Edited by:
DSL CONFERENCE

# ABSTRACT BOOK

26-30 June, 2016 / Split, Croatia / Radisson Blu Hotel

## 12th INTERNATIONAL CONFERENCE ON DIFFUSION IN SOLIDS AND LIQUIDS - DSL2016

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# PREFACE

It is our great pleasure to welcome you to the **12th INTERNATIONAL CONFERENCE ON DIFFUSION IN SOLIDS AND LIQUIDS: DSL2016** (Split, Croatia – from 26-30 June, 2016).

DSL-2016 aims at attracting a balanced portion of delegates from academia, industry and research institutions and laboratories involved with research and development work. In doing so, the conference provides a binding platform for academics and industrialists to network together, exchange ideas, provide new information and give new insights into overcoming the current challenges facing the academics and the industrialists relating to mass transfer, heat transfer, microstructure and properties, nanodiffusion and nanostructured materials.

I would like to thank the Organising Committee members and members of the Local Committee for their help in contributing to the successful organisation of this meeting and especially give thanks to **Prof. Hrvoje Gotovac (Split University)** for his support to help participants to get VISA's from the Croatia Embassies.

I would also like to sincerely thank the organisers of the SPECIAL SESSIONS for their great work!

A special "thank you" as well to Professor Graeme Murch, Professor Ali Shokuhfar and Professor João Delgado, co-chairs of DSL-2016, for the excellent work, significant inputs and support to this conference.

You all made the way to Split and I would like to personally thank you and all delegates for the decision to attend DSL 2016. I hope that you will find the meeting very useful for your work and business, as well as a useful forum for obtaining new knowledge.

Have fun learning and meeting new people!

See you again in 2017, in VIENNA, Austria!

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Professor Andreas Öchsner
**DSL CONFERENCES – Chairman**

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12th INTERNATIONAL CONFERENCE ON DIFFUSION IN SOLIDS AND LIQUIDS - DSL2016

# ABSTRACT BOOK

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# PLENARY LECTURE

**VIP008**
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## Thermodynamics beyond Equilibrium and its Application to Diffusion

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Thermodynamics is a science concerning the states of a system whether it is stable, metastable, unstable or any states between 1, far beyond the equilibrium states only believed by some. In this presentation, the thermodynamic fundamentals are reviewed and discussed in terms of theory, modeling, and applications along with the impact of first-principles calculations based on the density functional theory 2. Our recent activities on thermodynamic instability and the associated extraordinary physical properties are presented, including the 100 year old mystery of INVAR effects 3. Furthermore, the prediction of diffusivity in solid phases based on thermodynamic energetics is discussed 4,5.

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**VIP005**
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## Diffusion in Glassy and Quasicrystalline Metallic Alloys

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This talk reminds the reader at first to some basic facts about glassy and quasicrystalline states [1]. Glassy metals, also called metallic glasses, comprise conventional and bulk metallic glasses and are usually produced by vitrification of undercooled melts. Quasicrystalline alloys are ordered but with icosahedral,

decagonal and dodecagonal rotational symmetries, which are not compatible with rotational symmetries of classical crystallography. Quasicrystalline alloys usually occur as equilibrium phases in some multicomponent alloys. In some cases quasicrystalline single-crystals could be grown.

Our current understanding of diffusion in glassy metals and quasicrystalline alloys is reviewed by comparing the temperature dependence of diffusion -- its common features and its differences -- to that of corresponding crystalline metals. We mention available studies of the pressure dependence and the isotope effect and we discuss tracer diffusion and viscosity diffusion for a bulk metallic glass and its undercooled melt. Computer simulations of atomic jump processes indicate that the diffusion mechanism in metallic glasses differs from that in crystalline metals and involves thermally activated, highly collective (chain-like or caterpillar-like) diffusion jumps. We also remind the audience to positron annihilation studies, which – in addition to diffusion studies -- indicate that diffusion in quasicrystalline alloys is vacancy-mediated.

[1] H. Mehrer, Diffusion in Solids – Fundamentals, Materials, Diffusion-controlled Phenormena, Springer Series in Solid State Science 155, Springer Verlag, Berlin Heidelberg, (2010); see especially Chapter 21.

# SPECIAL SESSION 1
# FLUID FLOW, ENERGY TRANSFER & DESIGN (SS1)

**DSL107**

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## Cooling Capability of Water Blocks for High Power Applications

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We designed and analyzed various water block configurations for high power applications, particularly laser diodes operations. The heat dissipated from laser diodes is normally more than half of the electrical power supplied, this can be a demanding problem to deal with, especially in the high power field. In order to maintain good performance and long life time of laser diodes, it is highly recommended to operate them within the acceptable temperature range, i.e. +15 to 35 °C; therefore a good and efficient cooling system is desirable.

We focus on conventional block for easy fabrication yet able to handle high power within 1U constrain. First, we design a 3 dimensional (3D) model for the water cooling block that is capable of handling 30 laser diodes arranging in series and parallel fashion that dissipate a total heat load of 2.1 kW. The model incorporates various design parameters, such as number of water channels, channel width, and block thickness. We then use 3D computational fluid dynamics (CFD) analysis to simulate the heat transfer between the heat source and cooling surfaces as well as the 3D temperature distribution of the block.

The analysis has shown that the performance of the cooling block is inversely proportional to the channel width. With the same channel width and inlet water flow rate, an optimum number of the water channels can be obtained. Further increase the number of channels does not improve the block performance, which we believe primarily due to the limitation of the inlet flow rate.

With optimum design parameters, the water block is able to handle the kW heat load while maintain the temperature within the acceptable range. This analysis is important and useful for high power applications where the heat must be managed properly to ensure good system performance before the actual fabrication of the block.

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**DSL126**

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## Development of Heat Pipe Thermal Protection Studies for Hypersonic Leading Edges

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Leading edges of hypersonic vehicles are the critical design structures because of the severe thermal environment they subjected to during flight. The high local heating at the stagnation regions cause high temperature, severe thermal gradients and high stresses to the materials, which threatens its safe flight. As a semi-passive thermal management concept, the heat-pipe structure with high thermal conductance based on the phase change and flow of a working fluid is utilized to redistribute the heat from the leading edges,

thereby eliminating local hot spots. The principles and characteristics of heat pipe used in hypersonic leading edges are introduced in this paper. The fabrication and thermal performance of three types of heat pipe cooled leading edge (the heat pipe embedded model with a “D” cross section, the integral model with wedge geometry, and the platelet model with rectangular cross section) are discussed respectively. Besides, the utilization of neutron imaging to capture the startup operations of high temperature heat pipe is introduced as a new method of experiment researching. Finally, a numerical approach taken to investigate the capillary and heat transfer performance of heat pipe wick structures, which is the critical component to provide the driving force for the flow of working fluid, is also discussed.

**DSL127**
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### Research of Mixed Diffusion and Combustion Characteristic in Micro-Slit Plate Splash Injector

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Platelet injector is a new type of liquid rocket injector designed by Aerojet liquid rocket Company in the 1970s. It was widely used in low thrust attitude control engine because of several unique features including excellent response, simple construction, potential high performance with reduced chamber lengths. In order to investigate the performance of a single-element LO2/GCH4 splash platelet injector, the flowing and combustion processes of different nozzle width were numerically simulated by solving the Navier-Stokes equations, the chemical reaction was modeled with Arrhenius formulation, along with detailed 14-step chemical dynamic model. The results show that two small scale oxygen-rich and methane-rich recirculation zones are produced near the nozzle, it has an great influence on mixing and combustion, which also serves as a flame holder. When the nozzle width L1/L=0.6,0.7,0.8,0.9, the spray angleθ=52.2。,54.0。,34.5。,3.5。 respectively. Spray angle is helpful to the mixed diffusion and combustion, made a short combustion length, a higher heat flux of the wall. Combustion efficiency existed an optimum value when L1/L=0.7.

**DSL129**
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### Analysis of the Fluid Diffusion Characteristics in Platelet Pre-mixer

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Keywords: Pre-mixer, Platelet, Mixing uniformity, Diffusibility

The combustion on an air-heater of low total temperature is usually performed under the oxygen-enriched conditions, and the exceeded air will have adverse effects on the stability of ignition. In order to improve the mixing characteristics, the platelet mixer is used as the pre-mixer of air heater in the ground experiment systems of high thrust rocket engine. Platelet mixer is a type of high-efficient equipment which can mix fluids through platelet micro-channels cross-bedded injection technology. It overcomes disadvantages that the fluid is centralized around the inlet. Meanwhile it can improve the diffusibility prominently. In the paper, the diffusibility of liquid-oxygen and air in pre-mixer is analyzed. The strain rate is considered to characterize the diffusibility of fluid and the mixing uniformity is predicted by means of kinematics. The results indicate that there exists high strain rate in platelet profile. The fluids on the condition of high strain rate have strong diffusibility and diffuse rapidly in the mixer. Then, the strain rate and mixing uniformity with different flux are analyzed. The mixing uniformity increases with the increase of flow flux whose alteration regularity is consistent with the strain rate.

**DSL133**

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### Influence of Insulating Performance of the Liquid Oxygen Tank on Diffusion and Transpiration

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The liquid oxygen(LOX) is a kind of non-toxic and non-polluting clean oxidizing agent, so it become a prefect and widely used combustion improver of the carrier rocket in aerospace. Working at low temperature, the transpiration of the propellant brings two main problems: the mass loss of fuel and the pressure control diffculty of tank ullage. Considering the influence of heat leakage of the LOX tank wall and the transpiration at gas-liquid interface, the computational fluid dynamics (CFD) model to study the physical fields in different insulating ways of the LOX tank is set up in this paper. The gas part in the tank ullage is dealt with the ideal-gas model and the gas-liquid interface is dealt with the fluid volume function (VOF) multiphase flow model. The computational results show that, with the increase of the tank surface heat flux, temperature stratification in the tank ullage becomes more obvious, and the transpiration becomes stronger at gas-liquid interface. Once the heat flux reach to a certain extent, the LOX output flow rate is seriously affected. These results can provide an important theoretical support to the design of the propellant tank of carrier rocket.

**DSL135**

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### Heat Transfer across a Vertical Plate Separating Forced and Free Convection

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A theoretical study is presented in this paper to investigate the conjugate heat transfer across a vertical finite wall separating two forced and free convection flows at different temperatures. The heat conduction in the wall is in the transversal direction and countercurrent boundary layers are formed on the both sides of the wall. The governing equations of this problem and their corresponding boundary conditions are all cast into a dimensionless form by using a non-similarity transformation. These resultant equations with multiple singular points are solved numerically using a very efficient singular perturbation method. The effects of the resistance parameters and Prandtl numbers on heat transfer characteristics are investigated.

**DSL141**

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### Photothermaly Detected Heat Diffusion as a Tool to Study Materials Properties

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Biofuels and reservoir rocks have been largely studying nowadays for their importance as energy resources. One is an alternative to the other but both are still needful. Thus the knowledge of their thermal properties and behaviour is fundamental for those dealing technologically with them. In this work we are applying a methodology of detection of heat diffusion in liquids and porous solid materials based in some configurations of the photothermal techniques. To obtain the dynamic thermal properties, namely diffusivity, conductivity and effusivity, and the volumetric heat capacity of the systems, one have applied the Open Photoacoustic Cell, the Photopyroelectric configurations and the Temperature Detection under Continuous Illumination techniques. Knowing sample density one can also evaluate its specific heat. The methodology is corroborated with the studies on several biodiesel systems either from vegetable oils or animal fat, and biodiesel/diesel mixtures as well. For porous solid samples some sedimentary rocks, important as petroleum reservoir, namely sandstone, black shale and marlstone, were investigated. An overall analysis of the results presented in this work permit us to point the photothermal methodology as a useful tool in the study of solid and liquid materials.

[1] A.O. Guimarães, C.G. de Souza, E.C. da Silva, M.E. Soffner, A.M. Mansanares, H.J. S. Ribeiro, A.A.G. Carrasquilla, and H. Vargas, Int. J. Thermophys., 36, 1093 (2015).
[2] A.O. Guimarães, D.A. Viana, T.C. Cordeiro, J.A. Sampaio, E.C. da Silva, R. Toledo, H.J.P.S. Ribeiro, A.A.G. Carrasquilla and H. Vargas, Mar. Pet. Geol., 43, 121 (2013).
[3] A.O. Guimarães, F.A.L. Machado, E.C. da Silva and A.M. Mansanares, Thermochim. Acta, Vol. 527, 125 (2012).

**DSL174**

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## Ambient Blooming Behaviour and Mechanical Properties of Vulcanized Natural Rubber Loaded with Non-ionic Surfactants

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This study investigated the ambient blooming behaviour of additives and mechanical properties of vulcanized natural rubber (VNR) loaded with non-ionic surfactants coco diethanolamide (CDEA) and glycerol monostearate (GMS). Taguchi design of experiment (DOE) and analysis of variance (ANOVA) were used to determine the significant main effects of additives on bloom amount and mechanical properties of VNR. Optical images of VNR surface show evidence of blooming. Attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) spectra of VNR surface confirm the presence of chemical functional groups of stearic acid (SA), paraffin wax, used oil, CDEA, and GMS. The amount of bloomed additives (MB) versus square root of time (t1/2) yields two linear regions corresponding to distinct blooming rates [1]. High loadings of zinc oxide (ZnO), SA, paraffin wax, used oil, and mercaptobenzothiazole disulfide (MBTS) significantly increase the bloom amount due to migration of excess unreacted additives to the surface of VNR [2]. On the other hand, high amounts of sulfur (S) and diphenylguanidine (DPG) significantly decrease blooming. Sulfur and mercaptobenzothiazole (MBT) are found to significantly improve the Shore A hardness, tensile modulus, and compressive properties of VNR. Sulfur acts as crosslinking agent, while MBT serves as accelerator which could have increased the crosslink density and improve the mechanical properties of VNR [3]. Meanwhile, increased loadings of ZnO, used oil, and CDEA significantly lower the mechanical properties. Accumulation of migrated additives at the rubber surface [4] and matrix softening could result to decreased mechanical properties of VNR.

[1] H. Yoshio. Rubber Chem. Technol., 83, 46 (2010).
[2] J. Arabit and B. Pajarito, Adv. Mat. Res., 1125, 64 (2015).
[3] M. Akiba and S. Hashim, Prog.Polym. Sci., 22, 475 (1997).
[4] F. Saeed, A. Ansarifar, R. J. Ellis and Y. Haile-Meskel, Adv. Polym. Tech., 32, 153 (2013).

**DSL177**

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## HF and Hcl Diffusivity and Solubility in Polymers Constituting Silicon Wafer Handling and Storage Carriers

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Polymeric materials are naturally able to incorporate and/or release molecular compounds. This molecular transfer is, according to the solution-diffusion model (polymer membranes), driven by the multiple concentration gradients caused by the surrounding environment and occurs ideally until the system polymer-environment is fully balanced. The three parameters characterizing these processes are diffusivity, solubility and permeability; their magnitudes depend on the polymer itself.

In the microelectronic industry, the silicon substrates (also known as wafers) upon which microchips are manufactured inside a cleanroom are in fact stored and led to the different fabrication steps within polymer-made containers called FOUPs (Front Opening Unified Pod). Several widely used corrosive gases, such as hydrogen halides, are known to seriously impact sensitive metal layers causing corrosion. Wafers stored inside a FOUP immediately after a fabrication step may still contain residual levels of hydrogen halides that are quickly released to the inner atmosphere of the FOUP, thus penetrating its polymeric compounds. This contamination may subsequently be outgassed upon the introduction of other wafers presenting sensitive metal layers causing severe yield losses [2, 3].

In order to manage these industrial issues, sorption and desorption mechanisms of HF and HCl related to FOUP environment control employing different polymers have to be investigated. The solubility and diffusivity coefficients presented in this work were obtained by means of the sorption-kinetic method applied to polymer thin films, which are exposed to the environmental conditions commonly found in semiconductor fabs (1 atm, 40% RH, 21°C, [HX]air = 100 ppbv range). The contamination retained by the polymers is extracted using hot DIW extraction and then analyzed by ionic chromatography. In continuation of the previous works dealing with HF in pure polymers [4], this study focuses firstly on HF transport coefficients in 100 μm thickness composite polymers that put in evidence the influence of various carbon loads onto the pure polymers (carbon powder, nanotubes). Pure polymer films have also been exposed to HCl: the resulting solubility and diffusivity coefficients shown in this work are thus compared to those referred to the HF case. Furthermore, the desorption phase in pure polymers has been studied in order to assess the reversibility of the sorption process. In summary, HF and HCl diffusivity and solubility in pure and composite polymers for the sorption and desorption steps will be compared and discussed regarding sorption and outgassing behaviors and transfer to stored wafers.

[1] P. González-Aguirre et al. Microelectronic Engineering, 105 (2013), 113-118
[2] T. Kamoshima et al. ECS Transactions, vol. 21 (4), p. 573-577, 2008
[3] T.Q. Nguyen, H. Fontaine, Y. Borde, V. Jacob. Proceeding of SPCC 2011, Austin, Texas, USA, 2011
[4] P. González-Aguirre et al., Diffusion in Solids and Liquids Conference, Munich, 22-26 June 2015

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**DSL179**
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## Self- Diffusion in Ionic Liquids

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Ionic liquids play very important role in many fields such as chemical, industry, biology etc.

Therefore, it is quite important to study the self-diffusion coefficient as a function of temperature in ionic liquids. In the present paper, we have studied the self-diffusion coefficient in four ionic liquids in temperature range of 298.15-343.15 K based on a model. The computed results in case of each liquid is found in very good agreement with experimental data as the root mean square deviation representing the goodness of fit is of the order of 10-12 m2/s.

**DSL192**
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## Electroosmotic Flow in a Circular Microcapillary with two Immiscible Fluids

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The electroosmotic flow of two immiscible fluids within a uniform microcapillary is theoretically studied, where temperature effects are taken into account. There is an annular layer of a non-Newtonian liquid, whose rheological behavior follows the well-known power–law model, adjacent to the inside wall of the capillary. This layer surrounds an inner flow of a conducting liquid that is driven by electroosmotic forces. This inner fluid flow exerts an interfacial force, dragging the annular fluid. The viscosity coefficient of both fluids and the electrical conductivity of the inner fluid are temperature-dependent. For carrying out this study, the governing equations describing this flow are obtained by using the lubrication theory, which are presented in dimensionless form. At the interface between both fluids, the shear and Maxwell stresses are taken into account. The nondimensionalized governing equations are solved by a conducting a regular perturbation technique. It is shown that this electroosmotic flow is controlled by several the dimensionless parameters: the ratio of viscosities of both fluids, the competition between the thermal conductivity of the inner and that of the surrounding fluid, the index of the power-law fluid, the competition between the heat generated by Joule effect and the heat dissipated through the external wall of the capillary and geometrical parameters. For values of the dimensionless parameters involved in this analysis, it is found that the volumetric flow rate can be increased by about 7 % when taking into account the temperature effects, and from 7 to 20 % in comparison with the case as if there were no annular liquid film.

**DSL193**
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## Three-dimensional Thermal Effects during Mixed Convection Heat Transfer around a Confined Circular Cylinder in Contra-flow

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An experimental work is conducted on laminar opposing mixed convection to study the local and average heat transfer from a circular cylinder confined inside a vertical closed-loop downward rectangular water channel. The experimental setup uses an aluminum cylinder subjected to a constant wall heat flux boundary condition while the other bounding walls are insulated and adiabatic. The cylinder is placed horizontally at the mid-plane with a 40% blockage ratio (BR, ratio between the cylinder diameter and the thickness of the rectangular section) and a cylinder aspect ratio (AR, ratio between the length and diameter of the cylinder) of 6. The investigation covers the Reynolds number range from 170 to 260, Prandtl number of Pr = 7, and values of the modified Richardson number, Ri* = Gr*/Re2, from 3.6 to 142.5. Time-average local temperature distributions with circumferential position are obtained at nondimensional distances of 0.25, 0.5, and 0.75 of the cylinder span. The results indicate that at high opposing buoyancy, because of the three-dimensional behaviour, the space-averaged surface temperatures and overall Nusselt number vary along the span and are not symmetric with respect to the centerline of the cylinder for all values of the modified Richardson number studied. The analysis brings out the significance of the end conditions and buoyancy-induced secondary flow on the local and overall heat transfer characteristics of the bounded flow.

**DSL202**

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## Enhanced Mathematical Modeling of DOP Plasticizer Migration from PVC into Liquid (Methanol)

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Polyvinyl chloride, commonly abbreviated PVC is a rather hard and rigid material. In order to extend its use it requires additives during the manufacturing process which are referred to as plasticizers, among them the Dioctyl Phthalate (DOP). This latter has a tendency to escape from the PVC matrix specially when in contact with liquids. In order to analyse this phenomenon we use the classical equations of mass diffusion (Fick's second law). However this method leads to results which are not always in good agreement with experimental ones, especially when the concentration of plasticisers is high. This situation requires the elaboration of an enhanced mathematical model that can deal with this particular type of forced diffusion characterized by the contact of a liquid and particular boundary conditions. The validation of the proposed model was obtained by comparing the theoretical results obtained with the model and those obtained by experiments.

Key words: DOP, forced diffusion, mathematical model.

**DSL203**
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### Numerical Study of Laminar Natural Flow Transitions in Rectangular Cavity

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The present work is proposed a numerical parametric study of heat and mass transfer in a rotating vertical cylinder during the solidification of a binary metallic alloy. The aim of this paper is to present an enthalpy formulation based on the fixed grid methodology for the numerical solution of convective-diffusion during the phase change in the case of the steady crucible rotation. The extended Darcy model including the time derivative and Coriolis terms was applied as momentum equation. It was found that the buoyancy driven flow and solute distribution can be affected significantly by the rotating cylinder. The problem is governed by the Navier-Stokes equations coupled with the conservation laws of energy and solute. The resulting system was discretized by the control volume method and solved by the SIMPLER algorithm proposed by Patankar. A computer code was developed and validated by comparison with previous studies. It can be observed that the forced convection introduced by rotation, dramatically changes the flow and solute distribution at the interface (liquid-mushy zone). The effect of Reynolds number on the Nusselt number, flow and solute distribution is presented and discussed.

Keywords: Vertical Solidification; Finite Volume method; Numerical analysis; Heat and mass transfer; Phase Change; Bridgman Growth

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**DSL209**
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### Computational fluid dynamics analysis of Autonomous Underwater Vehicles

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An Autonomous Underwater Vehicle (AUV) is a robot that operates underwater without physical communication with the land and without real-time control by human operators. The technological and economical potential of AUVs are appreciable, especially in activities such as search and exploration of oil and natural gas in deep waters. Given this context and knowing that the study and development of these vehicles are essential to large areas such as modeling, identification and control of marine systems, this work aims at the study of computational fluid dynamics of Autonomous Underwater Vehicles.

This work focuses on the analysis of the parameters of the Myring Equations to describe the AUV's geometry aiming the reduction of the vehicle's drag force and consequently its energy consumption. The numerical study considers a single fluid flow analysis in different geometric configurations of an AUV hull. Results of speed, pressure distribution and drag coefficient are presented and analyzed. ANSYS CFX® software is used to perform the numerical study of fluid dynamics of the AUV hull. The methodology adopted and modeling obtained are validated with experimental data.

**DSL223**
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## Development of Automatic Grid Generation Program for Flow Analysis of Flow Passage between Turbine Blades with Geometric Periodicity

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In Computational Fluid Dynamics(CFD), governing equations of flow phenomena are solved numerically at a finite number of calculation points (or cells), and the accuracy of the resultant flow solutions as well as the time required to obtain them are strongly affected by how those calculation points(or cells) are distributed, that is what grid generation is for. Grid generation is still the most time-consuming and labor-intensive process among several entities composing CFD procedure to solve flow fields.

In this paper our research efforts to develop an automatic grid generation program based on problem-oriented approach would be described. [1] There can be many types and categories of problems, and our main interest in this research is focused on the case of flow passage between turbine blades, which can be found in the problems such as turbomachinery and wind-energy turbines. This type of problems is characterised as its geometric periodicity of physical domain in its circumferential direction, and this feature is utilised in automatically generating grid system for the target region. Grid type chosen in the current research is multi-block structured grids, and algorithmic approach and numerical procedure for automatic grid generation would be described. Some resultant grid system for sample problems and their flow solutions would be demonstrated and discussed.

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**VIP010**
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## Numerical Study of Heat Transfer and Pressure Drop Applied to Laminar Non-Newtonian Fluid Flow Over Ducts with Elliptic Cross-Section

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This work studies numerically the heat transfer and pressure drop in laminar non-newtonian fluid flow over ducts with elliptic cross-section. The objectives are to maximize the density of heat transfer and minimize the pressure drop of the flow. The fluid is viscoplastic and the relationship between shear stress and strain ratse follows the Herschel-Bulkley model. The flow is incompressible, laminar, two-dimensional and steady state. It is investigated the effect of the power-law index, n, and the ratio between the semi-axis of the elliptic cross-section in the heat transfer density and pressure drop. The Reynolds, Bingham and Prandtl numbers are kept constants and equal to 1, 1, and 0.72 respectively. The numerical solution is achieved using software based on the finite volume method. The results show that the maximal heat transfer density and the optimal ratio between the elliptic cross-section semi-axis decrease as the power-law index increases. The optimal ratio between the elliptic cross-section semi-axis which minimizes the flow pressure drop increases as the power-law index increases. However, the minimal dimensionless pressure drop decreases as the power-law index increases.

Keywords: geometric study; power-law index; viscoplastic fluid; elliptic cross-section, semi-axis ratio.

**VIP042**
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## Aerosol Transport in a Bifurcating Tube at Cyclic Flow

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Branching tube flow is a common feature of many fields of science and technology, and occurs both in animate and inanimate systems [1]. The transport of aerosol particles is of particular importance in industrial flow networks but also for the respiratory tree [2].

In this analysis a 3-D numerical study is performed to investigate transport and deposition of aerosol particles in branching tubes. Bifurcation tubes designed according to Hess-Murray law [3] but with different branching angles are analyzed. This study covers cyclic flow conditions at frequencies of 0.25 Hz, 0.50 Hz and 0.75 Hz, Stokes numbers ranging between 0.03 and 0.25, and Reynolds numbers up to 3000.

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**VIP053**
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## Constructal Self-Cooling Structures with Mechanical Strength

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In this paper, we show how the design of vascular channels affect the mechanical strength of a self-cooling structure which is simultaneously subjected to heating load and mechanical load. Radial circular and semi-circular vascular channel designs were compared. The effect of channel cross-section on the coolant mass flow rate, peak temperature and peak stresses are documented. The fluid flow resistances to the coolant flow is smaller with circular channel cross-section. However, the peak temperature is smaller with semi-circular channels. This result uncovers that even the convective resistances are the smallest with circular-cross section, overall thermal resistance is smaller in semi-circular channels for the compared designs. In addition, this paper documents how the channel cross-section and the pressure difference in between the inlet and outlet boundaries of the coolant fluid flowing affect the peak von Mises stress. The peak stress is smaller with semi-circular channels when the pressure drop is less than 500 Pa for the given designs in comparison with circular channels. In addition, the effect of mechanical load on the stress distribution is also documented. The chief result of this paper is that semi-circular channels provide smaller thermal resistance in comparison to the circular channels for the given parameters, and therefore eliminating some design parameters with previous experience is not always the best option. This result also in accord with the constructal theory because it uncovers that the prescribed design parameters does not always yield the best performing designs. In summary, this paper shows that the designs should be free to morph in order to find the best performing design, i.e. the constructal design.

**VIP039**
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## Evaluation for Long-Term Structural Shading Materials for use in Harsh Environments

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This paper evaluated the performance and durability of leading structural shading materials to be used in the Super Containerized Living Units (Super-CLU's) project to used in harsh environments. Fifteen unique tent fabrics were tested in five different experiments in order to evaluate their strength, resistance to wind, abrasion heat and their heat transmissivity and breathability. A material used by the United States Navy in other locations was used as a control material for the evaluation of the other tested materials. Each fabric was tensile tested in both its warp and weft orientation to create an ‘as received’ baseline condition. Then each fabric was exposed to wind, abrasion, or heat and subsequently tensile tested to observe the degradation that occurred compared to the ‘as-received’ condition samples.

Key Words: Shading Material, Tent Fabrics, Abrasion, Wear, Tensile Strength

**DSL247.1**
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### A Mathematical Model to Evaluate Heat and Mass Transfer in Vertical Channels with Joule Heating

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Accurate evaluation of heat transfer and subsequent mass flow in vertical channels is an immensely popular problem in power industries, where flow patterns determine the rate of heat dissipation and temperature distribution in windings of electrical transformers. Existing models assume uniform heat generation within the core and winding of transformers and therefore calculate the volumetric heat source (Qv), as constant power losses over winding blocks of known volume. However, non-linear coolant properties and complex varying geometrical specification limit these models to typically uncoupled thermal and flow analysis [1].
In this paper, calculation of heat source using coupled thermal and electromagnetic field equations helps in determining electrical conductivity, magnetic flux density, magnetic permeability and anisotropic thermal conductivities of insulating materials as non-linear temperature dependent functions [2]. These acts as input variables in coupled heat transfer and fluid flow models using Navier-Stoke equations to determine the temperature-velocity-pressure formulations in vertical channels with various flow patterns [3-4]. The presented mathematical model is validated against available literature data and is useful in optimization of thermal performance of electrical transformers.

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### Evaluation of Abrasion Resistance, Heat Transfer Effects, and Solar Reflectance of Roof Coatings

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Several acrylic coatings were evaluated as potential coatings for roofing areas on Containerized Living Units (CLU's) existing in harsh environments. This effort is part of a broader work which addresses the use of innovative coatings that can potentially cover the exterior surfaces of the CLU structures. Abrasion resistance, heat transfer, and solar reflectance properties were examined. The purpose of the research is to ultimately reduce the CLU buildings' need for air conditioning.

The abrasion resistance of these coatings to hard particles was tested on three substrates. The evaluation of heat transfer through coated metal containers was performed on the coatings which passed the abrasion test. The final five coatings were tested for solar reflectance, color and gloss. The coatings were analyzed before and after a brief period of environmental exposure and natural soiling at an environmentally harsh location.

The evaluation of all the tests reaffirms that temperature reduction, energy savings, and increased solar reflectance are due to the color of the coatings rather than to specialized additives, and that a substrate-appropriate "white" coating has the potential to significantly affect these attributes for CLU structures.

Keywords: Coating, Cool Roof, Solar Reflectance, Abrasion Testing, Heat Transfer.

# SPECIAL SESSION 10
# HYDROGEN-RELATED KINETICS IN MATERIALS (SS10)

**VIP066**
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## Hydrogen Storage and Thermal Transport in Magnesium- Carbon Nanotubes Composites

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The hydrogenation properties of magnesium – carbon nanotubes (CNTs) composites have been studied in a number of works, yet the impact of composite processing on morphology and atomistic structure of the CNTs has been rarely addressed. In our previous work we demonstrated that severe plastic deformation by equal channel angular pressing causes significant damage to the CNTs [1]. In the present work, we synthesized pelletized porous composites of Mg admixed with 2 wt.% of either multiwall CNTs or graphite. The composites were prepared by high energy ball-milling of Mg powder with carbonaceous additives, followed by uniaxial compression and sintering in hydrogen environment under mechanical constraint. The correlations between ball-milling conditions, composite microstructure, hydrogenation kinetics, and thermal conductivity of the pellets were established. The presence and condition of carbon additives controls the morphology of Mg particles and, consequently, the mechanical stability of the pellet upon hydrogenation cycling. The best combination of hydrogen desorption kinetics, thermal conductivity, and mechanical stability was obtained for the pellets synthesized from the mixture of Mg with 2 wt.% of CNTs processed by 4 h of co-milling. The milling transformed CNTs into carbon nano-particles/nano-onions. These carbonaceous species promote metal nucleation from the hydride phase and allow formation of Mg-Mg bonds between the Mg particles contributing to mechanical stability of the pellet.

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**VIP064**
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## Dynamics of Hydrogen in Nanocrystalline Palladium Studied by Neutron Scattering

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Palladium hydride ($PdH_x$) is one of the most popular metal hydride which has been studied extensively in both basic and industrial research fields for many decades. The remarkable properties are that Pd has a large absorption ability of H atoms and the H atoms are highly mobile in the Pd lattice. It is interesting to examine how the properties are changed as the particle size is reduced to a nanometer-scale. We have investigated the thermodynamic property by an adiabatic calorimetry [1], structure by a neutron diffraction, and diffusion/vibrational dynamics by means of neutron scattering techniques for both bulk and nanocrystalline $PdH_x$. Neutron scattering is a powerful tool to explore the dynamics of H atoms in an atomic scale owing to a large scattering cross section of H atoms. In this presentation, we will mainly show our neutron scattering works.

