# Book of Abstracts

# INTERM 2019

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# 6th International Congress on Microscopy & Spectroscopy

# (INTERM 2019)

**Oludeniz/Mugla - Turkey**

**May 12-18, 2019**

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# INVITED SPEAKERS

## Id-213

### Optical Fibers for Solar Radiation Transmission as an Alternative Scheme for Solid-State Laser Pumping

Z. A. S. RADHI, H. MANAA*, A. H. JAFAR, H. M. ALBUFLASA AND W. E. AL-NASER
Department of Physics, University of Bahrain, P.O. Box 32038, Sakheer, Kingdom of Bahrain
Corresponding author: hmanaa@uob.edu.bh, hmanaa@gmail.com

**Abstract:** To improve pumping schemes of solar-pumped solid-state lasers, optical fiber bundles for solar radiation transmission are proposed. The conventional laser radiation collectors consist generally on a heliostat and a paraboloidal mirror where the heliostat tracks the sun and reflects the sun radiation on the fixed parabolic mirror and the mirror concentrates the sun light on the laser rod in a few square centimeters area. The inconvenient of this architecture are the security issue, because tens of kilowatts of laser radiation are manipulated, the heaviness of the instruments and their impracticability. In this paper, we propose another alternative for sun light transportation that is based on the use of optical fiber bundles to transmit sun light. The sun light is collected with small parabolic mirrors they will track the sun and concentrate the radiation on the ends of several optical fiber bundles that are placed at the mirrors focus. The fibers transmit the sun light into the laser cavity from outdoors to indoors. This design is expected to overcome the disadvantages of the conventional methods.

**Keywords:** Solar energy, solar-pumped lasers, YAG:Nd, fiber optics.

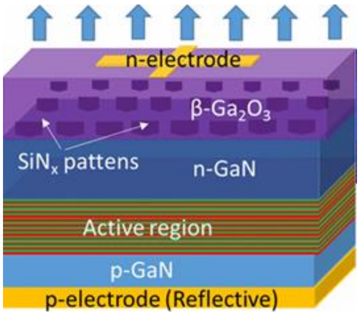
# INVITED SPEAKERS

## Id-214

### III-Nitride Emitting Devices Grown on (-201)-Oriented β-$Ga_2O_3$ Substrate

I. S. ROQAN
Physical Sciences and Engineering Division, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

**Abstract:** Wide bandgap III-nitride semiconductors possess several material properties that make them attractive for potential application in developing devices that emit and detect light in the spectrum between UV and visible wavelengths, as well as high-power electronic devices. Vertical-injection GaN-based light-emitting diodes (VLEDs) are of particular importance in this context, as they are promising candidates for high-efficiency and high-power devices. In this work, we report on high-quality III-nitride films and VLED based on multiple quantum wells (QWs) with high optical efficiency that were grown on β-$Ga_2O_3$ substrates by MOCVD, with high internal quantum efficiency (~ 86%). We show that (-201)-oriented β-$Ga_2O_3$ has a much lower lattice mismatch with GaN (~ 4.6%) compared to the currently utilized substrates, such as sapphire ($Al_2O_3$) and Si. This low lattice mismatch reduces threading dislocation density relative to that grown on sapphire, while also improving optical efficiency. InGaN/GaN and GaN/AlGaN multiple quantum wells have been structurally and optically investigated by X-ray diffraction, transmission electron microscopy, photoluminescence and time-resolved spectroscopy. Carrier dynamics analysis shows that all our samples are dominated by radiative recombination, indicating high optical and structural quality. The findings show that high optical efficiency can be produced by growing III-nitrides directly on (-201)-oriented β-$Ga_2O_3$ without the need for advanced and expensive fabrication methods. In this work, we confirm that this substrate can be employed in the production of large-scale, cost-effective and high-efficiency UV and visible vertical emitting device arrays.



VLED grown on (-201)-oriented β-$Ga_2O_3$

**Keywords**: III-nitride semiconductors, GaN-based light-emitting diodes, MOCVD

# INVITED SPEAKERS

## Id-216

### Application of Microscopy and Spectrometry for the Characterization of Various Materials: Powders, Films, Foams and Further their Investigation toward Alcohols Oxidation Reaction

V. KEPENIENE*, Z. SUKACKIENE, R. STAGNIUNAITE, J. VAICIUNIENE, A. NAUJOKAITIS, L. TAMASAUSKAITE – TAMASIUNAITE, E. NORKUS
Center for Physical Sciences and Technology, Lithuania
Corresponding author: virginalisk@gmail.com

**Abstract:** Various materials characterization is one of the most important investigations in materials science. It refers to the broad and general process by which a material's structure, morphology and properties are probed and measured. It is a fundamental process in the field of materials science, without which no scientific understanding of engineering materials could be ascertained.

While many characterization techniques have been practiced for centuries, such as basic optical microscopy, new techniques and methodologies are constantly emerging. In particular, the advent of the electron microscopes in the 20th century has revolutionized the field, allowing the imaging and analysis of structures and compositions on much smaller scales than was previously possible, leading to a huge increase in the level of understanding as to why different materials show different properties and behaviors.

In the present work, the surface analytical techniques such as Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS) and Inductively Coupled Plazma Optical Emission Spectrometry (ICP-OES) were used for the characterization of various materials: metals and metal oxides nanoparticles deposited on the surface of carbon powder; electroless deposited metals on Pt sheet or Ni foam. Further the examined materials were investigated towards ethylene glycol and glycerol oxidation by cyclic voltammetry.

Using various analytical techniques, the materials structure and particle size were determined and their influence on the activity of materials toward ethylene glycol and glycerol oxidation was demonstrated.

**Keywords:** Surface analytical techniques; Ethylene glycol; Glycerol oxidation.

# INVITED SPEAKERS

## Id-217

### Peculiarities of Auger Spectroscopy Study of Nanostructural Ceramic Composites Containing Light Elements

T. PRIKHNA
V. Bakul Institute for Superhard Materials of the National Academy of Sciences of Ukraine
Corresponding author: prikhna@ukr.net

**Abstract:** Auger spectroscopy allows to analyze thin surface layer (1-2 nm thick) and nanonclusions (10 nm in diameter); method is very sensitive for presence of light elements (N, O, B, C); it allows to distinguish whether nitrogen is present in the compound (even if the X-ray microprobe analysis cannot give the answer); the ion argon etching in the chamber of an analyzer gives possibility to remove surface oxidized layer in order to analyze “true” structure of the material and to perform the so-called analysis in depth (to estimate the variation of the elements distribution in the depth of the sample performing multiple etching and microprobe analyzing of the material composition). It should be noted that each definite material depending on the nature of the present elements needs the special approaches. The details of Auger analysis of nanostructural ceramic composites on the examples of MgB2 (with additions of carbon, silicon carbide (SiC), titanium and impurities of oxygen), B4C, B4C-SiC, AlB12, AlB12C2, Ti3AlC2, (Ti,Nb)3AlC2 and Ti2Al(C,O) ceramics by a JAMP−9500F (which combined SEM X-ray microprobe and Auger analyzers) will be under the discussion. The peculiarities of the materials etching, surface mapping, and estimation of their composition are considered as well as the intensity of electron beam during X-ray microprobe analysis of borides.

**Keywords:** Auger spectroscopy; Nanostructural ceramic composites; Light elements.

# INVITED SPEAKERS

## Id-219

### Glass-Clad Crystalline Fiber Based Broadband Light Sources for Optical Coherence Tomography

S. L. HUANG
Graduate Institute of Photonics and Optoelectronics, National Taiwan University, Taiwan
Corresponding author: shuang@ntu.edu.tw

**Abstract:** The recent advancement on the glass-clad crystalline fibers on broadband light emissions have shown superiority on the application for optical coherence tomography (OCT) where broadband, high brightness, continuous wave, and Gaussian-like spectral shape are crucial for in vivo and real-time cancer/disease diagnosis with cellular resolution.

Applying OCT on cells and tissues with sub-micron resolution could help unveil functions of living organisms and facilitate clinical disease/cancer diagnosis in the early stage. With cellular resolution, the lamellar structure of the epidermis and the vascular network in dermis could be resolved in both the cross-sectional and en face planes. Using Ti: sapphire crystalline fiber to generate broad and bright spontaneous emission, the OCT penetration depth can reach reticular dermis with appropriate image contrast. The melanin, fibrous connective tissue, capillary as well as small blood vessels were observed, and the vector flowing of the red blood cells were quantitatively analyzed. Image analysis algorithms have also been developed to automatically extract deterministic information from live tissue and single cells for discriminant analyses. The results show that OCT can be used to identify regions that suggest abnormalities and should be biopsied for histopathological examination. Both morphological recognition as well as parametric analysis using the back scattering from the subcellular structures will be addressed in the talk. With improved sensitivity/specificity, real-time data transmission and storage, and linking pathology to the treating physician, it is expected that the imaging advancement will eventually offer a see-and-treat paradigm, leading to improved patient care.

**Keywords:** Crystalline fiber; Optical coherence tomography.

# INVITED SPEAKERS

## Id-222

### Spectro-microscopy for Green Nano-materials/Devices

M. OSHIMA
The University of Tokyo, Japan
Corresponding author: oshima@sr.t.u-tokyo.ac.jp

**Abstract:** In order to develop green nano-devices for sustainable society, it is important to characterize electronic structure of nano-materials/devices such as 1) power generation devices like fuel cells and photocatalysts for hydrogen generation, 2) energy efficient devices like ReRAM, graphene FET, SiC power MOSFET, and GaN HEMT, and 3) energy storage devices like Li ion batteries on a nanometer scale. Therefore, we have developed soft X-ray scanning photoelectron microscopy (SPEM) with 70 nm spatial resolution [1]. We also modified our SPEM to apply different voltages independently to electrodes for gate, source and drain in a sample holder connected to a semiconductor parameter analyzer. Regarding power generation devices, we have analyzed each catalyst particle for novel water splitting photocatalysts La2Ti5CuS5O7 which can act as a p-type semiconductor electrode by SPEM. It is revealed that valence band spectra shift towards lower binding energy with Ga doping, suggesting successful hole doping probably into Cu 3d band by Ga doping [2]. Regarding energy efficient devices, the first example is graphene FET (GFET) where we discovered the linear band dispersion in graphene FET by operando SPEM analysis, showing the direct observation of p-type doping feature under back gate biasing [3, 4]. The second example is 4H-SiC trench MOSFETs for power devices. We conducted pin-point analysis of RIE (reactive ion etching)-processed trench sidewall for the MOS channel region directly affecting the FET performance. SPEM analysis revealed local band bending features caused by RIE damages. The estimated energy level was correlated with DFT calculations [5]. The third example is organic FETs for light weight and flexible electronics. OFETs for operando nano-spectroscopy were fabricated using ultrathin (3ML) single-crystalline C10-DNBDT-NW films. Operando measurements of line profiles of C 1s binding energy across the OFET channel from drain to source revealed that local threshold voltage in the nano-region can be determined by fitting the potential profile with gradual channel approximation (GCA) [6]. We also have applied this operando technique to GaN HEMT as the fourth example [7]. Furthermore, we have analyzed Lithium Ion Battery (LIB) as an example for energy storage devices using our SPEM [8]. We are also attempting operando SPEM analysis for all-solid-state LIB during charging/discharging processes. Future prospects of spectro-microscopy will be discussed.

**Keywords:** Spectro-microscopy; Synchrotron radiation; Green nano-devices; Operando analysis; Field effect transistors.

**Acknowledgement:** This work has been done in collaboration with H. Horiba, N. Nagamura, E. Sakai, H. Fukidome, K. Omika, M. Suemitsu, H. Matsui, J. Takeya, D. Mori, A. Takigawa, T. Hisatomi, K. Domen, A. Yamada and so on.

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# INVITED SPEAKERS

## Id-225

### Raman Hyperspectral Imaging of Transferrin-Bound Iron in Cancer Cells

T. C. KHOO, K. TUBBESING, A. SHARIKOVA, M. BARROSO, A. KHMALADZE
Department of Physics University at Albany, 1400 Washington Avenue Albany, NY 12222, USA
Corresponding author: akhmaladze@albany.edu

**Abstract:** Iron is an essential element required for human life. Iron is highly regulated in the body, as iron deficiency leads to many adverse health effects, such as anemias. Ferric iron (Fe3+) bound to serum transferrin (Tf) is internalized into cells via the transferrin receptor (TfR). Since the exact mechanisms of iron release in cells are not well known, a technique that allows detection of *Tf bound iron* inside intact human cells has been developed. Methods to determine when and where Tf releases iron inside a cell are required to better understand disease progression, including cancer. We have previously shown that Raman micro-spectroscopy is able to detect and quantify the Tf-bound iron in epithelial cells. In this work, we applied hyperspectral Raman imaging to visualize the spatial distribution of Tf-bound iron in human breast cancer T47D cells internalized with iron-loaded Tf, oxalate-Tf, a chemical Tf mutant unable to release iron, and iron-depleted Tf. We have also shown that Raman imaging can quantify the amount of iron under different times of Tf internalization, prior to fixation. Raman micro-spectroscopy provides a unique way to determine the amount of iron under different Tf internalization times by employing the Raman metric, which was used to quantify iron content in oxa-, apo-, and holo-Tf samples. Importantly, Raman microspectroscopy can be used to follow iron release from Tf in breast cancer cells. Determining the kinetics and location of iron release in cancer cells is key to further our understanding of iron metabolism during cancer progression.

