightly during the first 12 hours of the test. Perlite has the lowest heat flow among all SCMs which implied a slower hydration reaction.

Keywords: Natural SCMs, Compatibility, Superplasticizers, Rheology, Hydration analysis
Optimization of activator solution and heat treatment of ground lignite type fly ash geopolymers

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Geopolymers are inorganic polymers which can be produced by the reaction between silico aluminate oxides and alkali silicates in alkaline medium. Materials containing silica and alumina compounds are suitable for geopolymer production. These can be primary materials or industrial wastes, i.e. fly ash, metallurgical slag and red mud.

In this paper, the results of the systematic experimental series are presented which were carried out in order to optimize the geopolymer preparation process. Fly ash was ground for different residence time (0, 5, 10, 30, 60 min) in order to investigate the optimal specific surface area. NaOH activator solution concentration also varied (6, 8, 10, 12, 14 M). Furthermore, sodium silicate was added to NaOH as a network builder solution. In this last series different heat curing temperatures (30, 60, 90°C) were also applied. After seven days of ageing the physical properties of the geopolymer (compressive strength and specimen density) were measured. Chemical leaching tests on the raw material and the geopolymers were carried out to determine the elements which can be mobilized by different leaching solutions. It was found that the above mentioned parameters (fly ash fineness, molar concentration and composition of activator solution, heat curing) has great effect on the physical and chemical properties of geopolymer specimens. Optimal conditions were as follows: specific surface area of the fly ash above 2000 cm²/g, 10 M NaOH, 30°C heat curing temperature which resulted in 21 MPa compressive strength geopolymer.

Keywords: fly ash, grinding, NaOH activator solution, geopolymer, heat treatment
Cracks and delaminations are the common structural degradation mechanisms studied recently using numerous methods and techniques. Among them, numerical methods based on FEM analyses are in widespread commercial use. The scope of these methods has focused i.a. on energetic approach to linear elastic fracture mechanics (LEFM) theory, encompassing such quantities as the $J$-integral and the energy release rate $G$. This approach enables to introduce damage criteria of analyzed structures without dealing with the details of the physical singularities occurring at the crack tip. In this paper, two numerical methods based on LEFM are used to analyze both isotropic and orthotropic specimens and the results are compared with well-known analytical solutions as well as (in some cases) VCCT results. These methods are optimized for industrial use with simple, rectangular meshes; a new type of a refined mesh is also introduced and confronted. The verification is made based on two dimensional mode partitioning.

**Keywords:** $J$-integral, mode partitioning, methods comparison, laminate models
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The 2\textsuperscript{nd} International Symposium on Innovative Carbon and Carbon Based Materials
Raman spectroscopy in the ternary layered phases of Ti-Si-C system: from first principles to experiment

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Among the binary and ternary phases within the Ti-Si-C system, the layered MAX (Ti3SiC2) and Nowotny (Ti5Si3Cx) phases are especially attractive due to a number of extraordinary properties [1,2] making them very promising as high-temperature structural materials [3]. A close relationship between microstructures of the binary and ternary Ti-Si-C phases results in a complex XRD pattern and the interpretation of the multicomponent spectra from the multiphase samples may lead to controversial results. Here, the micro-Raman spectroscopy occurs useful for additional refinement of the phase composition. This contribution presents results of a combined experimental and theoretical Raman spectroscopy obtained for the multiphase Ti-Si-C samples [4,5]. The role of ab initio Raman spectroscopy, employing the recently developed tools based on the density functional theory and the direct method [6], for the identification/refinement of phase composition in the multiphase system will be highlighted.

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Studying The Modification of Construction Composites with Carbon Nanomaterials

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The present work considers one of the directions of using carbon nanomaterials (CNM) – construction materials science. The aim of the research was to experimentally study the mechanism of modifying construction composites with carbon nanomaterials. The review of numerous literature sources allowed to reveal a common pattern of the modification process based on the possibility of forming a nanocomposite structure.

Herein, the carbon nanomaterial (CNM) “Taunit” produced at NanoTechCenter Ltd (Tambov, Russia) was investigated. This CNM represents micrometric granules having a structure of entangled bunches of multiwalled carbon nanotubes (MWNTs). At present the CNM, “Taunit” has found its successful application at more than 150 scientific and industrial organizations in the Russian Federation and abroad.