Quasielastic neutron scattering (QENS) measurements were carried out to probe the diffusion of H atoms. In the bulk $PdH_{0.7}$, a diffusion process, which corresponds to a jump among the octahedral (O) sites (1/2,1/2,1/2), was clearly detected. From the temperature dependence of the relaxation time, the activation energy ($E_a$) was estimated to be 19.9 kJ/mol. As for the nanocrystalline $PdH_{0.5}$, we found a new faster diffusion process with the smaller $E_a$ (10.8 kJ/mol), in addition to the one observed in the bulk sample. Furthermore, our inelastic neutron scattering works revealed that the nanocrystalline $PdH_{0.5}$ exhibits two distinct vibrational excitations which can be roughly described as quantum harmonic oscillators. One is the same as the excitation with $h\square_0$ = 68 meV observed in the bulk sample and the other is the new one with $h\square_0$ = 80 meV. These results indicate that a part of H atoms exist in different environments in the nanocrystalline sample. The H atoms near the surface may sit in deeper potential wells and exhibit the faster diffusion process, while the H atoms in the core of the nanoparticle behave nearly as those in the bulk sample do. The H atoms near the surface may not be accommodated in the O sites but in different interstitial sites, possibly the tetrahedral (T) sites (1/4,1/4,1/4).

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## DSL106

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### Stability of MgH2 Samples after Thermodesorption.

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Mg is the metal useful for storage hydrogen because able to absorb reversibly a large amount of hydrogen and is environmentally harmless. However, high temperature of the dissociation (high stability) of MgH2 and slow kinetic of hydrogenation reaction both are not favourable for wide using of MgH2 for different practical applications. [1, 2]. At present work stability of the MgH2 samples was studied using of the X-ray diffraction method. Two sets of the samples were prepared by thermodesorption (vacuuming at temperature about 4000C) of the MgH2 powder. All obtained samples have two phases (Mg and MgH2) in the different proportion. The first set of the samples in air and at room temperature demonstrate a high stability and not decomposed for several months, containing both Mg and MgH2 phases unchanged. The second set of the samples was cooled down in liquid nitrogen (fast quenched) and heated back until to the room temperature. After several days it was revealed that second set of the samples decomposed and the samples contained practically only Mg phase. This behaviour indicates that at low temperature followed by heating may be occurred phase transition from MgH2 to Mg, coexisting in the samples. These phases have ordered and disordered hydrogen and due to the different diffusion of the hydrogen atoms the proportion of these phases starts to change at diverse temperature. This leads to decreasing of the stability of the second set samples.

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## DSL166

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### Hydrogen Trapping Sites and Hydrogen Embrittlement of Iron and Steels

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The identification of hydrogen trapping sites is important to clarify the function of hydrogen in embrittlement of iron and steels. In the present paper, the relationship between atomic-level changes in iron and steels and macroscopic mechanical properties was clarified. First, the hydrogen trapping sites such as vacancies, dislocations and grain boundaries were determined using thermal desorption spectrometry dheated from lower temperature of 73 K (L-TDS). TEM, positron annihilation lifetime measurement and EBSD were used to characterize dislocation, vacancy and grain boundary structures, respectively. For dislocation trapping in iron specimen, a peak temperature (Tp) at 293 K corresponds to hydrogen desorption from dislocations. The trap activation energies (Ea) between dislocations and hydrogen correspond to 47.5 kJ/mol for edge and 30.1 kJ/mol for screw. For vacancy trapping in iron specimen, the Tp at 383 K corresponds to hydrogen desorption from vacancy clusters (V10). The Ea between V10 and hydrogen corresponds to 68.9 kJ/mol. For grain boundary trapping in iron specimen, Tp at 303 K corresponds to hydrogen desorption from high-angle grain boundaries. The Ea corresponds to 13.2 kJ/mol for high-angle. The evaluation of strain-induced lattice defects associated with hydrogen and their relevance to hydrogen embrittlement are discussed [1], [2], [3].

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**DSL178**

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### The Influence of Plasma Immersion Titanium Implantation on Hydrogenation of Zr-1%Nb

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Zirconium-based alloys are widely used in nuclear industry. However, it is known that relatively small amount of hydrogen as solid solution can lead to hydrides precipitation, which in turn leads to degradation of the mechanical properties. The presence of high temperatures, pressure and mechanical loads induced hydrogen uptake [1]. The ion surface modification is one of the ways to protect zirconium alloys from hydrogen embrittlement. The plasma immersion ion implantation (PIII) has been widely used for ion implantation, thin film deposition, and modifying surface structure as well as material properties [2]. The accelerated ions penetrate into the lattice structure of the target material which resulted in the changing of the chemistry, the microstructure and material properties. In order to study the effect of titanium PIII on hydrogenation behavior of Zr-1Nb alloy, the samples were implanted with titanium ions for different implantation time using a filtered metal vapor vacuum arc source at the bias voltage of 1.5 kV. The surface

morphology and depth distribution of elements were analyzed by scanning electron microscopy (SEM) and atomic force microscopy (AFM), and glow discharge optical emission spectroscopy (GDOES), respectively. The samples were hydrogenated at 673 K temperature and 2 atm pressure for 60 min. The modified layer formed after PIII accumulates hydrogen and makes a hydrogen diffusion barrier. It was found that the hydrogen absorption rate of Zr-1%Nb alloy is decreased with increasing implantation time. The mechanism of hydrogenation of titanium-implanted zirconium was discussed.

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**DSL185**
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## Hydrogen and Hydrogen-Induced Defects in Aluminum Alloys and their Effects on Mechanical Properties

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The effects of diffusible or non-diffusible hydrogen and hydrogen-induced defects on degrading the mechanical properties of aluminum alloys were analyzed. Hydrogen was introduced into the material by electrochemical charging. Hydrogen desorption properties were measured by thermal desorption analysis to establish the state of hydrogen present in the material together with the amount of absorbed hydrogen. Mechanical properties were obtained by tensile tests. Micro-scale defects like vacancies were identified by positron annihilation lifetime analysis, and larger scale defects like voids or pores were observed by X-ray micro-computed tomography.

Prominent peaks around 70 °C and above 400 °C were commonly observed in the hydrogen desorption curves of aluminum alloys that absorbed hydrogen above the solubility limit. The diffusible or non-diffusible nature of absorbed hydrogen was identified on the basis of changes in the positions of the peaks and profile in the hydrogen desorption curves after exposure to room temperature. Vacancies were introduced accompanying hydrogen charging and later transformed into more stable divacancies or vacancy clusters. Stress-strain curves obtained in tensile tests showed that both elongation and flow stress during plastic deformation decreased with increasing hydrogen content. Diffusible hydrogen was found to be responsible for the decrease in flow stress and change in morphology of the fractured surface from brittle to ductile. After dissipation of diffusible hydrogen and transformation of hydrogen-induced defects, the level of flow stress returned to the original state and the fracture surface again became ductile with dimples of larger size. However, ductility was not recovered after the loss of diffusible hydrogen, while the volume fraction of cavities increased due to hydrogen charging. Therefore, the effect of diffusible hydrogen and hydrogen-induced defects on mechanical properties is to lower stress during plastic deformation, while that of non-diffusible hydrogen is to enhance damage to the material leading to the loss of ductility.

**DSL196**
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## On The Impact of Grain Boundary Character on the Hydrogen Segregation

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The context of this work falls within an approach to understand the mechanisms of diffusion and trapping of hydrogen in fcc grain boundaries (GB) and their influence on the GB microstructure. Recently, it has been shown experimentally [1] that the grain boundary character plays an important role in hydrogen diffusion and segregation processes. In this present study, atomistic simulations based on energy minimization method were employed to compute the structural and defect properties of four Ni Σ3 ⟨110⟩ tilt grain boundaries (GBs). The GB properties (energy, excess volume) are treated by the notion of the inclination angle between the two symmetric tilt grain boundaries (STGB): coherent twin boundary (CTB) and incoherent twin boundary (SITB) configurations, including two other asymmetric GBs: Σ3 (110)/(114) and Σ3 (221)/(001).

In order to assess the H absorption efficiency at grain boundaries, the distribution of the H segregation energy as a function of the direction normal to the GB plane will be presented for each GB configuration. Relatively, the hydrogen segregation profile displays a tiny width compared to the energetic distribution of the Ni vacancies within the GB core region. A linear correlation between the atomic volume and the segregation energy of the H interstitial site was established for all the GBs treated in this work, suggesting that GB interstitial sites with large local volume facilitate the hydrogen segregation. Beside this relevant result, more investigations on the mechanisms of H diffusion in Ni Σ3 GBs will also be performed by exploring static jump events and by quantifying barrier and activation energies between the relative stable GB interstitial sites.

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**DSL201**
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## Qualitative and Quantitative Analysis of Hydrogen Distribution in Zirconium Alloy by GD-OES

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Dissolution of hydrogen in metal is characterized by its non-uniform distribution from the surface to the depth. To study the hydrogen distribution in Zr-1Nb alloy glow discharge optical emission spectroscopy (GD-OES) was applied in this work. Qualitative analysis needs the calibration samples with hydrogen, but there are no Zr+H calibration samples. In this work method of Zr+H calibration samples production was created. Automated Complex Gas Reaction Controller was used for samples hydrogenation. To calculate the parameters of post-hydrogenation maintaining of the samples in an inert gas atmosphere the diffusion equations [2] were used. RF GD-OES was used to investigate hydrogen distribution uniformity from the surface to the depth. Absolute hydrogen concentrations in the samples under study were determined on the RHEN 602 produced by LECO. The depth of the craters was measured with the help of a Hommel-Etamic profilometer by Jenoptik Germany, 3D-profiles of the craters were investigated by three-dimensional non-contact profilometer MicroMeasure 3D Station.

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**DSL205**
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## A Critical Review and Experimental Analysis of the Equation Recommended by ASTM G148-97 and ISO 17081: 2004 for the Calculation of the Hydrogen Diffusivity in Metals and Alloys

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Since its discovery in 1864 by [1], the hydrogen embrittlement of metals was target of numerous researches, first being studied by injecting gaseous hydrogen in the investigated metals and alloys. In 1962 [2] developed an electrochemical technique of hydrogen permeation contributing to the advancement of this analysis. The experimental procedure is based on a double-cell, which in one of them it happens the hydrogen production and in another where H is detected. Therefore, three methods are used to produce hydrogen using this technique; the galvanostatic and potentiostatic methods and production under open circuit potential in an acid medium (PCAA). The mathematical equations for evaluate the diffusivity were deducted from the Fick's second law solution, according to the specific boundary conditions for each methodology. However, until the moment it is not known an equation that satisfies the PCAA method. On the other hand the standards [3] and [4] have standardized hydrogen permeation methodology only adopting the calculation of its diffusivity by double-potentiostatic method, not considering, therefore, the other mentioned above.

The present work aims to evaluate the use of mathematical models to determine the hydrogen diffusivity by electrochemical method. For this purpose we used the API 5L X65 and X70 alloys. Furthermore, an experimental methodology suitable for PCAA procedure was developed.

It concludes that: the use of standards proposed by equations [3] and [4], may lead to underestimated values of diffusivity. The sigmoid model proposed for the PCAA method showed a satisfactory fit of the experimental data. diffusivity values were calculated, permeability and solubility of hydrogen suited not only for this case but has also found its application to other cases.

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**DSL227**
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## Anomalous Behavior of Hydrogen Permeability at Low Temperature for Pd-Cu Alloy Membrane with B2 Crystal Structure

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Pd-based alloy membranes are widely used practically for hydrogen separation and purification. Among them, Pd-53mol%Cu alloy membrane with B2 crystal structure exhibits high hydrogen permeability, because the hydrogen diffusivity in bcc crystal is higher than in fcc crystal [1, 2]. Also, the activation energy for hydrogen diffusion is quite small for Pd-53mol%Cu alloy. Therefore, Pd-53mol%Cu alloy membrane with B2 crystal structure is expected to show high hydrogen permeability even at low temperature. However, the hydrogen permeability for Pd-Cu alloy membrane at low temperature has not been investigated in detail. In this study, the hydrogen permeability of Pd-53mol%Cu alloy membrane at around room temperature is focused on.

A plate of Pd-53mol%Cu alloy with a thickness of 60 mm is prepared, and cut into disks with a diameter of about f12 mm. It is confirmed by the XRD analysis that the sample is composed of a single phase with B2 crystal structure. The hydrogen permeation tests are performed from room temperature up to 673 K. The applied hydrogen pressures at feed and permeation sides of the membrane are 0.10 MPa and 0.01 MPa, respectively.

The value of the hydrogen permeation coefficient, f, at 673 K is about 1.3×10-8 mol H2 m-1 s-1 Pa-1/2. After the hydrogen permeation test at 673 K, the sample cell is air-cooled to room temperature, and the time dependence of f is measured. Just after cooled down to room temperature, the f value is about 6.7×10-10 mol H2 m-1 s-1 Pa-1/2. However, during for about 4 days at room temperature, the f value decreases in two steps. It first decreases to about 1.5×10-10 mol H2 m-1 s-1 Pa-1/2 and then becomes about 2.5×10-11 mol H2 m-1 s-1 Pa-1/2 in the second step. Such a slow and large decrement in hydrogen permeability at low temperature for Pd-Cu alloy membrane has not been reported. It is noted that after heating the sample cell again up to 673 K, the f value almost recovers to be the first value, 1.3×10-8 mol H2 m-1 s-1 Pa-1/2.

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**VIP012**
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## Hydrogen Transportation Properties of Palladium Surfaces: Structure Sensitivity and Control

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The transportation of hydrogen (H) between the H2 gas phase and the interior of metals is crucial for H storage and purification as well as for industrial hydrogenation catalysis [1], yet it is entirely undesirable in the context of embrittlement. Despite their technological importance, the atomic-level processes that occur at metal surfaces during H2 absorption and desorption are still not sufficiently understood as to allow controlling these phenomena. We therefore elucidate the microscopic reaction coordinate that connects H2 gas and H in the interior of H-absorbing metals by studying the H2 ingress into and H2 desorption from the (110) surface of a palladium (Pd) single crystal as a structurally well-defined model system. Through a unique combination of H depth profiling via 1H(15N,)12C nuclear reaction analysis (NRA) [2] and thermal desorption spectroscopy (TDS) with isotope-labeled (H, D) surface hydrogen, we demonstrate that the Pd (110) surface exhibits two H2 absorption pathways, which below 145 K precipitate Pd hydride with distinctly different depth distributions and associated TDS features. These two pathways can be attributed to surface penetration at defects and at regular terrace sites [3]. H2 absorption at regular Pd (110) terraces is remarkable in contrast to Pd (100 and Pd(111), where only defects contribute to absorption, suggesting that H2 absorption kinetics are surface structure-sensitive and that Pd(110) may promote H2 absorption due to its relative openness and atomic corrugation [3]. Recent experiments at even more open Pd (210), which shows even higher H2 absorption rates, corroborate this hypothesis. By exploiting this structure-sensitivity through the susceptibility of the Pd (110) surface to well-defined CO-induced (de-)reconstructions, we further demonstrate the possibility to control the desorption dynamics of Pd-dissolved hydrogen through deliberate surface structural modifications [4].

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**VIP049**

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## Kinetics of Hydrogen Isotope Exchange in Traps in Tungsten

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Numerical rate equation models describing the diffusion of hydrogen isotopes and their trapping at defects are the current workhorse for extrapolating results on H isotope uptake and release in W from laboratory experiments to current and future nuclear fusion experiments. Despite the general success of these codes, recent experiments revealed that trapped H or D is removed much more efficiently by isotope exchange than by isothermal annealing [1]. This effect cannot be reproduced by conventional diffusion-trapping codes. Currently, two different mechanisms are proposed that would preserve the reasonable agreement with single isotope experiments, but also accommodate isotope exchange. One is to introduce an exchange cross section, i.e., a process that has a negligible activation energy [1]. The other is the introduction of traps that can accommodate multiple H or D atoms, while the de-trapping energy depends on trap occupancy [2]. Both models can describe low temperature isotope exchange, but the transport behaviour particularly of the mobile, interstitial H respectively D atoms is qualitatively different.

In our contribution we present experiments where we created a large number of traps with a high de-trapping energy for H isotopes by implanting W foils with 20 MeV W ions [3, 4]. We then performed ion-driven permeation experiments where we sequentially loaded the samples with a H or D ion beam accelerated by 1 kV. We compared the measured fluxes of H2, D2 and HD on the permeation side with model calculations for each of the exchange mechanisms described above. The results clearly favor the multiple H trapping option.

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**VIP050**
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## Zooming in to Sub-Nanoscale: Molecular Hydrogen in Confinement Probed by Neutron Scattering

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In porous materials the molecular confinement is often realized by means of weak Van der Waals interactions between the stored molecule and the pore surface. Thus the interaction between guest and matrix can be expected to be sensitively influenced by structure of the confining host and, in particular, the dimension of the confinement cages. We have explored the microscopic mechanism of hydrogen storage in pores of sub-nanometer size in ice-based clathrates and recently synthesized metallic organic framework CAU-1 with help of neutron scattering techniques. The ability of neutrons to probe both microscopic structure and dynamics combined with high sensitivity to hydrogen makes neutron scattering a powerful tool in the exploration of the confined hydrogen behavior.

Thus, in clathrate hydrates we have found that by varying the size of the pore the diffusive mobility of confined hydrogen can be modified in both directions, i.e. reduced or enhanced compared to those in the bulk solid at the same temperatures. In the small cages with a mean crystallographic diameter of 0.8 nm the confinement reduces diffusive mobility by orders of magnitude. The observed localization of the hydrogen in the cage provides for the improved functional properties such as gas degassing temperature and the gas loading pressure. In contrast, in large cages with a mean radius of 0.9 nm hydrogen molecules displays diffusive jump motion between different equilibrium sites inside the cages, visible at temperatures where bulk H2 is solid. The sub-nanometer size of the pores in CAU-1 metallic organic framework promotes the adsorption of hydrogen on the organic linkers and drives the gas intake in CAU-1 even though all metal sites in this material are saturated. The formation of the hydrogen bonds between hydrogen and linkers lead to the shrinking of the host framework structure and as a result to changes in the electronic potential surface inside the pores. This in turn provides for the formation additional occupational positions and increase of hydrogen intake. The onset of guest-guest correlations at higher pressures results in further increase of storage capacity.

**VIP063**
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## Phase Transformations in Thin Metal-Hydrogen Films

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Physical properties of nano-systems such as thin films are different from their bulk counterparts, both in thermodynamics and in phase formation kinetics. In our work we try to separate the different contributions, originating from micro-structure, mechanical stress and the finite size. [1] The contribution of H-induced mechanical stress and the importance of stress-release is discussed in the present paper. We experimentally study this aspect using several in-situ methods like scanning tunneling microscopy [2,3], X-ray diffraction, electrical resistance measurments, electromotive force measurments and optical light transmittance ("hydrogenography").
For eptaxial Nb-H films several critical thicknesses were found. Below about 40 nm, the film transforms coherently into the hydride phase, while above that thickness, incoherent transformations are found. [4] The hydrides morphology during the phase transition changes at this thickness, from growth dominated large hydrides to nucleation dominated small hydrides. In this regime, stress-releasing dislocations are still present. Below a thickness of about 10 nm, ultrahigh stress arises [5] and no phase transition occurs upon hydrogen absorption. This can be attributed to the high stress state which contributes to the chemical potential of the system and reduces the critical temperature of the miscibility gap. We regard these results as beeing of general nature that account for many gas-atom absorbing nano-systems when fixed to rigid stabilizers.

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**VIP065**
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## Hydrogen at Pd Surfaces Studied by He Atom Scattering

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Low energy, neutral beam of He exhibit absolute surface sensitivity and thus is one of the ideal tools to study hydrogens at the solid surfaces. We utilize He atom scattering for the investigation of the hydrogen adsorption and absorption processes of Pd (110) surface, which is one of the most reactive surface for hydrogen absorption [1, 2]. We especially focus on the adsorption and absorption reaction of hydrogen at room temperature, which has not been well pursued so far.
From the in-situ observation of the the He reflectivity during the dose of hydrogen, surface process can be clearly separated into three regions, I. surface adsorption of hydrogen (< 100 L) , II. surface reconstruction of Pd(110) (100〜102 L), and III. hydrogen absorption (>102 L). After the surface reconstruction, surface become covered with missing and added rows showing 3x1 structure, which was confirmed by He-diffraction. Further change in the He reflectivity during the region III was also detected, suggesting the

changes in the surface upon the hydrogen absorption. We also found that this process is strongly dependent on the presence of atomic hydrogen. We discuss the relation between peculiar surface behavior of Pd(110) upon hydrogen doze and its superior hydrogen absorption properties.

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# SPECIAL SESSION 11
# MICROSCOPY, MICROANALYSIS AND THEIR APPLICATION ON MATERIALS (SS11)

**DSL219**
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## Structure and Magnetic Properties of Mechanically Alloyed Fealsn Powder

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$Fe_{72}Al_{26}Sn_2$ alloy is prepared by mechanical alloying of pure Fe, Al, Sn powder using a high energy ball mill, with increasing milling time ranging from 0 up to 24 h. The variation of the morphology and the elemental distribution were investigated at these different stages on various grains of the alloy using a scanning electron microscope with a dispersive energy analyzer. Microstructural changes during the mechanical alloying have been studied by X-ray diffractometry (XRD). Some magnetic properties at room temperature were measured by a vibration sample magnetometer. The electron microscope observations show morphology of powder particles and changes in chemical composition during mechanical treatment. The changes in composition are observed at samples formed by mechanical alloying. Experimental evidences indicate that Al and Sn gradually dissolve in Fe, finally forming a crystalline solid solution. According to the present experiments, nanostructured Fe-Al-Sn alloy were developed with an average crystallite size of about 5 nm by milling for 24 h. With increasing the milling time, the crystallite size decreased for all powders. Increase in microstrain is observed with increasing the milling time. The magnetic measurements show a contrasting saturation magnetization and coercivity (Hc) in Fe-Al-Sn alloys. These variations are explained on the basis of crystallite size and strain variations in the samples during milling.

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**DSL101**
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## Applications of in-Situ Electron Microscopy to Correlate the Materials Properties and the Microstructure

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In-situ electron microscopy techniques are to observe the microstructural change while applying external variables, which can provide enormous amount of information of materials research. In early days of in-situ experiment, external variables applied were limited to simple variables of heat and strain for qualitative observation, which, nowadays, made great progresses with the aid of new materials and fast electronics to obtain quantitative changes while applying external variables. It is now possible to measure physical

properties of materials to provide the fundamentals for the materials computation and other forecasting of material properties, with multiple environmental variables involved.
Talk will be mainly 3 topics, 1) Basic operation and constitution of the piezo system, 2) Combination of the piezo system to be fully working system, and 3) application of the system in the materials research. Piezo materials are the basic building block for the motion related in-situ experiment and “slide-and-stick” and the tube system are mainly used. Simple in-situ system, which can be easily built in a lab, will be introduced to be used in scanning electron microscopy (SEM), which can be extended to TEM. A simple in-situ system in SEM and transmission electron microscopy (TEM) will be demonstrated in measuring the stiffness from a single-crystal cementite sheet, and nano-scale probing to inject current in local area, respectively. Various design and application of in-situ systems, such as nano-grippers to handle nano-materials, electrical conduction system to investigate material transport under electrical bias, defect sensitive cathodoluminescence system, and nano-crystal growth and dissolution in the liquid cell system will be introduced in the later part of the in-situ application.

**DSL109**
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## Microstructure and Grain Boundaries Barrier Layer Effect in ZnO Based Multilayer Varistors

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Multilayer varistors are utilized for low voltage protection against surges caused by electrostatic discharge, atmospheric discharge, overvoltage in integrated circuits [1,2]. The varistor nonlinear current-voltage behavior is attributed to the formation of potential barriers at grain boundaries of semiconducting grains. In this work, multilayer varistors based on doped ZnO were fabricated in a process comprising: preparation of slurry, tape casting of varistor ceramic foils, laser cutting of green sheets, screen printing of internal electrodes, stacking of green sheets, isostatic lamination, dicing into individual varistors, firing of multilayer varistors at 1000-1120°C, deposition and firing of external parallel connections of internal electrodes. XRD, SEM and EDS methods were used for characterization of the microstructure and composition of multilayer varistors. The ceramic layers were well sintered and compatible with platinum based internal electrodes. Characteristic varistor microstructure was observed, comprising micrometer ZnO grains surrounded by continuous nanometric Bi2O3 rich films which are responsible for barrier layer effect and submicrometer spinel grains which are suppressing grain growth due to pinning of grain boundaries. It was found basing on impedance spectroscopic studies performed at frequencies 0.01 Hz-2 MHz in the temperature range from -55 to 450°C that the dominant dielectric response can be attributed to grain boundaries. The current-voltage characteristics of the multilayer varistors were nonlinear with high nonlinearity coefficient of 15-38 and breakdown voltage of 30-120 V, being dependent on temperature and the number and thickness of ceramic layers in the multilayer structure.

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**DSL137**
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## Faceting and Migration of Grain Boundaries and Grain Rotation in Aluminum Bicrystals

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Anisotropy of grain boundary properties can be essential for microstructure development during grain growth in polycrystals. The experiments on aluminum bicrystals and molecular statics simulations demonstrated that the inclinational anisotropy of grain boundary energy is not restricted to the “special” low S CSL boundaries only, but also applies to low-angle tilt boundaries with low index rotation axis: boundaries with misorientations lower 15° did not attain a curved shape (remained straight or formed facets) at any temperature and, thus, did not move under a capillary driving force. An impact of the energy anisotropy on the evolution of the boundary shape and boundary kinetics is confirmed by molecular dynamics simulations. Further simulations showed that the shrinkage of grains with pure tilt boundaries was accompanied by grain rotation. In contrast, grains with the tilt-twist boundaries did not rotate during their shrinkage. An analysis revealed that the boundary structure is crucial for the observed rotational behavior.

**DSL148**

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## Atomic Structures of Strained SrRuO3 (110) Thin Films

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SrRuO3 (SRO) has attracted attentions because of high thermal conductivity, strong chemical stability, and outstanding resistance to corrosion, which makes SRO thin films as valuable electrodes or thin barriers for various oxide devices [1]. On the other hand, its magnetic characteristics strongly depend on subtle changes in atomic configurations such as rotation and/or distortions of RuO6 octahedra, which is caused possibly by substrate strain [2]. Recent advance of annular bright field (ABF) scanning transmission electron microscopy combined with high angle annular dark field (HAADF) imaging allows visualizing light atoms [3].

In this study, we characterize and analyse the structures of SRO thin films epitaxially grown on GdScO3 (110) and SrTiO3 (100) substrates in atomic scales with varying SRO thickness. Thickness of the SRO films is monitored with in situ high-pressure reflection high-energy electron diffraction (RHEED). The crystalline quality and epitaxial arrangement are determined by high-resolution X-ray diffractometer (HRXRD) and transmission electron microscopy. Further quantitative information on octahedral rotation is obtained through ABF-HAADF technique. Moreover, electrical and magnetic properties of the SRO films are measured as well in order to explore structure-property relationship.

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**DSL170**

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## Prediction of Strain Rate of Aluminum and Aluminum/Silicon Carbide using Gray Level Run-Length Matrices

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The microstructural processes occurring in metals and alloys during hot deformation are: dynamic recrystallization (DRX), superplastic deformation, dynamic recovery, wedge cracking, void formation, inter-crystalline cracking, prior particle boundary (FFB) cracking, and flow instability processes. Deformation characteristics of materials are interpreted as follows; in the low temperature (T ≤ 0.25 of melting temperature) and high strain rate regime of 10 to 100 s-1, void formation occurs at hard particles and leads to ductile fracture.

Many researchers used the currently defined statistical approaches to characterize images and extract useful information from the captured images. On the other hand, some researchers still focus on introducing new texture features, which are more suitable for specific tasks [1]. Higher-order statistics (HOS) estimate properties of three or more pixels occurring at specific locations relative to each other. HOS have been already involved in the texture analysis of the early years by Galloway [2] These include interval cover and a series of statistics on the run-length methods. Tsatsanis and Giannakis [3] employed the HOS to solve the invariant texture classification and modeling problems. Gray level run-length matrices (GLRLMs) are popular method of HOS to extract texture features. This paper deals with the relation between texture features of GLRLMs and changes in strain rate.

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**DSL188**

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## Characterization of Micro Arc Oxidized Unalloyed Low-Carbon Steel After Hot-Dip Aluminizing Process

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In the present study, micro arc oxidation (MAO) process was applied to unalloyed low-carbon steel after hot-dip aluminizing (HDA) treatment. HDA treatment was performed in molten pure aluminum bath with a purity of 99.6% for a dipping time of 5 min at 800 °C. MAO process is an effective and efficient technique to modify the surface of valve metals such as Al, Mg, Ti etc., however, it is not a convenient method to form oxide coatings on the steel surface directly [1]. In order to overcome the limitations of MAO process for steel substrates, a thick layer of aluminum was first formed on steel by HDA treatment and later, aluminized steel was oxidized by MAO process in a silicate-based electrolyte. The successive applications of HDA and MAO processes produced a wear- and corrosion-resistant oxide (dominantly alumina phase) layer [2]. Characterization of surface layers formed by the combination of HDA and MAO treatments were made by SEM, EDS and XRD analyses. In order to mechanically characterize the surface layers, Vickers microhardness tests were performed through the thickness of the sample.

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**DSL199**
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## Characterisation of a Ball Milled Femnc Composite

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Fe-Mn-C alloy with (85.8-13-1.2%wt respectively) has been prepared by high energy ball milling method using the milling parameters such as time (30min-72h) and speed 250rpm.The phase composition and microstructure of the milled powders have been investigated by XRD and SEM, milled powders were uniaxially pressed into cylindrical pallets to study the micro hardness profile of each sample and its evolution during ball milling time.

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**DSL206**
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## Microstructural Study of Thin Films Cufe Obtained by Thermal Evaporation of Milled Powder

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Commercial copper and iron powders were used as starting materials. These powders were mixed to obtain Cu70Fe30 supersaturated mixture by mechanical alloying. The milling duration was chosen in such a way to obtain a nanostructured mixture and to form supersaturated solid solution of CuFe, Powder mixture was used to deposit CuFe films on Glass substrate. The deposition was carried out by thermal evaporation under 9 × 10-6 mbar vacuum from an electrically heated tungsten boat filled with supersaturated solutions Cu70 Fe30 powder.
The microscopic study and structural proprieties of nanocrystallin powder and the thin film of CuFe binary alloy were examined using transmission electron microscopy (TEM), scanning electron microscope (SEM), atomic force microscope (AFM) and X-ray diffraction. The milling of powder leads to the formation of nanocrystallin CuFe super saturated solid solution with about 16 nm of crystallite size after 24h of milling. Finally, the cross-sectional TEM images showed that the obtained CuFe layer was polycrystalline film of about 20 nm thick and 10 nm the average crystallites size.

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**DSL228**

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### Analysis of the Microstructure of the Weld Cracking on the Ni Based Super Alloy

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Nickel is an important alloying element that is good for increasing the toughness [1]. Also, nickel improves the low temperature toughness of ferritic steels. For example, 9% Ni steel is used for LNG handling and storage. Mainly, Ni based super alloy is used for filler metal in the welding of 9% Ni steel. However, the weld cracking is common in Ni based super alloy and it can cause the structural issue. In this study, we analyzed the weld cracking with Ni based super alloy. Inconel and Ni based super alloy were used for base material and filler metal, respectively. Phase transition and solidification mode were predicted by thermodynamic calculation and compared with experimental results. Microstructure of weld metal were analyzed scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX) mapping and its mechanical properties were tested. From EDX mapping, it was obtained that Mo and Nb alloying element were segregated around the weld crack and it is a result of the formation of Laves phase which is a cause of solidification cracking.

[1] John C. Lippold, Welding Metallurgy and Weldability, p. 112, (John Wiley & Sons, New Jersey, 2015).

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**VIP013**

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### Deformation Microstructures and Grain Refinement Mechanism of the

### Stainless Steels upon Shot Peening

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Various ferritic (T91, X20) and austenitic stainless steels (SS304, TP347H and super 304H), used widely for the heat exchanger in power plants, were shot peened with the objectives on the enhancement of high temperature corrosion resistance and global properties.