**Keywords:** Raman scattering spectroscopy; Hyperspectral imaging; Cellular iron; Cancer.

# INVITED SPEAKERS

## Id-229

### Manufacturing of Titanium/Hydroxyapatite Bilayer Coatings on Medical Implants by Robotic Microplasma Spraying

D. ALONTSEVA

D. Serikbayev East Kazakhstan State Technical University, 69 Protozanov St., Ust-Kamenogorsk, 070004, Kazakhstan

Corresponding author: dalontseva@mail,ru

**Abstract:** Nowadays, the main commercially viable method of deposition of suitable biocompatible coating on the medical implants surface is thermal spraying [1]. The porous structure of the coating, developed by thermal spraying method, promotes the effective intergrowth of bone tissue into the pores of the implant significantly, which in turn contributes to its reliable fixation in the bone [2]. Plasma coating of surfaces of complex configurations, such as the medical implants of endoprostheses, presents a challenge for the implementation of the plasma thermal spraying technology. It requires automated manipulations of the plasma jet along with robotic control for appropriate treatment of a surface [3]. Currently, D. Serikbayev East Kazakhstan State Technical University (EKSTU) and specialists from E. O. Paton Institute of Electric Welding (EWI) are running combined activities on developing compositions (titanium/hydroxyapatite) and robotic technologies of microplasma spraying (MPS) of biocompatible coatings on endoprostheses made from the medical alloy produced in Kazakhstan. Hydroxyapatite (HA) is the calcium phosphate mineral of the apatite group. In medical implantology synthetic HA is used as implants coating, promoting a new bone formation. In order to form a Ti/HA bilayer porous coating on the implant surface by MPS several stages of activities needs to be completed. It is necessary to synthesize the hydroxyapatite powder of the desired composition with particles of a certain size and shape, suitable for MPS. The other stage of process is to prepare the implant surface for spraying with desired roughness. Then the modes of robotic MPS of both titanium wire and HA powder should be selected. Each of these processes requires further development and optimization.

The objective of the work is to consider the advantages and challenges of application of robotic MPS for applying titanium/hydroxyapatite bilayer coatings on medical implants.

The research has been carried out at a pilot production site established at EKSTU with an industrial complex for plasma processing of materials on the basis of Kawasaki RS-010LA, an industrial robot (Kawasaki Robotics, Japan). The robot's arm is equipped with "MPN-004" microplasmatron produced by EWI (Ukraine) for MPS of wire or powder coatings. Experimental methods included X-ray diffractometric analysis and transmission electron microscopy for analysis of the structural-phase composition of research materials, electron scanning and optical microscopy for measuring the porosity of coatings, and roughness and adhesion strength tests.

The main results of this work are the following: 1) hydroxyapatite has been synthesized and HA powder for MPS has been manufactured; 2) the parameters of gas-abrasive surface treatment of titanium implants have been developed to provide the required surface roughness before MPS; 3) the composition and modes of robotic MPS of bilayer Ti/HA coatings for titanium implants have been developed; 4) it was established that

coatings have desired phase composition, porosity and thickness and an adhesion strength with substrates of 25 MPa, meeting the requirements of the ISO 13779-2 regulations.

The results of the research are of significance for a wide range of researchers developing the plasma technologies for manufacturing biocompatible coatings.

Acknowledgment

The study has been conducted with the financial support of the Science Committee of RK MES in the framework of the target financing program for 2017-2019 within the program 0006/PTF-17 “Production of titanium products for further use in medicine”.

**Keywords:** Biocompatible coatings; Robotic microplasma spraying; Titanium; Hydroxyapatite.

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# INVITED SPEAKERS

## Id-239

## Positron Annihilation Spectroscopy as an Advanced Non-Destructive Characterization Tool in Nanomaterials Science

O. SHPOTYUK[1,2], A. INGRAM[3], J.FILIPECKI[1], Y. SHPOTYUK[4]

[1] Jan Dlugosz University, al. Armii Krajowej, 13/15, 42-200, Czestochowa, Poland

[2] Vlokh Institute of Physical Optics, 23, Dragomanov str., 79-005, Lviv, Ukraine

[3] Opole University of Technology, 75, Ozimska str., 45-370, Opole, Poland

[4] University of Rzeszow, 1, Pigonia str., 35-959, Rzeszow, Poland

Corresponding author: olehshpotyuk@yahoo.com

**Abstract:** Methodological possibilities of positron annihilation spectroscopy (PAS) in a lifetime measuring mode applied to characterize different functional materials exploring a nsanoscale are analyzed.

This method is grounded on space-time continuum determination for electron interaction with its antiparticle (positron), i.e. phenomenon of positron-electron annihilation. The PAS is high informative indirect characterization tool to study atomistic imperfections such as extended free-volume defects (vacancies, vacancy-like clusters, voids and void agglomerates, pores, micro- and macroscopic cracks, etc.) in solids whatever their structural organization (crystals or glasses, fine- or coarse-grained powders, ceramics or bulk alloys, etc.). The PAL spectrum consisting of histogram of measured annihilation times for positrons after deconvolution with instrumental resolution of detecting system and fitting with a theoretical model (governing corresponding regularization strategies in data parameterization), carries a rich information on atomic- and molecular-scale positron- and positronium-trapping free-volume imperfections. Such imperfections evolve sub-nanometer low electron density spaces stretching far below character sizes a few interatomic bonds, a level which is beyond possibilities of direct probes, such as intrusion porosimetry and electron microscopy.

The first part of this report deals with time-size correlations followed from application of positron-electron interaction formalism to structurally homogeneous solid state media, such as those composing host matrices for embedded nanoparticles. The second part of report concerns fundamentals of the PAS method applied to inhomogeneous nanoparticle-embedded substances. Different algorithms to treat PAS data for such solids within three-state trapping model evolving channels of positron and positroniun (Ps, i.e. bound electron-positron state) annihilation are analyzed. The developed approach to the void-filling problem in “host-guest” chemistry will be justified for:

- organic-inorganic nanocomposites like polyvinylpyrollidone (PVP) nanosuspension of mechano-chemically-activated sulfides,
- crystallization/ceramization-affected nanoinclusions like “hot”- and “cold”-grown nano-crystallites in phase-separated glassy-like matrices and
- structurally-intrinsic nano-inhomogeneities in network glass formers in the form of different spatially-extended structural defects.

In application to nanostructurized materials, the possibilities of PAL spectroscopy treated within x3-term fitting are critically considered to parameterize the underlying free-volume sub-atomistic void-evolution processes. Unlike conventional x3-term analysis based on admixed positron trapping and Ps decaying, the nanostructurization is considered as occurring due to conversion from preferential positronium decaying in a *host* matrix to positron trapping in modified *host-guest* matrix. The developed approach referred to as the x3-x2-CDA (i.e. the coupling decomposition algorithm) allows estimation defect-free bulk and defect-specific positron lifetimes of free-volume atomic-deficient elements responsible for nanostructurization. The x3-x2-CDA can be used as test-indicator, which separates processes of *host-matrix* nanostructurization from uncorrelated changes in positron-trapping and Ps-decaying modes.

The applicability of this approach is comprehensively proved for a variety of solids, allowing free-volume mictrostructure evolution due to Ps-to-positron trapping conversion. The free-volume guided functionality of nanomaterials is analysed at the numerous examples of (i) nanocomposite systems, (ii) structure-modification processes and (iii) *host-guest* chemistry problems like rare-earth doping in the modern chalcogenide and chalcohalide photonics.

**Keywords:** Positron annihilation; Nanomaterial; Trapping; Free-volume voids

# INVITED SPEAKERS

## Id-244

### Advantages of Impedance Spectroscopy Application in Analysis of Charge Carrier Transport in Chalcogenide Glasses

M.V. ŠILJEGOVIĆ[1], D.L. SEKULIĆ[2], K. ČAJKO[1], S.R. LUKIĆ PETROVIĆ[1]

[1]University of Novi Sad, Faculty of Sciences, Department of Physics, Trg Dositeja Obradovića 4,
21000 Novi Sad, Serbia

[2]University of Novi Sad, Faculty of Techical Sciences, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia

Corresponding author: mirjana.siljegovic@df.uns.ac.rs

**Abstract:** The scope of this paper is to provide an insight on the application of impedance spectroscopy (IS) for the study of mechanisms of the charge transfer processes occurring in chalcogenide glasses. A general introduction of the technique will be given, followed by an indepth review of its application on electrical measurements data of chalcogenide samples. The significant advantage of this technique is that enables estimation of contributions of the particular structural elements to the overall impedance response. By applying this analysis, it is possible to create an equivalent electric circuit model whose elements consistently represent the electrical properties of investigated material that correspond to the physical-chemical elements of its structure. Namely, transport properties of chalcogenide glasses strongly depend on the nature and the degree of short range order and are not notably affected by the loss of long range order. However, when introducing dopant elements in the structure in order to improve some properties significant for application of these materials in practice, complexity of the network can influence the conductivity values and electrical transport properties in a way of: change in conductivity type, appearance of ionic component of conductivity and diffusion processes, phase separation effects, etc... Implementation of IS analysis in these cases enables qualitative and quantitative characterization of all the contributions separately.

Some of these phenomena will be illustrated on the examples of chalcogenide systems doped with some metals such as Ag, Cu and Bi. Also, a discussion of future research directions concerning use of different electrical circuit models in order to describe the influence of dopants on amorphous matrix structure will be presented.

**Keywords:** Impedance spectroscopy; Chalcogenide glasses; Electrical properties.

# INVITED SPEAKERS

## Id-246

### On the Degradation of Pt-based Nanocatalysts for PEM Fuel Cells: An Identical Location Aberration-corrected TEM Study

P. FERREIRA[1,2,3]
[1]Materials Science and Engineering Program, University of Texas at Austin, Austin TX 78712, USA
[2]INL – International. Iberian Nanotechnology Laboratory, Braga, Portugal.
[3]Mechanical Engineering Department and IDMEC, Instituto Superior Técnico, University of Lisbon, Lisboa, Portugal
Corresponding author: paulo.ferreira@inl.int

**Abstract:** Proton exchange membrane fuel cells (PEMFCs) are promising energy conversion devices for transport and stationary applications. Pt nanoparticles (NPs) are currently used as the catalyst to promote the kinetics of the hydrogen oxidation and oxygen reduction reactions in the anode and cathode of the fuel cell, respectively. However, Pt-based alloys are being investigated to replace Pt on the cathode as a way to improve the efficiency of the fuel cell, and reduce cost. Although the enhancement in the ORR activity of Pt alloys is well established, the durability of the catalysts remains the main issue for their commercialization.

In this context, the focus of this work is to understand the behavior of Pt-Ni nanoparticles during the various stages of fuel cell cycling. For this purpose, a set-up was developed to simulate the effect of voltage cycling on the cathode side of the fuel cell. In this set up, Pt-Ni catalyst NPs supported on carbon were deposited on a gold TEM grid attached to a gold plate, which is used as a working electrode in a three electrode electrochemical cell. The NPs were cycled between 0.6 and 1.0 V in a $N_2$ saturated 0.1M $HClO_4$ liquid electrolyte. In this fashion, pre-defined locations of the electrocatalyst on the TEM grid were analyzed before and after cycling using an aberration-corrected JEOL ARM 200F.

Single atoms and atomic clusters appeared on the surface of the carbon support after voltage cycling as a result of surface dissolution of nanoparticles. As a result, single atoms move on the surface of the carbon support and redeposit on the surface of larger particles through modified Ostwald ripening. The surface re-deposition of single atoms occurs on {111} type planes of large particles. In some cases, a NP exhibits both dissolution and re-deposition at the same time. Aoms on surface steps are dissolved during voltage cycling, while new (111) facets are formed on the surface of the NP.