In the experiments, various structures of concrete - fine-grained, foam, fibro-, asphalt, highly-siliceous, and special-purpose concrete - were used. Optimum technological parameters of the nanomodification of construction materials were determined. To distribute the CNM in the composite matrix, the following methods were implemented: mechanical dispersion, sonication, and magnetodynamic. The CNM concentration ranged from $10^{-4}$ to 10 % of the binder weight. The obtained results demonstrate a positive effect of the nanomodification of the construction materials with the CNM "Taunit", the nanocomposite structure becomes more dense due to a decrease in porosity which promotes an increase in the physicomechanical characteristics (frost and crack resistance, water tightness, durability, and radio absorption) of about 30-40 %. The active use of these methods to nanomodify different construction materials with the CNM “Taunit” will allow to obtain a high-quality construction product capable of satisfying at all levels needs of the ultimate consumer.

**Keywords:** carbon nanomaterials, construction materials, concrete, nanomodification, physicomechanical properties.
Preparation of SiC–MgAl$_2$O$_4$–Y$_3$Al$_5$O$_{12}$–MWCNTs nanocomposites by spark plasma sintering

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The combined insertion of carbon nanotubes and oxide additives to silicon carbide makes it possible to create new composite ceramic materials for use them at high temperatures in the presence of intense external actions of different nature. Nanocomposites based on silicon carbide reinforced by 6% vol. of multiwalled carbon nanotubes with addition of magnesium aluminate spinel, and yttrium aluminum garnet were prepared by spark plasma sintering in form of discs with 20 mm in diameter and 5 mm thick. Sintering of samples was performed at installation at 25 HP D (FCT Systeme, Germany) at a temperature in the range of 2000-2200 °C at a heating rate of 450 °C/min under pressure of 19 to 22 kN. Soaking time of samples at maximum temperature and pressure ranged from 10 to 15 minutes.

**Table 1. Averaged values of the mechanical properties of the samples**

<table>
<thead>
<tr>
<th>Material</th>
<th>Load, g</th>
<th>Microhardness, GPa</th>
<th>Fracture toughness, MPa / m$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC + 6% vol. MWCNT</td>
<td>300</td>
<td>28,3</td>
<td>4,3</td>
</tr>
<tr>
<td>SiC +6% vol. MWCNT +1% wt. MgAl$_2$O$_4$</td>
<td>300</td>
<td>31,4</td>
<td>5,1</td>
</tr>
<tr>
<td>SiC +6% vol. MWCNT +1% wt. MgAl$_2$O$_4$+ +1% wt. Y$_3$Al$<em>5$O$</em>{12}$</td>
<td>300</td>
<td>34,8</td>
<td>5,7</td>
</tr>
</tbody>
</table>

**Table 2. The thermomechanical properties of samples**

<table>
<thead>
<tr>
<th>Material</th>
<th>Test temperature, °C</th>
<th>Stress, N</th>
<th>Tensile strength, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC+6% vol. MWCNT</td>
<td>1500</td>
<td>400</td>
<td>152,3</td>
</tr>
<tr>
<td>SiC +6% vol. MWCNT +1% wt. MgAl$_2$O$_4$</td>
<td>1500</td>
<td>400</td>
<td>177,5</td>
</tr>
<tr>
<td>SiC +6% vol. MWCNT +1% wt. MgAl$_2$O$_4$+ +1% wt. Y$_3$Al$<em>5$O$</em>{12}$</td>
<td>1500</td>
<td>400</td>
<td>251,4</td>
</tr>
</tbody>
</table>

Adding magnesium aluminate spinel to silicon carbide facilitates process of sintering and increases the mechanical properties of the composite. The addition of yttrium-aluminum garnet leads to suppression of growth of silicon carbide grains during sintering. The grain size of SiC was 0.5-2 μm.

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Investigations on new carbon-based nanohybrids combining carbon nanotubes and metal oxide nanoparticles

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During the last decade composite materials have spurred a large interest and with the rising of nanotechnology, the development of new nanocomposite materials promoting new properties has taken a step forward. By combining different classes of materials, it is possible to obtain nanocomposites exhibiting properties of the individual materials along with new characteristics as a result of hybridization. Hybrid materials consisting of non-functionalized multi-walled carbon nanotubes (MWCNTs) and metal oxide nanoparticles (MONPs) (HfO2, ZnO) have been synthesized. Free-standing HfO2 with an average size of 2.6 nm have been synthesized and demonstrated unusual optical properties and a strong photoluminescence emission in the visible region originating from surface-defects.