Ferritic SS typically bears carbides (M23C6, ~200 nm) embedded in ferritic (a, ~400 nm) matrix. On the other hand, austenitic SS304 only has coarse g grains. The TP347H has extra distribution of NbC within the coarse g grain. In super 304H, the additional Cu precipitates as the nanosized Cu-particles during the exposure to elevated temperature, providing strain induced precipitation hardening (SIPH) upon service. Therefore, the focus was on the effect of carbides on the deformation microstructures during shot peening and subsequent aging (to simulate service condition).

The results indicate that dominant deformation structures in ferritic SS are the dense dislocation walls, which subsequently transfer to high angle grain boundary, resulting in refinement of the original coarse grain. In case of austenitic SS, the main deformation structures are the deformation twin and martensitic transformation (a¢- and e¢-martensite) [1], which refine the original coarse grain structure. In the process, the carbides (M23C6 or NbC) accelerated the grain refinement progress. The detailed effects of SP on the SIPH of NbC and Cu during post heating will be described in the talk.

[1] Y. He, K. Li, I.S. Cho, C.S. Lee, I.G. Park, J.-I. Song, C.-W. Yang, J.-H. Lee, K. Shin, Applied Microscopy 45 (2015).

**VIP070**
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### In-situ TEM and APT studies of the Local Crystallization of Amorphous NbO2 used in Switching Devices

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2. B.-G Chae; Pohang University of Science and Technology (POSTECH), Republic of Korea
3. C.-G Park; Pohang University of Science and Technology (POSTECH), National Institute for Nanomaterials Technology (NINT), Republic of Korea

Threshold switching is known to be the electrical or thermal-driven phase change mechanism of oxide layer. That is, some oxide can change their structure, and thus resistivity, from the level of insulators to that of metals at above a certain current density. Although the mechanism of threshold switching is not fully understood at present, it can be utilized for a switching device. For the application of the bipolar switching devices to a Resistive-switching Random Access Memory (RRAM), a selection device which can minimize the sneak leakage current is needed. Among various candidates, we chose amorphous Nb-oxide for the selection device due to its superior compatibility with semiconductor structure. We have elucidated the mechanism of threshold switching of the amorphous NbO2 layer by using in-situ TEM probing technique combined with atom probe tomography (APT).

In this study, we observed that, through an ex-situ experiment using TEM, the threshold switching of amorphous NbO2 accompanies local crystallization. The change in I-V characteristics after electroforming was examined by evaluating the composition profile. Atom probe tomography (APT) combined with in-situ TEM probing was performed to understand the threshold switching mechanism in amorphous NbO2. The local crystallization of amorphous NbO2 was validated by the difference in time-of-flight (ToF) between amorphous NbO2 (a-NbO2) and crystalline NbO2 (c-NbO2). We concluded that the slower ToF of a-NbO2 compared to that of crystalline c-NbO2 was caused by both the resistivity difference and trap-assisted recombination.

**VIP071**

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## Applications of in-Situ Electron Microscopy to Correlate the Materials Properties and the Microstructure

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In-situ electron microscopy techniques are to observe the microstructural change while applying external variables, which can provide enormous amount of information of materials research. In early days of in-situ experiment, external variables applied were limited to simple variables of heat and strain for qualitative observation, which, nowadays, made great progresses with the aid of new materials and fast electronics to obtain quantitative changes while applying external variables. It is now possible to measure physical properties of materials to provide the fundamentals for the materials computation and other forecasting of material properties, with multiple environmental variables involved.

Talk will be mainly 3 topics, 1) Basic operation and constitution of the piezo system, 2) Combination of the piezo system to be fully working system, and 3) application of the system in the materials research. Piezo materials are the basic building block for the motion related in-situ experiment and “slide-and-stick” and the tube system are mainly used. Simple in-situ system, which can be easily built in a lab, will be introduced to be used in scanning electron microscopy (SEM), which can be extended to TEM. A simple in-situ system in SEM and transmission electron microscopy (TEM) will be demonstrated in measuring the stiffness from a single-crystal cementite sheet, and nano-scale probing to inject current in local area, respectively. Various design and application of in-situ systems, such as nano-grippers to handle nano-materials, electrical conduction system to investigate material transport under electrical bias, defect sensitive cathodoluminescence system, and nano-crystal growth and dissolution in the liquid cell system will be introduced in the later part of the in-situ application

**DSL219**

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## Formation of a Fealsn Solid Solution by Mechanical Alloying

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This work investigates the alloying reaction undergone by Fe72Al26Sn2 powder mixtures submitted to mechanical processing by ball milling. X-ray diffraction, Mössbauer spectrometry, and electron microscopy have been applied to follow changes in the microstructure and phase composition in dependence on milling time. Experimental evidences indicate that Al and Sn gradually dissolve in Fe, finally forming a crystalline solid solution. According to the present experiments, nanostructured Fe-Al-Sn alloy were developed with an average crystallite size of about 5 nm by milling for 24 h. With increasing the milling time, the crystallite size decreased for all powders. Increase in microstrain is observed with increasing the milling time. SEM results show that welding of very small particles to the surfaces of larger particles occurred and that the powder particles tended to form a matrix of randomly welded thin layers of highly deformed particles.

[1] S. Souilah, S. Alleg, C. Djebbari, R. Bensalem, J. Sunol, (2012) Mater. Chem. Phys. 132 766–772.
[2] M.A. Meyers, A. Mishra, D.J. Benson (2006) Prog. Mater Sci. 51, 427–556.

**DSL239**
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## Prediction of the Local Properties of High Pressure Die Casting Products with Validation of Real Discontinuities by Computer Tomography

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It is known that the mechanical properties of the casting besides the type and distribution of the structure phases are considerably influenced by compactness of its structure, in that the porosity resulting from change of alloy state of matter, from liquid to solid. In the case of aluminum alloy castings made by gravity casting the porosity is shrinkage and gas (hydrogen) origin. Whereas, in the case of high pressure die casting (HPDC), appears also the problem of air entrapment [1], which result from the dynamic introduction of the liquid alloy from the shot sleeve of high pressure machine through the ingate into the mould cavity. The amount of these discontinuities (shrinkage/gas porosities) can be reduced and thereby improve the local properties of high pressure die casting product using a Vacuum system or/and others systems called as Local squeeze casting and the Jet cooling are applied both nearly in the casting hot spots zones.

In this paper, the simulation study was taken with an attempt to effective prediction of intensity and distribution of discontinuities with the validation studies using tomography of real casting. Porosity defects identified by the prediction based on the simulation and by computer tomography (CT) way, was introduced as the voids to the 3D geometry of the casting, which is then was subjected to a real exploitation load. Next stage was the simulation calculations of stress map (using mechanical simulation code). Applying the principles of fracture mechanics, there was defined the stress intensity factor (SIF), assuming the dealing with a linearly elastic material and further the material resistance to cracking $KIC = \sigma \sqrt{\pi a}$ ($a$   the length of discontinuity, $\sigma$   stress value on its interface). The value of this factor should not exceed the critical threshold that there has been no development of cracks from discontinuity present in the geometry. Such a design procedure of casting allows for defining acceptable discontinuities with keeping of the principle of "Tolerance of damage" [2].

[1] J. Campbell, Complete Casting Handbook. Metal Casting Processes, Metallurgy, Techniques and Design, Butterworth-Heinemann, 2015.
[2] Z. Ignaszak, J. Hajkowski, P. Popielarski, Mechanical Properties Gradient Existing in Real Castings Taken into Account during Design of Cast Components, Defect and Diffusion Forum, Vols. 334-335, pp. 314-321, 2013.

**DSL249**

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## Interface of Anorthite (CaAl2Si2O8) and Aluminium: Kinetics of High-Temperature Reactions

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This current research focuses on the kinetics of interfacial reactions between anorthite and molten aluminium. Very high density anorthite substrates were produced by melting a stoichiometric mixture of alumina, silica and calcia and these were then exposed with liquid aluminium for varying reaction times (0.5 to 250 h) at different temperatures (850°, 950°, 1050°, and 1150°C). The resultant ceramic-metal interface was investigated by optical microscopy (OM), scanning electron microscopy (SEM) equipped with energy dispersive spectrometry (EDS) and electron probe microanalyser (EPMA) and electron transmission microscopy (TEM). The thickness of the interfacial region showed a parabolic growth rate with increasing temperatures and this suggests that the interaction between anorthite and aluminum occurs through a diffusion controlled process. The analyses revealed that the interfacial phases from the metal to the ceramic side were alumina (Al2O3), calcium hexaaluminate (6CaO.Al2O3), calcium dialuminate (2CaO.Al2O3), calcium monoaluminate (CaO.Al2O3), gehlenite (2CaO.Al2O3.SiO2) and anorthite. The interdiffusion of Al3+-Si4+ and Al3+-Ca2+ are believed to be the major mechanisms responsible for the reactions of anorthite with Al alloys, with the activation energy for the complete corrosion reaction being 112 kJ/mol.

# SPECIAL SESSION 12
# NANOTECHNOLOGY IN OIL AND GAS (SS12)

**VIP030**
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## Effects of Surfactant-Polymer Interactions in Enhanced Oil Recovery

Theories of surfactant flooding and polymer flooding have been discussed individually. This study focuses on surfactant-polymer (SP) interactions used for an oil field to study the compatibility and optimization of surfactant-polymer injection. The methodology and even some conclusions in this research will be utilized and useful for other processes as well.

**DSL217**
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## Effect of Zinc Oxide Nanoparticle (ZnO NPs) Sizes on Viscosity and Interfacial of Nanofluid for Enhanced Oil Recovery (EOR) Application

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The effect of ZnO NPs on the viscosity and interfacial tension (IFT) of nanofluid in potential EOR application were investigated. First the nanosized ZnO was synthesized using sol-gel method by citric acid precursor with different pH. The nanomaterials with structure were annealed at same temperature which was 600 ºC based on the Thermogravimetric (TGA) Analysis. The structure and morphology were characterized by Powder X-Ray Diffraction (PXRD) and Transmission Electron Microscope (TEM), respectively. TEM analysis revealed the same tendency which the Zn NPs size increased with the pH of the precursor. The ZnO nanofluid was then prepared with different particles sizes with the same concentration. Results revealed that the viscosity of the nanofluid increased 50% when the particles size increases from 70 to 200 nm with conserved density. The interfacial tension (IFT) also increased when the particle size increases. Based on these analyses, it supports the theory claiming that nanoparticle can contribute for betterment in Oil and Gas industry especially in EOR [1].

[1] N.A. Ogolo, O.A. Olafuyi, and M.O. Onyekonwu, 8-11 April 2012, “Enhanced Oil Recovery Using Nanoparticles,” Paper SPE 160847, presented at the SPE Saudi Arabian Section Technical Symposium and Exhibition.

**VIP014**
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### Ferrite-Based Nanofluid as a New Viscosifying Agent for Enhanced Oil Recovery Application

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Magnetic fluid is proposed as a substitute for the application of polymer solution as a means to recover the residual oil left in the bypassed region in oil reservoirs. When subjected to magnetic field, the viscosity of magnetic fluids increases and enable flow control. In this study, the response of magnetic nanofluid with the applied magnetic field was observed as a function of shear rate. Two types of samples, namely magnetite and cobalt ferrite of 0.1% w/v of different polydispersity index, saturation magnetization and mean hydrodynamic particle size were used. The strength of the applied magnetic field was also varied to investigate the effect of magnetic field strength on the viscosity enhancement of magnetic fluid. Shear dependence response of the magnetic fluid exhibit non-Newtonian behavior when magnetic field of 20 to 40 mT was applied. Viscosity of the magnetic fluid reduced with increasing shear rates, showing shear thinning behavior. At a particular shear rate, viscosity remains constant when the strength of magnetic field increases indicating saturation in chain length even at low field. Magnetoviscous effect (MVE) is calculated as an indicator for a viscosity gain magnitude when magnetic field is applied. Cobalt ferrite sample shows larger MVE compared to magnetite that may be attributed to the higher polydispersity index. In conclusion, particle size distribution is the most dominant factor affecting MVE of the dilute magnetic fluid when magnetic field is applied.

[1] H. Soleimani, N. Yahya, N. R. A. Latiff, H. M. Zaid, B. Demiral and J. Amighian, Novel Enhanced Oil Recovery Method using Co2+ xFe2+ 1-xFe3+ 2O4 as Magnetic anoparticles Activated by Electromagnetic Waves J. Nano Research.26 (2014) 111-116.
[2] N. Yahya, M. Kashif, N. Nasir, M.N. Akhtar and N.M. Yusof: Cobalt Ferrite Nanoparticles: An Innovative Approach for Enhanced Oil Recovery Application, J. Nano. Res. Vol. 17 (2012), p. 115-126.

**VIP030**
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### Studies on the Stability of the Foamy Oil in Developing Heavy Oil Reservoirs

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In the process of natural energy depletion, foamy oil is characterized of low production gas oil ratio, high oil viscosity, high daily production rate and high primary recovery factor. The stability of the foam turns out to be the prevailing factor that governs the life of the ‘foamy oil’. To enumerate the main factors affecting the stability of the foam,–high-pressure visualized experiment model for foamy oil stability test was developed. A serial of experiments were conducted to evaluate the performance of the foam stability. The effects of oil viscosity, height of the oil column, dissolved gas content and dispersed gas were investigated and recorded. These experiments were conducted using a Jergusen cell, a high pressure cell. The volume of foamy oil produced, either by a step reduction in pressure or by a gradual (linear) reduction in pressure, and its subsequent decay was observed, visually. The experimental results show that foamy oil stability increases

with higher oil viscosity, higher oil column, higher dissolved gas content and higher pressure decline rate. Asphaltene content was not observed to increase the foamy oil stability significantly. The results also show that the foam quality of foamy oils is much lower than aqueous foams.

# SPECIAL SESSION 13
# SURFACE TREATMENTS OF ADVANCED MATERIALS (SS13)

**DSL090**
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### Influence Deposition of Zeolite on Corrosion Resistance of Aluminum Alloy 6082 T651

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One of the most widely processes adopted to protect metals from corrosion, involves metal surface coatings by various methods to form protective layers. Indeed the surface coatings of zeolites are becoming ecological alternative coatings for metal substrates [1], they are best known for their thermal, mechanical and chemical stability [2].

The excellent mechanical properties of aluminum alloys are in fact the origin of their domains as qualifications for demanding construction. They particularly involved in the development of all modern means of transport and especially aviation, mainly because of their lightness (density about 2.7). Nevertheless, the chemical stability of their surfaces is insufficient against aggressive media. It is known that aluminum and such alloys are resistant to corrosion in a chemically neutral environment due to the surface oxide layer naturally formed by the oxygen in the air (alumina $Al_2O_3$). However, chloride, acid or alkaline medium, the thin alumina layer formed is soluble giving rise to damage by corrosion; for example the aggressive gel of zeolite (basic pH), it may deteriorate the properties of the surface of the substrate resulting in pitting. At this stage, a reflection for passivation and protection is necessary and indispensable, to successfully optimize the coating and protection against corrosion. [3].

In this context, zeolite films composed of various types of zeolites were synthesized according to a general protocol in two parts. The first part is divided into two steps, the first one tries to coat this material with a highly siliceous zeolite (hydrophobic zeolite), has neutral pH to passivate our material against corrosion of aggressive synthetic gels, taken as a second stage, either by a superposition of a layer of the same MFI structure (Mobil Five), estimated for the inhibitory property against corrosion aggressive environments, called ZSM-5; or another zeolite called Y. The second part, our material without being passive, we try to cover our material separately, one with ZSM 5 and the other with zeolite Y, at the end, an investigation of the various layers deposited is capital. [4].

The electrochemical characteristics of coated materials have been studied in artificial seawater by: (1) Open Circuit Potential (2) potentiodynamic polarization. The structures before and after deposition were analyzed by microscopy and RX. The results indicated that the coated aluminum alloy showed considerable corrosion resistance compared to uncoated.

[1] P. Chakraborty Banerjee, Ren Ping Woo, Sam Matthew Grayson, Amrita Majumder, R. K. Singh Raman. Influence of Zeolite Coating on the Corrosion Resistance of AZ91D magnesium Alloy.2014.
[2] Dalei Song, Xiaoyan Jing, Jun Wang, Yanli Wang, Piaoping Yang, Ming Zhao, Milin Zhang. Corrosion-resistant ZSM-5 zeolite coatings formed on Mg–Li alloy by hot pressing. 2011.
[3] Mayuri N. Katariya, Arun K. Jana, Parimal A. Parikh. Journal of industrial and Engineering Chemistry 19 (2013) 286-291.
[4] Lauridant N. Doctoral thesis. 2012, p 79, 80.

**DSL094**
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### Characterization and Study Hard Layer on steels grade Machinery

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This paper presents the growth and formation of boride coating in steel AISI 9840 used in Plastic Industry; the growth of FeB and Fe2B hard layers, plus the effect of time of thermochemical treatment paste boron, as well as the incubation time for FeB and Fe2B phases, besides the growth kinetics of the layers boride at the material surface in AISI 9840 steels with boron dehydrated past [1-2]. This technique generates hard coating on the material characteristics of two bilayers FeB/Fe2B and a transition zone, called diffusion zone at the interface of coating / substrate. The thermochemical treatment was performed at three different temperatures: 1173, 1223 and 1273 K, with treatment times of 2, 4, 6 and 8 h. Using the law of growth parabolic; also mobility of boron was determined on study of this material. The potential influence of boron, the treatment time and temperature is clearly seen on the growth kinetics of boride layers. Moreover optical microscopy, scanning electron microscopy (EDS) and XRD analysis present the type of layer formation and presence FeB/Fe2B sawn and two phases FeB/Fe2B, for the steel grade machinery; On the other hand, by the nanoindentation technique is obtained modulus and hardness of the phases present steels boriding.

[1]N. López-Perrusquia, M.A. Doñu-Ruiz, C. R. Torres San-Miguel, M. Flores-Báez, I. Flores-Báez, Defect and Diffusion Forum, 365, 122-127 (2015).
[2] N. López-Perrusquia, I. Campos-Silva, J. Martínez-Trinidad, A. Avilés, E. Alvárez-Castañeda, S. Juárez-Torres, Advanced Materials Research, 65, 47-52 (2009).

**DSL096**

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### Effect of Boride Coatings on Hydrogen Embrittlement on AISI 4140

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The present work studied the effect of boriding coatings on hydrogen embrittlement on AISI 4140 by means the mechanical behavior. The samples were boride by paste dehydrated pack [1] at temperatures 1203 and 1233 K for 7 hours, after that all samples at a temperature of 1143 K for 1 hour and tempering 473 K were exposed for 1 hour 30 minutes. The microstructural characterization were examined by scanning electron microscopy and it was observed saw-tooth morphology of boride coatings type FeB and Fe2B [2]. The mechanical properties were evaluated by nanoindentation technique along the depth of boride coatings at

a constant load of 200 mN to determine the modulus elastic and the hardness. Hydrogen was introduce into samples through cathodic charging applying a current density of 50 mA/cm2 by .5 M sulphuric acid solution kept at a room temperature. Three-point bend test was used to study the effect of hydrogen cathodic charging on the mechanical behavior of AISI 4140 with boride coatings. Microcracks are formed base steel in the presence of hydrogen, the boride coatings on AISI 4140 steel are effective to prevent hydrogen diffuse into the material. With boride coatings, hydrogen entry into the base metal was delayed [3]. The results showed a reduction of ductility and index of embrittlement.

[1] M.A. Doñu Ruiz, N. López Perrusquia, D. Sánchez Huerta, C.R. Torres San Miguel, G.M. Urriolagoitia Calderón, E.A. Cerillo Moreno, J.V. Cortes Suarez, Thin Solid Films, 20 August 2015, ISSN 0040-6090
[2] N. López-Perrusquia, M. A. Doñu-Ruíz, F. Vasquez, O. Trujillo-Alfaro, Y. E. Vargas-Oliva, Advanced Materials Research, Vols. 535-537, pp. 513-516, Jun. 2012.
[3] Quadrini, E., & Badini, C. (1989). Materials chemistry and physics,24(1), 111-122.

**DSL105**
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## High-temperature Corrosion of Inconel alloy 800 in N2/H2S Gas

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Inconel alloy 800 (33Ni, 44Fe, 21Cr, 0.05C with less than 2% of Cu, Mo, Nb, Al, Ti, Si, and Mn) is a nickel-iron-chromium alloy that has high strength and resist oxidation, carburization, and other harmful effects of high-temperature exposure. The high nickel content maintains an austenitic structure so that the alloy is ductile. Nickel makes the alloy highly resistant to both chloride stress-corrosion cracking and to embrittlement from precipitation of sigma phase. Inconel alloy 800 used material of construction for equipment that must have high strength and resist sulfidation, carburization, and other harmful effects of high-temperature exposure. It is used for heat-treating equipment, chemical and petrochemical processing systems, and nuclear power plants. Although the general corrosion resistance of Inconel alloy 800 is excellent, its corrosion behavior in N2-H2S-H2O containing atmospheres was not yet adequately examined. They corroded at 600 and 800 oC for up to 70 h under a total gas pressure of 1 atm. Each test coupon was suspended by a Pt wire in a quartz reaction tube within the hot zone of an electrical furnace. The corrosion atmosphere was 0.9448 atm of N2 plus 0.031 atm of H2O plus 0.0242 atm of H2S. Test coupons were characterized by an X-ray diffractometer (XRD) with Cu-Kα radiation, a scanning electron microscope (SEM), and an electron probe microanalyzer (EPMA).

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**DSL119**
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## Improving the Hardness of the Surface Layer of Ferrous and Nonferrous Metals by the Tribofinishing Process

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This work investigates the influence of vibratory finishing process on the surface layer of the material.
The tribofinishing process is a treatment method of chemical and mechanical surface that is to use the low frequency vibration technology in the treatment of metal parts in the presence of abrasive and chemical additives. The treatment regime and its parameters have a great influence on the metallographic characteristics of the treated surfaces. The tribofinishing process has many technical and economic advantages for the treatment of mechanical parts with very complex geometrical shapes.

Keywords: tribofinishing process, hardening, ferrous and non-ferrous, amplitude, frequency.

**DSL156**
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## Estimating and Extracting of the Surface Profile Waviness by Use of the Spatial Notch Filters in the Roughness Profile Analysis

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Classical surveying of the machined surface quality is performed very often with using roughness profile recording by means of contact mechanical profilometer. Also classical attitude to the profile run elaboration is to regard it as to a curve composed of the three main components: shape – treated mostly as deterministic, waviness – treated essentially as deterministic with some probabilistic component, and roughness itself – treated to as purely probabilistic. The question is: how to estimate this three components quantitatively and extract for individual evaluation. In our works we have developed methodical procedures of profile elaboration divided into three major steps: shape estimation and extraction using polynomials of the order fitted to the evaluated profile, waviness estimation and extraction with using spatial filtering methods employing so called notch filters, statistical roughness estimation with using the set of tests and methods devoted especially to roughness runs of the purely probabilistic type. The notch filters are filters cutting out one, well determined spatial frequency l0 from the spectrum of frequencies related with investigated profile. The second but not less important advantage of this choose, was the phase characteristics of this filter, which tends rapidly to zero outside the “notch” band, not distorting unfiltered spatial signal. The width of cutting “notch” is controlled with using one independent variable Q. Our methodology is based upon cutting out a set of frequencies from the profile, tuned separately for to achieve as well reflection of the periodic impulse-like signal as possible, due to the well known theorem of decomposing physical, periodical signals into their harmonic components. In our researches we have employed the set of 8 filters providing us with good results even with profiles “scared” with substantial tracks of periodically acting tool. Of course, it is still

possible to employ even greater number of filters, due to their low numerical complexness. Also the comparison has been performed with the profile not affected by waviness for to convince, that this type of filter simply do not affect the probabilistic roughness component. All of the profile elaboration stages have been checked due to their spatial spectrum with using classical tool – FFT transform of the roughness profile autocovariance function. As the major check of the proposed procedure correctness, i.e. proper extraction of the waviness component without disturbing of the statistical roughness parameters we assumed checking of the shape factor Rq/Ra before and after waviness extraction, which turned to be the same at the good level of accuracy. Our results, basing on simple and fundamental frequency analysis seems to be the good alternative to much complicated and time consuming analysis based on wavelet transforms in different forms.

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**DSL183**
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## Influence of the Recrystallization Annealing Gas Atmosphere on the Oxidation State of a TRIP Mn-Al Steel Grade

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Recrystallization annealing is required for setting up the mechanical properties of automotive steels after cold rolling. This treatment is done under controlled gas atmospheres ($N_2$-$H_2$, dew point DP < -20°C) in order to obtain a metallic iron surface which is compulsory for a good coating adherence during the subsequent Hot-Dip Galvanizing process. Although reducing for iron, these gas atmospheres are oxidizing for the alloying elements. Hence, selective oxides form on the steel surface and generally lead to a poor wettability by liquid zinc. However, the DP can be adjusted to minimize the external oxidation and promote internal oxides. To date, TRIP Mn-Si steels have been extensively studied, but only few data are available for TRIP Mn-Al steels.

Therefore, in this study, we have chosen to examine the effect of annealing gas atmosphere DP on the selective oxidation behaviour of a commercial cold rolled TRIP 780 steel (0.2 wt.% C, 1.7 wt.% Mn and 1.5 wt.% Al). The annealing experiments were performed at T = 860°C in a $N_2$-3vol.% $H_2$ gas atmosphere with DP of -50, -40, -30, -20, -10, 0 and +10°C. The obtained selective oxides were analysed by complementary techniques such as Scanning Electron Microscopy (SEM), Energy-Dispersive X-Ray Spectroscopy (EDS), Glow Discharge Optical Emission Spectroscopy (GDOES) and Auger Electron Spectroscopy (AES).

The results show that the morphology of external selective oxides changed from film-like oxides at low DP to nodular ones at high DP. The chemical composition of selective oxides was also affected by the DP conditions. Indeed, by increasing the DP, and thus the oxygen partial pressure, Al exhibited a transition from external to internal oxidation, whereas Mn significantly precipitated on the steel surface. These observations will be discussed not only from a thermodynamic point of view, but also in terms of diffusivity of oxygen and alloying elements.

**DSL184**
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## Oxidation Behavior in Air and Liquid Pb-Bi Alloy of a Reduced Activation Ferritic/ Martensitic Steel Sample with a Pre-Formed Nanostructured Surface Layer

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Reduced activation ferritic/martensitic (RAFM) steel is the primary candidate material for the first wall and blanket structures of accelerator-driven systems (ADS). And liquid lead–bismuth eutectic (LBE) alloy has been considered as the potential coolant and neutron spallation source in ADS. To improve the compatibility between RAFM steel with LBE melt becomes one of the most concerned issues for ADS because of the high corrosiveness of liquid LBE to conventional steels.

By means of surface mechanical rolling treatment (SMRT), a gradient nanostructured surface layer has been produced on a 9Cr2WVTa RAFM steel sample. The average grain size is ~50 nm on the topmost surface layer and increases gradually with increasing depth. In comparison with the initial sample, oxidation experiments at 600 °C in air showed that the oxidation resistance of the SMRT sample has been significantly enhanced, due to the formation of a more continuous, homogenous, and compact oxide scale. Subsequently, oxidation experiments at 550 °C in the oxygen-saturated liquid LBE alloy showed that the oxidation resistance has been also markedly improved on the SMRT sample after pre-oxidation in air at 600 °C, due to depressed diffusivities of both alloy elements from the steel and O from the LBE alloy in the pre-formed oxide scale.

**DSL230**
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## Nitrogen Diffusion and Stresses during Nitriding of Metals and Alloys

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Nitriding of metals and alloys is an effective way of producing surface layer of unique and mutually complementary properties for applications in machinery and tool industry. A formation of the nitrided zone composed of the layers of tailored phase compositions and given thicknesses ensures an increase of the operational parameters of the element subjected to the modification, including: corrosion and wear

resistance, hardness and resistance to thermal shock. Depending on the base alloy composition and the processing conditions, the diffusion zone can have complex morphology. In complex multicomponent systems (alloys) the multiphase layers can grow together with single phase layers and solid solutions.

In this work we present a kinetic model of nitriding. The objective here is for the first time to combine the bi-velocity method and the stress field factor. The model couples the mechano-chemical effects and allows simulating reactive diffusion in multi-component system leading to the formation of the nitrided diffusion zone of complex morphology.

The main postulates of the model and the initial and boundary conditions (metal composition, temperature, and processing time) are discussed and some exemplary results presented. Based on the model, the basis for designing nitrided layers are formulated.

Acknowledgements
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**DSL244**

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## Adsorption of Lead (Ii) In Liquid-Solid Interfaces of Natural Hydroxyapatite. A Batch Study

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The removal of lead from aqueous solutions was studied using the liquid-solid extraction method. The solid used is the crude phosphocalcichydroxapatite [Ca10(PO4)6(OH)2] noted (HAPc), and the treated with a solution of lactic acid (HAPal).

The extraction results showed the influence of parameters: the initial concentration of Pb(II) and the temperature of the suspensions. The application of Langmuir adsorption model showed maximum adsorption capacities of each material (HAPc and HAPal). The One specific to hydroxiapatite treated with a solution of lactic acid, was the most important (41.84mg/g). Isotherms established at different temperatures showed that this parameter affects adsorption. The best results were obtained for a temperature of 25°C compared with those recorded at 50°C.

Finally, the results of this study allow us to note that the hydroxiapatite material can be a good extractant solid, for heavy metals, especially when it is modified with lactic acid

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# SPECIAL SESSION 14
# BIOMEDICAL IMPLANTS AND DEVICES (SS14)

**DSL115**

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## Analysis of Bone Plate with Different Material in Terms of Stress Distribution

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One of the most used in the field of medicine for the treatment of tibial shaft fractures internal fixation methods is by osteosynthesis plates, the most common plate limited contact dynamic compression (DCP-LC) [1]. This paper presents the results of the fracture site grade 1, where the contact plate and the bone callus area on plates made of bone (LVM stainless steel, titanium alloy different biomedical materials and cobalt alloy), in recovery conditions 1% (one week of recovery), 50% (three weeks of recovery), 75% (six weeks of recovery) and 100%. The fractured tibia bone was modeled with an ideal geometry in CAD [2], modeling of commercial LC-DCP plate was obtained by parameterization of the part using a coordinate machine equipment for the exact geometry.

Finite element method for the analysis of each case under the same loads and boundary conditions are used, the results were used to determine stress concentrations in the displacement plate and the fracture callus in load direction, to have a starting point in the optimization of the geometry from a commercial plate minimizing mass while determining that the material has faster and better biocompatibility with the human body recovery.

The compression and tension strength that occur in the intact part of the bone and the tibial fracture interface at different stages of osseous healing have been investigated, the results indicate that the titanium plate is the one with the lower shielding effect [3] according to the distribution of contact stresses according to the recovery period in the part of callus, making it the material of which the best adaptability to the bone is obtained.

Keywords: Finite Element Analysis, plate fracture diaphysis and tibia

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**DSL155**

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### Nano-structural Constraints for Ultrafast Excitation Energy Diffusion and Transport in Photosynthetic Membranes

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In photosynthesis solar photons are converted into molecular excitations in the so-called antenna pigment-protein complexes and the absorbed energy is eventually transported to the reaction center pigment-protein complex, where a conversion of the excitation energy into chemical energy occurs. The light harvesting efficiency is considered to be significantly enhanced by exciton interactions present between the pigments of the antenna complexes, which broadens their energy spectrum and also, by virtue of forming a suitable energetic ladder of exciton states, allows ultrafast energy diffusion and transfer in the photosynthetic membrane. Recent advances in atomic force microscopy combined with innovative synthetic biochemistry have provided evidence for nanoscale structural adaptation of photosynthetic membranes in response to changing habitats. Examples include mutable stoichiometry of the antenna and reaction center complexes with the light intensity experienced during the membrane development as well as varying architecture of the protein complexes due to the presence/absence of certain chemicals.

Here, we report on the following fundamental question: how the nano-structural remodeling of the photosynthetic membrane influence the rate of the delivery of photo-excitations into the reaction center traps and the efficiency of charge separation at the reaction centers. The answer was sought by analyzing steady state and picosecond time-resolved fluorescence spectra of photosynthetic membranes extracted from differently treated purple bacteria Rhodobacter sphaeroides. The data [1] imply a robust photosynthetic apparatus that functions surprisingly effectively under a wide variety of conditions.

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**DSL222**
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### Cancer Cell Behaviour in Magnetic Fields

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Biological cells have special properties. Among the biological cells, cancer cells attracted more attentions during last few years. According to our studies in this article, cancer cells have electrical and magnetical properties. Cancer cells make magnetic fields around themselves, so, by calculating the magnetic fields made by suspending cancer cells flowing in microchannel filled by fluid we can estimate existing the cancer cells. Cancer cells rarely exist in patient’s blood. By this method we will be hope to forecast cancer in primary levels.