These experiments also showed the heterogeneous deposition of Pt on NPs. Initially, as there are no other particles nearby, the deposition of single atoms should have occurred, instead of coalescence. This heterogeneous deposition is likely due to the type of available surfaces in each NP. EDS mapping of this particle confirms that the atomic re-deposition that occurred consisted of Pt and not Ni. Due to the low $Ni/Ni^{2+}$ redox potential and low enthalpy of mixing of Pt-Ni, re-deposition of Ni on the particles is not thermodynamically favorable.

**Keywords:** Aberration corrected electron microscopy; Fuel cells; Catalysts.

# INVITED SPEAKERS

## Id-248

# Temperature Dependent Features of Low-Frequency Spectra of Ionic Liquids Studied by Femtosecond Raman-Induced Kerr Effect Spectroscopy

H. SHIROTA
Department of Chemistry, Chiba University
1-33 Yayoi, Inage-ku, Chiba 263-8522, JAPAN
Corresponding author: shirota@faculty.chiba-u.jp

**Abstract:** Femtosecond Raman-induced Kerr effect spectroscopy (fs-RIKES) is a powerful technique to investigate the molecular motions of condensed phases in the low-frequency or terahertz region (ca. 0.3 – 700 cm-1 or ca. 0.01 – 20 THz) where is difficult to observe using stead-state spectroscopic techniques [1]. The molecular motions of condensed phases in the low-frequency region have often key roles on elementary steps of chemical reactions in solution and controlling the materials properties. Essentially, the low-frequency motions are intra- and intermolecular vibrations and orientational dynamics. Our group has extensively studied the low-frequency motions of ionic liquids (ILs) by fs-RIKES [2,3]. ILs are molten salts at room temperature and getting more attention for their unique properties, such as nonvolatility, less flammability, and high electric conductivity. Recently, we have rather focused on the temperature dependence of the low-frequency spectra of ILs. In this talk, I am going to show the results of the temperature dependence of the low-frequency spectra of ILs including anion dependence of (i) imidazolium-based ILs [4] and (ii) pyrrolidinium-based ILs [5], (iii) cation dependence [6], and (iv) effects of aromatic rings [7].

**Keywords:** Femtosecond Raman-Induced Kerr Effect Spectroscopy; Ionic liquid.

**Acknowledgement:** The works were partially supported by JSPS KAKENHI (15K05377), Chiba University (Global Prominent Research Program: Soft Molecular Activation), and the Takahashi Industrial and Economic Research Foundation.

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# INVITED SPEAKERS

## Id-252

### Microscopy Investigation of Bacterial Biofilm Development in Flow Systems

L.C. GOMES, P. ALVES, A. SOARES, F.J. MERGULHÃO*

LEPABE – Department of Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias s/n, 4200-465 Porto, Portugal

Corresponding author: filipem@fe.up.pt

**Abstract:** Biofilms can be described as structured communities of microbial cells attached to surfaces and enclosed in a self-produced matrix of extracellular polymeric substances. Bacterial biofilms are often regarded as a problem in industrial and biomedical settings since their formation entails high costs and health risks. However, they can also be used advantageously in engineered systems such as in the production of recombinant proteins and industrial chemicals, and in wastewater treatment.

Bacterial adhesion and subsequent biofilm growth can be affected by several factors including the surface properties, which prompted the development of novel materials with the potential of either delaying or promoting biofilm formation. An important step in the development of these materials is the availability of testing platforms where the surface performance can be assayed in defined hydrodynamic conditions, since in nature, including human hosts, and in artificial environments, biofilms are often immersed in an aqueous fluid in motion and subjected to hydrodynamic forces. This presentation reviews the most widely used microscopy techniques to evaluate surface properties, gene expression, biofilm amount and architecture, and cell viability. Particular attention is given to the microscopy methods that can be applied to parallel plate flow chambers that enable real-time monitoring of cell adhesion, and to flow cells that can be used for kinetic long-time biofilm formation assays. These methods include bright-field microscopy (BM), epifluorescence microscopy (FM), confocal laser scanning microscopy (CLSM), scanning electron microscopy (SEM) and atomic force microscopy (AFM). This work shows that different microscopy techniques provide valuable and complementary information about different aspects of the complex structure of biofilms formed under flow conditions. A combined approach is therefore recommended for a comprehensive biofilm analysis.

**Keywords:** Biofilms, mcroscopy, flow systems.

# INVITED SPEAKERS

## Id-262

### Quantitative Phase Imaging of Plasmonic Metasurfaces

V. KŘÁPEK[1], P. DVOŘÁK[1], A. FAßBENDER[2], P. BOUCHAL[1], M. HRTOŇ[1], J. BABOCKÝ[1], F. LIGMAJER[1], R. CHMELÍK[1], S. LINDEN[2], T. ŠIKOLA[1]

[1] Central European Institute of Technology, Brno University of Technology, Purkyňova 123,
612 00 Brno, Czech Republic

[2] Physikalisches Institut, Universität Bonn, Nussallee 12, 53115 Bonn, Germany

Corresponding author: vlastimil.krapek@ceitec.vutbr.cz

**Abstract:** Plasmonic metasurfaces have emerged as a new generation of ultrathin optical devices, capable of all possible functionalities including lenses, wave plates, polarizers, holography, or optical vortex beam converters. The metasurfaces are made of structured arrays of subwavelength-spaced plasmonic nanoantennas that act as light scatterers with controlled phase response. Wavefront shaping of the light transmitted or reflected by plasmonic metasurfaces is ensured by the spatial distribution of the scattering phase. For studies on metasurfaces the high-resolution quantitative imaging of the phase directly in the metasurface plane is highly appreciated.

We present coherence-controlled holographic microscopy (CCHM) as a real-time, wide-field, and quantitative light-microscopy technique enabling three-dimensional (3D) imaging of electromagnetic fields, providing complete information about both their intensity and phase [1]. We demonstrate its ability to obtain phase information from the whole field of view in a single measurement on a prototypical sample consisting of silver nanodisc arrays. We employed it in the analysis of a simple metasurface represented by a plasmonic zone plate [2]. By scanning the sample along the optical axis we were able to create a quantitative 3D phase map of fields transmitted through the zone plate. Polarization-filtered CCHM has been used to map the phase of broadband Laguerre-Gaussian vortex beam converters based on Pancharatnam-Berry geometric phase [3].

Finally, we introduce quantitative 4th generation optical microscopy as the first wide-field (non-scanning) phase imaging technique that approaches resolution limits of optical microscopy and indicates the response of a single nanoantenna. Its unprecedented spatial resolution and light sensitivity are achieved by taking full advantage of the polarization selective control of light through the geometric phase. The measurement is carried out in an inherently stable common-path setup composed of a standard optical microscope and an add-on imaging module. We demonstrated the wide-field measurement of the phase altered by a single nanoantenna, while maintaining the precision well below 0.15 rad [4].

**Keywords:** Plasmonic metasurfaces; Phase imaging; Holographic microscopy.

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# INVITED SPEAKERS

## Id-263

### Single-Particle FRET Microscopy in the Studies of Nucleosome Structure and Interactions

A.V. FEOFANOV[1,2], N.V. MALUCHENKO[1], E.Y. KOTOVA[3], V.M. STUDITSKY[1,3], M.P. KIRPICHNIKOV[1,2]
[1]Biological Faculty, Lomonosov Moscow State University, Leninskie Gory 1, 119992, Moscow, Russia
[2]Shemyakin-Ovchinnikov Institute of Bioorganic Chemistry, Russian Academy of Sciences, ul. Miklukho-Maklaya 16/10, 117997, Moscow, Russia
[3]Cancer Epigenetics Program, Fox Chase Cancer Center, Philadelphia, USA
Corresponding author: avfeofanov@yandex.ru, Vasily.Studitsky@fccc.edu

**Abstract:** The structural basis of nucleosome formation, controlled unwrapping of nucleosomal DNA during different nuclear processes (i.e. transcription, replication, DNA repair) as well as recovery of nucleosome structure after DNA processing requires detailed investigation in order to understand, manipulate with and reproduce genome machinery. Nucleosomes assembled *in vitro* from core histones and short DNA are widely and successfully used to investigate this machinery. We contribute to this investigation by developing and applying single particle Förster resonance energy transfer (spFRET) microscopy [1-8].

We developed several experimental systems based on mononucleosomes labeled with a donor-acceptor pair of fluorophores: (i) core nucleosomes (CN) assembled using the 147 bp DNA templates; (ii) nucleosomes with a single linker (LN) reconstructed with the 167 bp DNA templates and having the 20 bp-long linker arm; (iii) linker nucleosomes (L2N) assembled with the 227 bp DNA templates and having two linker arms of 40 bp length each [1-3]. Introducing a donor-acceptor pair of fluorophores in neighboring DNA gyres and varying their positions along nucleosomal DNA we systematically probe DNA structure (namely, its packing, unwrapping, looping, etc) of the entire nucleosome. Introducing a donor-acceptor pair of fluorophores in neighboring DNA linkers and varying label positions along them we analyze the structure of the linker DNA regions.

Single nucleosome studies complemented with statistical analysis allow us to reveal and characterize structurally distinct subpopulations of nucleosomes that cannot be detected in conventional ensemble-based studies.

Poly-(ADP-ribose)-polymerase 1 (PARP1) is involved in DNA repair and transcription, and the mechanism of its action in chromatin requires detailed investigation. Our studies of nucleosome interactions with a poly-(ADP-ribose)-polymerase 1 (PARP1) before and during poly(ADP)-ribosylation as well as in the presence of PARP1 inhibitors employing spFRET microscopy will be presented.

**Keywords:** Single molecule; FRET; Nucleosome; PARP1; Fluorescence; Microscopy.

**Acknowledgements:** The studies were supported by RFBR (grant 17-54-33045).

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# INVITED SPEAKERS

## Id-265

### Mössbauer Spectroscopy: Applications in Microbiology and Biochemistry

A. KAMNEV
Laboratory of Biochemistry, Institute of Biochemistry and Physiology of Plants and Microorganisms, Russian Academy of Sciences, Prosp. Entuziastov, 13, 410049, Saratov, Russia
Corresponding author: aakamnev@ibppm.ru; a.a.kamnev@mail.ru

**Abstract:** The Mössbauer effect (recoilless emission or absorption of γ-radiation by nuclei of some elements/isotopes), discovered in 1957, soon emerged into a unique spectroscopic technique, Mössbauer spectroscopy, which has found diverse applications in materials science. Dominating applications utilise the stable isotope $^{57}$Fe (~2.2% in natural iron). In the $^{57}$Fe transmission variant of Mössbauer spectroscopy (**$^{57}$Fe-TMS**), the nuclear γ-resonance position is finely "tuned" by the $^{57}$Fe chemical state and coordination microenvironment (within an extremely narrow energy range, about ±0.6 μeV). It is equalised with the γ-quantum energy of the source ($^{57}$Co which decays via electron capture by the nucleus forming $^{57}$Fe and emitting a 14.4-keV γ-quantum) using the Doppler effect by vibrating the sample at varying speed versus the source (±12 mm/s cover the whole range of possible resonances). Conversely, in $^{57}$Co emission Mössbauer spectroscopy (**$^{57}$Co-EMS**), $^{57}$Co compounds are contained in (or added as a probe to) a sample under study, with a standard $^{57}$Fe-containing absorber. Thus, a $^{57}$Co emission Mössbauer spectrum provides information on the chemical state and coordination microenvironment of the *nucleogenic* ("daughter") $^{57}$Fe cation (stabilised after the $^{57}$Co→$^{57}$Fe decay largely in the same coordination and charge states) which *substitutes* for the "parent" $^{57}$Co cation.

Both $^{57}$Fe-TMS and $^{57}$Co-EMS can be useful in monitoring the chemical state, coordination and metabolic transformations of iron and cobalt, respectively, in various biological samples (see, e.g. recent reviews [1,2] and experimental reports on TMS [3] and EMS [4,5]). However, $^{57}$Co-EMS has so far been scarcely used in biological studies [1,2,4,5], although it is ~$10^4$-fold more sensitive than $^{57}$Fe-TMS. In this lecture, the possibilities of $^{57}$Fe-TMS will be shown in monitoring the state and time-dependent changes of the microbial intracellular Fe pool [1,3], as well as some recent representative examples reflecting the sensitivity of $^{57}$Co-EMS in studying cobalt(II) binding (with subsequent metabolic transformations) in live bacteria [1,2,5] or in the active centres of a metalloenzyme [1,2]. Interrelation of $^{57}$Co-EMS and $^{57}$Fe-TMS will be illustrated by the involvement of $Co^{2+}$ in reactions with labile [Fe-S] clusters (found in many proteins) during their *de novo* biosynthesis and/or repair as a basis of $Co^{2+}$ toxicity in bacteria [1].

**Keywords:** Mössbauer spectroscopy, biological applications, microbiology, biochemistry, iron metabolism, cobalt biocomplexes.