Transmission electron microscopy studies show that these NP decorate the MWCNTs on topological defect sites. The electronic structure of the C-K edge in the nanocomposites was probed by Electron Energy Loss Spectroscopy, highlighting the key role of the MWCNT growth-defects in anchoring MONPs. A combined optical emission and absorption spectroscopy approach illustrated that in contrast to HfO2 NP, the metallic MWCNTs do not emit light but instead expose their discrete electronic structure in the absorption spectra. The photoluminescence of the nanocomposites ZnO_MWCNTs, HfO2_MWCNTs and ZnO:HfO2_MWCNTs have been studied. The photoluminescence of the nanocomposites indicates features attributed to combined effects of charge desaturation and charge transfer to the MWCNTs with an overall reduction of radiative recombination. Finally, photocurrent generation under UV-visible illumination suggests these hybrid system have promising features for being used as flexible nanodevices for light harvesting applications.

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Keywords: Carbon nanotubes, Nanoparticles, Photoluminescence, Photocurrent, ZnO, HfO2
Electron paramagnetic resonance (EPR) investigated samples were obtained by sintering powders of detonation nanodiamonds (DND) at a temperature of 1800 °C and a pressure of 8 GPa for 20 seconds. Register of the EPR spectra was carried out with a modified spectrometer «RadiPan» SE / X-2543 X-band.

In the studied samples sintered tablets of detonation nanodiamond powder was observed asymmetric EPR signal Dyson form (Fig. 1 shows a signal from the sample №1), which may indicate the presence of conductive structures in the microwave range. Signal parameters vary depending on the technological conditions of synthesis of initial nanodiamond powder. The width of the EPR signal lines for different samples varies (6 - 9) ± 0,01 G. The value of the g-factor ranges 2,00063 - 2,00119. The original signal is isotropic, i.e. the EPR spectra parameters (g-factor, line width, amplitude) are not changed by the rotation of the sample in a magnetic field.

After one year of storage sintered nanodiamond powders under laboratory conditions, EPR spectra parameters were changed. Thus, the sample №1 line width was reduced to 8.4 ± 0.01 G, g-factor decreased to 2.00072 and 2.00065 while the polarizing direction of the magnetic field parallel and perpendicular, respectively, the compression of the sample plane. The difference in the g-factor in two perpendicular directions shown that paramagnetic centers giving the signal are in the anisotropic environment.

Figure 2 shows the change in the spectroscopic splitting factor in the test (b) samples sintered nanodiamonds from baseline samples (a) of nanodiamond powders (axis of paramagnetic centers concentration corresponds to the of the initial sample of nanodiamonds).
3D Computer Model of Ti-C-V T-x-y Diagrams with Three-Phase Reaction Type Changing

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Among the 3-phase regions in T-x-y the diagrams are encountered such, in which a change in the type of reaction occurs. The fact that change of the phase reaction type is not at constant temperature, but in a certain temperature interval, was for the first time proven in [1,2]. Later it was shown [3,4], that the special surface corresponds to the mass increment sign change in the of each of three coexisting phases. This surface is ruled and is formed with the horizontal generating line (since the reaction from the 3-phase is converted into the 2-phase on this surface, i.e. it is possible to name it as the 2-phase reaction surface). The algorithm of the construction of the 2-phase reaction surfaces in the 3-phase regions is included to the software of the T-x-y diagrams 3D models design [5].

Change of the reaction type in the system Ti-C-V along the univariant curve, which corresponds to interaction of liquid and solid solutions Ti(V) and TiC(C0.9V), with the minimum with 1575°C was fixed experimentally [6]. However, in contrast to the assertion into [6], that “the tie-line, that corresponds to this minimum, is the boundary between the peritectic part of the diagram and the eutectic”, in reality, the temperature of this transformation is not constant (1575°C) for the different alloys, but it changes over the surface of 2-phase reaction with L and Ti(V) with zero increment of the mass of TiC(C0.9V). Furthermore, with the aid of the Ti-C-V T-x-y diagram 3D model it was determined that the change in the phase reaction type from the peritectoid to the eutectoid has a place three times in the region Ti(V)+TiC(C0.9V)+CV2 with three surfaces of 2-phase reactions.

Keywords: reaction type changing, carbides, phase diagram, titanium, vanadium, mass balances

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