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**VIP020**

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## Surface Treatments and Dental Implant Infections.

Titanium and its alloys have shown to fulfill most physical, mechanical and chemical properties for an ideal biomaterial. However, the existing lifespan of a dental implant has shown to be limited due to biological contamination from the oral cavity. The prevalence of peri-implant infection in patients with dental implants has shown to range from 28–56%. Clinical cases showing the current treatment to address peri-implant infection and treatment shortcomings will be presented. Ongoing studies using the approach of local delivery of antibiotics using a nanotube treated dental implant surfaces, will be presented. The ultimate goal of these studies are to propose the development of a novel strategy to promote the development of a self-efficient biological implant that will ultimately provide clinicians with another tool to rehabilitate patients with history of chronic and acute periodontitis.

**DSL229**

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## Characterization of Barrier Performance of Biomedical Polyurethanes

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The choice of an appropriate material for biomedical devices, such as artificial heart and ventricular assist devices (VAD), is of prime importance as specific properties are required, namely resistance to thrombosis, fatigue and degradation, biostability, excellent blood compatibility, and low fluids and gases permeability [1]. For example, significant water permeation through these materials could induce the corrosion of electric circuits, wires and other metal components resulting in the degradation of the device performance and, therefore, in the decrease of the device lifetime. For this reason it is essential to use the material with high barrier properties.

There is a growing interest in using polyurethanes (PUs) for medical devices due to their suitable mechanical properties and ease of processing. However, like majority of polymers, they are known to be easily permeable towards water and gases [2], in particular due to their amorphous rubbery state. Moreover, the water and oxygen permeability of PUs is strongly influenced by its chemical composition. Thus, the establishment of a correlation between the structure/morphology and the resulting properties is necessary in order to better understand how the structural and morphological parameters influence the functional properties of PUs.

The physicochemical properties of two commercial PUs, i.e. Chronoflex®ARLT (CFARLT) and Bionate®II (BNII), were studied and compared in terms of their morphology and the thermal stability. In addition, the water and oxygen transport properties of PUs membranes were analyzed as a function of temperature and relative humidity. FTIR results reveal that CFARLT membrane has a polyurethane-urea-type structure, while BNII is pure polyurethane. It is known that the urea groups favor a high degree of hydrogen bonding between the hard and soft segments of PU compared to the pure urethane groups [3,4,5]. Such hydrogen bonding, confirmed by DSC analysis, promotes the phase separation between these segments. The water and oxygen permeation measurements have shown that the diffusion coefficients are lower for BNII, i.e. when the phase separation is less pronounced. It is found that BNII has barrier properties about 2 times higher than CFARLT. As it was expected, the increase of the water and oxygen diffusion coefficients (22-54% and 11-49%, respectively) with the temperature increase (from 25 to 42 °C) was observed. Moreover, the increase of the permeability coefficient was obtained for the both types of PUs due to the water plasticization effect. However, no effect of the relative humidity on the oxygen diffusion was found testifying that the PU plasticization by water was not sufficient to promote the oxygen transfer. In conclusion, the obtained results suggest that the pure polyurethane (BNII) is a more suitable material for the design of biomedical devices as compared with polyurethane-urea (CFARLT).

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**VIP073**

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## Real-Time Observations of Electrochemically-Driven Diffiusion Reactions Nanoscale Materials

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Electrodes in rechargeable batteries undergo complex electrochemically-driven phase transformations upon driving Li ions into their structure. Such phase transitions in turn affect the reversibility and stability of the battery. This presentation gives an overview of the PI's research program on in-situ transmission electron microscopy (TEM) of battery materials. In-situ TEM has been shown to be a very powerful technique in shedding light to some of the mysteries in electrochemical performance of new materials. Various anode materials including SnO2, MnO2, ZnSb were subjected to lithiation process and the transport of Li ions was visualized within their atomic structure. For SnO2 nanowires, it was observed that the Li ion transport results in local strain development preferably along (200) or (020) plans and [001] crystallographic directions. The lithiation behavior in the presence of twin boundary defects was completely different compared to pristine state with no twin boundary defect. We showed that twin boundaries in general provide a more accessible pathway for Li ion transport. Anisotropic plastic deformation was also observed along [010] directions of $MnO_2$ nanowires. Sb-based intermetallics, such as Zn-Sb system, which have been proved to be promising anode materials for Li-ion batteries, are also capable of storing of sodium ions. We investigated the microstructural changes and phase evolution of the Zn-Sb intermetallic nanowires using in-situ TEM. Zn-Sb alloys also exhibit a new cubic alloying phase $Li_2ZnSb$ that form by intermixing of the ABAB atomic ordering in hexagonal LiZnSb due to Li inclusion in their lattices. Our results indicate that the reaction between Zn-Sb and sodium proceeds through a different pathway during the first compared to the subsequent cycles. Atomic resolution imaging shows that NaZnSb has a layered structure, which provides channels for fast $Na^+$ diffusion.

Keywords: Energy Storage, Rechargeable Batteries, In Situ Transmission Electron Microscopy, Nanomaterials, Nanowires.

**VIP019**

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## Nanomedicine Towards Infection-free Cardiovascular Devices

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In the past two decades, the estimated number of cardiac implantable electronic devices (CIEDs), has greatly increased. While CIEDs are currently an effective choice for many patients, unfortunately CIED system infection has become a serious complication of cardiac device implantation and is associated with up to an 18% mortality rate. An average 1-1.3% increase in the infection rate of such devices has become a huge concern resulting in an unfortunate 10.5% in-hospital mortality rates [1]. Thus CIED infection problem significantly reduces the longevity, performance and survivability of the device, and frequently requires device extraction or removal, which is more complicated and twice as costly as the original implant procedure. Therefore, there exist a pressing need for a more attractive and effective approach such as application of antibacterial envelopes into the device pocket especially for the high-risk patients. Herein, we report on custom-designed engineered antibacterial surfaces and envelopes for Infection-free CIEDs that can integrate better with tissue, elute antibiotics, possess long-term antimicrobial benefits and thereby reduces infection risk. This multifunctionality will be achieved by coating the surface of CIEDs with conductive Hollow Nanofilaments and intercalating antibiotics within them to provide prolonged antibacterial benefits, while at the same time encouraging device integration and survival. The coating occurs without harmful chemicals, takes place in a single setup of apparatus, in a very short time, and with low-cost and non- hazardous materials for intercalation of antibiotic drugs (minocycline and rifampin). Our drug-intercalation method is acquired at room-temperature through a diffusion-driven process inside the conductive hollow nanofilaments.

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# SPECIAL SESSION 15
# TRANSPORT IN MEMBRANES (SS15)

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## Transport in Membranes: From Diffusion to Convection and Adsorption

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The transport phenomena in membranes depends on the microstructural and macrostructural membrane characteristics. This work proposes to describe, brief and clearly, the phenomenon's involved in the transport that occur within a membrane; compares the efficiency of different membranes and shows different applications with technical and commercial impact.

Dialysis are based on concentration differences and membrane characteristics. The constant interest of the industry and clinicians, in the new class of dialysis membranes and dialysis technics aimed at enhancing the transport capabilities of middle, large and even protein-bound molecules has evolved from technics that uses almost only diffusion to technics that are using all the available membrane separation phenomena: diffusion, convection and adsorption. Recently the development of high-performance "high-flux" membranes and "super-flux" membranes are opening new fields for medical use of membranes as the treatment of myeloma.

The basic functions of membranes are: separation - water purification and waste water treatments, seawater desalination and brackish water purification, petroleum and petrochemical industries for separations, purification and recovery involving solvents; sterilization - removal of viruses, bacteria, fungi and unwanted microbes; discrimination - batteries, fuel cells and sensors: pH meter, blood glucose meters; selective transport -contact lenses, artificial liver and artificial pancreas, artificial lung, dialysis and hemodialysis; and delivery - control drug delivery, patches such as nicotine patches.

# SPECIAL SESSION 2
# FUNDAMENTAL DIFFUSION PROCESSES AND REACTIONS IN ENGINEERING MATERIALS (SS2)

**VIP031**
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## The Oxidizing Roasting and Reducing Melting of Titaniferous Iron Ores

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The influence mechanism of the initial properties of the iron ores and mode of oxidizing roasting in the agglomerative machine on the metallurgical characteristics of agglomerate (the reducibility and durability) and also on the heat exchange and reduction processes of agglomerate in the blast furnace and therefore on the main technical and economic indices of the blast furnace smelting (the coke consumption and productivity) are considered. Objects of examination: the iron ore concentrates for production of agglomerate and also agglomerate – raw materials for the pig iron production. The technical systems, for which the selected objects are researched by methods of the experimental and mathematical simulation, are the main metallurgical aggregates - the agglomerative machine and blast furnace (the scheme “the blast furnace - the converter”) [1, 2]. The scheme “metallization - electromelting” is also considered.

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**DSL097**
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## Phonons Scattering by Substitutional Impurities in a Quasi-3D Nanostructure

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In the present work we investigate the influence of reticular defect on the elastic waves propagating through a crystalline nanostructure modelled by two infinite monatomic plans. The study is confined to the elastic scattering in the harmonic approximation framework. The problem is tackled from a semi-classical point of view, using scattering boundary conditions and applying the matching method formalism. This theoretical approach, based on the Landauer-Büttiker principle, permits to determine the dynamics of the perfect waveguide and allows one to carry out the phonons localized induced states, the displacements of the perturbed region atoms, the transmission and reflection coefficients of the perturbed system. This matching method combined with the Green's functions theory allows one also to determine the densities of states (DOS) of all irreducible atoms in the perturbed region.

Numerical results show that the presence of defect in the nanostructure modifies particularly its mechanical and vibrational properties by the creation of new localized states by bulk and surface phonons scattering. Its influence results in a general decrease of the transmission probability amplitude accentuated by Fano-like resonances and/or Fabry-Pérot oscillations. The transmission spectra, obtained by scattering experiments, could then be regarded as fingerprints of the specific defect structure. The results could be

also useful for the design of phonon devices and provide a basis for the understanding of conductance spectroscopy experiments in mesoscopic systems.

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**DSL110**

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## Influence of the Composition on Lowering of Dielectric Constant of Glass-Ceramic Composites

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Low dielectric constant materials are of great importance for intensively developed microwave integrated circuits. Using of a low dielectric constant substrate enables diminishing of signal propagation delay in high speed, high frequency circuits [1-3]. In this work, glass-ceramic composites destined for low dielectric constant substrates were fabricated. The influence of the composition of glass and crystalline components on dielectric properties of the composites was investigated. A few glasses from the systems SiO2-B2O3-BaO-Al2O3-ZnO-ZrO2, SiO2-B2O3-CaO-Al2O3-MgO and B2O3-CaO-Al2O3-ZnO-ZrO2, SiO2 glass, and cordierite and mullite as crystalline phases were used as main components of the glass-ceramic composites. The substrates were obtained by ball-milling of starting powders, pressing and sintering in the temperature range 850-1200°C. Addition of sintering aids, like Cu2Bi2O4, was helpful in lowering of sintering temperature due to liquid phase formation at grain boundaries. The properties of the substrates were characterized by XRD, SEM, heating microscope observations and impedance spectroscopy. The lowest values of dielectric constant at a level of 5-7 at 1 MHz were found for the composites with high SiO2 and B2O3 concentrations. The presence of ZnO and ZrO2 in the glass composition resulted in partial glass crystallization which was advantageous for mechanical and thermal properties. The fabricated glass-ceramics were dense and fine-grained and showed good compatibility with commercial Ag, Ag-Pd and Pt thick film pastes.

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**DSL122**

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## The Kinetics of Diffusion-Controlled Recombination of Radiation-Induced Defects in Ionic Solids

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It is well known that the radiation resistance of ionic materials is controlled by the concentration and mobility of primary radiation defects which recombine upon encounter [1,2]. In binary oxides (e.g., MgO, Al2O3, MgF2) a Frenkel pair of complementary defects consist of an F center (oxygen or halogen vacancy) and an interstitial Oi (Fi) anion. We have calculated the kinetics of diffusion-controlled bimolecular recombination of these defects and for the first time suggested interpretation of the experimental data on F center annealing upon heating of a number of ionic solids, where defects were produced by different types of irradiation (electrons, heavy ions, neutrons).

In particular, we have demonstrated that this process is controlled by the interstitial oxygen (halogen) ion migration, whose mobility is much higher than that of the F centers. It is shown that the shape of the F-annealing curves is determined by two control parameters: migration energy Ea and the effective pre-exponential factor X = N0 ´ a0 ´ D0/β, where N0 is initial defect concentration, a0 - recombination radius, D0 - diffusion pre-exponent and β is the heating rate [3].

A comparison of the recombination kinetics for the same material under electron irradiation (homogeneous defect formation over the sample volume) or under fast neutrons and swift heavy ions (high defect densities along the particle trajectory) shows considerable differences. The latter are attributed to the change of initially perfect crystalline structure with embedded point defects for locally amorphous one, with considerably reduced migration barriers.

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**DSL132**
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## Random Alloy Kinetics Theory for Analysing Tracer Diffusion and Interdiffusion in Binary Alloy Systems

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The binary alloy case is analysed where the interdiffusion coefficient and one of the self- or tracer diffusion coefficient is known. The missing tracer diffusion coefficient for the same composition and temperature will have to be found via the Darken-Manning equation. This is a commonly encountered problem for diffusion in binary alloys. The problem is analysed for the random alloy model by applying three formalisms: Darken; Manning; and Moleko, Allnatt and Allnatt (MAA). The results of these formalisms show different levels of accuracy that sometimes are reflected in high sensitivity of the vacancy-wind factor. It is found that there is a difference in the number of self-diffusion coefficient roots depending on whether the tracer diffusion coefficient is available for the faster diffuser or for the slower diffuser and also depending on how close the composition is to the percolation threshold.

**DSL138**
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## Study of Atomic Motion in Rubidium Borate Glasses

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Atomic Scale X-ray Photon Correlation Spectroscopy (aXPCS) utilizes coherent X-rays to probe the dynamics of materials on an atomic scale. It was used with great success to study atomic diffusion in crystals [1], but its application was recently extended to glasses as well [2]. Rubidium borate glasses serve as an example for fast ion-conducting alkali borate glasses and are a promising candidate for future applications in energy storage as well as in many other fields. In this work the ionic motion in rubidium borate glasses was studied via the aXPCS method.

Correct data analysis of an aXPCS measurement of amorphous materials requires the knowledge of the partial structure function of the diffusing atomic species, i.e. in this case the alkali ions. We used the pair distribution function (PDF) method to investigate the structure of rubidium borate glasses and Metropolis Monte Carlo simulation to extract the required partial structure factor. The parameters of a Born-Mayer-Huggins potential used by Verhoef and den Hartog [3] for structural simulations of alkali borate glasses were modified to fit the experimental scattering data.

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**DSL144**
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## Molecular Dynamics Study of Mass and Heat Transport Properties of Liquid Cu-Ag Alloys

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In this study, mass and heat transport properties of liquid Cu-Ag alloys are investigated in wide temperature and composition ranges. The calculations are performed within the framework of the Green-Kubo (GK) formalism by using equilibrium molecular dynamics (MD) simulations along with one of the most reliable embedded-atom method potentials for this system developed in [1]. The employed approach allows for evaluation of all kinetic constants of the binary system in liquid state. These include two tracer diffusion coefficients D_1 and D_2 as well as three phenomenological coefficients L_cc, L_cq and L_qq. The coefficients L_cc and L_qq relate conjugated mass and heat fluxes and forces, respectively, while the coefficient L_cq describe cross-correlation between mass and heat transport.

The results obtained in this study give a unique opportunity to predict the kinetics of non-equilibrium solidification of real liquid Cu-Ag alloys. In particular, on the basis of these calculations, one can estimate the possibilities of building up temperature and composition gradients at the crystal-melt interface during non-equilibrium solidification of real Cu-Ag alloys.

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**DSL145**
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### Arc Fusion Welding of Mg-Al2Ca Added Al 5xxx Alloys

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When melting or casting Mg alloys, small amount of beryllium or toxic SF6 gas are often used to prevent the Mg ignition. Recently so-called “ECO Mg” alloys have been introduced by adding Mg-Al2Ca into Mg alloys [1] and the improved weldability of ECO Mg alloys was demonstrated in authors’ previous study [2]. Similarly, “ECO Al” alloys are also devised using Mg-Al2Ca in place of Mg element [3] and several researches are ongoing to reveal their mechanical and metallurgical properties.

In this research, arc fusion weldability of ECO Al 5052, 5056 and 5083 alloys was investigated by using gas metal arc welding process as one of most economic joining processes. Al 5356 and 5183 wires were used as filler wires and the results were compared with ones using commercial Al 5052 alloy as a base metal. The strengths of weldments were examined after BOP welding and lap-filler welding. When welding ECO Al alloys, the almost full joint efficiencies were achieved while it was only 82% for conventional Al 5052 alloy.

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**DSL149**
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### The Study of Nitrogen Content on Magnetic Properties of AISI 316L and 316LN Welded Stainless Steels

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Austenitic stainless steels with ferrite content have magnetic properties that may cause interference at electron beam of synchrotron radiation machines. To produce the synchrotron light, electro-magnets and radio frequency waves are used to accelerate electrons near to the speed of light in a storage ring [1]. Since some austenitic alloys have low ferrite content, these materials should be used as an alternative to this application considering the material cost. Using elements that have austenite stabilizing effect on the gas protection during the welding process, it is possible to reduce or prevent ferrite formation even in regular austenitic alloys. Using different nitrogen concentration on the shield gas at GTAW process it is possible to reduce the ferrite phase formation after welding process [2]. The specimens was studied to determine the magnetic permeability, microstructure including X ray diffraction by synchrotron radiation and mechanical properties. The inclusion of Nitrogen on shield gas of an autogenous GTAW process prevent the ferrite phase formation. Large areas are reported without ferrite formation on welds performed with nitrogen concentration at 10%. The mechanical properties of samples have not changed significantly. The welding of stainless steel Type 316L reached satisfactory magnetic permeability levels, although lower than stainless steel Type 316LN, that allows an application for the booster synchrotron production.

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**DSL150**

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### Determination of Transport Properties in Proton-Conducting Oxides

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Since initial reports by Iwahara et al. about proton conductivity in (Ba,Sr) CeO3-δ oxides at high temperatures [1], a great interest in research of proton-conducting ceramic materials begun [2]. Importance of these materials originates from their possible, numerous applications in gas sensors, fuel cells, hydrogen separation membranes or high-temperature electrolyzes [3]. Among candidate compounds, B-site substituted AB1-xLnxO3-δ/2 (A: Sr, Ba; B: Zr, Ce, Sn, In) perovskite-type oxides are of interest, with Ln3+ cations (Y3+, lanthanides) introduced, in order to induce presence of the oxygen nonstoichiometry δ. Oxygen vacancies are required for incorporation of the hydrogen into the oxide lattice, with OH•O defect being reversibly formed. Introduction of +3 cations at the A-site is less studied, as it is expected to cause decrease of values of δ and shrinkage of the unit cell volume. However, concurrent substitution at the A-site (donor-type) and B-site (acceptor-type) can be used to study influence of the chemical composition and structural parameters on the protonic conductivity, as well as may inhibit unwanted reactivity of the compounds in CO2-containing atmospheres.

In this work kinetics of hydration and dehydration of the materials were measured on thermobalance in dry, H2O- and D2O-containing artificial air, with data evaluated using mass relaxation technique. Electrical conductivity of the compounds was measured on dense sinters by electrochemical impedance method. The measurements were conducted in dry, H2O- and D2O-containing artificial air, in order to establish the isotope effect and proton transference numbers. Apart from AB1-xLnxO3-δ/2, also doped NdBaInO4-type novel oxides were evaluated in terms of their transport properties. The obtained results suggest possibility of further optimization of protonic conductivity in the studied systems.

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**DSL152**
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## Influence of Additive Elements on Diffusion and Magnetic Properties of Iron Nitride Thin Films

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Iron nitrides (Fe-N) are an important class of materials, both from the basic physics and application point of view 1-6]. However, due to the weak Fe and N bonding, thermal stability of Fe-N compounds is poor, that makes these compounds unsuitable for device applications. To overcome this issue, the addition of a small amount of third element X (X = Al, Ti, Zr etc), which is having high affinity for N and low heat of formation of X-N, was suggested in the literature [4-5]. It was argued that presence of X modifies local diffusion activation energy of N which leads in the enhancement of thermal stability of Fe-X-N thin films. However, no experimental verification is available to support this argument. Moreover, in the available literature, the choice of X is arbitrary and the role of Fe self-diffusion is not clear.

In the present work, we address these issues by systematically studying Fe and N self-diffusion in Fe-X-N (X=Al, Ti, and Zr) thin films deposited using reactive sputtering. Using neutron reflectivity, and various structural and magnetic characterization techniques, we observed that (i) thermal stability of Fe-X-N films gets improved significantly with additives. (ii) A strong correlation exists between Fe self-diffusion and structural and magnetic stability of this compound. (iii) On contrary to the general persuasion, it was found that N diffusion plays only a trivial role in improving the thermal stability. (iv) On the basis of obtained results, we propose that Al suites, most efficient in the enhancement of thermal stability of Fe-X-N thin films [2-5].

In another part, Fe and N self-diffusion study was performed on iron mononitride thin films. Interestingly, an anomaly was observed in the Fe and N self-diffusion measurements in this compound. We found that N diffuses slower than Fe, although the atomic size of iron is larger than that of nitrogen [6]. From secondary ion mass spectroscopy measurements we found that grain boundary diffusion of Fe leads to its faster diffusion as compared to N. Moreover, using Le Clair’s analysis distinct kinetic regimes of grain boundary diffusion of Fe was revealed that helps to understand observed structure and magnetic phase transformation in this compound [1]. These results can be extended to understand the kinetics of phase transformation in similar metal nitride systems.

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**DSL159**
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## Novel Method for Determining Frequency Factor and Activation Energy of Solid Interdiffusion Coefficients from a Single Diffusion Experiment

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An experimental method employing temperature and concentration gradients is presented that is suitable to determine solid interdiffusion coefficients including their temperature dependence in a single experimental cycle. The method consists of a diffusion experiment in a temperature gradient and fitting of the generated concentration distribution by a distribution calculated in combination with an optimization algorithm.

The Al-Cu system is used to illustrate the feasibility of the method. In a single phase α-Al solid solution, concentration gradients are generated by exposing a cylindrical sample to steep temperature gradients and by annealing until the initially formed solid/liquid mushy zone is resolidified. The annealing is performed in two steps to generate a ramp with increasing Cu concentration and adjacent a ramp of decreasing Cu concentration along the sample axis. This sample with a concentration profile in form of a “roof” is then again exposed to a temperature gradient for diffusion heat treatment. Due to the varying diffusivity along the sample axis the concentration profile evolves locally different during the diffusion heat treatment, and the formerly symmetric “roof” profile flattens to an asymmetric diffusion profile.

Starting with the initial concentration profile, the evolution of the local concentration during the diffusion heat treatment is calculated using assumed values of the frequency factor D0 and the activation energy Q for the given experimental conditions. The optimization algorithm adapts D0 and Q by minimizing the total deviation between the concentration profiles obtained from the diffusion experiment and the diffusion calculation. Thus, reliable diffusion parameters, D0 and Q, are determined with distinctly reduced experimental effort. The experimental procedure is generally applicable to alloy systems with finite solubility.

**DSL160**
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## Thermodiffusion in Liquid Al-Ni Measured by X-Ray Radiography

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Thermodiffusion (or Soret effect) describes the formation of a concentration gradient induced by a temperature gradient in binary or multicomponent mixtures. This cross-coupling effect of heat and mass transfer influences the microstructure formation of alloys because during the solidification process temperature gradients are present.

In order to determine the thermodiffusion coefficient in liquid alloys experimentally, the concentration profile is usually analysed after solidification of the sample. We present a method using X-ray radiography to determine the concentration profile in a vertical capillary. In alloys with sufficient contrast in X-ray absorption,

this technique allows for in-situ observation which excludes disturbances by solidification and reveals possible error sources as e.g. free surfaces. In addition, the development in time can be analysed.
We study Al-Ni as a highly non-ideal binary system. Nickel was found to migrate to the cold side in Al78,5Ni21,5.
Evteev et al. [1] found in molecular Dynamics simulations of Al50Ni50 that nickel diffuses to the hot side. The direction of thermodiffusion possibly changes as a function of concentration like it is the case in liquid Na-K [2].

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**DSL161**
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## Numerical Modeling and Validation of Solid Melting with Natural Convection Using Lattice Boltzmann Method

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Convection melting is associated with applications such as metal processing, environmental engineering, thermal energy storage, nuclear reactor core safety, etc. Models of the convection melting need to account for thermally-driven flow coupled with a moving solid-liquid interface (melt front) where latent heat is absorbed during melting. The major variable interested is the melt front movement which describes the evolution of the system geometry [1]. In the present paper, a lattice Boltzmann method (LBM) [2] is formulated by using D2Q9 lattice model to couple pure solid melting and thermal convection in liquefied region and thus to describe momentum and heat transfer processes taking place in the liquid, mushy and solid regions as well as the melt front advancement. The LBM uses single set of formulations rather than complicated Navier-Stokes equations coupled with heat diffusion equation. Also, compared with previous melt-front tracking methods such as adaptive grid or level set methods, the LBM can track the melt front by simply imposing positions with a specified liquid fraction as the interface. The liquid fraction is estimated from the Godunov enthalpy method using the temperature field obtained from the LBM. The flow boundary condition at the interface is treated with bounce-back mechanism widely adopted in the LBM. Existing typical two-dimensional experiment for Gallium melting [3] is used for validating the melt front movement. Also, the transient flow field and the melt front movement analyzed are also compared with those from rigorous computations using finite element method (FEM) and finite volume method (FVM). Reasonable agreement is achieved and it is concluded that the LBM approach is well-suited to study convection melting processes in geometrically complex media with many engineering applications.

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**DSL162**

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### Ternary Interdiffusion in Fe-Al-Mn System

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Formation of brittle intermetallics at the interface is a major issue in the joining of aluminum alloys to steels. Manganese is an important alloying element in both steels and aluminum alloys and it has been observed to affect the growth of intermetallic layer. In order to model the effect of Mn on the interdiffusion and growth kinetics of intermetallics during joining processes, it is necessary to develop understanding of diffusion interactions in the Fe-Al-Mn system. This report presents the investigations of interdiffusion carried out with diffusion couples assembled with ternary Fe-Al-Mn alloys. Interdiffusion fluxes and coefficients are evaluated from the concentration profiles developed within the diffusion zone based on Boltzmann Matano analysis and Dayandanda-Sohn analysis. Significance of diffusional interactions in this system will be highlighted and discussed in reference to their effect on growth of intermetallic layers during joining of steel to aluminum alloys.

**DSL164**

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### Investigation of Solid-State Diffusion Processes Involved in Metal Oxide Sulfidation

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Natural gases generally contain huge amounts of H2S which must be removed to avoid environmental issues, corrosion at industrial scale, or metal catalysts poisoning [1]. Deep gases desulfurization can be achieved using metal oxide based sorbents, such as ZnO, CuO and NiO commonly used at industrial scale for this purpose, as they are able to react with H2S to form corresponding metal sulfides. However, despite sulfidation thermodynamics is highly favored, reaction kinetic rate is strongly limited. Previous studies related to ZnO evidenced the occurrence of solid-state diffusion limitations, both through the ZnS product layer formed during the reaction, and at ZnO/ZnS interface [2]. In the present study, sulfidation reactions and solid-state transport have been studied through combined experimental methods (thermo-gravimetric analyses under reactive atmosphere) and molecular modeling approaches (molecular dynamics studies performed via LAMMPS package). For this purpose, reference materials with controlled particle morphologies and impurity contents (doping) have been considered. Specific elements were selected for doping to achieve modification of ZnO & ZnS point defects content and structural properties (Al3+, …). These materials modifications are expected to affect solid-state diffusion processes, and therefore to influence reaction mechanisms and kinetics. This contributes to a better understanding of heterogeneous

gas-solid reactions via a multi-technique approach, which highlights the role of solid-state diffusion processes on reaction mechanisms and on the evolution of materials properties.

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**DSL173**
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## Universal Scaling Laws for Diffusivity in Liquid Metals

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Understanding how dynamic properties depend on the structure and thermodynamics in liquids is a long-standing open problem in condensed matter physics. Our current knowledge is based on two different contributions developed for model fluids in which universal scaling relationships between the excess entropy of the fluid and its reduced diffusion coefficient is considered. Difference holds in the way to obtain reduction parameters, using either macroscopic arguments as proposed by Rosenfeld [1] or microscopic ones as in the Dzugutov scheme [2]. However their applications to "real" liquids still remains elusive due to the ability of model reference fluids to describe more complex interactions that may occur in the real systems. The purpose of this contribution is use ab initio molecular dynamics simulations to calculate both structural and dynamic properties at different temperatures for a wide series of liquid metals including Al, Au, Cu, Li, Ni, Ta, Ti, Zn as well as liquid Si and B [3-5]. From this analysis, we demonstrate that the Dzugutov scheme remains successful only if a self-consistent method to determine the packing fraction of the hard sphere reference fluid as well as an appropriate approach to express the excess entropy are used. We compare our results with original Rosenfeld's scaling law [1] as well as with its generalized version proposed very recently [6].

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**DSL181**
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### Analytical Solution for Coupled Non-Fickian Diffusion-Thermoelasticity Problem Subjected to Shock Loading

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The generalized coupled non-Fickian diffusion-thermoelasticity analysis are studied using an analytical method. The transient behaviors of field variables, including mass concentration, temperature and displacement in a strip, which is subjected to shock loading are discussed in details. Based on Lord-Shulman theory of coupled thermoelasticity, the governing equations are derived using generalized coupled non-Fickian diffusion-thermoelasticity theory. To solve the problem the governing equations are transferred to the frequency domain using Laplace transform technique and then the field variables are obtained in analytical forms using the series solution method. The field variables are eventually determined in time domain by employing the Talbot technique. The dynamic behaviors of mass concentration, temperature and displacement are studied in details. High capability for simulating the wave propagation with finite speed is observed in mass concentration field as well as for tracking thermoelastic waves. Furthermore, it is shown that the obtained results are more realistic than that of others.

Keywords
Non-Fickian diffusion; wave propagation; mass concentration; thermoelasticity; analytical method; coupled problems.

**DSL189**
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### Hydrodynamic Forces on a Submerged Breakwater

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In this work, we carried out an asymptotic analysis, up to the second order in a regular expansion, of the interaction of linear long waves with an impermeable, submerged breakwater composed of wavy surfaces. The undulating surfaces are described by sinusoidal profiles. The effects of three different geometric parameters — the amplitude of the wavy surfaces and the submerged length and width of the structure — on the free surface elevation and the hydrodynamic forces, that are acting on the breakwater, are analyzed. Using the domain perturbation method, the small wavy surfaces of the breakwater are linearized. The wavy surfaces of the breakwater generate values of the hydrodynamic forces that are 10% greater than those obtained for breakwater with flat surfaces, and the largest values of this hydrodynamic force is obtained when the length of the breakwater is of the same order of magnitude as the wavelength. The effects of the number of undulations on the hydrodynamic forces are also studied. The asymptotic solution is compared with a numerical solution. The present theory provides basis for comparison with other approximate theories

based on shallow water flows and serves as a prelude to characterize submerged breakwaters of undulating surfaces.

**DSL190**
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### Structural and Magnetic Properties at High Temperature of the Fe-Mg Alloys Prepared by Mechanical Alloying

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Nanocrystalline Fe-Mg alloy samples were prepared by mechanical alloying process using planetary high energy ball mill. The prepared powders were characterized using X-ray diffraction technique (XRD) at high temperature, differential thermal analysis (DTA), transmission electron microscopy (TEM), and the vibrating sample magnetometer (VSM). Obtained results are discussed according to milling time. XRD results also indicated that when the milling time increases, the lattice parameter, whereas the grain size decreases. Connections between the temperatures, activation energies and thermodynamic driving forces were considered. Coercive fields derived and Saturation magnetization derived from the hysteresis curves are discussed as a function of milling time.

**DSL194**
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### Transport Phenomena at the Interface Between Fe-Cu-Ni Alloys and Oxide under Current at High Temperature

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Transport phenomena occurring when two materials are in contact at high temperature under electrical current is a combination of diffusion, reactive diffusion, redox reactions and electromigration of each species [1,2]. Diffusion coefficients can be measured precisely in monocristals or in perfect grain boundaries only. Nevertheless, it is important to estimate transport phenomena in the case of more complex materials and operating conditions. The aim of this study is to obtain a general description of the mechanisms involved in the contact between multiphase metallic materials and oxides composed of iron, copper and nickel.