**Acknowledgement:** Supported in part by The Russian Foundation for Basic Research, Grant 17-08-01696-a.

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# INVITED SPEAKERS

## Id-266

### Application of Blood Serum Fluorescence and CD Spectroscopy Studies in Normal Locomotory and in Movement Dysfunction People

Z. DRZAZGA[1], I. SCHISLER[1], I. POKORA[2], E. SADOWSKA-KREPA[3]
[1] Department of Medical Physics, A. Chełkowski Institute of Physics, University of Silesia,
ul. 75 Pułku Piechoty 1A, 41-500 Chorzów, Poland
[2] Department of Physiological and Medical Sciences, Department of Physiology, The Jerzy Kukuczka Academy of Physical Education in Katowice, ul. Mikołowska 72a, 40-065 Katowice, Poland
[3] Department of Physiological and Medical Sciences, Department of Biochemistry, The Jerzy Kukuczka Academy of Physical Education in Katowice, ul. Mikołowska 72a, 40-065 Katowice, Poland
Corresponding author: zofia.k.drzazga@gmail.com

**Abstract:** Fluorescence spectroscopy as a sensitive, non-invasive method, which measures the emission intensity of the sample at very low concentration of fluorophores in biological systems, plays an important role in medical diagnostic of cancer (PDD and PDT). Our earlier studies indicated that fluorescence of serum can be also helpful in evaluation of the efficiency level of sportsmen. The aim of this study is spectroscopic assessment of blood serum in three groups of subjects with completely other motor ability: cross-country skiers, patients with Parkinson's disease (PD) categorized according to the Hoehn & Yahr classification and healthy volunteers.

Serum samples from a group of elite cross-country skiers (n=6), 24 PD patients and 11 age-matched healthy volunteers were studied using fluorescence and circular dichroism measurements at room temperature. The selected physiological and biochemical parameters of plasma samples were determined for the athletes due to a running exercise model performed at individual anaerobic threshold. Data were collected before the exercise, 3 min after exercise and after 1h and 24h of passive recovery. In addition, CD spectra as a function of temperature were registered for PD patients and control subjects. The statistical analysis was performed with Statistica software 13 and the level of statistical significance was set at $p<0.05$.

Two well-separated parts of the emission in the ultraviolet and visible regions have been detected for all the studied serum samples. A strong autofluorescence originated from structural aromatic amino acids emission included in blood serum proteins, mainly from tryptophan (Trp) and tyrosine (Tyr) in the ultraviolet region was observed. In the visible region markedly, weaker fluorescence contribution with broad maximum at about 505 nm and with the marked elongated shoulder towards the shorter wavelengths (about 460nm) was registered. Shape of the curves and maximum of emission peaks were dependent on the subject's group. Emission of amino acids in athlete's serum markedly decreased after treadmill exercise but Tyr fluorescence was more influenced than Trp emission. Decrease of fluorescence intensity after physical exercise may be related to the fact that muscles during exercise use large amounts of energy for intercellular processes that are activated by tyrosine. 24-h period of rest was sufficient to return to the pre-exercise state after training session. Significant correlations of the fluorescence intensity with the selected physiological and biochemical factors were obtained. For PD patients some deviations in the amino acid emission was observed. Significant correlations of fluorescence intensity with age, PD disease duration and H&Y rating were found.

CD measurements have shown that secondary structure of serum proteins dominated by *alpha-helix* structure is slightly more modified by PD that the effort of athletes. Temperature CD spectra of PD patients revealed decrease of stability of *α-helix* structure in favor of *beta-turn* contribution.

The fluorescence occurring in the visible region is attributed to closely spaced overlapping emissions generated by number of low molecular mass fluorescent compounds, mainly metabolic coenzymes (NAD(P)H, flawins). Non-enzymatic pathway of advanced glycation and products (AGE) could be also considered due to enhancement of emission at about 460 nm. Treadmill exercise for cross-country skiers caused a significant decrease of fluorescence intensity in the visible region but 24-h period of rest was sufficient to return to the pre-exercise state after training session. Fluorescence assay of metabolic pathways was also used in characterizing the severity of PD disease. Significant differences were found between healthy and HY4 groups unlike HY2 one.

Results of the study indicate that fluorescence and CD spectroscopy based on blood serum can be helpful in evaluation of the human movement abilities. However, further research is needed for validation of results.

**Keywords:** Fluorescence spectroscopy, CD spectroscopy, cross-country skiers, parkinson’s disease, human blood serum, biochemical/physiological factors.

# INVITED SPEAKERS

## Id-269

### WWOX Drives UV/Cold Shock-Induced Bubbling Cell Death in a Calcium-Dependent Manner whereas Pop-Out Death Occurs without WWOX

C. C. TSAI, N. S. CHANG*
Institute of Molecular Medicine, National Cheng Kung University, Tainan, Taiwan, ROC
Corresponding author: wox1world@gmail.com

**Abstract:** Bubbling cell death (BCD) is known as release of a nitric oxide-containing nuclear bubble from the cell surface in response to UV irradiation and cold shock. BCD, which is not apoptosis, necrosis and necroptosis, contributes to the severity of frostbite and UV. Unlike membrane blebbing for generating apoptosis, bubbling death is irreversible. Here, we determined that tumor suppressor WWOX drives the BCD. Mechanistically, UV energy is rapidly taken up by the nuclear DNA that transmits signals to cause rapid influx of calcium ion ($Ca^{2+}$), followed by nuclear accumulation of proteins, including p53, WWOX, TRAF2, NOS2, NFκB-p50, C1q and Hyal-2. Following release of the nucleolar content, irreversible bubble formation and death occurs. No cell shrinkage is shown. Depletion of $Ca^{2+}$ by EGTA retards BCD. In contrast, WWOX-deficient cells undergo shrinkage, then nuclear bubble formation and finally whole cell pop-out. By time-lapse holographic microscopy, the thickness of each bubble is about half of the cell height. In contrast, apoptotic and dividing cells have a significantly increased height in each cell. UV/cold shock significantly downregulates housekeeping proteins (e.g. α-tubulin). Gene expression profiling revealed that mRNA processing, mRNA metabolic process and RNA splicing were down-regulated, whereas sensory perception of smell, sensory perception of chemical stimulus and G-protein coupled receptor protein signaling pathway were up-regulated in L929S cells. Together, WWOX drives UV/cold shock-induced bubbling cell death in a calcium-dependent manner whereas pop-out death occurs without WWOX.

**Keywords:** Bubbling cell death; Pop-out death; WWOX; UV; Cold shock; Imaging.

**Acknowledgement:** Supported by MOST and NHRI, Taiwan.

# INVITED SPEAKERS

## Id-276

### Advanced Optical Microscopy Techniques in Studies of Cell Migration Mechanobiology

Z. RAJFUR, Z. BASTER, T. KOLODZIEJ, T. WITKO, D. SOLARZ, O. ADAMCZYK
Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science,
Jagiellonian University, Kraków, Poland
Corresponding author: zenon.rajfur@uj.edu.pl

**Abstract:** Cell migration is connected to several important biological phenomena like embryonic development, neural development, wound healing and immunological processes. Also many pathological processes like arthritis, osteoporosis and cancer metastasis are the result of impaired regulation of cell migration. For these reasons studies of cell migration regulatory mechanisms are of particular interest for science.

Recently, a new research direction, which studies the influence of the mechanical parameters of cellular microenvironment on the cell homeostasis and cellular phenomena, called mechanobiology, emerged. One of the phenomena, which are very sensitive to the mechanical properties of the environment, is cell migration. Migrating cells sense external forces exerted on them, elastic properties of their environment or its dimensionality. Then, they convert these mechanical clues into internal biochemical signals which, in turn, regulate the cells' response to such factors. Such process is called mechanotransduction. The studies of this phenomenon require new microscopic techniques which allow dissecting the molecular basis of cellular mechanotransduction.

Many advanced optical microscopy methods were developed which enable not only visualization of cellular mechanotransduction processes but can also employ light to elicit controlled changes in cellular processes or cell architecture. Optical tweezers, photoactivation of “caged” molecules, cell culture substrate with given elasticity, endogenous FRET biosensors, cell traction and confocal microscopies are examples of such techniques. Here, we present the results of our research where we employed these techniques to study the molecular pathways regulating the mechanotransduction process in migrating normal and cancer cells. Our studies focused on the mechanobiology of these cells migrating on elastic substrates such as polyacrylamide hydrogels with controlled stiffness or poly(3-hydroxyoctanoate) (PHO). We clearly show that elastic properties of the cell culture substrate heavily influence the cytoskeletal architecture and other biophysical parameters of migrating cells. Our results also indicate that employment of such advanced microscopy techniques is necessary in studies of dynamic, biological processes.

**Keywords:** Mechanobiology; Cellular mechanotransduction; Advanced optical microscopy; Cell migration.

# INVITED SPEAKERS

## Id-280

### Fluorescence Study of Interaction between Silicone and Lignin Model Compound

D. DJIKANOVIC, J. DRAGISIC MAKSIMOVIC, V. MAKSIMOVIĆ, K. RADOTIC
Institute for Multidisciplinary Research, University of Belgrade, Kneza Viseslava 1,
11030 Belgrade, Serbia
Corresponding author: djikanovic.daniela@gmail.com

**Abstract:** Silicon (Si) is the second most aboundant element on earth and it is considered as beneficial element for plants. In plants,Si promotes strengthening of the cell walls and provides increased mechanical support and exerts alleviative effects on various biotic and abiotic stresses. Previous investigation showed that Si occurs in the cell wall, where it is firmly associated with wall macromolecules in the form of silica ($SiO_2$). Lignin is one of the main components of the plant cell wall and it is a natural phenolic polymer with high molecular weight, complex composition and structure. Simillar to Si, lignin extensively contributes to mechanical properties in plant, growth, tissue/organ development, lodging resistance and the responses to a variety of biotic and abiotic stresses. Our physico-chemical investigation was focused on interaction between silicone and lignin model compound related to fluorescence as intrinsic property of lignin.

Dehydrogenative polymer (DHP), as representative lignin model, was synthesized with different concentration of $SiO_2$ (0.1mM, 0.6mM and 6mM). Fluorescence emission spectra were analyzed by the deconvolution method and time-resolved analysis, combined with HPLC data. We deconvoluted fluorescence spectra to three and four components. Approximation of the probability density (APD) results for component positions showed that Si induced structural changes in lignin model compound depending on Si concentrations. Low Si concentrations (0.1mM, 0.6mM) significantly disturbed, while high Si concentration (6mM) slightly affected ligning macromolecule.

The results of this work provide new perspective for manipulation of Si and lignin concentrations in the cell wall related process as well as new materials.

**Keywords:** Si; Lignin model compound; Deconvolution; Fluorescence.

# INVITED SPEAKERS

## Id-284

## 2D Correlation Analysis of Lysozyme Fibrillation Studied by Raman Optical Activity and Raman Spectroscopy

V. KOPECKÝ JR., T. PAZDERKA, K. HOFBAUEROVÁ, V. BAUMRUK
Institute of Physics, Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5,
121 16 Prague 2, Czech Republic
Corresponding author: kopecky@karlov.mff.cuni.cz

**Abstract:** Understanding of processes of amyloid fibrils formation is one of the key tasks in searching for proteins structural origin of human neurodegenerative diseases. Therefore, hen egg white lysozyme (HEWL) can serve as a good model of amyloid fibril formation. Furthermore, this protein is homologous to human lysozyme, which is one of the proteins that cause amyloid diseases [1]. Despite the intense scientific research, studying mechanisms in homologous proteins to lysozyme, the detailed mechanism of fibril formation is still far from complete understanding. Raman spectroscopy and Raman optical activity (ROA) can be used as very powerful techniques for studies of unfolded proteins and promising experiments on lysozyme have been already performed [2].

Here we present Raman and ROA study using 2D correlation spectroscopy (2DCoS) [3]. Firstly, we model changes of ROA band shapes and positions and investigate characteristic patterns in 2DCoS because the origin of 2D patterns for spectra with positive and negative bands has not been investigated yet [4]. Subsequently, temporal and thermal (from 20 °C to 60 °C) spectral changes in ROA and Raman spectra of HEWL were analyzed by means of factor analysis and 2DCoS. It gave us an opportunity to study delicate details of HEWL fibrillation and denaturation, i.e., the sequence of the secondary structure changes upon fibrils formation. Moreover, application of heterospectral 2DCoS enabled us to transfer band assignment from Raman spectroscopy to ROA. Spectral changes reflecting alterations in the conformation of S–S bridges were identified for the first time in protein ROA spectra and new ROA band assignment for Trp was found.

**Keywords:** Raman spectroscopy; Raman optical activity; lysozyme.

**Acknowledgments:** The Czech Science Foundation is acknowledged for support (Nos. 305/09/0457 and P205/10/1276).

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# INVITED SPEAKERS

## Id-285

### Ferromagnetic Co Films on Silicene

R. ZDYB, T. JAROCH, M. KOPCIUSZYŃSKI, M. KRAWIEC, M. DACHNIEWICZ, A. STĘPNIAK-DYBALA, M. JAŁOCHOWSKI

Institute of Physics, Maria Curie-Sklodowska University, Pl. M. Curie-Sklodowskiej 1, 20-031 Lublin, Poland

Corresponding author: ryszard.zdyb@umcs.pl

**Abstract:** Silicen is silicon analogue of graphene. It consists of one layer of Si atoms arranged in a 2D honeycomb lattice and as that, it possesses most of the fascinating properties of graphene. Beside many similarities with graphene silicene reveals additional features which make it even more interesting. It has been shown that it is possible to induce various quantum phases of matter and phenomena in silicene even at room temperature e.g. quantum spin Hall Effect, quantum anomalous Hall effect, band insulator, valley polarized metal and others [1]. These phases/phenomena can be brought into existence by applying and proper tuning eiher magnetic or electric fields. One of the ways for introduction of magnetic field is induction of internal magnetic field in silicene by adsorption of transition metal atoms on its top [2-5].

Bringing technology of making ferromagnetic silicene and/or making ferromagnetic contacts on silicene under control would allow preparation of spintronics devices like e.g. spin transistor. In this way, it would cause integration of spintronics with existing silicon technology.

In this report we present results of experimental and theoretical studies of the formation of silicene on the Au (111) ultrathin films grown on silicon substrate and the following growth of ferromagnetic ultrathin Co films on its top. We report crystallographic, electronic and magnetic properties of the grown heterostructures.

Deposition of ultrathin gold films on the Si(111) surface with subsequent mild annealing leads to the appearance of a new order on the Au surface in a form of 2D honeycomb lattice as revealed by STM images. The presence of silicon atoms on the top of Au is confirmed with the Auger electron spectroscopy. The electronic structure of the film measured with angle-resolved photoemission spectroscopy shows linear bands crossing each other 0.8 eV below the Fermi level. Results of the calculations performed within density functional theory indicate that the observed linear bands are associated with the formation of silicene on the top of the Au layer [6].

The following deposition of cobalt films on silicene is studied with low energy electron diffraction, low energy electron microscopy (LEEM) and spin-polarized LEEM. It is shown that the Co atoms form a continuous layer on silicene from the very begining of the growth and that the Co film becomes ferromagnetic at a thickness of about 3 ML at room temperature.

**Keywords:** Silicene; Cobalt; ARPES; LEED; LEEM; SPLEEM.

**Acknowledgement:** This work has been supported by the National Science Center under Grant No. 2016/21/B/ST3/01294

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# INVITED SPEAKERS

## Id-291

### Analysis of Nano-Scale Precipitates in Metallic Materials through Conventional and Advanced TEM Techniques

R. P. BABU
KTH Royal Institute of Technology, Brinellvägen 23, 114 28 Stockholm, Sweden
Corresponding author: pbrrajan@kth.se

**Abstract:** Nano-scale precipitation is one of the many strengthening mechanisms promoted in structural metallic materials through alloying and heat treatments, for applications in load bearing environments. The type of precipitates, distribution and their temporal evolution are the main governing factors which decide the strength and degradation of properties during prolonged application. Identifying the type of precipitate require both chemistry and crystallography studies. Volumetric distribution of such precipitates is analysed through imaging and spectroscopic techniques. Among the most advanced techniques of material analysis, transmission electron microscopy (TEM) is the most versatile technique for the nanoscale microstructural analysis in combination with energy dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS). This discussion covers a number of case studies on the techniques used in analyzing nano-scale precipitates in aluminium, magnesium, stainless steel, zirconium and titanium alloys through multiple imaging, diffraction and spectroscopic techniques combined with suitable analysis tools. Challenges in sample preparation and acquisition of useful information from both microscopy and spectroscopy and the methods used to tackle the issues will be elaborated. Examples of how the information of the nano-scale precipitate distribution is used for building a thermodynamic and physical based predictive material design models for developing a reliable understanding of material genome is also discussed.

**Keywords:** Transmission Electron Microscopy; Metallic materials; Nano-scale precipitates; Strengthening; Predictive models.

# INVITED SPEAKERS

## Id-292

## High Resolution Spectroscopy of Weak Molecular Lines in an Optical Cavity

K. BIELSKA
Institute of Physics, Faculty of Physics, Astronomy and Informatics, Nicolaus Copernicus University in Toruń,
ul. Grudziadzka 5, 87-100 Torun, Poland
Corresponding author: kasia@fizyka.umk.pl

**Abstract:** Weak molecular lines are crucial for remote sensing and astrophysics. Some of applications already require accuracy of reference line intensities as good as 0.1%, which is usually unobtainable in laboratory conditions. In many cases, such as in the oxygen B band, these lines are too weak to be carefully investigated with traditionally used techniques such as Fourier transform spectroscopy. However, it can be done with cavity enhanced techniques, such as well-established cavity ring-down spectroscopy (CRDS) and two novel techniques: cavity mode-width spectroscopy (CMWS) [1] and cavity mode-dispersion spectroscopy (CMDS) [2].

CRDS is based on measurement of the photon lifetime in a high finesse cavity. It has been used for determination of the lowest line intensities ever measured in laboratory conditions [3]. It was also used for verification *ab initio* calculations of line intensities for $CO_2$ molecule where an average, relative agreement at 0.33% level was found [4]. It is the best agreement ever obtained between theory and experiment on molecular line intensities. In our laboratory we use frequency stabilized, Pound-Drever-Hall locked cavity ring-down spectroscopy (PDH-locked FS-CRDS). The spectrometer is linked to the atomic frequency standard, UTC(AOS) having the relative stability of $10^{-15}$ [5]. We performed several studies on oxygen B band occurring near 689 nm and determined full sets of lineshape parameters for self-perturbed transitions [6]. The link to UTC(AOS) enabled us to determine frequency of unperturbed transition in the Doppler regime with relative uncertainty of $2\times10^{-11}$, which is typical for the Doppler-free techniques [7]. We have also simultaneously observed the speed-dependence and the Dicke narrowing in the pressure range as low as 10 Torr [8].

A technique complementary to CRDS is CMWS. CMWS relies on measurement of the cavity resonance width. With the increase of absorption, the time decay constant measured in CRDS decreases and thus nonlinearities of the detection system limit the measurement accuracy. In contrast to this, the mode width increases. Here high bandwidth of the detection system is not required, and thus absorption higher than in CRDS can be accurately measured.

Instead of absorption, the dispersion can be measured in an optical cavity (CMDS). It is related to absorption by Kramers-Krönig relations and it is manifested by the change of cavity mode positions. This results in purely frequency-based measurements, which is very attractive for metrological applications. We have shown that the technique enables determination of line intensity with sub-per-mile accuracy [9]. The comparison between results obtained with three cavity enhanced techniques will be presented.

**Keywords:** Cavity ring-down spectroscopy; Cavity mode-width spectroscopy; Cavity mode-dispersion spectroscopy; High resolution molecular spectroscopy.

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# INVITED SPEAKERS

## Id-293

### Optical Harmonics Spectroscopy of Semiconductors and Dielectrics

V.V. PAVLOV
Ioffe Institute, the Russian Academy of Sciences, St. Petersburg, Russia
Corresponding author: pavlov@mail.ioffe.ru

**Abstract:** Among various optical phenomena associated with a frequency conversion of electromagnetic waves, the second harmonic generation (SHG) is the simplest nonlinear process of second order. The next important nonlinear third-order optical process is the third harmonic generation (THG). Distinctive features of the SHG and THG processes are their high sensitivity to the crystal symmetry, local type of crystallographic environment, and spin order in transition-metal systems. Nonlinear spectroscopy based on the SHG and THG methods makes it possible to obtain a fundamentally new information about solids as compared with studies using linear optical techniques. This fact is due to the in the selection-rule difference for single-photon and multi-photon processes [1].

In this lecture, nonlinear magneto-optical phenomena associated with the SHG and THG processes in various classes of materials, such as magnetic dielectrics – ferrite-garnets, hexagonal multiferroic manganites, and antiferromagnetic oxides, magnetic, diluted and diamagnetic semiconductors ($A^{II}B^{VI}$ and $A^{III}B^{V}$) will be considered. Various aspects of nonlinear optical spectroscopy such as polarization, temperature and field dependences; revealing crystallographic, ferroelectric, ferromagnetic and antiferromagnetic contributions will be discussed [1-5].

The unique possibility of visualization of antiferromagnetic domains the SHG technique indistinguishable by linear optics methods will be demonstrated. The possibilities of the SHG and THG methods for establishing of new mechanisms for nonlinear optical interaction at exciton states in bulk semiconductors will be revealed.

**Keywords:** Optical harmonics spectroscopy; Second harmonic generation; Third harmonic generation.

**Acknowledgement:** The Russian Foundation for Basic Research (RFBR-DFG) and the German Scientific Foundation DFG (TRR142) are acknowledged.

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# INVITED SPEAKERS

## Id-300

### Photodissociation Dynamics of $CF_2I_2$ in Solution Probed by Femtosecond IR Spectroscopy

M. LIM
Department of Chemistry, Pusan National University, Busan, Korea
Corresponding author: mhlim@pusan.ac.kr

**Abstract:** Although chemical reaction dynamics of isolated molecules has been extensively explored and understood, that in liquid phase is much less comprehended due to the complication of solvent-solute interaction. One strategy to study reaction dynamics in solution is comparing solution-phase reactions with their gas-phase counterparts. The comparison helps us to explore which ideas from the study of gas phase reaction can be applied to that in solution [1]. Recent advances in ultrafast spectroscopic techniques make it possible to obtain a detailed information on the reaction dynamics in liquids. In particular, time-resolved infrared (IR) spectroscopy can probe transient molecular structures involved in chemical reaction pathways, providing critical information needed in solving the mechanisms of chemical reactions [2]. Here, photodissociation dynamics of $CF_2I_2$ in $CCl_4$ and c-$C_6H_{12}$ were probed by time-resolved IR spectroscopy in the time range from sub picosecond to microsecond. We were able to compose a complete photolysis dynamic of $CF_2I_2$ in solution including the fate of all the produced intermediates.

**Keywords:** Femtosecond IR spectroscopy; Photodissociation dynamics in solution.

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# INVITED SPEAKERS

## Id-305

### Development of Iron Oxide Based Adsorbent for $CO_2$ Capture Studies

A. H. LAHURI
Department of Basic Science and Engineering, Faculty of Agriculture and Food Sciences, Universiti Putra Malaysia
Kampus Bintulu, P.O Box 396, Nyabau Road, 970008 Bintulu, Sarawak, Malaysia.
Corresponding author: azizulhakim@upm.edu.my

**Abstract:** Various type iron oxides of FeO, $Fe_2O_3$, and $Fe_3O_4$ were used for carbon dioxide ($CO_2$) capture at room temperature and pressure by studying its adsorption−desorption properties. Higher adsorption performance was conducted by impregnating iron oxide on activated carbon (AC) to further study the adsorption isotherms and kinetics. This works aims to obtain most efficient type of iron oxide for $CO_2$ capture and to study the adsorption isotherms and kinetics for iron oxide impregnated on AC. These adsorbents were characterized by using XRD, FTIR, SEM, $N_2$ adsorption-desorption isotherm.

Several interactions of carbonate species were detected on iron oxides surface which similar to the hematite/ carbonate complexes. Furthermore, surface hydroxyl groups formed easily that interacted with $CO_2$ to generate bicarbonate species. Trace amounts of moisture content of approximately 20 ppm from the feeding gas $CO_2$ have the potential to be a precursor and enhance the formation of more surface hydroxyl groups on the ready presence of the hydroxyl before adsorption. Adsorption at room temperature and pressure shows no adsorption bands for monodentate and bidentate carbonate on $Fe_2O_3$. The morphology of carbonate formation through SEM images shows different structures on FeO (grooves-like), $Fe_2O_3$ (fine sharp particles), and $Fe_3O_4$ (aggregated nanoparticles). $CO_2$ chemisorption discovered a potential adsorbent of $Fe_2O_3$ with an adsorption capacity of 3.95 mg $CO_2$/g adsorbent. $\mathrm{Max}_{DT}$ 575 °C is lower than that of other common materials used such as CaO (900 °C). The adsorption capacity increased up to 62.8 % by using concentrated 99.9% $CO_2$ for adsorption. $Fe_2O_3$ possessed the highest basicity strength (1.26 $cm^3/g$), and the adsorption capacity after four cycles was not significantly reduced by 8.6 % indicating an effective chemical or physical adsorption in $CO_2$ capture.