Diffusion couples formed by rods of the different materials tested are heated at high temperature (900-1000 °C) under an Ar atmosphere containing low oxygen partial pressure (PO2 ≈ 10-7 Pa). In order to distinguish phenomena due to voltage, two diffusion couples are tested at the same time in the furnace and an electrical current (15 A) is applied to one of them only. The diffusion time (i.e., time at high temperature with current) ranges from 200 to 500 h. After the heat treatment, the rods are cut in order to analyse the interfaces by means of Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDS). The proportion and composition of each phase are measured on both sides of rods interface. Thermodynamic data (Po2, activity, oxidation number…) are also calculated using FactsageTM software. The data permit to obtain mass balance charts, concentration profiles and so to calculate global diffusion coefficients and electromigration coefficients.

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**DSL197**
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## H-Induced Changes of Displacement Threshold Distributions in Tungsten: A Molecular Dynamics Simulation Based Study

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Tungsten has been chosen as divertor material in the next step fusion device ITER and is also considered as one of the most promising materials for future fusion devices. The benefits of tungsten are the low sputtering yield under low-to medium energy (~200 eV) hydrogen isotope bombardment due to its high displacement energy (>38 eV) and the ineffective kinematic energy transfer due to the large mass-ratio of hydrogen isotopes and tungsten. In addition tungsten exhibits a low tritium retention [1]. However, recent experimental results of low-temperature deuterium plasma exposure of tungsten samples challenge that view and indicate the possibility of a damage mechanism active already below the estimates of binary collision approximation based damage thresholds [2]. Here we present a comprehensive molecular dynamics study of the influence of hydrogen in the tungsten lattice on the displacement energy threshold. The static effects of the hydrogen on the tungsten lattice (ie. weakened next-neighbour or second next-neighbour interaction) are studied in detail and are compared to the dynamic effects. In the latter the probability of defect creation is strongly influenced by recombination processes, thus that even for high recoil energies the net amount of created defects can be zero. The MD simulations reveal that the presence of hydrogen alters the angular dependent defect creation probability distribution. The resulting consequences for hydrogen permeation properties under high hydrogen fluxes (ie. those relevant for the first wall in fusion devices) will be outlined.

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**DSL211**
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## Methods for Early Stage Detection of B - A Transformation in Sn

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b®a transformation in Sn (tin pest) is the solid-state transformation of metallic white b-Sn to a semiconductor powder of grey a-Sn below +13.2°C. The phase transformation into grey tin (a-Sn) demands the overcooling of the b-Sn solid solution to tens of degrees below 13.2°C. White tin (b-Sn) with a stable bct (body-centered tetragonal) structure crystallizes in the space-group symmetry I41/amd (No. 141) with lattice parameters a = 5.8316 Å and c = 3.1815 Å at a temperature above 13.2°C. A displacive-type transformation concurrently rearranging the whole groups of Sn atoms leads to the formation of a diamond structure (grey a-Sn) with cubic symmetry Fd3m (No. 227), lattice parameter a = 6.4892 Å and a much larger volume (26.3%) with respect to b-Sn structure . The identification of b®a transformation in tin-rich samples can be crucial for some electronic devices working in sub-zero temperatures in aeronautical, aerospace and automobile applications. There are several methods for identifying a-Sn growth including electrical measurements [1], XRD diffraction [2], SEM/EDS analysis [3] and others. All of them are based on different physiochemical properties of tin and they resulting in different levels of measurement sensitivity. This work presents the results of the investigation evaluating the sensitivity of different methods applied for b®a transformation identification.

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**DSL218**
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## Anisotropy of Telluride Diffusion Coefficient in, Bismuth Selenium

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We present results for the anisotropic diffusion coefficient of telluride, isoelectronic impurity, in Bi2Se3 along the solidus. The single crystals were prepared by travelling heater method and they have a high crystalline quality. The experimental conditions permitted us to obtain a definite stoichiometric deviation, and diffusion profiles were obtained by SIMS. We propose a mechanism of atomic diffusion by antisite defects and thermal vacancies taking into account of the anisotropy of the diffusion coefficients.

Keywords
A. Semi-conducteurs; D. Diffusion; D. Defects.

**DSL220**

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## An Atomic Simulation of Atomic Jumps with Help of the Constant Temperature Molecular Dnamics Method

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We study features of atomic jumps by simulation and associated problems are discussed. At the first we present a new approach of the constant temperature molecular dynamics that fully takes into account the fluctuating character of this phenomenon. In addition the combination of molecular dynamics and previously developed molecular statics variant [1,2] allowed within this model to take into account thermal expansion of the lattice and long-range elastic displacement of the atoms in the vicinity of defects. Then by the developed model, we study diffusion features of vacancy in iron and carbon in iron.

In particular we simulate vacancy diffusion at various temperatures and pressures for bcc iron and calculate the diffusion coefficient, migration volume and its alteration with increasing a temperature. Migration energy and pre-exponential factor for vacancy diffusion are calculated. The results are compared with results of other works obtained by molecular dynamics [3] and molecular statics [1,2].

In the case of carbon diffusion in iron, analysis of a large number of diffusion jump trajectories allowed us to estimate the contribution of double jumps in the diffusion coefficient. The diffusion mobility of carbon atoms was studied at different temperatures. We evaluated the influence of anharmonic effects on the diffusivity at high temperatures. Migration energy and pre-exponential factor are calculated. Also, temperature dependence was found of the migration volume. An isotope effect and its temperature dependence are calculated. We analyze the possible reasons for the acceleration of the diffusion of carbon in α-iron at high temperatures.

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**DSL221**

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## Advanced Method for the Constant Temperature Molecular Dynamics Simulation of Vacancy Diffusion in Metals

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We developed an advanced approach and MD model based on it. This approach allows to implement conditions in the simulation cell closest to those being realized in a natural experiment using a thermostat. Thus, in this model used closest to the reality implementation of the canonical ensemble. Within the framework of our model one can obtain a fluctuation spectrum at a given temperature, furthermore, in contrast to other models, this spectrum is independent of parameters which cannot be found in these models.

Additionally, in our model elastic displacements in the layer of atoms around the simulation cell are taken into account. Moreover, we take into account the effects of thermal expansion and external pressure on the system. The model allows us to investigate the characteristics of each diffusion jump and its trajectories.

During testing the model, the amplitude of the average kinetic energy fluctuations was evaluated. Then we found the number of atoms in the system for which changes of the amplitude are negligible.

The model advantages, described above, allow to obtain the characteristics that determine the effect of pressure on the diffusion of defects.

Using our model and many-body potential [1] we simulated vacancy diffusion at various temperatures and pressures for bcc iron. Also, we calculated the diffusion coefficient, migration volume and its alteration with varying the temperature. The results compared with results of other works obtained by molecular dynamics method [2].

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**DSL226**

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## Thermal Stability of CaZnSO Oxysulfide

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As zinc-calcium oxysulfide is one of the most promising materials for creating luminophores it follows that understanding its physicochemical properties is important both for the synthesis and further processing of materials and products.

Information about the heat capacity and melting temperature of CaZnSO oxysulfide was combined with the data on the differential scanning calorimetry and that allowed us to conduct thermodynamic calculations of phase equilibriums in the CaZnSO-CO and CaZnSO-O2 systems in a temperature range up to 1270 K. The determining influence of the partial oxygen pressure (PO2) on the composition of the condensed products in reducing (CaS) and oxidizing (CaSO4, CaO, ZnO) media was shown. The results of the calculations were revealed experimentally by means of the oxidizing (O2-N2) and (CO-N2) reducing heat treatment. A

mechanism for the reduction processes during the heating of CaZnSO mixtures with metallic iron powder was set as following:
CaZnSO + xFe = CaFexZn1-xSO + Zn↑.
Unit cell parameters for the CaFexZn1-xSO at x from 0.4 to 0.75 were defined. The data received can be used for the functional materials creation.

**DSL231**
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### Interdiffusion in the FCC-Structured High Entropy Alloys on the Example of Al-Co-Cr-Fe-Ni and Mn-Co-Cr-Fe-Ni Metallic Systems

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A group of Al-Co-Cr-Fe-Ni alloys was synthesized by arc-melting method for diffusion couple experiments. The annealing of the couples was conducted in the temperature range of 1273 - 1373 K. Concentration profiles were measured using the EDS wide-line scan method. Basing on the experimental profiles, the tracer diffusion coefficients were determined by combinatorial approach, with Darken-Manning based formalism being applied. Two independent methods of optimization were used: Levenberg-Marquardt and genetic algorithm method, with both giving nearly the same results. The approach was also validated against literature data obtained for diffusion in Co-Cr-Fe-Ni-Mn alloys. The results for both systems confirm the theory of sluggish diffusion in High Entropy Alloys, with the values of diffusion coefficients being significantly lower than in the case of conventional alloys. The differences in energies of activation, resulting from different chemical interactions in both systems, were observed.

**DSL232**
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### Influence of Local Equilibrium Assumption on the Diffusive Interaction in the Two-Phase Zone of the Ternary Alloy; Numerical Experiments and Experimental Results

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The complexity of diffusion interactions in multi-phase ternary system leads to the consideration of models based on different assumptions. Many assume diffusion in the matrix-phase at negligible diffusion in

precipitates. Essentially all models base on the local equilibrium assumption. The problems of the pseudo-interface, coarsening, influence of drift and Kirkendall effect on phase separation, movement of inclusions and the prediction of the final morphology of the two-phase zone are not completely solved.
We compare the Gusak [ ], Morral [ ], multi-multi and the dual-scale [ ] models. The comparison is given for the model and real Ni-Cr-Al alloy. Different diffusion paths, zigzags, horns, Kirkendall flow and various sequences of two-phase and single-phase regions are discussed in view of initial conditions, components mobilities and partial volumes. The comparison with dual-scale model coupled with thermodynamic constraints allows tracing deviations from the local equilibrium condition due to limited rate of precipitate growth/shrinking. The results confirm that models taking into account the diffusion fluxes in both phases are useful in predicting interdiffusion in multi-component and multiphase systems at various boundary conditions and the results are in satisfactory agreement with experiment. The use of thermodynamic data allows self-consistent describing the diffusion and thermodynamic properties of ternary systems. To build the closed system of equations describing the formation and growth of multi-phase zones, it is possible to use additional thermodynamic principles.

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**VIP001**
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### Interdiffusion and Thermotransport in Binary Liquid Alloys

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Diffusion-based phenomenon, such as interdiffusion and thermotransport, in liquid binary alloys is the main focus of this presentation. A clear and convincing understanding of thermotransport is necessary for providing a satisfactory description of the properties of liquid metal alloys. This in turn will be very useful for enhancing technological progress involving these alloys. Thermotransport is generally known from its resulting segregation of the liquid components in a temperature gradient (the Soret effect), or for its contribution to heat flow in liquids (the Dufour effect). Thermotransport in liquids does not have a general theory that can both marshal the experimental facts together and provide an understanding that has predictive power. The major shortcomings involving thermotransport have recently come to a head. As material property information associated with the Materials Genome Initiative for fast-tracking materials development is scrutinized it has been noted that information on thermotransport in liquid metal alloys is largely lacking. A reason for this is that experiments on thermotransport in liquid metal alloys are difficult because of the need to perform temperature gradient experiments in such a way as to avoid the confounding effects of convection and sedimentation. In this presentation a new formalism for describing thermotransport in a liquid metallic alloy with examples of molecular dynamics evaluation of the thermotransport and interdiffusion and tracer diffusion parameters for a range of compositions in Ni-Al and Ag-Cu liquid alloys will be presented.

**VIP007**
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## Isotope-Free Experimental Method for Obtaining Simultaneously Tracer Diffusion and Interdiffusion Information in Alloys

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A novel isotope-based experimental method with sandwich geometry for obtaining simultaneously interdiffusion and self-diffusion coefficients as a function of compositions has recently been developed in [1]. A further, isotope-free version of this experiment is now developed. The sandwich now consists only of a local ‘spike’ in the composition of one or more of the alloy components. Its annealing out at the same time as interdiffusion proceeds also provides immediate access (but only for relatively short diffusion times) to the required self-diffusivities as a function of composition in the diffusion zone. A similar experimental method has been employed to measure self-diffusion coefficients in liquid alloys. The outcomes of this type of experiment can be analysed using the ‘spike’ approach. Results of this type analysis are presented in this paper with extensive discussion.

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**VIP033**
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## Direct Interdiffusion: Monte Carlo Simulations Implemented with Vacancy Thermodynamics

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Vacancy-mediated interdiffusion in binary diffusion couples is simulated by means of a Kinetic Monte Carlo (KMC) algorithm accounting for the assumption that equilibrium vacancy concentration in the system is achieved much faster than the equilibrium atomic configuration. This means that the process of interdiffusion proceeds in under conditions of the locally equlibrated vacancy concentration, whose evolution is strictly coupled with the atomic-migration-induced evolution of the atomic configuration. These conditions are simulated on the base of an Semi Grand Cannonical Monte Carlo (SGCMC) routine implemented in the

KMC code which generates on-line vacancy concentrations according to the virtual atomic configuration in the sample.

Presented are first results of the simulations of diffusion couples composed of binary disordered systems with similar or dissimilar energy barriers for the component-atom jumps to nearest neighbours vacancies. The results are analysed in terms of interdiffusion coefficients calculated either phenomenologically by analysing the MC-determined correlation factors, or directly within the Boltzmann-Matano analysis on MC-generated concentration profiles. The MC-generation of the vacancy wind and the Kirkendall effect is also discussed.

**VIP034**

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## Studies of Atomic Scale Diffusion in Ordered Intermetallics and Glasses by X-Ray Photon Correlation Spectroscopy

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Macroscopic tracer methods achieve incredible sensitivity and allow for drawing conclusions about atomistic diffusion mechanisms. Sometimes, however, it is an advantage to use methods measuring atomic movements directly in space and in time. These studies have been particularly challenging. Only a few scattering methods are sensitive enough to resolve jump vectors on the atomic scale but they are limited to a restricted number of suitable isotopes. Moreover, they are always restricted in the time/energy resolution and thus limited to very fast diffusion processes.

It was, therefore, especially important to search for new methods which would allow to overcome such experimental obstacles and enable to measure a wide range of ordered or short-range ordered alloys as well as amorphous solids. The advent of modern synchrotron sources opened the way for a new experimental technique which is not restricted to particular isotopes and even allows studying diffusivities as slow as the lower diffusivity limit accessible to tracer methods (approximately 10-23 m2s-1). Our group managed to develop the relatively new technique of X-ray photon correlation spectroscopy to work on the atomic scale. This technique operates in the time regime and measures chemical fluctuations rather than self diffusion. Atomic-scale X-ray photon correlation spectroscopy (aXPCS) is not subject to the limitations mentioned above. The time resolution towards faster dynamics is only limited by the readout time of the detector and the intensity of the X-ray beam. Towards slower dynamics it is limited by the measurement time restrictions and the persistence of the experimenter.

The first successful aXPCS experiment was carried out only few years ago by our group [1]. This talk will give an overview of experiments starting from disordered Au-Cu [1] and Ni-Pt [2] alloys to well ordered B2 intermetallics like Fe-Al and Ag-Mg, as well as new application as a direct observation of single atomic motion in lead silicate glass [3] and in fast ionic conductors like alkali borate glasses.

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**VIP035**
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## Marking the Centennial of the Discovery of Alpha Silver Iodide

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In 2014, present-day scientists had the opportunity of marking the centennial of a discovery that triggered the development of a new field of research, which is now called Solid State Ionics. In their 1914 paper, Tubandt and Lorenz reported on the extraordinary properties of the α-phase of silver iodide [1]. Although α-AgI is a crystalline material, it resembles a molten salt with regard to its ionic conductivity. As proved by Tubandt and Lorenz, the current is completely carried by the silver ions, while the iodide ions form a rigid lattice.

For several decades, α-AgI, known as the fast ion conductor par excellence, defied attempts to understand its puzzling features. Around 1980, experiments using neutrons and single crystals eventually led to the emergence of a self-consistent picture of the structure and dynamics of the highly mobile silver ions [2,3].

More recently, confinement in glass has been employed as a technique to preserve the α-phase of AgI at temperatures far below its regular stability range [4]. As shown by measurements of its frequency-dependent conductivity, the silver ions display a more solid-like behavior in low-temperature α-AgI [5]. Indeed, a conjecture made by Jost in 1937 has now been corroborated: The liquid-like activation energy for ionic transport is found to be replaced by a larger value, although the anion structure and, therefore, the barriers for elementary displacements of the cations remain essentially unchanged. The underlying mechanism is outlined.

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**VIP036**
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## Diffusion of Mass in Multicomponent Alloys

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The measurement of diffusion coefficients in multi-component metallic liquids represents an experimental challenge. Measured data are often hampered by convective contributions to mass flow, as well as artifacts during melting and solidification of the sample. Recently we expanded the field through the establishment of versatile, in-situ measurement techniques: QuasiElastic Neutron Scattering (QENS) for the study of self-diffusion [1] and X-Ray and neutron radiography for the study of interdiffusion experiments [2]. These in-situ experiments allow circumventing many additional contributions to mass transport and hence, enabling accurate measurements of transport coefficients with good precision. In multicomponent alloys these measurements are complemented by shear-cell experiments and a subsequent chemical analysis of the diffusion profile.
In this presentation results on self- and chemical diffusion of new experiments on ternary Al-Cu-Ag and Zr-Al-Ni alloys are discussed and set in context to chemical driving forces for interdiffusion, viscous flow and dense packing of the liquid.

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**VIP041**
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## Diffusion Processes in Proton Conducting Materials

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Interest in materials exhibiting proton conduction has increased during the last years owing to their great importance for energy and environmental applications, such as solid oxide fuel cells for converting chemical to electrical energy, solid oxide electrolyzer cells for high-temperature electrolysis of water, or hydrogen permeation membranes for separating hydrogen.
BaZrO3-based oxides are proto-type proton conductors. Using density-functional theory (DFT), we have investigated proton migration energies in Y-doped BaZrO3. The macroscopic proton conductivity was then investigated by means of Kinetic Monte Carlo (KMC) simulations. We analyze the resulting proton conductivities concerning special percolation pathways for protons.
Lanthanum tungstate, LaWO54, is a mixed conductor where oxygen ions, protons and electron holes are mobile. Using the conductivity relaxation technique, we find during hydration a two-fold relaxation kinetics. From the formal analysis of the relaxation curves, we obtain different chemical diffusion coefficients for oxygen and hydrogen. These results show that during incorporation of H2O both hydrogen and oxygen move independently.

**VIP043**
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### Interdiffusion, Crystallography and Mechanical Properties of Ni-Mn-Ga Alloys

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The ferromagnetic shape memory or mangetocaloric properties of NiMnGa alloys are closely related to the martensitic transformation from high temperature austenitic phase to low temperature martensitic phase. The transformation temperature and the resulting microstructure and crystallography of the martensites can be very complex, but are crucial to the optimization of the material performance. A combinatorial study with a series of solid-to-solid diffusion couples and various characterization techniques, including SEM, FIB, TEM, EPMA, and nano-indentation, was carried out to investigate phase equilibria, ternary interdiffusion, microstructural and crystallographic development, and mechanical properties in NiMnGa alloys. Both austenitic and martensitic phases were found at room temperature in each diffusion couple with a clear interphase boundary. The compositions at the interfaces corresponded closely to a valence electron concentration (e/a) of 7.6. Ni was observed to interdiffuse the fastest, followed by Mn then Ga. The interdiffusion flux of Ni also has strong influences on the interdiffusion of Mn and Ga with large and negative cross interdiffusion coefficients. Crystallographic variations in martensitic phase, including non-modulated (NM) martensite and modulated (5M or 7M) martensite, were found in the diffusion couples. The 5M and 7M martensites were only found near the interface between austenite and martensite, corresponding to compositions with lower e/a ratios. The NM martensites were found mostly away from the interface region, with high e/a ratios. All martensitic microstructure consists of twinned variants with different orientations and the twinning relationship, along with the c/a ratio, was correlated to the martensitic transformation temperature. Additionally, the pre-martensitic state has been clearly observed in the cubic austenitic phase region, with distinctive tweed microstructure originating from the local lattice distortions. A decrease of Er and H was observed with Mn or Ni substituting Ga, and Ni substituting Mn for the austenitic phase. However, an opposite trend was found for the martensitic phase. The softening of the elastic constants near the vicinity of martensitic transformation contributed to sharp decreases in Er and H near the interface region. The measured Er and H had larger scatter for the martensitic phase than for the austenitic phase.

**VIP067**
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### Controlling Lattice Thermal Conductivity of Thermoelectric Materials Via Microstructure Modifications

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Thermoelectric (TE) materials are involved in a variety of devices converting waste heat into electrical energy, as well as for solid-state refrigeration and are, therefore, of utmost technological importance. The energy conversion efficiency is determined by the dimensionless TE figure of merit, ZT. Attempts to optimize

ZT require reducing the lattice thermal conductivity, while maintaining relatively high values of electrical conductivity.

We investigate the PbTe- and AgSbTe2- compounds, which exhibit good TE performance at the mid-temperature range. Introducing second-phase precipitates as well as lattice point defects to these base alloys is expected to reduce their lattice thermal conductivity, thereby increasing ZT. We, accordingly, focus on these two avenues. Supplemented by theoretical calculations of phonon scattering, we predict that precipitate number density larger than ca. 1019 m-3 is demanded for reducing lattice thermal conductivity of PbTe. Additionally, lattice dynamics density functional theory (DFT) calculations for AgSbTe2 predict that La substitution defects at the Ag-sublattice sites should result in reduction of the average sound velocity from 1684 to 1563 m·s-1, which is manifested by ca. 14 % reduction of thermal conductivity.

Motivated by these predictions, we manage to reduce thermal conductivity of PbTe by controlled nucleation of Ag2Te-precipitates, and obtain a maximum number density value of 2·1020 m-3 after 6 h aging at 380 °C. This yields ZT value of ca. 1.4 at 450 °C, which is one on the largest values reported for n-type PbTe compounds. Similarly, laser flash analysis (LFA) of AgSbTe2 compounds reveals that La-alloying reduce thermal conductivity from a value of 0.92 to 0.71 W·m-1·K-1 at 573 K. This exemplifies the design of TE properties by microstructure modificaions.

**VIP068**
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## Dual-Scale Model of Diffusive Interactions in Two-Phase Ternary Alloy

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Formation and growth of precipitates and interdiffusion in the two-phase zone of ternary alloy remains one of not well understood diffusion-controlled processes. In the work a dual-scale model of interdiffusion between two-phase ternary alloys is shown. It combines the coarsened scale for overall concentrations and phases redistribution with the fine scale for growth or shrinking of the minority phase precipitates [ ]. The analytical scheme of the model is coupled with thermodynamic constraints. The model is implemented numerically and exemplary results for ternary Ni-Cr-Al alloy are presented. The various types of diffusion paths are discussed in view of initial conditions and mobilities of the components. The discussion focuses on zigzags, horns, redistribution of precipitates due to Kirkendall flow and various sequences of two-phase and single-phase regions. An effect of different molar volumes of the components is discussed. The model allows tracing deviations from the local equilibrium condition due to limited rate of precipitate growth/shrinking. The results confirm that the model is useful in analyzing and predicting complicated interdiffusion microstructures in multi-component and multiphase systems of various boundary conditions.

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**VIP069**
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## Diffusion in Neutron-Irradiated U-Bearing Intermetallics and Alloys

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Neutron irradiation can have a dramatic impact on the interdiffusion behavior of fuel constituents in a nuclear fuel material. The fission process produces many defects in the fuel microstructure and the local compositions change over time due to the production of fission products, and this can result in an increased mobility of constituents at relatively low temperatures. The results can be phase changes, grain growth, swelling, creep, etc. As part of international programs to develop low-enriched uranium fuels for application in research and test reactors [1], a variety of U-bearing fuel materials have been irradiated at relatively low temperatures (<200˚C) and microstructurally examined. Some of these materials include: U3Si, U3Si2, U6Fe, U6Mn, U-Mo alloys (up to 12 wt.%), U-Nb-Zr alloy, U-Mo-Pt alloy, U-Mo-Os alloy, etc.
This presentation will describe the interdiffusion behavior that has been observed for different fuel materials that have been irradiated in the Advanced Test Reactor, located at the Idaho National Laboratory. Focus will be given to the as-irradiated microstructures that have been observed using optical metallography, scanning elctron microscopy with energy dispersive spectroscopy (EDS), and transmission electron microscopy with EDS, and how diffusion processes have changed the microstructures vis-a-vis as-fabricated microstructures. Many samples have been produced for characterization using a focused ion beam, which allows for some very detailed observations to be made. Using a focused ion beam to make samples from irradiated fuel is a very unique capabililty that will enable the interdiffusion behavior in irradiated fuels to effectively be investigated into the future. Comments will be made about amorphization, fission gas bubble growth, creep, swelling, precipitate growth, and the general interdiffusion behavior of alloy and fission product constuents of some of the different U-bearing materials that have been irradiated over the past few decades.

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**VIP031**
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## The Thermophysical Bases of Monitoring of the Fireproof Lining Wear in the Blast Furnace Hearth

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The monitoring system of the fireproof lining wear of the blast furnace hearth is offered. The mathematical description, algorithm and program of calculation the temperature fields in any vertical and horizontal cross-section of the hearth lining are developed on base of the heat conductivity equations with use of the indications of the temperature sensing transducer in the hearth lining. The calculation algorithm is developed regarding the account of an intricate profile of blast furnace hearth with use of a Nyquist theorem. The continuous control of the temperature change in each point allows to define the remained thickness the firebrick lining and to warn the furnace personnel about the beginning of the firebrick lining deterioration. The technique of definition of the optimum quantity and arrangement thermocouples in lining of the furnace hearth is made. The technique of determination of thermophysical properties of refractory materials according to the temperatures diagram at blowing-in of the furnace is developed. This monitoring system is established on five blast furnaces of metallurgical plants of China [1-3].

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**DSL242**

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## An in Situ High-Temperature X-Ray Diffraction Study of Phase Transformations in Maraging 300 Steel

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The phase transitions in the maraging 300 steel were studied using high temperature X-ray diffractometry. Maraging are ultra-high resistant steels with Ni-Co-Mo-Ti presenting a broad range of application in key areas such as nuclear and aerospace industries [1]. Prolonged high temperature exposure would lead to the formation of equilibrium austenite and ferrite phases [2]. Solution annealed maraging 300 steel was continuously heated at a 10 ºC/min rate from ambient temperature until 900 ºC and X-Ray measurements were done at 25, 400, 500, 600, 650, 700, 800 and 900 ºC. Dilatometric curve at the same heating rate and temperature range were performed and the results were compared with high temperature X-Ray diffraction. The martensite to austenite transformation and vice-versa could be detected by both techniques, although the precipitation could be detected only by dilatometry, associated to low volume of precipitates to be detected by X-Ray diffraction. The effect of austenite reversion at 600ºC was also studied after at 1, 2, 3 and 4 hour of exposure, showing that the amount of reverted austenite is proportional of time exposure at high temperature.

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**DSL236**

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### Crystallization Kinetics in $Cu_{50}Zr_{43}Al_7$ Bulk Metallic Glass

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Crystallization kinetics of a $Cu_{50}Zr_{43}Al_7$ bulk metallic glass was investigated through differential scanning calorimetry (DSC) in isothermal mode using Johnson–Mehl–Avrami model. Samples were prepared by copper-mold casting method. Amorphousness of the as-cast samples was confirmed by XRD. Each DSC curve of the BMG exhibited an incubation period followed by an exothermic peak corresponding to the crystallization of the amorphous phase indicating that the devitrification of $Cu_{50}Zr_{43}Al_7$ BMG has followed a one-stage process. The activation energy for crystallization was about 471 kJ/mol obtained by the Arrhenius equation. The Avrami exponent was determined to be 2.2 implying that the crystallization is governed by three-dimentional interface-controlled growth. At the end, fragility index was also calculated and discussed.

Keywords: Bulk metallic glasses, Crystallization kinetics, Avrami exponent, differential scanning calorimetry

**DSL237**

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### Co-Segregation of Boron and Carbon at Dislocations in a Dual Phase Steel

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It is well known that solute atoms tend to segregate to defects of the crystal structure of alloys. Their presence at the defects can significantly change the physical and mechanical properties of materials [1]. In steels, for example, segregation has different effects. One of them is the reduction of heterogeneous precipitation at grain boundaries: in the case of boron-added dual phase steels, boron segregates at austenite grain boundaries, decreasing their interface energy. In this way, boron retards the austenite-to-ferrite transformation [2]. High levels of carbon and boron enrichments at grains boundaries in steels have been reported. Carbon segregation at dislocations was also reported, but there was no evidence of boron segregation at dislocations. As boron segregation plays an important role on the hardenability of boron-bearing steels, quantifying the levels of boron segregation at dislocations is a necessary task in this context. A dual-phase Nb-Ti micro-alloyed steel was investigated by Atom Probe Tomography (APT) to quantify solute segregation levels at dislocations before and after intercritical annealing. These results showed boron and carbon accumulation along a dislocation in two phases, ferritic phase before annealing and martensitic phase after intercritical annealing. This study presented a high level of boron at dislocation, which could considerably reduce the boron quantity available to segregation at austenite grain boundary. Therefore, B segregation at dislocation has a non-negligible effect on the hardenability of low carbon steels. The segregation levels of C and B and their co-segregation mechanisms will be discussed.

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**DSL241**
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## Motion Equations of Helium Atom Through Disordered Quartz Structure

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In order to describe Helium atom through disordered quartz structure is used local chains method [1,2]. Number of local chains equals to number of nearest neighbours. Each local chain include He-atom certainly. Transition of He-atom from origin place to neighbor one imply two opportunity: (1) local order is kept, (2) local order is changed. Last situation means the structure element of quartz, $SiO_4$, is removed / added with respect to He-atom. Distortion of local order creates defect of order. Distortion of local order means turning on additional average field proportional to internal “surface square” of the defect. Transition He-atom from origin place to neighbor one generates space-periodical field, which acts on He-atom. By the way a problem of He-atom motion through disordered quartz structure is reduced to Frenkel-Kontorova model. It allows to take into account mass, size and quantum states of atom at diffusion process.

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**DSL243**
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## Prediction of Boride Layer Depth Obtained on XC38 Steel in Molten Salts using Adaptive Neuro-Fuzzy Inference System

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This paper discusses an application of neuro fuzzy inference system (ANFIS) on the performance prediction of boride layer thickness. Boronizing treatment was carried out in three different molten salts consisting of borax ($Na_2B_4O_7$) added to boron carbide ($B_4C$), aluminum (Al) and silicon carbide (SiC). The substrate used in this study was XC38. Boride layers involved in this work were obtained from boriding treatments at temperature range of 800-1050 °C with 50 °C interval for 2, 4 and 6 h. Temperature of treatment and duration time are taken as input, boride thickness as the output of the model. Variation of the layer depth has been modelled using ANFIS, and the experimental results have been compared to each other’s. In addition, we

compared the proposed ANFIS model with artificial neural network (ANN) approach and fuzzy logic approach. The results have shown that the ANFIS model is more accurate and reliable compared to the ANN approach.

Keywords: Adaptive neuro-fuzzy inference system; Borides; layer depth; Molten salts; XC38 steel

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**DSL245**
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## Morphological Evolution of Bi-Crystalline Thin Film Surfaces Due to Combined Surface and Grain Boundary Diffusion Driven by Capillarity and Thermal – Stress Gradients

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Evolution of thin film surfaces is a problem of technological importance hence it provides a mechanism for VLSI interconnect failure. In this study, we examine the morphological evolution of a bi-crystalline thin film surface by combined surface and grain boundary (GB) diffusion under the action of capillary forces, and thermal stress gradients associated with a steady state heat flow. It is assumed that the GB remains perpendicular to the free surface during the overall process and the evolution dynamics of surfaces was simulated by using front tracking method that strictly follows an irreversible thermodynamics model to describe the non-equilibrium nature of GB triple junction motion. The displacements due to the presence of the thermal stresses, induced by the applied uniform temperature gradient, were computed at the interior (GBs) and at the boundary (free surface and film/substrate interfaces) points using the indirect boundary element method. The motion of the triple junction and the evolution of stress/strain fields within the film were followed while systematically considering the effect of elastic dipole tensor interaction between the stress field and the mobile atomic species situated at the surfaces and GB layers.