Chemically treated AC with loaded iron oxide are compared at various concentration ratios of $KMnO_4$ treatment (0.1, 0.5 and 1 M) and iron oxide loading (0.1, 0.5 and 1M). Thermal gravimetric analysis experimental setup was used for $CO_2$ adsorption kinetics measurement and the experimental data is subsequently modelled using pseudo first and second order kinetics models. Gas sorption analyzer was used to measure the multilayer $CO_2$ adsorbed and subsequently modelled using Langmuir and Freundlich isotherms. The adsorption capacity revealed that the 0.1K:0.1Fe is most efficient adsorbent with the presence of $Fe_2O_3$ peaks are dominant rather than $Fe_3O_4$ through XRD diffractogram. The capture capacity at 25 and 30 °C were measured to obtain 108.60 mg/g and 41.12 mg/g respectively. The $CO_2$ adsorption kinetics data for the adsorbents were varied depends on the surface morphology condition of the adsorbents. The efficient adsorbent of 0.1K:0.1Fe well fitted with pseudo second kinetics model suggesting chemisorption process involving exchange of electrons between adsorbent and adsorbate. The intra-particle diffusion model indicates intra-particle diffusion is not the only controlling mechanism in the adsorption process. The adsorption isotherms show that all adsorbents well fitted with Freundlich isotherm indicating the iron oxide loading on AC creates a heterogeneous surface binding with exhibiting a non-uniform distribution of adsorption energy.

**Keywords**: $CO_2$ capture, iron oxide, activated carbon, adsorption isotherm, adsorption kinetics.

# INVITED SPEAKERS

## Id-307

### FTIR Spectroscopy Study of Nanosurface Phenomena

I. MARKOVA-DENEVA
University of Chemical Technology and Metallurgy, 8, St. Kl. Ohridski blvd., 1756 Sofia, Bulgaria
Corresponding author: vania@uctm.edu

**Abstract:** The nanoscale state is a characteristic of the animate and inanimate nature objects, including the space. A specific characteristic of the nanoscale state is a size of the order of several to 100 nm (1 nm=$10^{-9}$ m, 1 nm=10Å). This is a state of shrinking matter, which is really proved. It is a remarkable special feature of the nanoscale state and is expressed as a change (decreasing) in the internucleus distance between two closest neighboring atoms, taking part in forming of diatomic molecules (dimmers), clusters and nucleus of nanoscale phases without an injuring of the atom nucleus. The conception “kinds of chemical bonds” (such as ion, covalent, metal, Van deer Waals, hydrogen, donor-acceptor and other bonds) at the nanostate loses its concrete definition. The chemical bonds ought to be considered regarding to length and energy. For all nanomaterials the so-called quantum-sized effect is observed, which is due to the decreasing of the internucleus distance between two closest neighboring atoms. The nanoparticles are characterized by a high developed specific surface area (SSA) and surface-active properties. The ratio of the surface atom number to the volume atom number is high. There are a lot of free chemical bonds on the nanoparticle surface, which is the reason for their adsorption affinity towards various ions and molecules.

In 1999 our team [I. Markova and I. Dragieva - J. of Bulgarian Chemistry and Industry, v. 70, No 2 (1999) 55-58] investigating Co nanoparticles by FTIR spectroscopy proved the shrinking effect of the synthesized nanoscale materials – a decrease in volume of the unit cell was established and confirmed, as deduced from a frequency shift of the stretching asymmetric and symmetric vibrations to the high frequency region characteristic for a vibration of shorter and stronger bonds. This shrinking effect of the nanoscale state for the synthesized Co nanoparticles was also proved by XRD investigations of the obtained Co nanomaterials.

We have used a chemical reduction method with $NaBH_4$ in various aqueous solutions of different metal salts with a different anion composition ($SO^{2-}_4$, $NO_3^+$, $Cl^-$, $Br^-$), respectively in their mixtures to synthesize metallic and intermetallic nanostructures at the relevant ratio between the metallic components chosen according the corresponding binary systems phase diagrams such as Cu, Co, Ni, Sn, Cu-Sn, Co-Sn, Ni-Sn, Co-Ni nanoparticles. Metallic (Cu, Co Ni) and intermetallic (Cu-Sn, Co-Sn, Ni-Sn, Co-Ni) nanoparticles have been synthesized through the same reduction method with $NaBH_4$, but applying the so-called “template” technique using different supports (ceramic supports $SiO_2$ and MCM-41 such as SiMCM, AlMCM, and respectively carbon support such as graphite and carbon powder) with a different chemical nature (element composition) and different specific surface area, SSA [$m^2/g$]. In such way hybride nanoparticles have been synthesized, respectively carbon nanocomposies have been obtained in situ.

The IR spectroscopy is used in both the research and industry as a simple and reliable technique for measurement, quality control and dynamic measurement. It is also successful for applications in the organic and inorganic chemistry. Our team considers that the IR spectroscopy (FTIR spectroscopy) is a sensitive and correct method to investigate the nucleation phenomena and creation of bonds on the nanomaterial surface, respectively molecule structure of the nanoparticles synthesized through a chemical reduction. The FTIR spectroscopy is mainly used in the mid-IR region of the visible spectrum to study the fundamental vibrations of the nanostructures. By means of the FTIR spectroscopy different atom groups formed on the nanoscale material surface have been successfully investigated on the basis of their natural vibrations at fixed frequencies. This spectroscopic study of the synthesized nanomaterials in our work was carried out with a Fourier Transform IR spectrophotometer EQUINOX 55 (Bruker) in 4000 to 400 cm$^{-1}$ frequency regions using KBr matrix.

As it is known, the IR spectroscopy exploits the fact that the molecules have specific frequencies at which they rotate or vibrate corresponding to discrete energy levels. The simple diatomic molecules have only one bond, which may stretch. The more complex molecules have many bonds, and the vibrations can be conjugated, leading to infrared absorptions at characteristic frequencies that may be related to chemical groups. For example, the atoms in $CH_2$ group, commonly found in the organic compounds can vibrate in six different ways: symmetric stretching (valence) vibrations, asymmetric stretching vibrations, deformation (bending) vibrations such as scissoring, rocking, wagging, twisting vibrations.

The FTIR spectroscopy method establishes exactly the differences between the FTIR spectra due to the different way of the support introduction and its surface wetting and the initial salt used (metallic sulphates or chlorides) for the nanostructure synthesis. The experimental data for the stretching and bending vibrations of the O-H, B-O, B-H, C-H, C-O, C=C, Me-O bonds are similar to those reported in the literature. The carried-out study of the nanomaterials in the mid-IR region (4000–400 cm$^{-1}$) demonstrates that the FTIR spectroscopy is a suitable, successful and powerful method for investigation of nanosurface phenomena and surface atom groups formed on the nanomaterial interface. From the FTIR spectra it can be received information about the technological conditions: initial salts, type of reactors used, ceramic and carbon supports used, different ways of a support introduction and the support surface wetting.

The FTIR investigations, respectively the taken FTIR spectra prove the creation of different chemical bonds (B-O, B-H, O-H, Si-O, C-O, C=C, Me-O) and the formation of different atom groups on the nanomaterial surface, the change of the bond length, and the nanosurface phenomena that occur on the solid-solid, solid-liquid, solid-gas nanointerface such as reduction, oxidation and molecule adsorption that are directly depended on the synthesis conditions. The FTIR spectra mode is different because of the stretching and bending vibrations of the created chemical bonds in different surface atom groups formed on the nanoscale material surface.

The results obtained based on the taken FTIR spectra for the characteristic vibrations of the chemical bonds (such as C=C, C-C, C-H, C-O, B-O, O-H, Me-O, where Me=Co, Ni ) in atomic groups formed on the surface of the nanoparticles complement the literary data concerning the nanomaterials. It is established the characteristic frequencies (wavelengths) at which bands of absorption occur referring to different types of vibrations of the generated chemical bonds (symmetrical and asymmetric stretching, bending) in different atomic groups and structural units (such as $BO_3$, $BO_4$, OH , $H_2O$, $CH_2$, $CH_2OH$, CO-OH, C-OH, CoO, NiO) covering the surface of nanoparticles.

Based on the different types of the taken FTIR spectra (position and intensity of the absorption bands) of the nanomaterials synthesized through a borohydride reduction method, as well as by a template synthesis using a carbon support, it has been shown that this spectral method for structural characterization of the chemical compounds is a sensitive and accurate method for studying nanomaterials as well. FTIR spectroscopy is a suitable research method for investigation of the phenomena and processes occurring at the interface nanosurface, including formation of atomic groups enveloping the surface of metallic (Co, Ni)/intermetallic (Co-Ni) nanoparticles.

The proven formation of different chemical groups located on the nanoparticle surface based on the absorption bands observed on the FTIR spectra at the respective characteristic frequencies will contribute to elucidating the particle formation mechanism depending on the elemental composition of the starting salts used. The data obtained in the IR spectroscopic examination of the surface state of the synthesized metal/intermetallic nanodimensional materials with sufficient reliability can be used by other researchers involved in IR spectroscopic characterization of nanosized objects.

**Keywords:** Metallic/intermetallic nanoparticles; Nanosurface phenomena; FTIR spectroscopy.

# INVITED SPEAKERS

## Id-308

### The Use of Raman Microspectroscopy in Polymer Research

Z. MORÁVKOVÁ*, P. BOBER, M. DUŠKOVÁ, V. CIMROVÁ
Institute of Macromolecular Chemistry, Czech Academy of Sciences, Heyrovsky Sq. 2,
162 06 Prague, Czech Republic
Corresponding author: moravkova@imc.cas.cz

**Abstract:** Raman microspectroscopy is a valuable technique in polymer research due to its good chemical and spatial resolution. In my contribution, I would like to illustrate the potential of this technique on several applications.

- Raman spectroscopy is a relatively surface sensitive technique – especially on dark and colored samples. This combined with the use of the microscope enabled us to verify if a substrate, carbonized nanotubes in our case, is uniformly coated with a polymer layer (polyaniline), or if a simple mixture of carbonized nanotubes and chunks of polymer is formed.
- When Raman spectroscopy is combined with confocal microscopy, depth profiling of optically clear samples is possible. We have studied a room temperature (RT) cured high-solid paint for automotive industry. Gradient of chemical composition is an inherent property of the RT-cured systems and influences the durability of the coating. Its main cause is inhomogeneous solvent evaporation – it leaves faster from upper layers and may get trapped in bottom layers, in addition, some components may sediment. To understand these processes, the knowledge of the chemical depth-profile is crucial.
- Low-band gap copolymers blends with fullerenes can be used as active layers in organic photovoltaic (PV) devices. The quality of the blend film is important for the PV device performance. Raman microspectroscopy is a useful tool to monitor local polymer order and enable us mapping of chemical composition of blends for bulk-heterojunction structure. By the use of two excitation wavelengths we were able to analyze the phase-separation of the blend and determine the changing content of the two components in different points of the film.
- Microscopy has its use also in combination with spectroelectrochemistry. In a Raman spectroelectrochemical experiment, Raman spectra are measured on a sample located on a working electrode at a certain potential, allowing the study of various redox forms of the sample and their transformations. So far we use the microscope only for easy and effective focusing of the laser beam on the sample unevenly spread on the electrode. However, mapping electrode surfaces can be useful for example in battery research.

**Keywords:** Raman spectroscopy; Conducting polymers; Depth profiling; Mapping.

# INVITED SPEAKERS

## Id-311

### Research on the Intelligent Technology of the Atomic Force Microscope

Y. ZHANG[1,3], Y. LI[2,3], J. QIAN[2,3]

[1]School of Instrumentation and Optoelectronic Engineering, Beihang University, Xueyuan Road *No*.37, Haidian District, Beijing, 100191, China

[2]School of Physics, Beihang University, Xueyuan Road *No*.37, Haidian Discritic, Beijing, 100191, China

[3]Key Laboratory of Micro-nano Measurement-manipulation and Physics (Ministry of Education), Beihang University, Xueyuan Road *No*.37, Haidian District, Beijing, 100191, China

Corresponding author: liyingzi@buaa.edu.cn

**Abstract:** Atomic force microscope (AFM) is a powerful tool observing nano-characteristics of materials and biology samples in various environment. However, to obtain high-quality AFM images, professional experience is required for operators to set proper controller parameters and appropriate scan speed. In this study, we have proposed novel methods for intelligent PID parameters tuning and real-time scan speed control of the AFM. Firstly, to obtain the proper PID parameters for different imaging situations, an intelligent tuning method based on iterative learning control is proposed. A pre-scanning before normal imaging is applied to acquire the information of the sample topography. Then the pre-scanning information is used to calculate a specific set of PID parameters. In addition, a real-time imaging rate control algorithm is proposed to balance the image quality with the imaging rate. Differently with traditional scanning methods, the proposed method adjusts the scan speed linearly according to the real-time tracking error of each scanning point, which is mainly determined by the sample topography. Simulations and experiments have been carried out to verify the proposed methods. The proposed intelligent PID parameters tuning method can be used to obtain proper PID parameters for different samples to ensure high-quality imaging. And the real-time scanning speed control algorithm can increase the scan speed of the AFM for samples with strongly fluctuant topography up to about 3 times without any imaging quality decrease. The proposed methods greatly reduce the professional requirement for AFM operators to obtain a high-quality image.