**DSL246**
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## Stochastic Kinetic Mean Field Model

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We introduce a new model for calculating the change in time of three-dimensional atomic configurations. The method is based on the kinetic mean field (KMF) approach, however we have transformed that model into a stochastic approach by introducing dynamic Langevin noise. The result is a stochastic kinetic mean field model (SKMF) which produces results similar to lattice kinetic Monte Carlo (KMC). SKMF is, however, far more cost-effective and easier to implement the algorithm (we also made an open source program code available). [1]

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**DSL248**
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## Evaluation of Metallurgical and Mechanical Properties of Dissimilar Metals Welds between Duplex Stainless Steel and HSLA Steel

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Dissimilar material joining is generally more challenging than that of similar materials, due to various factors such as the differences in chemical compositions and the physical and mechanical properties of the base metals welded

In this study, the metallurgical characteristics, and mechanical properties of dissimilar metals welds between duplex stainless and Low alloy steel have been evaluated. Duplex E2209 and Austenitic E309L filler metals were used to join this combination using multipass gas tungsten arc welding process.

The microstructure investigation was conducted with optical microscope, scanning electron microscope, Energy dispersive scanning and X-ray diffraction.

EDS line scanning performed across the interface low alloy steel/weld metals reveals a variation of alloying elements (Cr, Ni, Mo) across this interface.

A higher value of hardness is recorded in a narrow zone between the fusion boundary and type II boundary, which is attributed to the presence of harder phases in this region, due to the migration of carbon from the HSLA side to the weld metal. Both of welds gave acceptable values of tensile strength and impact toughness

**DSL242**
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## Microstructure and Properties of Composite WC-8(Co, Ni): Effect of the Addition of SiC

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The WC-based cemented carbides, also called hard metals, are a family of composite materials consisting of carbide ceramic particles embedded in a metallic binder. They are classified as metal matrix composites (MMCs) because the metallic binder is the matrix that holds the bulk material together [1]. WC based composites are used in applications where a good combination of hardness and toughness are necessary [2]. It is usual to add more components as the tungsten carbide in a binder (Co, Ni) to build the microstructure. The hardness for the cemented carbides based on nickel, because of the addition of reinforcements SiC nano-whisker increases significantly [3]. In this work, the SiC was researched as an additional component for composite WC-8(Co, Ni). Four mixtures were prepared with SiC content ranging from 0 to 3.0 wt%. These mixtures were pressed (200 MPa) and green samples with 25.2 mm of diameter and 40g were produced. Sintered were carried out in Sinter-HIP furnace (20 bar). Two sintering cycles were investigated with 1380 and 1420°C, and the sintering time considered was 60 minutes. The relative density, hardness, linear and volumetric shrinkage were determined. Microstructural evaluation was performed by optical microscopy and scanning electron microscopy (SEM-FEG). The results showed that the addition of SiC promoted higher densification and grain growth. The hardness was higher for samples with SiC, so solid solution hardening of the binder was more effective than WC grain growth.

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# SPECIAL SESSION 4
# ALTERNATIVES ENERGY(SS4)

**VIP032**
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## On The Use of Genetic Algorithms for Parameter Identification in Anaerobic Digestion Modelling

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Anaerobic digestion (AD) of wastewater is a very interesting option for waste valorization, energy production and environment protection [1]. It is a complex, naturally occurring process that can take place inside bioreactors. The capability of predicting the operation of such bioreactors is important to optimize the design and the operation conditions of the reactors, which, in part, justifies the numerous AD models presently available. The existing AD models are not universal, have to be inferred from prior knowledge and rely on existing experimental data [2]. Among the tasks involved in the process of developing a dynamical model for AD, the estimation of parameters is one of the most challenging.

This paper presents the identifiability analysis of a nonlinear dynamical model for a batch reactor. Particular attention is given to the structural identifiability of the model, which considers the uniqueness of the estimated parameters. To perform this analysis, the GenSSI toolbox was used [3]. The estimation of the model parameters is achieved with genetic algorithms (GA) which have already been used in the context of AD modelling, although not commonly. The paper discusses its advantages and disadvantages.

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**DSL089**
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## Investigation of Sunlight Behavior Through Multilayer’s SWCNT-Tio2 Silicon Solar Cells

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Reflection of the incident photons by the silicon surface is a major source of losses for photovoltaic solar cell. However, these losses can be minimized by depositing an antireflection layer. Due to their particular atomic structure, carbon nanotubes have fascinating chemical and physical properties as indicated by

graphite and diamond characteristics. They have acquired critical achievements in various fields such as materials, electronic devices, energy storage, separation, sensors, and so on. Recently, antireflective coatings (ARCs) with self-cleaning properties attract critical consideration for both their fundamental aspects and wide practical applications. To optimize the antireflection multilayer, we have developed a numerical simulation code with Matlab software package where we have used the Transfer Matrix Method (TMM) to solve the optical equation. These solutions permit us to plot the optical reflectivity and the absorption versus wavelengths and layer thicknesses. The optical refractive index and thicknesses of considered materials, which allowed us to have the lowest reflection, can be used to simulate the electrical properties of the cell with PC1D and Silvaco software in the future.

**DSL095**

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## Application of Chemical Treatments in Sisal Bagasse for Cellulose Conversion to Glucose by Enzymatic Hydrolysis

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To mitigate the search for renewable energy sources, emerged a growing interest in the production of alternative fuels from lignocellulosic materials. Although abundant, these materials need pretreatment to reduce its resistance to hydrolysis, which occurs mainly due to the presence of lignin. In this context, there is a need to develop methods allowing delignification biomass, leaving free the pulp necessary for the production of ethanol. In this work we were applied chemical treatments (Fenton and acid followed by basic), where it was intended to produce glycosidic liquor, of Sisal bagasse (Agave Sisalana) previously treated in order to evaluate its potential in the conversion of cellulose to glucose by enzymatic hydrolysis. The proposed methodologies obtain significant results as regards the solubilization of the hemicellulose and lignin removal following of the proposed pre-treatment, obtaining good conversion results.

**DSL099**

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## Role of Substrate Temperature on the Structural and Morphological Properties of Cu2ZnSnS4 Thin Films deposited by Ultrasonic Spray Pyrolysis

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Keywords- CZTS, spray technique, thin films, photoluminescence, Transmission, XRD.

The influence of substrate temperature on the properties of Cu2ZnSnS4 thin films elaborated by spray ultrasonic method has been investigated. Samples are deposited at various substrate temperature ranged from 280 to 360 °C about 45 min. the results of X-ray diffraction analyses indicated that Cu2ZnSnS4 films have nanocrystalline structure with (112) preferential orientation and reveals the formation of ZnSnO3 and Cu2ZnSnS4 phases. The crystalline size is varied from 20 to 45 nm with increasing substrate temperature. The optical films characterization was carried out by the measurement of UV-visible transmission. The optical gap was deduced from the absorption spectra. The photoluminescence spectrum measured at 77 K showed a broad emission around 1.27 eV

**DSL112**

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## Experimental Study of Pollutant Emissions of Dual Fuel (Diesel, Biodiesel and Natural Gas) in Diesel Engines

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Fossil fuels are in continuous process of reduction. Find alternative fuels to reduce emissions of pollutants, it is necessary. Biodiesel is one of the best renewable resources available that have appeared recently.
The gaseous fuel used in engines of the diesel cycle has been much studied in recent years, due to the characteristics positive of these fuels when the focus is the level of emissions launched in the atmosphere. The challenge is to transform the engine diesel into dual engine by using adequate mixtures of diesel, biodiesel and natural gas to obtain correct ratio of each fuel and to maintain the original characteristics of diesel engine and good performance [1,2].
In this sense, the objective of this research is to study the emission characteristics of a commercial diesel engine working with biodiesel (B40, B50 and B60) and natural gas mixture. The fuel used in motor consists of 15% mixture of diesel and biodiesel (liquid fuel) and 85% natural gas; experiments were carried out in powers 80, 100 and 120 kW. The engine was instrumented in order to obtain temperature, mass flow rate, pressure and concentration of combustion gases during the experimental test. From the experimental results it was verified that NOx, NO, and CO2 concentrations decrease. It was also verified an elevated reduction on level the emissions of these exhaustion gases compared to diesel standard.

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**DSL124**

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### Impact of Renewable Energy on Economic Growth

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Clearly Fuel fossil reserves are finite, and lead to an untenable situation, due to its side effects on the environment, and the concentration of carbon dioxide in the atmosphere.

In order to resolve this problem, the economies intervened and proposed the alternative energies as a solution. But these alternative energies satisfy only 25% of our needs.

For this reason, we propose a study of an economic model that guarantees the needs of the world energy.

**DSL176**

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### Harvest of Cladding Light of Fibre Laser Systems

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Near-infrared (NIR) fibre lasers have been widely used in many applications, however, their residual, i.e. unabsorbed pump light or known as cladding light can amount to 5 – 30 % of the total output power. This will not only sacrifice the efficiency of the system, but also create the thermal issue when the power of interest increases. In this paper, we propose to harvest this wastage by integrating the fiber laser system with photovoltaic (PV) cells and convert the residual into energy for cooling purposes.

We investigate various commercially available PV cells, with different dimensions, rating and material structures such as amorphous and crystalline PV cells under different illuminations covering from visible to NIR range. As predicted, amorphous PV cells show little or no response to the NIR emissions, while monocrystalline silicon PV cells show a much more significant response. Hence it has been chosen as the candidate for the task.

We also study the placement configurations of the PV cells for better absorption of the cladding light from the fiber laser. The PV cells are arranged to surround the cladding mode stripper of the fiber laser, and in a manner that the reflected light from other cells can be captured.

With proper selection of the low cost commercially available PV cells and placement configurations, we obtained an optical-to-electrical efficiency of about 16% under the illumination of fiber laser residual.

We have demonstrated that the cladding light of fiber laser systems can be harvested, however more cells and cells with high efficiency are needed in order to drive a practical cooling system such as fan or TEC cooler for the laser.

**DSL216**

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## Twice Optimized Evaporative Cooling System with Vortex Tube

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Indirect evaporative cooling is currently seen as the best available technology for energy efficiency enhancing of air conditioning in buildings. With a potential market growth valued at 20% for the next two decades, the evaporative cooling technology is certainly an interesting bet for significant savings in energy consumption and consequent reduction of carbon dioxide emissions. A new hybrid system for evaporative cooling with Tube Vortex (IEC/ VT cooler) is presented for a better approach toward a solution to control the great dependency on the ambient air conditions of performance of the evaporative cooling that leads to very limited cooling capacity in damp or mild climates. The functional characteristics and performance of the newly presented IEC/ VT cooler system are evaluated when assuming that heat and mass transfer processes occur within fixed control volumes with geometric configurations optimized according to the Constructal theory [1, 2]. The two operating regimes studied were defined by considering a minimum temperature between the adiabatic saturation temperature and the temperature of dew point ( ) and when temperatures below the dew point are needed. Numerical values of the coefficient of performance were determined in the range. In this study has also been considered the possibility of recovering the residual energy from pressurized gaseous effluents, whose cost is almost zero, to check the economic feasibility of the novel IEC/ VT cooler system for industrial applications.

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**DSL224**

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## Development of Novel Electrode Materials for Symmetrical Sofcs

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The technology of Solid Oxide Fuel Cells fueled by carbon-containing fuels is particularly of interest, as the usage of fuels, such as methane, natural gas instead of pure hydrogen can considerably decrease the cost related with fuels, therefore, significantly accelerate the commercialization step [1-4]. When such fuels applied, carbon deposition can occur on the typical used Ni-YSZ anode, and consequently substantially reduces the SOFC performance. Sulfur poisoning on the anode is also unfavourable, as such fuels usually

contain impurities, such as sulfur with only very small amount can noticeably deteriorate the cell performance [1, 2]. Application of symmetrical SOFCs with identical electrodes can allow to remove the deposited carbon or sulfur on the anode just by switch between anode and cathode. Among novel anode materials, A2M (Mo,W)O6-δ (A: Sr, Ba; M: 3d metals)-type perovskites seem to be particularly of interest [1-4].
In this study, Mo-containing perovskites Sr(Fe,Ti,Mo)O3 and W-containing materials with SrFe1-xWxO3 formula were proposed as novel anode and cathode materials for symmetrical SOFCs. The physicochemical properties including: the crystal structure change function of temperature, thermal expansion under reducing and oxidizing atmospheres, transport properties, oxygen nonstoichiometry and chemical stability in operation conditions were investigated, and electrochemical properties of proposed electrode materials were studied in hydrogen and methane. Elucidation of the coking mechanism on the anodes is also addressed.

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**DSL235**

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## Hydrogen Permeability of Ultrafine-Grained Vanadium

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The grain refinement is important technique to create the new hydrogen structural and functional materials. As for the vanadium, it may contribute to the improvement in resistance to hydrogen embrittlement promising as a candidate membrane for hydrogen separation and purification technology. In recent study [1], the hydrogen permeation test is reported for fcc palladium under a controlled temperature in the range of 100-500°C. In that case, hydrogen diffusion is almost the same between coarse- and ultrafine-grained states at temperatures above 200°C. However, it is increased at temperatures below 200°C. Following this result, the hydrogen permeability of ultrafine-grained bcc vanadium is evaluated by the following experimental methods.
In this presentation, the importance of measurement of the pressure-composition-isotherm (PCT) curve for estimate the hydrogen content in the vanadium matrix before performing a hydrogen permeation test will be informed. The difference between coarse- and ultrafine-grained states for vanadium under a temperature range of 300-500°C hardly appeared concerning the PCT curves. Also, the conditions of hydrogen pressures of inlet/outlet sides are decided based on corresponding PCT curve and, then the hydrogen permeation test are performed. A series of experiments, the ultrafine-grained vanadium show even 300°C of 2.5 to 5 times as high values as that of the hydrogen permeation coefficient, f, of virgin (=coarse-grained) vanadium. Since the influence of the crystal grain size exerted on a PCT curve is not observed, it seems that the enhancement in f results in the increase in the diffusion coefficient of hydrogen. The GB area which increased with the ultrafine structures controlled by giant straining process may have functioned as a rapid diffusion path of hydrogen.

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**VIP023**
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## On Transfer Processes at Phase Transition in Porous Structures

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The lecture represents analysis of fundamental principles of heat transfer processes in heat pipes. High heat transfer coefficients due to phase transition in porous structures performed by these devices is one of the crucial factors making heat recovery and renewable energy applications and the development and use of other alternative energy technologies with low heat potential highly effective. Phase transition in porous medium of complex geometry with many length scales, surface phenomena, effective transport coefficients compared to phase change without porous medium is of interest from physical point of view as well.

Results of extensive experimental studies and physical modelling of evaporation from porous media saturated with water and film boiling at high heat fluxes resulting in turbulent filtration of vapour films at the surface imbedded in porous media are summarised in the lecture. The correlation between the porosity and surface area of the porous structures on the evaporation rate is discussed. Porous media is made up from spherical brass and glass balls of different sizes.

The second fundamental process studied is film condensation at the surface imbedded in porous media. The lecture represents results of systematic studies of film condensation on surfaces embedded in porous media and physical models which take into account the most significant effects and parameters of phenomena covering the Darcy regime, non-linear filtration and the near-wall zone effect due to the change of porosity in comparison with experimental results. Particular attention is given to the existence of two-phase zone above the film dramatically changing the scenario of heat transfer. A recently developed asymptotic analysis of this zone contribution allowing an analytical solution is discussed. Experimental data are used for validation of physical models developed.

**VIP032**
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## Assessment of the Availability of Forest Biomass for Bioenergy Production in Southwest Portugal

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The use of biomass for energy production presents several benefits and is generally foreseen as playing a key role in a future, more sustainable energy supply. The Portuguese solid biomass targets by 2020 set in the National Renewable Energy Action Plan are: 367 MW installed capacity for electricity production, 126 ktoe of electricity produced and 1484 ktoe of thermal energy produced. With an increasing use of biomass in the future Portuguese energy production, it is necessary to exploit more available biomass in a sustainable way. This is reinforced by the fact that, presently, Portugal is one of the larger exporters of industrial pellets in Europe. A good assessment of the biomass availability for energy purposes at both country and regional levels is, therefore, required.

This work focuses on the assessment of the biomass availability in a region in Southwest Portugal. The main purpose of this work is to access if in the area there is enough biomass to feed a local pellet production plant. An allometric function was used for the estimation of maritime pine above ground biomass, applied in a remote sensing data and Geographical Information Systems (GIS) environment. The study area is a forest stand of about 1000 ha. The available biomass for energy was estimated according to the forest species and stand composition and structure. The analyzed components related to the biomass potential are: effective biomass area (in ha), biomass availability (in dry t/year) and energy potential (in GJ/year).

# Special Session 5

# CARBON AND OXIDE NANOSTRUCTURES (SS5)

**VIP038**
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## Graphite/Metal Electrodes for Electrochemical Exfoliation: Few Layers Graphene with Low Defects

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The electrochemical approaches for exfoliation of graphite to obtain graphene have been intensively studied in the last decade, since they are still considered as industrially attractive, due to several advantages over other liquid phase processing methods. In this work graphene material was obtained by electrochemical exfoliation of graphite and metal/graphite electrodes of different compositions and electrical conductivities. The concentration of the electrolyte in liquid solution was also varied in order to study the reaction and velocity oxidation process of the graphene flakes [1]. Metal/graphite electrodes were prepared using high purity copper and nickel precursor and commercial graphite [2]. The source electrodes were disposed to work both, via cathodic reduction as well as anodic oxidation. Processes of rapid expansion and direct exfoliation of graphite in a H2SO4 solution were observed using voltages from 2V to 15V and currents of 0.03mA to 0.08mA. The total time for each process was one hour and the maximal concentration of few layers graphene flakes was 0.002mg/mL. The oxidation degree varies in dependence of electrolyte concentration. X rays powder diffraction of the expanded electrodes showed the effect of the electrochemical process in the crystallinity and the increasing of interlayer distance. A characterization of a large amount of graphene flakes was performed by Raman spectroscopy and optical microscopy [3]. Typical size of the flakes are between 1μm and 10μm and the Raman spectra indicate number of layers from single or bilayers to approximately ten layers. Size and thickness are related to the processes of ion intercalation and expansion and the greatest variations in thickness of flakes occur when the intercalation process concludes before the expansion of the layers. It was also observed that the few layers flakes form agglomerates, at higher concentrations of acid. Raman spectroscopy is essential to distinguish these agglomerates from remanent graphite. A low degree of oxidation and of structural defects was characteristic of the experiments of lower acid concentracion.

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**DSL091**
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## Synthesis and Characterization of Tin Oxide Sno2 Nanoparticles-A Potential Candidate for Gas Sensors

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Tin oxide (SnO2) nanoparticles/ nanomaterial-the candidate for gas sensing- are synthesized by Sol-Gel and Chemical precipitation methods and are characterized using X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray spectroscopy(EDXS). The outcome shows the Tetragonal Rutile structure of SnO2 nano-particles having the mean sizes of 9.63nm and 15.65nm by Sol-Gel and Chemical precipitation method respectively. A comparison study of this Metal oxide semiconductor concludes that Sol-Gel technique is suitable for gas sensing; having fine grain structure and good surface morphology.

**DSL104**
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## Analysis of the Effect of Heating Temperature on the Fabrication of Al Nanowire Array using Electromigration

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Al nanowires can be used as important components for micro/nanoelectro-mechanical system (MEMS/NEMS) and small devices, therefore, it is very attractive to fabricate Al nanowire array in an effective approach [1]. Traditional approach for forming Al nanowire is based on deposition and etch processes, however, this approach has its limitations due to the wavelength used to expose resist and the etching precision. Some specific techniques such as focused-ion-beam (FIB) etching and fast-atom-beam (FAB) etching can be used to fabricate Al nanowire, however, these processes are very time-consuming and need high cost. It is noted that the approaches based on techniques to control atoms and molecules [2], such as the vapor-liquid-solid mechanism and electrochemical deposition, can be employed for making nanostructures. However, these techniques are somewhat inefficient. Recently, it was reported that electromigration (EM), which is a physical phenomenon, can be used to fabricate metallic micro/nanomaterials. Al thin wires and micro-spheres have been fabricated on a Si wafer by using EM, and high current density and high heating temperature were used as the driving force [3]. However, it is still a great challenge to obtain Al nanowire array in a short time because the combination of current density and heating temperature was difficult to be optimized for Al nanowire array fabrication.

In this work, the effect of heating temperature on the growth of Al nanowire array was investigated by using finite element analysis technique. By using same current density, atomic diffusion was enhanced by increasing heating temperature, which should be controlled in a suitable range to avoid failure due to fast atomic accumulation. A optimized combination of current density and heating temperature was proposed for fabricating Al nanowire array.

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**DSL157**
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## Electrospun Fibers of Luminescent Eu(Tta)3(Phen)/ PVK Nanocomposites

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Luminescence in polymers can be induced by doping them with rare-earth complexes such as Eu(tta)3(phen) (tta=2-thenoyltrifluoracetonato; phen=1,10-phe3nantroline). Europium complex nanoparticles are interesting due to their extremely sharp emission bands and long fluorescence lifetime [1]. Previous works have shown that poly(vinylcarbazole) (PVK) is an electrospinnable photoconductor polymer that exhibits interesting electrical and optical properties [2]. In this work the morphology and photoluminescence properties of electrospun Eu(tta)3(phen)/ PVK nanocomposites fibers were investigated.

The fibers exhibited leaflike and ribbon structures, with an average width of ~ 6 µm. TEM micrograph indicated Eu(tta)3(phen) nanoparticles (~150-300 nm) uniformly dispersed in the PVK matrix. The electrospun nanocomposites fibers showed a bright red fluorescence, with five characteristic bands due to the electronic transitions of the Eu3+ ions [3]. A band peaking at 410 nm, attributable to the PVK, was also observed suggesting that energy transfer from PVK to the Eu(tta)3Phen complex is not efficient. On the other hand, in the excitation spectra of the nanocomposite fibers, a notorious peak appeared on the last part of the broad absorption band of the neat Eu complex, which height depended on the solvent used. This behaviour could be ascribed to a distortion of the ligand filed arising from the interaction with the PVK and/or solvents.

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**DSL163**
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## Optical Characterization of Inhomogeneous TiO2 Thin Films

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In this contribution the optical characterization of inhomogeneous TiO2 thin films is presented. Atomic force microscopy (AFM), spectroscopic ellipsometry and reflectometry are used for this characterization [1]. It is shown that the combination of these methods enables us to determine the parameters describing the optical properties and the inhomogeneity of TiO2 thin films. Moreover, the values of roughness parameters of the TiO2 films are determined [2]. The limitations of this characterization are discussed too.

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**DSL188.1**

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## Tio2 Nanotube Fabrication by DC Anodization on Cp-Ti Samples After Preoxidation in an Alkaline Electrolyte

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Semiconductor TiO2 nanotube arrays, attracted large attention and investigated in detail lately for applications such as solar energy conversion, sensing, photocatalysis. One of the main applications for TiO2 nanotube arrays is dye-sensitized solar cells (DSSCs). For increasing the efficiency of DSSCs, it is very important to optimize TiO2 nanostructures which are used in photoanodes [1]. Most common way to produce well arranged TiO2 nanotube arrays is electrochemical anodic oxidation of titanium. It was reported that anodic oxidation of titanium in fluoride containing electrolytes produced TiO2 nanotube morphology on the surface [2]. After that many reports have been made to optimize nanotube structures by using different electrolyte compositions and electrical parameters.

In this work TiO2 nanotubes arrays were prepared on electropolished Cp-Ti foils via two step anodization by using a unipolar DC power supply. In the first step, Cp-Ti foils were anodized in an alkaline electrolyte to form an initial oxide layer. In the second step the preoxidized samples were anodized in 1% HF electrolyte to produce nanotube morphology. Different levels of voltages were applied in both steps of anodic oxidation. Following anodization, structural and morphological characterizations were performed by Scanning Electron Microscope (SEM), qualitative X-ray diffraction analysis (XRD) and contact angle measurements.

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**DSL198**

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## Elaboration of Ag3Sn Nanoparticles with Polyol Process: Effect of Synthesis Parameters

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Electronic chip is connected to a copper substrate using an interconnection material, which usually limits thermal and mechanical stresses between both [1]. Interconnection materials are nowadays often composed of alloys containing lead [2]. They will be phased out for respecting environmental European regulations due to lead toxicity. The elaboration of lead-free interconnection materials is a new challenge. Ag3Sn phase shall likely presents better performances for electronic applications, it was chosen according to its high melting point (480°C). In the literature, Ag3Sn samples are prepared from bulk rods of tin and silver [2]. The main objective of this study is to synthesize pure Ag3Sn alloy nanoparticles with polyol process.

Polyol process is a soft chemistry method to elaborate particles with a controlled morphology [3, 4]. This method was already used to synthesize nano-objects with various composition and morphology: tin nanoparticles [4], silver nanowires [5]. This process follows several steps [6]: metallic precursors, often salts, are dissolved in a polyol solvent. The solution is heated at a moderate temperature in presence of other reagents, like protective agent Polyvinylpyrrolidone (PVP) and reducing agent. Metallic precursors are reduced in solution, and Ag3Sn nanoparticles precipitate. Hence, an adapted polyol method has been chosen to study the effects of varying experimental conditions (heating time, PVP mass, concentration of reducing agent, etc) with an initial molar composition of tin and silver. Samples were characterized with XRD, SEM, DTA/TGA and DSC. The results have shown that a heating time of six hours and a high concentration of reducing agent were more appropriate to reach a pure Ag3Sn phase in presence of PVP as protecting agent.

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**DSL200**

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## Investigation of Mechanical properties and Fracture of Polymer-clay Nanocomposites

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Classical polymer-based composites have improved strength and modulus over pure polymers, but they notably possess lower ductility and elongation, especially at high volume fractions, which are needed to attain better mechanical properties. Polymer-clay nanocomposites (PCNC), on the other hand, do offer better properties at very low volume fraction of the nano-filler, which minimizes the effect on other favored properties of the polymer [1]. An extensive work on mechanical and fracture behavior of epoxy-clay nanocomposites presented an explanation of mechanisms leading to fracture starting from micro-cracking at clay epoxy interface to coalescence and catastrophic failure [2]

This work investigates mechanical and fracture behavior of polymer-clay nano-composites, and the improvements in different responses. It builds on previous efforts to explain the improvements due to the presence of the second phase of clay nano-particles in very small amounts whereby proper distribution of particles is assumed. Mechanical response to stressing and fracture behavior are modeled using finite element method and the mechanisms for improved response are investigated. As the high dispersion of clay platelets is evident from experimental work, tortious path of the crack propagation is assumed to be the dominating mechanism for fracture and energy dissipation.

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**DSL204**
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## Synthesis and Characterisation of Nanocomposite MW Carbon Nanotubes – Nanocrystalline Nay Zeolite

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Zeolite Y, a highly versatile member of the faujasite family, plays a great role in the petrochemical industry [1, 2]. Nanocrystalline zeolites are zeolites with discrete, uniform crystals with dimensions of less than 100 nm that have higher external surface areas and reduced diffusion path lengths relative to conventional micrometer-sized zeolite crystals [3]. Zeolites with particle size in the nanometer range have been recently subject of intense research [4].

The present study covers the synthesis of nanocrystalline zeolite Y by applying a two-stage varying temperature synthesis without template under hydrothermal conditions through crystallization of gel in mesoporous system of multi-wall carbon nanotubes (MWCNT) with an internal diameter of 10~30 nm. Those, sodium aluminate and colloidal silica are used as Al and Si sources, respectively, to synthesize the small and uniform zeolite crystals. The crystal size of zeolite Y is influenced by temperature, aging time, alkalinity, water content and mesoporous system. The synthesized nanocomposite (NaY nanocristalline-MWCNT) is characterized by X-ray diffraction (XRD), Fourier Transmission Infrared Sprectroscopy (FTIR), Transmission Electron Microscope (TEM), Scanning Electron Microscope (SEM) and Nitrogen Adsorption.

These characterizations show that the obtained nanocrystals possess the typical nanosized NaY zeolite structural characteristics combined with multi-wall carbon nanotubes.

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**DSL234**

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## Effect of Coating on Heating Curve of FeCoV Magnetic Nanoparticles

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The effect of coating on heating curve of FeCoV magnetic nanoparticles was studied. Iron cobalt vanadium (FeCoV) nanoparticles with an average diameter of 30 nm were synthesized by thermal plasma processing method. Two types of coating had been pursued: 10 nm thick graphite (C) coating uniformly added to create overall 50 nm in diameter FeCoV/C nanoparticles and 5 nm thick oxide (CoO·Fe2O3) coating. The latter coating was established by subjecting original 30 nm in diameter FeCoV nanoparticles to oxidation which resulted in 20 nm core diameter of FeCoV nanoparticles embedded in 5 nm thick oxide layer of CoO·Fe2O3. These magnetic nanoparticles were exposed to ac applied magnetic field and their heating responses were measured. The measurements were done at frequency of 175 kHz and intensity of the magnetic field for three different current values of 5 A, 10 A, and 15 A. Heating capabilities of the magnetic nanoparticles described by Specific Absorption Rate (SAR) were calculated by finding the initial slope of the heating curve with respect to time based on experimental data. It was found that the maximum value of SAR was obtained when applied frequency and current were at 175 kHz and 15 A, respectively. Results were analyzed to find the coating effect on the heating rate. The most significant result based on our study is that FeCoV/C magnetic nanoparticles with graphite coating had higher heating power than FeCoV/CoO·Fe2O3 with oxide coating.

# SPECIAL SESSION 6
# DRYING AND WETTING OF SOLIDS AND PARTICULATE MATERIALS (SS6)

**DSL100**
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## Elaboration and Characterization of Thin Water Molecule Sensitive Layers Deposited from Hexamethyldisiloxane

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Keywords: Hexamethyldisiloxane; Thin film; Humidity sensors; Electrical characterization.

This paper reports the study of electrical properties of plasma polymerization of hexamethyldisiloxane (pp-HMDSO) thin film based sensors. Thin water molecule sensitive layers were deposited from hexaméthyldisiloxane precursor on tow-interdigited electrode at low frequency power (19 KHz) plasma conditions. The sensor was calibrated in terms of impedance as a function of relative humidity, using a Frequency Response Analyzer. The electrical properties of the sensor are measured. The deposited film sensor exhibited a accepted sensitivity (impedance change from 106 to 104 Ω in the humidity range of 30–95% RH), fast response (8 and 34 s for adsorption and desorption between 35% RH and 95% RH, respectively) [1-2], and the current intensity increased from 10-10 to 10-6 A in the explored range of RH (25-95% RH). The change in the current of pp-HMDSO with respect to increasing and decreasing RH (hysteresis) of no more than 3% has been observed. Structural analysis of the sensitive layer was carried out by Fourier Transform Infrared spectroscopy (FTIR). The films showed good sensitivity to water molecule due to the presence of hydroxyl groups OH. These groupments provide the adsorption sites for water and play an important role in the humidity sensor properties, make the elaborated layer a promising candidate for humidity sensors devellopement.

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**DSL113**
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## Study on Infrared Drying of Cocoa Beans (Theobroma cacao L.) assisted by Non-heated Air flow using an Coupled Heat and Mass Transfer Model

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The mature fruit of cocoa (Theobroma cacao L.) contains 30 to 50 beans, which are covered by a mucilaginous pulp containing 10 to 15% sugar. The "nibs", which are fragmented cotyledons of beans, are the economically important part. The chocolate flavor is developed in the fermentation step, after which, the fermented material, called “wet beans”, contains about 55% moisture and is not suitable for storage. The moisture content should be reduced to about 6%. Drying of the beans is not simply the reduction of moisture. Excessive heating must be avoided in order to prevent quality deterioration, but a very slow drying is also unsuitable, as it can lead to mold formation [1]. By presenting several advantages such as: uniform heating of the product, easy control, low maintenance cost and high efficiency in converting electrical energy into heat [2], infrared (IR) drying appears as an interesting alternative for drying cocoa beans. Its use combined to the forced convection of unheated air aims to cool the bean surface, in order to prevent overheating. The objective of this work was then to investigate the IR drying of cocoa beans assisted by non-heated air flow, evaluating the influence of the IR heat flux and air velocity on the heat and mass transfer parameters. To this end, a drying model based on coupled mass and energy balances was applied. Tests were conducted at IR source temperatures of 70, 85 and 100°C, distances between the heat source and the sample of 10, 15 and 20 cm, and air velocities of 0, 1, 2 and 3 m/s. The non-heated air flow contributed to prevent case-hardening, thus facilitating mass transfer through the bean coat. Results in terms of the Biot numbers for heat and mass transfer allowed to better understand the behavior of the internal and external resistances during IR drying.