**Keywords:** Photodissociation; IR spectroscopy.

**Acknowledgement:** This research is supported by the National Science Foundation of China (No. 61771033 and 61371008).

# INVITED SPEAKERS

## Id-312

### Biological Fate of Engineered Nanomaterials: Tracing Aggregation/Degradation and Nanomaterial Dose in Vitro and in Vivo

S.E. MOYA
CIC biomaGUNE, Paseo Miramón 182 C, 20006 San Sebastian, Spain
Corresponding author: smoya@cicbiomagune.es

**Abstract:** There is an urgent need for a deeper understanding of the impact of engineered nanomaterials (ENMs) on human health resulting from deliberate exposure to ENMs, such as in nanomedicine, or from accidental exposure due to handling or using devices or products containing ENMs. The characteristics of ENMs, such as shape, size, degradability, aggregation, surface and core chemistry determine their interaction with biomolecules and the ENMs fate both intracellularly and at body level. Therefore, for the assessment of ENMs toxicity is necessary to correlate ENMs characteristics with their fate and biological interactions. ENMs fate *in vivo*, distribution per organ, accumulation, biodurability and dose are fundamental to assess how ENMs affect biological functions. The physical state of the ENMs, including aggregation, the interaction with biomolecules in different cellular environments, and guide the intracellular action of nanomaterials. A fundamental aspect for understanding toxicity is to establish the relation between exposure dose of ENMs and the intracellular dose or the dose per organ. The actual dose following an exposure route is the result of the translocation of ENMs across different barriers.

Tracing ENMs in biological matrixes and moreover, determining the intracellular or organ dose of ENMs pose several challenges since ENMs are not easy to visualize and to quantify once in a biological matrix. In this presentation we will focus on the methodologies and experimental techniques, mainly from Molecular Imaging and Biophysics, which are combined to address these issues.

Several aspects of ENMs fate *in vitro* and *in vivo* will be discussed mainly in relation with ENM quantification and ENMs stability. Cell uptake and intracellular fate of ENMs will be presented. The intracellular dose for metal oxides nanoparticles will be measured with Ion Beam Microscopy. Relations between exposure dose, intracellular dose and cell viability will be established.

Protein corona formation and the aggregation behavior of gold nanoparticles (Au NPs) will be investigated by means of Fluorescence Correlation Spectroscopy (FCS) in cell culture media and in live cells. The behavior in vitro will be compared with the level of aggregation of the NPs intracellularly. Diffusion coefficients of the NPs will be measured following NP trafficking at different positions in the cell: the endoplasmatic reticulum, the endocytic vesicles, the cytosol and in intracellular vesicles. Fluorescence Cross Correlation Spectroscopy (FCCS) will be applied to study the intracellular stability of protein corona.

The bio distribution, organ accumulation and fate of radiolabelled ENMs will be studied in animal models by means of Positron Emission Tomography (PET). NPs dose per organ will be evaluated. A dual radiolabelling strategy of nanoparticle core and coating will be presented using gamma emitters with non overlapping emission bands. After intravenous administration into rats, energy-discriminant Single-Photon Emission

Computerised Tomography (SPECT) resolve each radioisotope independently revealing different fate in vivo for the core and coating, which will be used to evaluate NP integrity.

**Keywords:** Radiolabelling; In vivo imaging; Nanoparticle translocation; Fluorescence correlation spectroscopy; Nanoparticle dose; Nanotoxicology

# INVITED SPEAKERS

## Id-317

### High-Resolution Episcopic Microscopy 3D analysis in Animal and Plant Research

Y. CINNAMON[1], O. GENIN[1], A. IZHAKI[2]
Agricultural Research Organization, The Volcani Center, Institute of Animal Science
Poultry and Aquaculture Sci. Dept.[1], Plant Sciences, Fruit Tree Sciences[2]. HaMaccabim Road, Rishon LeTsiyon, P.O.B 15159, 7528809, Israel
Corresponding author: yuval.cinnamon@mail.huji.ac.il

**Abstract:** Three dimensional imagine is a powerful tool to study morphology and anatomy of biological samples. In the last decade's various methods for 3D imagine such as confocal laser scanning microscopy, high-resolution X-ray based µCT, Magnetic Resonance Imaging (MRI), Positron Emission Tomography (PET), Optical Projection Tomography (OPT) and 3D reconstruction of histological sections were adopted and applied both in animal and plant research.

Initially developed by Prof. Tim Mohun (The Francis Crick Institute, London, UK), the High Resolution Episcopic Microscopy (HREM) is a technique for visualizing the morphology of materials embedded in methacrylate resin JB4. In recent years the system became commercially available (Indigo Scientific, Baldock, UK) with growing number of worldwide users, allowing them to exploit the benefits of the technique. While cutting through the sample, series of inherently perfectly aligned digital images are created from subsequently exposed block surfaces. These images are stacked and converted to volume data of high resolution and quality. HREM volume data fit excellently for in-situ visualization, volumetric, planar and mathematical analysis of the analyzed specimen.

HREM is a microscope-microtome based imaging system which generates hundreds-thousands of perfectly aligned thin section images of an embedded tissue. The fluorescence stereomicroscope and a digital camera facilitate a block-face image capture of fluorescent dyes mixture which stains the embedded specimen in the block. Following every section (~1-5 µm thick) the camera captures the image of the block cut surface. The images are stacked and processed using 3D visualization software to generate 3D model of the specimen at a near histology resolution (~2 $\mu m^3$ per voxel). The 3D model can then be virtually sectioned in any plane and enables metric analysis of the visualized structures. Since its first introduction (Nat Genet. 2002 Jan;30(1):59-65.) the HREM method was used in various medical application such as morphology analysis of model organisms and human tissue visualization. The HREM method was shown to obtain 3D images with higher resolution compared to µCT, µMRI, and OPT techniques. Though HREM fails to reach confocal, light sheet or electron microscopy resolutions, it can provide 3D imagine of much bigger specimens (effectively up to 10X10X15 mm).

Using the cutting-edge technology of the HREM, the presented studies aim at creating a comprehensive 3D chick embryo atlas, in sequential time resolution of 4 developmental hours apart, starting at oviposition until 6.5 days of incubation. Altogether, 40 models are included in a developmental time frame which ovetlap the developmental stages 1-30 HH. This atlas benefits from near-histological spatial resolution in all plans highlighting complex morphological features. Additionally, we demonstrate the usefulness of the system in analyzing tough-to-process various types of plant tissues such as in a study of the relationship between stem anatomy and rooting capacity in easy and difficult-to-root persimmon rootstock. These were analyzed by HREM and detailed 3D models were generated.

**Keywords:** High-resolution episcopic microscopy; Chick embryo; 3D plant tissue imaging**.**

# INVITED SPEAKERS

## Id-318

### Highly Mismatched CdMnTe/Si Heterostructures: Characterization by AFM, HR-TEM and HR-XRD

S. OLAVO FERREIRA
Federal University of Viçosa, Physics Dep. - Av.P. H. Rolfs, sn, 36570-900 - Viçosa/Brazil
Corresponding author: sukarno@ufv.br

**Abstract:** Cadmium Telluride and Cadmium Manganese Telluride have been studied for a long time due to their optoelectronic properties and potential application in solar cells, x-ray detectors and other devices. This work describes the growth of quantum dots and thin films on Silicon (111) substrates using molecular beam epitaxy with CdTe and Mn solid sources. The obtained samples were characterized by high resolution x-ray diffraction (HR-XRD), high resolution transmission microscopy (HR-TEM) and atomic force microscopy (AFM). Our results show that this system obeys the Volmer-Weber growth mode, nucleating tri-dimensional islands without formation of a wetting layer. Despite a lattice mismatch of almost 19%, almost perfect, completely relaxed quantum dots are obtained, and the mismatch is probably accommodated by a Tellurium interfacial layer. However, the dots have a high in-plane mosaicity, with 30 degrees' preferential orientation. Up to 50% Mn can be incorporated in the epitaxial layer. This causes the appearance of a dense defect network near substrate interface during coalescence and the resultant thin films show a roughness which increases as the growth proceeds. Besides that, thick films present good optical properties.

**Keywords:** CdTe; CdMnTe; Epitaxy; MBE; Characterization.

# INVITED SPEAKERS

## Id-321

### Application of Fourier Transform Infrared Spectroscopy in Reaction Engineering and Heterogeneous Catalysis

S. UPADHYAYULA
Department of Chemical Engineering, Indian Institute of Technology Delhi, Hauz Khas, New Delhi-110016, India
Corresponding author: sreedevi@chemical.iitd.ac.in

**Abstract:** Fourier transform infrared spectroscopy (FTIR) can be effectively used to gain insights into both catalytic and non-catalytic reactions that are of engineering importance to the Chemical industry. Herein, two examples one each of from a catalytic reaction and a non-catalytic reaction have been chosen to explain the importance of FTIR application. Among the catalytical reactions, the well-known Fischer-Tropsch synthesis converting syngas (CO/$H_2$) to fuel range hydrocarbons over a bimetallic catalyst is considered while the oxidative stabilization of mesophase pitch in a carbon composites is considered among the non-catalytic reactions. The effect of noble metal promotion over the Fischer-Tropsch (FT) activity of silica supported Fe-Co bimetallic catalyst was investigated via detailed experimental study. *In-situ* Diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) was used to investigate the adsorption properties of CO on Rh and Ru promoted catalyst with CO and $H_2$ as probe molecules. Photo-acoustic sampling technique in FTIR was used to analyse the oxidative stabilization of mesophase pitch matrix in carbon-carbon composites, with respect to oxygen permeability and crosslinking.

**Keywords:** Fourier transform infrared spectroscopy; Reaction Engineering; Heterogeneous; Catalysis; DRIFTS.

# INVITED SPEAKERS

## Id-331

### Gate-Controlled Chemical Reactions at Graphene Surface

R. NOUCHI
Department of Physics and Electronics, Osaka Prefecture University, Sakai 599-8570, Japan
PRESTO, Japan Science and Technology Agency, Kawaguchi 332-0012, Japan
Corresponding author: r-nouchi@pe.osakafu-u.ac.jp

**Abstract:** Two-dimensional (2D) materials that can be obtained by exfoliation of layered crystals are very sensitive to surface phenomena owing to their ultimate thinness. Their ultrathin body enables us to control the whole body by means of a field-effect-transistor (FET) configuration because the gate electric field is not completely screened. Thus, it is expected that surface phenomena are controllable by means of FETs with a channel of 2D materials. I will talk on such gate-controlled surface phenomena, especially on gate-controlled chemical reactions at 2D materials surfaces.

FET-related parameters such as the drain voltage and the gate voltage are shown to control surface chemical reactions. The drain voltage should raise the temperature of the 2D channel through Joule heat generation. The gate voltage can tune the charge carrier density/type in the 2D channel, and control surface adsorption phenomena by the gate electric field. If reactants come from the surrounding environment (e.g., oxygen molecules in oxidation reactions), the adsorption of the reactants onto 2D channels can be a rate-limiting process. In this case, we can control the whole reaction by controlling the adsorption process [1].

In this talk, such controllability of FET-related parameters will be discussed based on various surface reactions at surfaces of graphene, an archetypal 2D material [1-3]..

**Keywords:** Graphene; Raman scattering spectroscopy; Gate-controlled chemical modification; Photo-oxidation; Graphene nanoribbon; Electron-transfer chemistry.

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[3]. R. Nouchi and K. Ikeda, submitted.