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**DSL114**
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## Fluidization and Drying Characteristics of Biomass Particles from Cocoa Husk in a Fluidized Bed

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In the chocolate manufacturing process is generated a large quantity of residues. Among these, cocoa husk presents potential application as biomass for power generation. However, the high moisture content of the cocoa husk limits its application as an alternative fuel because of the resultant high transportation costs, storage difficulties and reduced thermal efficiency during energy conversion [1]. Thus, the moisture content of biomass particles from cocoa husk must be reduced to 8-10% wt by means of a suitable drying technique. The fluidized bed dryer has been reported as a potential compact dryer for biomass, but studying the effect of the physical properties of each biomass on fluidization characteristics is of fundamental importance. Experimental and theoretical studies to understand the fluid dynamics of cocoa husk is needed so that the processing parameters and the drying system can be optimized to avoid problems at the large scale. Within this context, the objectives of this work were to determine the fluidization and drying characteristics of particles from cocoa husk in a conventional fluidized bed. The effects of particle size, moisture content and bed height on the minimum fluidization velocity are presented and discussed. The influence of the bed load, air temperature and velocity on the drying kinetics is also evaluated. From the changes in the fluidization behavior of the particles during drying strategies are studied aiming to improve the energy efficiency of the process.

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**DSL116**

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## Applying Lumped Model to Predict Drying Process in Solid with Complex Shape

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Drying is perhaps the oldest and most common unit operation (thermal method) found in several industrial that occur in the chemical, pharmaceutical, paper, mineral, polymer, ceramic, textile, food and agricultural industries. Drying is a thermodynamic process that occurs in the presence of the simultaneous heat transfer and moisture removal of a porous material [1,2]. As the heat transfer is due to movement of hot air around the solid, drying is named convective drying. Different materials when exposed to drying without control can suffer cracks and deformations, reducing its quality post-drying, specially heat sensitive materials.

Thus, this work aims to study the holed solid drying with arbitrary shape using the global capacitance method [2,3]. The analytical solution of the governing equations was done using the method of separation of variables. Results of the mass loss and heating of the materials along the process are presented and analyzed. It was observed that the moisture loss process occurs at a lower speed than the heating of the ceramic material because its thermal diffusivity is much greater than the mass diffusivity and that the area / volume relationship strongly affects the phenomena of heat and mass transport.

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**DSL117**

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## Drying of Banana Slices using Liquid Diffusion Model: A Theoretical Investigation

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Fresh fruits are dried after harvesting, in order to reduce the waste and the spoilage and to extend their shelf life. From the viewpoint of consumption, bananas are fruits with aromatic flavor which are naturally sweet. They are essential for the human diet providing vitamins, minerals and energy. Further, with the sequential growth of banana cultures and the amount of harvested banana, losses of these fruits has increased and thus new studies for fruits conservation play important role [1,2]. However during drying of materials with high moisture content such as fruits and vegetables, for example banana, occurs big variations of the volume and surface area of the product simultaneously with the moisture removal and heating. So, to control drying process contribute to increase quality of product post-drying.

In this sense, this work presents a theoretical drying study of banana fruit using diffusion model (Fick´s law) considering liquid movement as dominant mechanism of moisture removal. The governing partial differential equation was solved by analytical method (separation of variables technique) [3]. For validation, whole banana were peeled and cutted in longitudinal slices manually. Following, the samples were dried in an oven at different drying conditions. Predicted results were obtained and fitted to experimental data using the least square error technique. Results revealed which air temperature affect significantly moisture migration rate of banana slices. The fitted results presented good agreement with experimental data.

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**DSL118**

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## Convective and Microwave Drying of Prolate Spheroidal Solids: A Numerical Investigation

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Drying is the usual process for moisture removal of a wet product up to certain thereshold value. The goal is to minimize effects of the water activity and production of microorganisms inside the product which reduces quality of it. So, the effectiveness in drying techniques can be evaluated on two levels: cost of the process and final product quality post-drying. There is different drying techniques used for drying, such as fixed-bed, spray drying, convective drying, infrared drying, microwave drying, and many others. Microwave drying offers to shorten the drying time without degradation of final quality of the dried product [1].

This work aims to study theorically drying of prolate spheroidal solid when exposed to microwave. The heat and mass transfer mathematical model considers convective boundary condition and constant thermophysical properties of the solid during drying process. The numerical solution (finite-volume method) has been implemented in a computational code for evaluation of moisture content and temperature distributions inside the solid at different instants of drying process. The mathematical model predicts that

the moisture content is reduced more quickly on the tip of the solids, thus this region is most favorable to fracture and warping of the product due to high moisture and temperature gradients. Moreover, it is observed that the temperature decreases from the surface to the center of the solid and increased drying time.

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**DSL121**

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## Water Absorption in Polymer Composites Reinforced with Fiber using Langmuir- Type Model

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Polymeric materials are considered indispensable to modern life. However, due to the development of new technologies it is increasingly difficult to find a polymer that fully meets all the needs for a particular application, whether for reasons of mechanical properties, characteristics, and ways to made or cost. Then, polymers are increasingly used as matrices to development of composites. The main aim of use of fiber reinforced polymer composites is to added new properties to the material mainly from the viewpoint of the mechanical behavior: mechanical resistance combined with the minimum weight. Compared to traditional synthetic reinforcement agents, vegetable fibers have a lower density, abrasiveness, low cost and are biodegradable and renewable. Its thermal and mechanical performances can be compared with those of synthetic fibers.

One big challenges of using these fibers is correlated to its hydrophilic nature which adversely strongly affect mechanical properties of the composite thus, it is essential to understand the moisture absorption rate. It is important to study in detail the behavior of water absorption not only to estimate its consequences, but also the durability of composites reinforced with natural fibers [1]. The behavior of water diffusion in polymeric composites can be classified according to the relative mobility of the water in polymer segments, which are closely related to Fickian, non-Fickian, anomalous and intermediaries between Fickian and non-Fickian models [2].

In present day, Fick´s diffusion model is the most widely used process, especially for the initial linear region uptake, and few authors have used the Langmuir absorption law to analyze this phenomenon. In this sense, this work aim to study water uptake in natural fiber-reinforced polymer composites on the basis of Langmuir-type model. Exact solution of the governing equation has been done using the method of the Laplace transform and applied to a parallelepiped solid. Results of the moisture content spatial distribution and water absorption kinetic during the transient process are presented and discussed.

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**DSL130**
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### Problems of the Theory of Electrocapillarity for Solid-Liquid Interface

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The main problem in electrocapillarity of solid electrodes is the lack of clarity in determining the surface stress and basic equations. Within the framework of the Gibbs concept of geometrical dividing surface, the "surface stress" cannot be defined because methods of continuum mechanics can be applied to a physical surface layer (of finite thickness), but not to a mathematical surface. Gibbs never used the concept of surface stress, introducing only "surface tension" for a liquid electrode and "closely related quantity" for a solid electrode. Revisiting the derivation of the Gibbs adsorption equation, we prove its applicability to solid surfaces without the limiting requirement of constant state of strain, which was undeservedly interpreted by Eriksson as a shortcoming of the Gibbs theory caused to look for other approaches to surface stress problem.

A critical analysis [1-3] shows that the attempts (Shuttleworth, Eriksson, Couchman, Gokhstein, Weissmüller, etc.) to create a thermodynamic definition of the surface stress (as well as the formulation of fundamental thermodynamic equations and Maxwell relations operating with surface stresses) contain mathematical defects. It is shown that confusing interpretations of some Gibbs' concepts encountered in the literature have led to "modifications" of the Lippmann equation based on the critical error in the Gibbs–Duhem relation due to the occurrence of an extensive variable, which is inadmissible. The famous Lippmann equation should not be modified, and it remains a unique electrocapillary relation applicable to liquid and solid electrodes.

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**DSL134**
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### A New Diffusion Model to Describe Intermittent Drying of Rough Rice

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Energy saving and good product after drying always are necessary in any drying process, mainly in materials very sensitive to heat and moisture several like rice. In the drying of rice grains, usually is used the intermittent method that switches drying periods with periods of rest or relaxation in order to reduce cracks

past-drying. Several mathematical models have been used to describe intermittent drying of rough rice grains [1, 2, 3]. In the present work, experimental data for continuous and intermittent drying of rough rice grains long-thin (BRSMG CONAI variety) at a temperature of 40°C and tempering periods ranging from 0 to 1 h were compared with simulated results by means of the liquid diffusion model. For this purpose, the diffusion equation, written in cylindrical coordinates, was resolved via Galerkin-based integral method considering the constant diffusion coefficient and equilibrium boundary condition. The geometry used to represent the rice grain was prolate spheroid. It was verified good agreement between modelling and experimental data. It was also possible to observe that the highest moisture gradients occur at the tip of the grain, so this region is more susceptible to thermal and hydric effect. According to the results, the drying rate increases with increasing tempering time. It was also possible to simulate optimization tests to develop an energy-saving strategy in the intermittent drying process.

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**DSL146**
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## Drying of Castor Bean Fruits (Ricinus communis L., "Br Energia" variety): An Experimental Investigation

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Castor (Ricinus communis L) is cultivated around the world because of the commercial importance of its oil which is used in the manufacture of a number of industrial chemicals like surfactants, greases and lubricants, specialty soaps, surface coatings, cosmetics and personal care products, pharmaceuticals, etc [1]. The India is the world largest producer of Castor oil seed with annual production of 1.73 million tons, followed of the Mozambique, China, Mainland China and Brazil with productions of approximately 62, 40, 40 and 38 mil tons, respectively [2]. Castor bean is an oilseed relevant economic importance to Brazil, being traditionally produced on small and medium farms, generating jobs and income due to its numerous application possibilities in industry area [3]. Drying is a complex operation involving simultaneous transient transfer of heat and mass that provokes physical and chemical transformations in the product, which, in turn, may cause changes in product quality as well as in the mechanisms of heat and mass transfer [4]. The objective of this study was to conduct an experimental investigation of the drying of castor bean fruits (Ricinus communis L., "BRS Energia" variety), using oven on the air temperatures 40, 50, 60, 70, 80, 90 and 100°C and relative humidity values 28.03, 16.34, 10.62, 6.24, 4.69, 2.67 and 2.06%, respectively. The average moisture content data as function of time were analyzed for different drying air temperatures (40, 50, 60, 70, 80, 90 and 100°C). Results shows that with increasing temperature and decreasing of the drying air humidity increases the drying rate and more quickly the castor fruits reaches its equilibrium temperature and humidity. It was found that high drying rates generates hydric and thermal stresses inside the product, causing cracks and deformations in the grains.

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**DSL165**
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## EPR Study of Diffusion of Nitroxide Radicals in Normal and Supercooled Water

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Electron paramagnetic resonance (EPR) spectroscopy is sensitive technique for detecting radicals in liquid solution. The EPR spectrum of radicals is affected by their relative motion, which modulates the Heisenberg spin exchange (HSE) and dipole-dipole (DD) interactions between radicals. Therefore, a diffusion coefficient of radical in a given liquid can be obtained from the concentration dependence of EPR spectral parameters, such as linewidths, hyperfine splittings and coherence transfer. However, this procedure demands a reliable spectral fitting to find experimental concentration constants and a valid theoretical expression relating them to the diffusion coefficient. The effect of HSE interaction on EPR spectrum, which is dominant effect at higher temperatures, was calculated by solving kinetic equations for spin evolution of interacting radical pairs whose distance varies according to continuous diffusion model [1].

We extended the spin evolution equations by including the DD interaction, whose effect increases by lowering temperature. Since no analytical solution exists for these equations, an iterative method was applied assuming a weak effect of DD interaction. The obtained theoretical expressions were tested by measuring concentration dependences of EPR spectra of two nitroxide radicals in the normal (T>0°C) and supercooled (T<0°C) states of water. The nitroxide radicals were 14N- and 15N-labelled perdeuterated TEMPONE, which were chosen because their EPR spectra strongly differ, while their size and diffusion coefficient are expected to be the same.

The experimental concentration constants derived by fitting the EPR spectra correlate with theoretical expressions, allowing us to calculate diffusion coefficients. The calculated diffusion coefficients have similar values for both radicals and the calculated Stokes-Einstein radius decreases by lowering temperature in the supercooled state.

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**VIP026**
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## On the use of CFD in drying technology – Cyclonic dryer

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Drying is one of the oldest and one of most popular process used to promote moisture removal and heating of the porous materials in order to avoid or minimize deterioration by water action inside them. Fundamentally, drying consists in a simultaneous heat and mass transfer phenomenon coupled with dimensions variations of the particle being dried. This process is realized using one device so called dryer. Depending on the material nature, operation conditions and amount of the material to be processed there are different types of dryers which can be used in ways batch as continuous operation, such as, band, conveyor, drum, fluid-bed, rotary, spray and cyclone dryers.

Convective drying is one technique in which heated air and material (cold and moist) being dried are in an intimate contact continuously. Thus, heat is supplied to material, being responsible to increase the temperature and evaporate moisture of it. Despite of the importance to study the different types of dryers, herein, special attention is given to more recent one, the cyclonic dryer. Cyclone is simple mechanical equipment with no moving parts, which operates by the action of fluid dynamic or external-field forces (centrifugal, gravitational and drag forces). Mathematical modeling and numerical prediction of the drying process by using cyclone dryer is a difficult task [1]. In this case drying process requires rigorous multiple and coupled nonlinear conservation and constitutive equation solutions. Fortunately, in present day, computational fluid dynamics (CFD) have now created the possibility to new related and accurate studies of complex flows, including coupled heat and mass transfer, three-dimensional effects, swirl and rotation, and particle-particle and fluid-particles interactions given great improvements in modeling and in understanding of physical phenomena [1,2].

In this sense this work aim to applied CFD in drying process with particular reference to cyclone as dryer. Results os the pressure and fluid flow fields, particle trajectory, residence time and gas-particle interactions (heat and mass transfer, dimensions variations and force effects) are presented and discussed.

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**VIP038**

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## An Improved Method to Extract Crystalline Cellulose from Alternatives Sources

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Crystalline Cellulose (CC) is a good material for the new biopolymer composites industry. Crystalline cellulose has a greater axial elastic modulus (Young's modulus) than Kevlar [1] and its mechanical properties are within the range of other reinforcement materials. The natural source of cellulose fibers is wood for its abundance and as a renewable source of raw material. Said fibers have amorphous parts and crystalline parts. There are four polymorphs of crystalline cellulose. Each has been extensively studied but Cellulose I is the crystalline cellulose that is naturally produced by a variety of organisms like trees and plants and it is sometimes referred to as ‘‘natural’’ cellulose [1]. The selective elimination of the amorphous part is usually made by acid hydrolysis by using strong acids like hydrochloric or sulfuric acid [2]. This paper presents the preparation of crystalline cellulose from wood pulp by acid hydrolysis with nitric acid. Crystallinity indexes were obtained between 80.0 % and 82.0 % which represent a great result. The X-ray diffractometric showed peaks at 2 Θ of 15º, 22,7º and 35º indicating the presence of cellulose crystals [3]. The method described herein is on a laboratory scale. Further studies need to create industrial processors for the application of cellulose crystals on a large scale [4].

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# SPECIAL SESSION 7
# HEAT AND MASS TRANSFER IN POROUS MEDIA (SS7)

**DSL092**
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## Encapsulation of Nanostructures by Diffusion Through Porous Layers

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Nanostructured optical elements are essential components in diffractive optics, being widely used in laser systems, as polarisers, in X-ray optics, etc. [1-3]. The main drawback of optical gratings is their mechanical instability and susceptibility to damage due to contaminants or cleaning solvents. Therefore, the encapsulation of these nanostructures is highly desirable. Additionally, encapsulated grating structures allow optical designs with higher efficiency than conventional binary structures.

In this contribution we present a general method to encapsulate nanostructures for protecting against environmental influences. This encapsulation process relies on the diffusion of liquids or gases through porous layers. First, the grooves of the nanostructures have been filled with a sacrificial material. Either an organic (e.g. a polymer) or an inorganic (e.g. $Al_2O_3$) has been applied as sacrificial material. A planarization step has followed to remove excess sacrificial material and extricate the top of the nanostructures. Then, a capping layer was deposited using atomic layer deposition (ALD) or molecular layer deposition (MLD). The capping layer is chemically bound to the top of the nanostructures providing high stability of the ensemble. Finally, thermal annealing or wet chemical etching has been applied. During this subsequent treatment, the capping layer becomes porous to allow for the diffusion of the gaseous combustion products of the polymeric sacrificial material or the etching solution and reaction products. The capping layer can be further reinforced by physical vapor deposition.

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**DSL139**
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## Monolithic Versus Multilayered Stone Walls: Capillary Absorption

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The knowledge of moisture transfer in the inner parts of the materials and construction building elements and components is of great importance for its behaviors characterization, especially for its durability, pathology, waterproofing, degradation appearance and thermal behavior, causing changes with difficult performance prediction.

Having regard to the awareness of the scientific community to the importance of this knowledge, studies on moisture transport in porous materials are increasingly being carried out. The multifaceted aspects of moisture migration phenomenon tended to encompass monolithic building elements, since the existence of joints or layers is a complex contribution to the change of moisture transfer along the respective building element as on the mass transfer law.

CONSTRUCT-LFC, of FEUP, has devoted a significant portion of its resources to experimental investigation in the moisture field. In recent years has developed, validated, characterized and scaled a wall base ventilation system technique in order to accelerate constructive elements drying, controlling problems such as rising damp or the consequences of floods occurrences. The system was specially designed for constructive elements of high thickness and heterogeneity, as is the current case in monumental heritage. Even this system was sized considering monolithic walls.

Portuguese building elements as walls, roofs and floors, are usually multilayer with joints which justify the moisture transfer knowledge about the continuity between layers. Although, current numerical simulation programs usually used for moisture transfer in materials and constructive elements shows limitations associated with the existence of interfaces.

In this paper it is intended to present some numerical simulations with WUFI 2D program, usually used for modelling moisture transport in building materials, elements and components to analyze the transport of liquid water along a monolithic and a multilayer stone building element. Then, a comparison with laboratory results will be made. The idea is to evaluate the simulations limitations associated with the consideration of the interface to appreciate the necessity of correctly feature the interface phenomenon.

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**DSL142**
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## Numerical Study of Droplet Evaporation in a Porous Medium

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Droplet evaporation in a porous medium is an important phenomenon in inkjet applications on porous surfaces, such as papers and bio materials, and in treatment of chemical agents in porous soil and fabrics. However, its general predictive model has not yet been developed due to the complexity of the liquid-gas-solid multiphase phenomena coupled to heat and mass transfer.

Recently, numerical simulation of droplet evaporation on non-porous surfaces has been performed using a finite-element method (FEM) [1], a body-fitted moving-grid method [2], and a level-set (LS) method [3,4]. However, the numerical methods were not extended to the porous cases where the multiphase characteristics are much more complicated by the porous solid structures.

In this study, the LS method for tracking the liquid-gas interface is further extended for computation of droplet evaporation in a porous medium. The volume averaged conservation equations of mass, momentum, energy and vapor fraction are employed for the porous medium including the effects of porosity, drag force and conjugated heat transfer caused by the porous solid. The conservation equations are coupled with those in the external region of the porous medium through the matching conditions of velocity, pressure, temperature and vapor fraction at the porous surface. The numerical simulation of droplet evaporation demonstrated the droplet deformation during the evaporation is significantly different from the non-porous case. The effects of porosity and particle size on the droplet evaporation rate are quantified.

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**DSL158**
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## Experimental Study of the Klinkenberg Effect on Gas Permeability of Coal

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Slip flow, i.e. the Klinkenberg phenomenon occurs in porous media where the mean free path length of the gas molecules is of the order of the average size of pore diameters. This results in an acceleration of individual gas molecules along the flow path. Such phenomenon typically occurs in the laboratory when gas flow experiments are performed at low pressures. Existing empirical relationships for correcting the measured permeability in natural porous media have been predominantly obtained on conventional reservoir rocks where pore-throat size is larger than 0.03 μm under atmospheric flow, where the mean pressure and flow rate were only controlled by adjusting the injection pressure. This paper presents the results of an experimental investigation on gas flow and Klinkenberg effect in coal, in which micro-porosity dominates, yielding an average pore diameter of less than 2 nm. A high rank coal from the South Wales coalfield is employed. A single-gas flow test using helium is performed under steady-state conditions. Backpressure flow mode with constant differential pressure across the sample is applied to improve the flow and pressure control, extending the range of mean pressures applied. The anthracite coal sample tested, is subjected to a range of effective stress conditions, in order to investigate a general trend of the coal permeability reduction with an increase in effective stress. A continuous measurement of both radial and axial strains is performed which allowed further insights into the coal's mechanical deformation under different effective stress conditions. In addition, through strain observation, it is proved that the change in permeability under constant effective stress condition was purely attributed to the change in mean gas pressure, i.e. the slip flow effect. Based on the measured values of permeability at different mean gas pressures for constant effective stress conditions, intrinsic values of permeability in the range 0.1 – 1 mD were determined using the Klinkenberg plot. Using measured permeability and calculated intrinsic permeability, an assessment of mean gas pressure required to avoid the Klinkenberg effect was conducted

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## Impact of Reinforcement on Gas Transfer in Concrete

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The durability of reinforced concrete structures is largely impacted by transfer properties which can be evaluated through permeability measurement. Usually, the concrete permeability is studied on plain specimens without reinforcement and the effect of the presence of steel bars on permeability into reinforced concrete is little studied in the literature. However, the steel-concrete interface is known to present a larger porosity than plain concrete and can thus be the cause of preferential percolation paths for fluids. Such percolation paths create a lower resistance transfer of fluid and could modify transfer kinetics. For reinforced and prestressed structures with large reinforcement content, such as nuclear power plant containment, it is necessary to assess the impact of reinforcement on gas permeability for better understanding of the flow within the structure, in the case of a reactor pressure rise (maintenance or accident).

The aim of this experimental study is to characterize the effect of the presence of reinforcement on flows by measuring: leakage rates, permeability and time to reach the steady state. Measurements are made with Cembureau permeameter on cylindrical concrete specimens with or without a steel bar in the center. Several configurations with different lengths of steel bar are studied: the bar is 20, 30 and 50 mm long in 50 mm thick specimens. Since transfer into concrete depends on the state of saturation of the material, the specimens are tested at four degrees of saturation: 0%, 10%, 30% and 60%. The analysis quantified the impact of the defects created by the steel bar for each state of saturation. The material composed by concrete and reinforcement has two distinct permeability zones that can be associated in series or in parallel according to the configuration. Consequences on permeability measurement for reinforced structures are finally drawn.

**DSL187**
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## Gas Diffusion Inside L-Leucine-L-Serine Dipeptide Nanopores. A Non-Equilibrium Molecular Dynamics Study

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Gas storage and gas separation using porous solids are important technologies that have been attracting great attention because of their environmental and energetic applications [1]. To investigate the ability of the gas separation by dipeptide nanopores, the diffusion of small gases (e.g. O2, CO, CO2) inside L-Leucine-L-Serine (LS) crystals was studied by Non-Equilibrium Molecular Dynamics (NEMD) simulations using the GROMACS 4.6 software, employing the General Amber Force Field (GAFF).

The molecular model of the LS nanopores was built based on the X-ray crystallographic data. The MD simulations were performed in vacuum with periodic boundary conditions (PBC). The investigated system consists of 9 dipeptide nanotubes, each formed of 54 molecules of LS. For all the gas-LS nanopores systems considered, three collinear boxes were prepared: Box 1 containing 1000 gas molecules, Box 2 containing

the LS nanopores and finally an empty box (Box3) with the same dimensions as Box 1. Different simulations with different pulling force constants were performed for each gas-LS system.

The results show that the rate of increasing density for oxygen behind the nanopores (in Box3) is higher than for others gases using three different values of force constant. Moreover, the presented study suggests that LS nanopores can be used for separation purposes.

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**DSL195**
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## Permeability, Diffusivity and Solubility of SF6 Gas in Various Rubber Materials and their Correlation with Free Volume Parameters

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Permeation of SF6 gas through set of basic sealing elastomers, namely EPDM, NBR and FKM, was measured by means of a gas accumulation method. The time dependence of SF6 concentration measured in dynamic non-equilibrium phase was fitted with the analytical solution of second Fick's law, which allow to extract solubility and diffusivity separately. Resulted permeability was subsequently compared with the experimental value measured at the equilibrium. This way of determining gas transport properties was reported as a method for achieving a considerable reduction in measurement time [1].
Not much data are available regarding SF6 permeation in various materials. Due to high kinetic diameter of SF6 molecule presented results can be used for validating several theories describing diffusion dependence on kinetic properties of permeant [2] and gas transport dependence on material properties [3]. To show the later one the free volume radius and free volume fraction were measured for three investigated elastomers using the positron annihilation method. The correlation of these parameters with solubility and diffusivity is presented. It has been found that for three studied materials, free volume radius correlates well with permeability whereas free volume fraction is in good correlation with diffusivity.

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**DSL212**
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### Experimental Study of Effects of the Wettability Alteration on Relative Permeability Curves and Oil recovery in Carbonate Porous Media

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Among many Enhanced Oil Recovery(EOR) methods, chemical floodingfor wettability alteration of carbonate oil-wet rock is one of the most noticeable method amid researchers. However, several crucial aspects concerning wettability alterationare still unknown and unanswered to date. This research focused on effects of wettability alteration on relative permeability and oil recovery. For the study, three different chemical solutions (made from different concentrations of brine divalent ions $Ca^{2+}$,$Mg^{2+}$ and $SO_4^{2-}$)with different abilities to change the wettability conditions of limestone core sample were used. The cores were subjected to dynamic (core flooding) and static (natural imbibition) tests. Furthermore, simulation results of the processes using CMG at laboratory scale were reported and compared to the tests. The comparison of bothdynamic and static tests and simulation results show that wettability has strong effects on relative permeability curves. Moreover, a change in wettability from oil wet to water-wet causes an increase in oil relative permeability, thus improving the oil recovery from carbonate rocks.

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**DSL225**

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### Construction Materials with Embedded PCM’s for Capillary Damp Control

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Construction materials of walls and foundations that are in contact with the ground are face up to capillary damp. Construction materials like: brick, stone, mortar or concrete, are to a greater or lesser extent porous, through them, that is through microcapillaries existing in their structure, damp gradually moves upward from the soil. Due to capillary action, water climbs up the wall, and while moving, creates an additional electromagnetic field in the wall, which enhances the capillary effect. This phenomenon occurs due to the polarization of water molecules, slowly decreasing in an upward wall direction. The height reached depends on several aspects, namely: the climatic conditions of the environment, the wall thickness and the porosity of the materials. The derived rising damp is a major cause of degradation with important damaging effects on the appearance and durability of construction materials.

The uses of Phase Change Materials (PCM) in constructive elements are mainly used for increasing thermal comfort owing to its heat storage capacity in the form of latent heat. These materials (PCM) has been studied and tested for several solutions in order to increased energy efficiency of buildings.
This paper aims to assess the possibility of using PCM to diminish the rising damp in construction elements in contact with the ground. The impregnation of PCM in the porous structure construction materials can potentially avoid the capillary damp, or the water movement thought microcapillaries.
Experimental campaigns and numerical simulations are used to predict the water ascension through the wall due to material characteristics and climatic conditions. The influence of PCM on rising damp mitigation is assessed and the global results are presented with the goal of highlighting behavioural differences in regard to common construction materials.

**VIP003**

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### Nanotechnology for Buildings: Energy Efficiency Improvement

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Building components with incorporated Phase Change Materials (PCM) are meant to increase heat storage capacity, enable stabilization of interior buildings surface temperatures whereby influencing the thermal comfort sensation and the stabilization of the interior ambient temperatures.
The potential of advanced simulation tools to evaluate and optimize the usage of PCM in the control of indoor temperature, allowing for an improvement in the comfort conditions and/or in the cooling energy demand, was explored. This paper presents a numerical analysis of the enthalpy and melting temperature effect on the inside building comfort sensation potential of the plastering PCM.

**VIP009**

**Prof. Lizhi Zhang**
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### Lattice Boltzmann Simulation Modeling (LBM) Technique as a Tool for Mass Transfer Intensification in Porous Membranes

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Porous membranes either with finger-like holes or with porous medium are one sort of promising membrane materials for energy and environment control. Mass transport in these materials are the key factor influencing system performance. To overcome the difficulties in modeling meso-scale mass transfer in these materials, in this investigation, a lattice Boltzmann simulation (LBM) methodology is proposed to model the pore-scale gas flow and mass transfer in the porous membranes with finger-like holes. A typical membrane is classified into three sub layers: a porous support layer, a layer with finger holes, and a denser skin layer. Simulated annealing technique is used to re-construct the calculating domain. Then fluid flow and mass transfer in the membrane are predicted with LBM. Permeability and effective diffusivity are evaluated. It is found that the existence of finger holes in the matrix have dramatically enhanced the overall mass transfer in the membranes. Besides, the inhomogeneity in membrane structures makes the macro-scale lumped parameter prediction of membrane performance questionable. Results different from previous macro-scale multi-layer analysis are found. The dominant resistance is in the skin layer. The directions for membrane optimization should be changed from the optimization of porous layer to the skin layer.

**Keywords**
Indoor humidity control, Moisture transport, Asymmetric porous membranes, Finger-like holes, Lattice Boltzmann Method (LBM)

**VIP061**
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## Moisture Transfer in Porous Media under Conditions of High Temperature Heat Shock

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The porous ceramic materials used in the metallurgical industry under high-temperature thermal shock can be divided into two groups. First group consist of sintered porous materials, dry or with a minimal amount of water. They are used repeatedly in quasi-stationary state, at a temperature lower than the sintering temperature with usage of lining metallurgical melting furnaces or heat treatment furnaces. Moisture is removed from the materials only during the primary drying and sintering process. Second group of porous ceramics such as mould bonded sands is widely used in the foundry industry, especially in the cast iron foundry. This one-time poured mould material is called a “green sand”. It is a mixture of granular material (such as quartz sand) with approximately 8-10% of bentonite binder (kind of clay). The amount of water added during the mixing process does not exceed 4%. The physical properties of the mould material, to which the liquid cast iron is poured, have a decisive influence on the course of the intensity of physical–chemical metal-mould phenomena which also affect heat and mass transfer in the mould material. There are two main factors that determine the final casting quality: the heat transfer rate from the casting (especially during the initial period of thermal mould-casting contact) and metal-mould interfacial reactions. The experiments of pouring the cast iron plate into green bentonite-sand mould are presented in the paper, applying the simplified modeling of heat and mass transfer, with usage of the simulation codes dedicated to the applications which are used in the foundry industry. Important phenomena which accompany the heating process of the green sand mould with only one casting-mould contact surface by poured metal is the production of water vapour and its condensation in the so-called “over-moisture zone” consequently moving in the deeper layers of the mould which were modeled using simulation code, which does not include direct mass transfer model. Taking such a simplified modeling of pseudo coupled heat and mass transport with the usage of simulation codes typical to the foundry application into consideration, it’s proposed to use the substitute thermophysical coefficients [1] that characterize green sand properties.

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**VIP062**

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## Flow of Yield Stress Fluids Through Porous Media: Simulations, Experiments and Applications

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A Yield Stress fluids injection porosimetry Method (YSM) has recently been developed as a simple potential alternative to the extensively used Mercury Intrusion Porosimetry (MIP). Its main advantage is the use of a nontoxic fluid instead of mercury used in MIP. Using this method, the Pore Size Distribution (PSD) of a porous medium is obtained by measuring the flow rate / pressure gradient relationship obtained by injecting a yield stress fluid in the porous medium. The principle of the method and some experimental results obtained using this technique will be presented and will be compared to those obtained by Mercury Intrusion Porosimetry (MIP).

In the Yield Stress fluid injection porosimetry method, the main assumption is that the porous medium is described as a bundle of straight capillaries of circular cross-section following a given pore size distribution. This simple model is revisited by introducing both non-circular and axially varying cross-sections. Two key points are tackled using numerical simulations: the flow onset at minimal pressure drop and the variation of the flow rate vs the pressure gradient. These results are finally used to show that the flow rate / pressure gradient relationship of a yield stress fluid through a porous medium can be more closely predicted using a bundle of capillaries of irregular cross-sections rather than using the classical bundle of straight circular capillaries.

# SPECIAL SESSION 8
# FUNDAMENTALS TO APPLICATIONS IN 2D MATERIALS (SS8)

**DSL147**
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## Electronic Structure „Engineering” in the Development of 2D Materials for Na-Ion Batteries

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Transition metal compounds with a general formula AxMaXb (A=Li, Na, M= transition metal, X= O, S) constitute a group of potential electrode materials for a new generation of alkaline batteries. This application is related to the fact that these compounds can reversibly intercalate high amounts of alkaline ions (1 or more moles per mole of MaXb) already at room temperature, without significant changes in their crystallographic structure. The author of this work basing on her own investigations of AxMaXb (A=Li, Na; M=3d, 4d, 5d; X=O, S) has demonstrated that the electronic structure of these materials plays an important role in the intercalation process.

Nowadays, further development of rechargeable batteries is focused on the discovery of new, high-performance and low-cost electrode materials. Recently, Na-ion batteries have attracted much attention due to their many advantages, such as: high abundance of sodium in the Earth’s crust, its low cost and suitable redox potential (only 0.3 V above that of lithium). Among possible electrode materials for Na-ion batteries, layered NaXCoO2-y oxide has attracted much attention due to its interesting electrochemical properties. This paper shows new approach to an explanation of the step-like character of discharge/charge curve of Na/Na+/NaxCoO2-y battery. This is still an open problem, which until now had no proper description in the literature. On a basis of comprehensive experimental studies of physicochemical properties of NaxCoO2-y cathode material (XRD, electrical conductivity, thermoelectric power, electronic specific heat) supported by calculations performed using DFT method with accounting for chemical disorder, it has been shown that the observed step-like character of the discharge curve reflects the step-like variation of the chemical potential of electrons (Fermi level) in the density of states of NaxCoO2-y, which is anomalously perturbed by a presence of the oxygen vacancy defects and sodium ordering [1,2]. In this work we undertaken to improve electrochemical properties of NaXCoO2-y by partial substitution of Co with Mn in order to achieved continuous density of states, monotonic variation of the Fermi level and consequently monotonic character of discharge curve.

Our studies show that electronic structure “engineering” is excellent method of controlling properties of the cathode materials for Na-ion batteries, changing their unfavorable character of the discharge curve, from step-like to monotonic, through modification and control density of states function of a cathode material.

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**VIP054**
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## Epitaxial Graphene Nanostructures for Future Carbon Based Electronics

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Keywords: 4-tip STM/SEM, surface transport, ballistic transport, Klein tunneling, graphene nanostructures

Functionalization of graphene is an essential task for any future carbon based electronics. Among others, the control of the edges, the chemical potential, the geometry and orientation of the nanostructures are the key parameters to enable and control the transport properties. In this talk I will highlight recent results in the field of epitaxially graphene nanoribbons which can be directly grown on insulting supports without the need of challenging transfer processes.

Graphene ribbons grown on pre-structured SiC(0001) surfaces exhibit promising transport properties, e.g. high temperature annealing of appropriately designed SiC-Mesa structures results in growth of sidewall nanoribbons revealing robust ballistic transport channels with mean free path lengths up to 16µm at 300K [1]. The existence of edge states on zig-zag oriented ribbons is confirmed by Raman, STM and STS measurements [2,3]. Moreover, by means of STM lithography, we have recently fabricated nano-constrictions within these wires revealing Fabry-Perot like resonance features. Moreover, npn-structures with Klein tunneling barriers have been realized by functionalizing the buffer layer on top of the SiC-mesas via Ge-intercalation. Depending on the local Ge coverage the chemical potential is either shifted above (+340 meV, n-type) or below (-290 meV, p-type) the Dirac point correlating nicely with the morphology as deduced from scanning tunneling microscopy and spectroscopy. The length of a single pn-junction is around 5 nm as revealed by spatially resolved STS measurements and therefore, significantly lower than those induced by field effects. In case of bipolar structures (npn and pnp) the resistance strongly depends on the inner barrier length D. For short barriers (D < 200 nm) the second junction appears almost transparent, a clear signature of Klein tunneling [4].

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**VIP055**

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## Introducing 2D Materials for Magnetic Tunnel Junctions

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The recent discovery of graphene, and other 2D materials, has opened novel exciting opportunities in terms of functionalities and performances for spintronics devices. While to date, it is mainly graphene properties for efficient spin transport which have been put forward, we will present here experimental results on another avenue for 2D materials in spintronics. We will show that a thin graphene passivation layer, directly integrated by low temperature catalyzed chemical vapor deposition (CVD), can prevent the oxidation of a ferromagnet [1]. This in turn enables the use of novel humide/ambient low-cost processes for spintronics devices, which would usually lead to oxidation during the fabrication and thus a quenching of the spintronic performances. We will illustrate this property by demonstrating the use of ozone based ALD processes to fabricate efficient spin valves protected with graphene [2]. Importantly, the use of graphene on ferromagnets allows to preserve a highly surface sensitive spin current polarizer/analyzer behavior and adds new enhanced spin filtering property [3]. Finaly, we will present results concerning another 2D material isomorph to graphene : the atomically thin insulator hexagonal boron nitride (h-BN). Caracterizations of complete spin valves making use of monolayer h-BN tunnel barriers grown by CVD will be presented [4]. These different experiments unveil promising uses of 2D materials for spintronics.

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**VIP056**
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## Efficient Spin Transport with Graphene

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Spintronics is a paradigm focusing on spin as the information vector. Ranging from quantum information to zero-power non-volatile magnetism, the spin information can be also translated from electronics to optics. Several spintronics devices (logic gates, spin FET, etc) are based on spin transport in a lateral channel between spin polarized contacts. We want to discuss, with experiments in support, the potential of graphene for the transport of spin currents over long distances in such types of devices.
We will present magneto-transport experiments on epitaxial graphene multilayers on SiC [1]. The measured spin signals are in the mega-ohms range, and the analysis of the results in the framework of drift/diffusion equations leads to large spin diffusion length in graphene in the 100 microns range. The high spin transport efficiency of graphene can also be acknowledged up to 75% in our devices. These results will be compared to previous studies on carbon nanotubes and to our on-going study on large scale CVD grown graphene making use of h-BN tunnel barriers [2]. Our latest results of spin precession in graphene channels obtained at room temperature will be also discussed. A unified picture of spin transport in nanotubes and graphene will be presented [3].

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**VIP057**

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## Ultrafast Atomic Diffusion Inducing a Reversible Phase Transition on Sn/Si (111):B

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Ultrathin metallic films on semiconductor surfaces are prone to phase transitions [1] and exotic phenomena as low-dimensional superconductivity [2], magnetic orderings [3] and strong electronic correlation [4]. In temperature-induced transitions, dynamical fluctuations of surface atoms often trigger changes on the structural and/or electronic properties of the system. These dynamical fluctuations are evidenced by a different symmetry on the system when studied with slow and fast probes. The apparent symmetry of the surface with slow probes is just the averaging of different instantaneous configurations of the system. These dynamical fluctuations correspond usually to the motion of single atoms around an equilibrium position, in a way that the system explores the different energetically degenerated configurations. Most of these transitions involve thus soft distortions from an ideal geometry due to the movement of atoms around their equilibrium position.

We have recently observed by low energy electron diffraction a new reversible transition explained with a novel diffusive mechanism. There dynamical fluctuations are not associated to motion of single atoms but to the motion of clusters exploring different quantum mechanical ground states in a sub-picosecond time-scale. Our theoretical calculations describe the dynamics of the Sn clusters and the onset of the observed transition. The Sn clusters move on the surface without coalescing in bigger clusters that would reduce the active region for eventual catalytic reactions, and their motion increases their cross section for capturing eventual reactants. We believe that the understanding of this model surface may open a route to seek for catalytic interfaces with high capture rates.

Fig.1. (a) Low energy electron diffraction patterns of the ground state at room temperature (left) and the fluctuating state above 520 K (b) Top view of the motion of the six Sn atoms (different colors) from DFT molecular dynamics simulations at 700 K. The open circles indicate the (ideal) positions of the Si surface atoms. The solid green lines indicate the initial and final configurations for the Sn tetramers.

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**VIP058**

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## Epitaxial Growth of Stanine and Topological Superconductor by Proximity Effects

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Stanene is composed of tin atoms arranged in a single layer honeycomb structure, in a manner similar to graphene. Stanene and its derivatives were proposed to be 2D topological insulator with a very large band gap and support enhanced thermoelectric performance, topological superconductivity and the near-room-temperature quantum anomalous Hall effect. In the first part of my talk, I will report a successful fabrication of 2D stanene by MBE. The atomic and electronic structures were determined by STM and ARPES in combination with first-principles calculations. In the second part, I will discuss our recent efforts to make topological superconductor by proximity effects and identify the Majorana mode in the vortex core. We systematically investigated the spatial profile of the Majorana mode and the bound quasiparticle states within a vortex in a thin layer of Bi2Te3 epitaxially grown on a superconducting NbSe2 substrate. While the zero bias peak in the local conductance splits right off the vortex center in conventional superconductors, it splits off at a finite distance ~20 nm away from the vortex center in Bi2Te3/NbSe2, primarily due to the Majorana fermion zero mode. While the Majorana mode is destroyed by reducing the distance between the vortices, the zero bias peak split of the conventional superconductor reappears. This work provides strong evidences for the existence of Majorana fermions and also suggests a possible route to manipulating them.

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**VIP059**

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## Formation of Graphene Nanoribbons and Sheets by Diffusion of Carbon in Liquid Metals Induced by Electrocharging Assisted Process*

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Graphene nano-ribbons and nano-sheets are produced in the crystal lattice of metals by an electrocharging assisted process at high temperatures. In this process the metal is heated above its melting temperature, particles of activated carbon are stirred in the liquid metal, and a high current is applied between graphite

electrode and the crucible containing the mixture. Raman scattering of the resulting materials, called covetics, reveals the presence of graphitic carbon with mostly sp2 bonding while transmission electron microscopy, in combination with electron energy loss spectroscopy, indicate that these graphitic regions are graphene nanoribbons and nanosheets which have a preferred orientation with the lattice of the metal [1] The electrical conductivity of copper with carbon first decreases and then increases with carbon content. Al 6061 and Al 7075 covetics show higher ultimate tensile strength and higher conductivity than the pure alloys.[2] Films of copper covetic deposited by e-beam evaporation and pulsed laser deposition using bulk copper covetic as target show higher transmittance than pure copper films of the same thickness and higher resistance to oxidation under ambient conditions.[3] Density functional theory and first principles calculations of the phonon density of states indicate strong C-metal covalent bonding at edges of graphene nanoribbons and sheets. The C-metal bonds are Raman active and produce weak signals in the Raman spectra.

* Funded by ONR Award No N000141410042, DARPA/ARL under Grant No. W911NF-13-1-0058, and ANL/DOE subcontract No. 6F-30062

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**VIP060**
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## Fabricating Novel Nanostructures in Situ with Electron Beams

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In this presentation, I will show how one can use an electron beam to irradiate material residing on freestanding graphene to form new nano-structures. For example, one can exploit amorphous carbon species to form new graphene layers. I will also show how carbon nanotubes stemming from graphene can release themselves and form a giant fullerene while irradiated by electrons. The released fullerene is then observed to diffuse over the graphene surface and is finally trapped at a graphene edge.

In addition, I will show how metallic species on the surface of graphene can be driven into graphene pores and form single atom thick membranes. In one case single atom thick Fe membranes are shown to form with a square lattice. In another case, graphene-like ZnO single atom thick membranes are formed. In both cases there are the first observations of these 2D materials in a free standing form.

In addition, the dynamic behavior of ZnO and CuO nanoparticles trapped in a graphene fold are investigated. Upon extended irradiation the graphene folds are seen to develop small holes. The material is then observed to effuse out. However, the etching rates of graphene by the oxide particles and their effusion behavior are rather different and in part this is attributed to differences in graphene gasification by the oxide particles.

**DSL238**
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## Molecular Dynamics Simulation of Carbon Nanolayer Grown on Silicon (001) Substrate

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Carbon nanolayers coatings are employed to yield important benefits in applications such as for optical lenses, microelectronics, engineering, and cutting tools. Several parameters must be investigated to get good quality nanolayer on the Si substrate [1]. The aim of this research was to study of morphology of carbon nanolayer at the different temperature of silicon (001) substrate and deposition rate. The molecular dynamics (MD) simulation technique based on Tersoff [2] many body potential was used to study growth of carbon by physical vapor deposition (PVD). PVD uses physical process such as heating to produce a vapor of material, which is then deposited on the object which requires coating. Periodic bindery condition enforced in the parallel to the surface of Si and mirror bindery condition enforced in the perpendicular to surface direction. The temperature of carbon vapor decreases from 11000K to substrate temperature during different cooling rate. Tow cooling rate including 2.5 and 5 K/Ps were employed to cool carbon vapor on the substrate with 600, 700, 800, 1100, 1300, 1500, 1800, and 2300K. The MD results show that, we can’t observed ordered layer with these physical condition.

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**VIP072**
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## Pristine Epitaxial Graphene and Metal Contacts

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In contrast to conventional three-dimensional materials which retain their bulk properties when patterned to form electronic devices, anything that interacts with the two-dimensional graphene surface during device fabrication modifies its properties. Such surface sensitivity poses significant difficulties for the fabrication of devices with controlled and reproducible performance, especially those for molecular sensing applications, as well as impedes understanding the intrinsic interaction of graphene with other materials such as metals. Here, we describe a femto-second laser assisted, large-area, resist-free lithography technique to fabricate electrically isolated, pristine graphene surfaces. Epitaxial graphene is formed on semi-insulating (0001)6H-SiC surfaces under an Ar ambient. For patterning, we select the laser parameters, i.e., wavelength, beam

shape, optical fluence, pulse width, focus, and repetition rate, to yield sufficient non-equilibrium heating to ablate graphene on a sub-micron scale and form a precisely defined region without damaging the unilluminated and/or partially illuminated material or the underlying substrate. Using these pristine graphene surfaces, we studied the interaction of Ni and discovered the interaction on epitaxial graphene is quite different from CVD formed graphene. Using these results, we propose a carrier transmission probability model to understand key parameters affecting metal contact resistance to graphene and explains the wide range of results found in the literature.

# SPECIAL SESSION 9
# NANO/BIO MATERIALS SYNTHESIS, CHARACTERIZATION, MODELING AND APPLICATIONS (SS9)

**DSL120**
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## Synthesis and Characterization of TiO2 doped CeO2 Nanopowders by a Hydrothermal Process

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Hydrothermal processes have the potential for the direct preparation of crystalline ceramic powders and offer a low-temperature alternative to conventional powder synthesis techniques in the production of oxide powders [1]. These processes can produce fine, high-purity, stoichiometric particles of single and multi-component metal oxides. Furthermore, if process conditions such as solute concentration, reaction temperature, reaction time and the type of solvent are carefully controlled, the desired shape and size of particles can be produced [2]. Uniform distribution of the particles is key for optimal control of grain size and microstructure in order to maintain high reliability. It has been demonstrated that such powders are composed of much softer agglomerates and sinter much better than those prepared by calcination decomposition of the same oxides [3]. These powders could be sintered at low temperature without calcination and milling steps [4]. The objective of this study was to synthesis TiO2-CeO2 nanosized crystalline particles by a hydrothermal process.

TiO2-CeO2 nanosized powders were prepared under high temperature and pressure conditions by precipitation from metal nitrates with aqueous potassium hydroxide. The TiO2-CeO2 nanosized powder was obtained at 185 ℃ and 6 h. The average size and size distribution of the synthesized particles were below 10±5 nm and narrow, respectively. The XRD diffraction pattern shows that the synthesized particles were crystalline. This study has shown that the synthesis of TiO2-CeO2 nanosized crystalline particles is possible under hydrothermal conditions in aqueous solution.

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**DSL088**
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## Effects of Milling Time and Impact Force on the Crystallite size of Nanostructured Al-45%Zn Alloy via Mechanical Alloying Process

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Fabrication of alloys in the solid state via mechanical alloying (MA) process has been studied by the earlier researchers. The effects of milling time and impact force, defined as the ball-to-powder weight ratio (BPR), on the crystallite size of nanostructured Al-45wt.%Zn alloy via MA process were evaluated in the current work. The mechanical milling was performed in a high-energy ball milling Fritsch P-6 planetary mill at different milling times and BPRs using stainless steel container and balls. X-ray diffraction (XRD) technique and scanning electron microscopy (SEM) were utilized for evaluating the structure, crystallite size and elemental diffusion of the milled powders. Results revealed that increasing the milling time and impact force give rise to decreasing the crystallite size during the MA process. The crystallite size after 10h milling at the BPR of 20:1 was ≈27nm. The interpretation of data resulted have been discussed in details.

Keywords: Nanostructure, Al-45wt.%Zn, Mechanical Alloying.

**DSL103**

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## Silver and Titanium Nanoparticles used as Coating on Polyurethane Catheters

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Silver nanoparticles have been used in the medical area due to their remarkable antimicrobial properties [1]. In this sense titanium dioxide nanoparticles obtained by the sol-gel method were used as coating of catheters for subsequent impregnation of silver nanoparticles with gamma irradiation and electron beam at 25 and 50 kGy [2]. This work aimed to study the use of the silver nanoparticles and titanium dioxide as coating of polyurethane Central Venous Catheter (CVC) for antimicrobial activity. Furthermore the amounts of titanium and silver present in the coated catheters had been evaluated by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP OES). Therefore the Raman spectrometry was used to identify the polymorph of titanium oxide, rutile.

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**DSL125**

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## Study of the Ball Milling Device for Synthesizing Nanocristalline Powder

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This study is based on kinematic modeling of the mechanical ball mill which operates with a single ball in motion, and it is actuated by crank and connecting rod system for producing nanocrystalline powders by the process of ball milling. The influence of the geometric and dynamic parameters play an important role on the variation of the impact forces generated during the impact of the ball with the inner wall of the jar, which result a transfer of energy required for grinding process.

The determination of these forces enables us to know their particular importance's on the intensity and the efficiency of milling, with the advantage of low operating power consumption of the mill and absence of the contamination problem. In addition, we have defined a model for calculating the temperature of the powder trapped between the ball and the wall of the jar of the mechanical mill whose start-up is provided by an electric motor.

**DSL153**
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### Time Distribution of Sampling Times in Diffusion Experiments in Biological Materials

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In diffusion experiments in biological materials, as in the case of food materials, the kinetics are usually characterized using data of concentration with respect to time. The amount and timing of sampling are usually restricted to the availability of the substrate to be analyzed and constrained to the availability of lab analysis. This constrains the number of data points obtained in a single diffusion experiment. Generally the time intervals between measurements are arbitrary, and usually uniformly spaced. Here we present a method to improve the timing distribution between each measurement to reduce the uncertainty in the diffusion parameters, i.e., diffusion coefficients. This method can reduce the number of experiments to characterize the diffusion process, proposing a distribution of the sampling times based on the relevance of each data point to the diffusion parameter. This distribution is based on the covariance matrix, which associates the information given by each data point to the estimated diffusivity [1]. As a case study, the osmotic dehydration of granny smith apples in sucralose solution was used. The cases for Fickian and anomalous diffusion are analyzed [2]. The estimation of the diffusion parameters in each case was improvedvia a simple procedure for time allocation of sampling. The method presented here can help to improve the design of diffusion experiments and to obtain better estimates for diffusion parameters.

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**DSL154**
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### Synthesis of the RGO/TiO2 Core-Shell Nanocomposite Flakes and Characterization of Their Unique Electrostatic Properties using Zeta Potential Measurements

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The study aimed at describing the influence of the modification of electrostatic properties of graphene oxide (GO) by in situ deposition of TiO2 nanoparticles. The RGO/TiO2 core-shell nanocomposite flakes were synthesized using simplified sol-gel method and subsequent thermal decomposition. The intermediate product was characterized by TGA/FTIR, FTIR and Raman spectroscopy whereas RGO/TiO2 (40 wt%) core-shell nanocomposite flakes (final product), GO flakes and TiO2 nanoparticles were characterized using SEM, HRTEM, XPS spectroscopy, N2 sorption analysis as well as helium pycnometer. The results of FTIR, Raman, XPS as well as electron diffraction were convergent and complementary. They confirmed that titanium dioxide existed mostly in anatase phase and was covalently bonded to GO surface. The spherical TiO2 nanoparticles (ca. 94 nm) completely covered the surface of curly-shaped RGO flakes and acted as a spreader between individual flakes of high BET specific surface area. We have also proved that after dense covering of the whole surface of GO flakes by TiO2 nanoparticles and formation of core-shell system, the influence of core composed of RGO on the measured zeta potential is highly visible in the distilled water and NaCl solution and the modification of electrostatic charge of GO can be clearly observed.

**DSL171**
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### An Investigation on Biocompatibility and Mechanical Properties of Polyetheretherketone Nanocomposites Reinforced by Bergman Phase Mg32 (Al ,Zn )49

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Polyetheretherketone is one of the most widely used thermoplastic polymer. Due to the use of thermoplastics in medical application is significantly increasing, the purpose of this study is to investigate biological performance and mechanical properties of PEEK with various content of Bergman phase with chemical composition of Mg32 (Al ,Zn )49. Bergman phase is known as one of complex metallic alloys which is produced by using ball milling technique. PEEK consists of 162 atoms with lattice parameter of a=1.416nm and coordination number Z=12. Complex metallic alloys (CMAs) which are characterized by their giant unit cells have recently suggested promising characteristic. Nanocomposite samples were produced by hot pressing. Morphology, distribution of particle into matrix and crystallographic structure were characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD). In vitro biocompatibility of these

nanocomposites has been investigate by in vitro cell tests. SEM images were obtained after the tests, in case of appearing any indication of cytotoxicity. Bioactivity of PEEK/ Mg32 (Al ,Zn )49 was compared with pure PEEK. Crystallinity evaluation of these nanocomposites carried out by differential scanning calorimeter (DSC). Tensile strength tests were performed in order to investigate mechanical properties of PEEK/ Mg32 (Al ,Zn )49.

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**DSL182**

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## Diffusion of Water Molecules in Microporous Caa Zeolite-Based Adsorbents. Effect of Zn-Ag Bimetal Ion Exchange

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Zeolites are microporous crystalline aluminosilicates generally used as size selective adsorbents in the chemical, petrochemical and pharmaceutical industries [1]. Recently, A zeolites type have been used to prepare new mineral-based hemostatic dressings applied to control heavy bleeding resulted from both military and civilian trauma [2]. The mineral materials act as adsorbents that dehydrate the hemorrhaging blood by adsorbing water molecules, modify the local electrolyte conditions, and induce hemostasis. In spite of the success of zeolite-based hemostatic agents in controlling bleeding and reducing the amount of heat release during water adsoption, the effect of porous properties on water diffusion in microporous media is limited and the associated phenomena are not well understood. In the present study, bimetallic Zn-Ag-exchanged A zeolites are successfully prepared via the liquid ion exchange method following a procedure reported in our previous study [3]. Thermodravimetry (ATG), differential scanning calorimetry (DSC) and thermal activity monitor (TAM) techniques clearly highlights the complexity of the adsorption and diffusion mechanisms of water molecules within the exchanged zeolite micropores. The diffusion kinetics and hydration heat appear to be controlled mainly by the prehydration rate, ion exchange degrees and location of Ca, Ag and Zn species in the cationic position of the A zeolite framework.

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**DSL186**

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## Characterization of Polystyrene Nanocomposites Containing Nanoparticles of Pseudoboehmite Obtained by Sol-Gel Process

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Polymer nanocomposites are hybrid materials in which inorganic substances of nanometric dimensions are dispersed in a polymeric matrix. These substances have high surface area allowing a better interaction with the polymeric matrix and consequently promote changes in the physical properties of the final composite with small additions of the same. The preparation of nanocomposites with polymer matrix allows in many cases to find a relationship between a low cost due to the use of lower amount of charge, reaching high level of performance. In this work, were obtained polystyrene nanocomposites with pseudoboehmite synthesized by the sol-gel process with different concentrations of pseudoboehmite using octadecylamine as a coupling agent. The nanocomposites were prepared by the melt intercalation technique and characterized by differential thermal analysis, thermogravimetric analysis, heat deflection temperature, Vicat softening point, mechanical and rheological tests. The results showed an increase in the thermal properties, hardness and tensile strength values and decrease in the melt index, impact resistance and tensile elongation, showing the interaction of the filler with the polymer matrix.

Keywords: Nanocomposite. Pseudoboehmite. Polyestyrene. Characterization of nanostructured polymer composites.

**DSL191**

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## Use of Gamma-Alumina Nanoparticles for Drug Delivery System for Releasing Acyclovir

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The sol-gel process is a method for preparing porous ceramics, vitreous and crystalline materials from molecular precursors. In this process, a lattice of oxide compounds is made through reaction of inorganic materials in aqueous solution. The sol-gel process may be used to produce nanoparticles of pseudoboehmite, which are employed in drug delivery systems, production of nanocomposites and in the synthesis of gamma-alumina [1,2,3]. The firing of pseudoboehmite produces pure gamma-alumina [4,5]. The use of nanoparticles in drug delivery systems is advantageous because it prevents repeated doses and also decreases the amount of drug intake, which not only enhance the therapeutic effect, but also reduces the risks of plasma concentration reaching toxic levels. The use of synthetic nanoparticles have attracted great interest for applications in drug delivery systems. Pure gamma-alumina obtained from

pseudoboehmite is a synthetic aluminum compound with excellent adsorbent properties. In this work, gamma-alumina was obtained through pseudoboehmite firing to be used for in vitro release of acyclovir. The gamma-alumina was characterized by X-ray diffraction (XRD), Differential Thermal Analysis (DTA), Thermogravimetry Analysis (TG), Scanning Electron Microscopy (SEM) using secondary electron detector and EDS detector. The release profile was obtained by UV/Vis spectroscopy for in vitro analysis. The results indicate gamma-alumina is appropriate for release of acyclovir.

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**DSL210**

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## Spark Plasma Sintering of Ti1-xAlxN Nano-powders Synthesized by High-Energy Ball Milling

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The present study focused on the fabrication of bulk materials from nano-powders using a spark plasma sintering (SPS) apparatus. Super-saturated solid solutions with various AlN fractions (10, 20, 30 and 50mole%) were synthesized by high-energy ball milling (HEBM) of pure nitrides. The complete dissolution of AlN in TiN was achieved after 100 h of milling. The milled powders were characterized by X-ray diffraction, SEM, energy-filtered transmission electron microscopy spectra imaging and energy dispersive x-ray spectroscopy. The crystalline size of the mechanically alloyed powders after 100 hours of milling was about 12-14 nm. powders of various compositions were sintered by SPS under 63MPa pressure at 1673K. The maximal values of hardness and bending strength (610MPa and 18.6GPa) were obtained for the composites with 20% mol%AlN and this powder was consolidated in the 1273-1423K temperature range by high pressure (500MPa) spark plasma sintering (HPSPS). Fully dense nanostructured HPSPS-processed at 1423K specimens displayed Young modulus 420GPa, hardness 20.5 GPa, bending strength 670MPa and fracture toughness 7.1 MPa·m0.5.

**VIP037**
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## Twinned Boundaries in Nanomaterials

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This overview outlines the current state of the nanotwinned structures studies in metallic nanomaterials. The various processes of twin generation, such as the pulse electrodeposition technique, magnetron sputtering and different variants of severe plastic deformation (equal channel angular pressing, high pressure torsion, accumulative roll bonding, and surface mechanical grinding/rolling treatment, etc.), are described. The structural characterization of the growth/deformation twins by transmission electron microscopy methods, including high-resolution one, is discussed. Special attention is given to a surface mechanical grinding/rolling treatment for the gradient structure formation with a high content of low-angle twinned boundaries. The influence of nanotwinned structure on the nanomaterials strength, ductility, fatigue properties, electrical conductivity, especially under extreme conditions (such as high temperatures, irradiation and corrosion actions) is discussed in details. A significant increase of these properties as compared with conventional nanomaterials is underlined. In many cases, the nanomaterials with the nanotwinned and gradient structure remain tolerant to the various extreme conditions, such as high temperature heating, irradiation, deformation and corrosion actions. Some poorly researched aspects are also put into considerations.

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**VIP052**
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## The microstructure of Ag/TiO2 and Au/TiO2 made by Single-Step Synthesis by Ultrasonic Spray Pyrolysis

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In this lecture, Ultrasonic Spray Pyrolysis (USP) will be presented – which was used for the synthesis of the complex Ag/TiO2 and Au/TiO2 nanoparticles. USP is a continuous method based on the Droplet-to-Particle (D-T-P) principle. The particle formation occurs through the thermal decomposition of the aerosol of the precursor solution (droplets 1-15 µm) and runs over the following intermediate steps: Evaporation, precipitation, reaction/decomposition, drying and final particle formation. The preparation of the precursor solutions for nano Ag and nano Au synthesis was done by direct dissolution of a defined mass of AgNO3 or HAuCl4 in distillate water. Since both solutions were used for synthesis of metal/oxide nanoparticles, a prepared noble metal precursor solution was mixed in defined ratio with the above explained prepared C16H36O4Ti solution.

The characterization of the obtained nanoparticles was done with SEM and TEM, as well as the FIB technique in order to determine the structural characteristics of the obtained nanoparticles.

The size of the Au nanoparticles is much smaller than the size of the Ag nanoparticles (the Au nanoparticles were in the size range 7 nm to 50 nm, where most of the particles were around 10 nm in size, whereas the average size of the Ag nanoparticles ranged from around 20 nm to 80 nm); the Ag nanoparticles are mostly ideally spherical in shape (hexagonal in the case of the defect formation, twinning, etc.), whereas the Au nanoparticles had various forms from spherical, cylindrical to an irregular shape; on the FIB analysis results it is clear to see that, in the case of Ag/TiO2 nanoparticles, almost all the Ag nanoparticles are formed on the oxide surface (>90%) whereas in the case of Au/TiO2 nanoparticles, some of the Au nanoparticles are formed in the volume of the TiO2 nanoparticles (~30%), and distributed randomly in the oxide matrix.

Ref.: BOGOVIC, J., RUDOLF, Rebeka, FRIEDRICH, Bernd. The controlled single-step synthesis of Ag/TiO2 and Au/TiO2 by ultrasonic spray pyrolysis (USP). JOM, ISSN 1047-4838, May 2015, vol., iss., p. [1-6]

**DSL120**

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## Synthesis and Characterization of YCrxAl1-xO3Particles by a Reverse Micelle Processing

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YCrxAl1-xO3 nanoparticle was synthesized by a reverse micelle processing for inorganic pigment. Y(NO3)2·6H2O, Cr(NO3)2·6H2O and Al(NO3)3·9H2O are used for precursor in order to synthesis YCrxAl1-xO3 nanoparticles. The aqueous solution is consist of mixing molar ratio of Y/Cr/Al was 1:x:1-x and heat treated at 800~1100°C for 2h. The average size and distribution of synthesized YCrxAl1-xO3 powders was in the range of 10-20nm and narrow, respectively. The average size of the synthesized YCrxAl1-xO3 powders increased with increasing water to surfactant molar ratio and heating temperature. The crystallinity of synthesized YCrxAl1-xO3 powder increased with increasing heating temperature. The synthesized YCrxAl1-xO3 powders were characterized by x-ray diffraction analysis (XRD), field emission scanning electron microscopy (FE-SEM) and color spectrophotometer. The properties of synthesized powders were affects as a function of such as molar ratio, heating temperature etc.

**DSL240**

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## Phase Behaviour of Cellulose Nanocrystal Dispersion in Aqueous Sulphuric Acid and Development of an Energy Efficient Separation Technique for the Acid-Cellulose Nanocrystal

## System

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In this work, the phase behaviour of cellulose nanocrystal (CNXL) dispersion in aqueous sulphuric acid is reported. In particular, this study focuses on a dispersion containing 12.6 mg/ml of CNXLs in sulphuric acid of 30 wt%. This mixture is commonly the result of the hydrolysis process of native cellulose to extract CNXLs, and which the liquid acid requires separation from. With a careful study of the phase behaviour and properties of this mixture and other similar ones, a very energy efficient mechanised separation technique have developed to perform the acid/CNXL separation. The energy input to accomplish this separation, normalised to the process volume/mass of CNXLs, is at least one thousand times less than the currently employed centrifuge process.

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**DSL250**
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## Synthesis, Characterization and Biocompatibility Studies of Double-Walled Carbon Nanotubes /Chitosan–Polyvinyl Alcohol Biocomposites

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Bio-nanocomposite films based on chitosan/polyvinyl alcohol (CS/PVA) with different double-walled carbon nanotube values were processed using the casting/evaporation technique. Blend solution with different mass ratio (CS/PVA = 30/70, 50/50,70/30) were prepared. It has been found that the three components of bio-nanocomposites can be easily mixed in controlled conditions enabling the formation of thick films with high quality, smooth surface and good flexibility.
The morphology, diameter and structure of nanotubes were characterized by Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FT-IR) and Thermo Gravimetery Analysis (TGA). Also, the biocompatibilities of scaffolds were investigated by fibroblast culture after 24 h and 48 h.

Keywords: Bio-nanocomposites, Carbon nanotubes, Chitosan-Polyvinyl Alcohol, Cell culture