# INVITED SPEAKERS

## Id-332

### Laser Plasma Source for Soft X-Ray Microscope

S. AMANO
3-1-2 Koto, Kamigori, Ako, Hyogo 678-1205 JAPAN
University of Hyogo
Corresponding author: sho@lasti.u-hyogo.ac.jp

**Abstract:** Soft X-rays (SXRs) in the "water window" region between 2.3 nm and 4.4 nm are used in high-contrast SXR microscope imaging. Many studies have been performed with the aim of producing water window SXRs using laser plasma X-ray (LPX) sources, which are both compact and inexpensive when compared with conventional synchrotron radiation (SR) SXR sources. Various materials, including gold, ytterbium, copper, Bismuth, carbon, mylar, ethanol, nitrogen and argon, have been studied as potential targets for these LPX sources. We have studied an LPX source in the water window that uses a cryogenic Ar plasma target; this source can generate continuously repeated pulses on a long-term basis with high efficiency, i.e., it maintains high average pulse power. Rare gas such as Ar is considered to be ideal deposition-free target because it is inert gas and, unlike metal targets, is thus chemically inactive and will not be deposited on the optics located near the plasma. This reduction in plasma debris is a major advantage for continuous source operation. We also decided to use a cryogenic Ar solid target because it provides both higher conversion efficiency and higher brightness as a result of the target's high solid density. In addition, a smaller gas load for evacuation by the exhaust pump system was also expected in the solid state when compared with gas and liquid jets. We therefore originally developed a cryogenic target translation system to supply the solid Ar target continuously. Using this system, we achieved high conversion efficiency (CE) of 14% in the water window. After we surveyed the operational parameters in the target system, we have successfully achieved average powers of up to 0.5 W at 3.3 Hz. The characteristics of the Ar ion debris from the plasma were also studied and it was found that the kinetic energies of the main ions were of the order of a few keV. Using the measured ion spectrum, the sputtering damage caused by Ar ions on the silicon nitride membrane was studied and the result indicated that the damage should cause no problems in the application of contact SXR microscope. Our laser plasma X-ray source will provide a compact and convenient source that is suitable for soft X-ray microscopy in place of conventional synchrotron facilities. In this paper, we report on the performance of our water window SXR source when using the cryogenic Ar target system for SXR microscopy.

**Keywords:** Soft X-rays, water window, LPX sources, laser plasma X-ray source, Ar ions on

# INVITED SPEAKERS

## Id-333

### Determination of Band Diagram for GaN Power Devices Using Hard X-ray Photoelectron Spectroscopy

K. KATAOKA*, T. NARITA, D. KIKUTA, KENJI ITO, S. TAJIMA, M. UMEHARA, N. TAKAHASHI, N. ISOMURA
Toyota Central R&D Labs., Inc., Nagakute, Aichi 480-1192, Japan
Corresponding author: e1354@mosk.tytlabs.co.jp

**Abstract:** A gallium nitride (GaN) has high potential as a semiconductor material for power devices due to its large bandgap energy, the high breakdown voltage, and the high electron mobility. GaN-based vertical metal-oxide-semiconductor field-effect-transistors (MOSFETs) are next-generation key components for efficient energy use, and therefore can be applied in an in-vehicle inverter. In order to achieve the high-performance devices, it is required to establish several process technologies without spoiling the advantage of GaN.

For dry etching process, the etching-induced defects near the surface region can degrade the electric properties. Thus, low damage etching techniques and/or defects recovery techniques are crucial although the observable defects are generally limited. Also, the MOS stack process must be controlled to get the optimal threshold voltage, the stability of bias stress and the long oxide lifetime, simultaneously. However, the gate oxide formation process with superior MOS properties has not been established yet.

To investigate these problems related to the device processes, the energy spectrum analysis using x-ray as an excitation source is effective in some cases, because it gives direct signals of the electronic structures, such as elements and energy band diagrams. It can provide useful guideline to improve device processes, and can contribute to reduction of the development period.

Hard x-ray photoelectron spectroscopy (HAXPES) can measure the device structures close to the actual products due to larger analysis depth than the conventional x-ray photoelectron spectroscopy (XPS). The HAXPES analysis is not only utilized to measure the elemental composition or the chemical binding states, but also allows the determination of the energy band diagram directly connected to the device properties.

We introduce several results regarding the energy band analysis by HAXPES. The changes of band bending near the surface of p-type GaN are successfully determined before and after the inductively-coupled plasma (ICP) dry etching [1]. The sample after the ICP etching showed that the depth of downward band bending to the surface increased due to the etching-induced donor-type defects, which indicates that suppression of the donor-type defects is significant for the dry etching process for GaN. In the gate oxide formation process, the band offsets between $Al_xSi_{1-x}O$ film formed by atomic layer deposition (ALD) and GaN have been quantitatively analyzed [2]. We demonstrate that the conduction band offset can be continuously changed by adjusting the Al/Si ratio in the $Al_xSi_{1-x}O$ film, allowing optimization of the MOS properties.

**Keywords:** HAXPES; Band diagram; GaN; Power device.

**Acknowledgement:** The synchrotron radiation experiments were performed at the BL46XU and the BL47XU of SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2009B2008, 2010A1805, 2010A1713, 2010B1781, 2014A1312 and 2015B1344).

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# INVITED SPEAKERS

## Id-338

### Optical and Mössbauer spectroscopy of Ion-Doped Metals and Alloys

V. V. OVCHINNIKOV
Institute of Electrophysics, Ural Branch of Russian Academy of Sciences, Russia
Corresponding author: viae05@rambler.ru

**Abstract:** In the studies of the author and his colleagues, nanoscale dynamic effects in condensed media were detected in the course of their corpuscular irradiation [1]. Some key results concerning nanoscale and atomic scale processes were obtained by optical (in situ) and Mössbauer spectroscopy. Methods of electron, field ion, and atomic force microscopy were additionally used. The recorded effects are associated with processes of explosive energy release in areas of dense cascades of atomic displacements, thermalized for ~ 10^–12 s (thermal spikes), heated to 103–104 K. Thermal pressures reach 5-40 GPa. These effects have not been considered by classical radiation physics.

The presentation takes a brief look at the phenomenological model that takes into account the emission of powerful elastic and shock solitary waves by heated cascade regions, which initiate self-propagating structural-and-phase transformations in metastable media at unlimited distances (in practice, at least at a depth of a few millimeters with surface ion irradiation [1]; the run of ions Rp <1 μm).

The applied part of the report contains a review of more than a dozen articles by the author and his colleagues (see, in particular, [2-6]). In these works, the temperatures were measured and the pressures in nanoscale thermal spikes with a lifetime of only 10^–12 s were estimated (based on the analysis of the spectral composition of targets glow). It was studied the effect of ion beams on the phase composition, atomic distribution, the grain and magnetic domain structure, as well as on the magnetic properties of soft magnetic materials such as transformer steel (bands 0.1-0.35 mm thick), finemets (ribbons 25 microns thick), permalloy and carbonyl iron powders. Along with technical alloys, a number of model iron alloys containing Si, Mn, Cr, Ni, Pd, Au, subjected to irradiation with ions of Ar+, Xe+, Cr+, was also investigated. The use of the local-nuclear-nature method (57Fe Mössbauer spectroscopy), optical spectroscopy and atomic-scale microscopic methods served as a basis of this message. The work also used the methods of X-ray diffraction, resistometry, magnetic measurements, and mechanical tests. A series of new, not yet published results are included in the presentation.

A gigantic decrease in the temperature of processes during ion irradiation as compared with thermally activated processes (by 100–250 K), a multiple (10^2-10^3 times) increase in their speed, as well as a multiple increase in the exposure depth, noted above, was found.

This work was performed as part of State Task.

**Keywords:** Ion irradiation; Condensed media; Nanoscale dynamic effects; Optical spectroscopy; Mössbauer spectroscopy.

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# INVITED SPEAKERS

## Id-341

### Drug Discovery for Brain Cancer: Imaging Approaches as Key Tools

C. RAPÔSO[1,2*], A. P. BONFANTI[1,2], M. CABALLERO[1,2], J. MUNHOZ[1,2], T. ROCHA - E - SILVA[3], R. SUTTI[4], F. DO N. MOURA[5], S. Q. BRUNETTO[6], C. D. RAMOS[7], R. THOMÉ[8], L. VERINAUD[2], N. BARRETO[1,2]
[1]Faculdade de Ciências Farmacêuticas, Universidade Estadual de Campinas (UNICAMP), Campinas, SP, Brazil,
[2]Departamento de Biologia Estrutural e Funcional, Instituto de Biologia, UNICAMP,
[3]Faculdade Israelita de Ciências da Saúde Albert Einstein, São Paulo, SP, Brazil,
[4]Faculdade de Ciências Médicas, Santa Casa de São Paulo, São Paulo, SP, Brazil,
[5]Faculdade de Ciências Médicas, UNICAMP; [6]Centro de Engenharia Biomédica, UNICAMP,
[7]Serviço de Medicina Nuclear, Hospital das Clínicas, UNICAMP,
[8]Department of Neurology, Thomas Jefferson University, Philadelphia, PA, USA
Corresponding author: raposo@g.unicamp.br

**Abstract:** The behavior and characteristics of cells (such as migration, adhesion, proliferation and death) are important hallmarks, which are taken into account in the discovery of new anticancer drugs. The most common types of primary brain tumors (40-60%) are represented by transformed glial cells (mainly astrocytes) and are classified as gliomas. The median survival time is 14-15 months for patients with grade IV glioma (glioblastoma multiforme). Considering the limitations of current treatment modalities, attempts are being made to develop new chemotherapeutic agents. Animal venoms are a mixture of biologically active molecules, which represent promising guiding compounds for drugs development. The venom of the South American spider *Phoneutria nigriventer* (PnV) (Ctenidae, Araneomorphae) has been shown to induce morphological changes in astrocytes, suggesting that PnV molecules could target glioma cells. The purpose of this study was therefore to screen the *in vitro* antitumor effects of PnV on cell adhesion, migration/invasion and morphology, and to select the PnV-isolated molecule with the highest effects, also contributing to clarify its mechanisms. In addition, PnV action was evaluated in a xerographic tumor model. Cultured human glioblastoma (NG97) cells were treated with PnV (14 µg/ml) or twelve PnV-isolated (HPLC) toxins (called F1-F12; 1 µg/ml) for 12, 24, 48 and/or 72 h (control was in IMDM). Some cells were pretreated with Y-27632 (10 µM), an inhibitor of RhoA-ROCK signaling pathway, known to be involved in migration changes during glioma development. Cell adhesion was assessed using a commercial colorimetric kit (fibronectin-coated wells); migration/invasion were assessed by scratch-wound healing and transwell assays; cellular morphology was demonstrated by GFAP immunofluorescence and phalloidin labeling. For *in vivo* analyses, NG97 cells were inoculated (s.c.) on the back of RAG$^{-/-}$ mice, female, 6-8-weeks-old (n = 5/group). After 7 days, controls received sterile saline (vehicle; 100 µl, i.p.) while PnV group received venom (100 µg/Kg, i.p.), every 48 h, for 14 days. MicroPET-CT images were acquired and, after euthanasia, tumors were dissected for histopathology. *In vitro* results showed that PnV significantly decreased cell adhesion compared to untreated cells. Control cells filled the scratch after 48 h, while PnV-treated cells were not able to fill it; significantly fewer PnV-cells migrated through the transwell, compared to control. In addition, PnV treated cells exhibited morphological alteration, with shorter processes. Toxins F1

and F11 have the highest effects considering all parameters. Pretreatment with Y-27632 abolished or modified the effects of PnV and both toxins, suggesting that the mechanism involves RhoA-ROCK signaling. PET-CT showed a 18F-FDG-positive tumor mass in all control animals; PnV-treated mice, however, did not present tumor, or had only a 18F-FDG-negative small tumor mass. Tumor weight and volume of the PnV-treated animals were significantly lower, compared to control. Histopathological analysis revealed that PnV-tumors exhibited extensive necrosis, explaining the absence of 18F-FDG in such tumors. Therefore, PnV components are potential candidates for glioma treatment. Experiments to elucidate the chemical nature of F1 and F11 toxins and to determine preclinical parameters, such as their *in vivo* effects on cancer development, pharmacokinetics and side effects will be the next steps.

**Keywords:** Glioblastoma; Spider venom; Migration; Metastasis; Microscopy; PET-CT.

**Acknowledgement:** FAPESP #2015/04194-0, #2016/15827-6; CNPq #431465/2016-9.

# INVITED SPEAKERS

## Id-344

### X-ray Absorption Spectroscopy and Resonant X-ray Inelastic Scattering in $FeSe_{1-x}Te_x$

J. M. DE LEÓN[1], D. M. GÓMEZ[2]

[1]Departamento de Física Aplicada, Cinvestav-Mérida, Mérida, Yucatán, México and Cinvestav, Mexico City, México

[2]Instituto Tecnológico de Estudios Superiores de Occidente, Guadalajara, Jalisco, México

Corresponding Author: jmustre@cinvestav.mx

**Abstract:** The superconducting transition temperature. Tc, in the FeSexTe1-x under Te substitution presents a maximum for intermediate Te content. This observation suggests phase separation between FeSe and FeTe. We present X-ray absorption near edge spectroscopy (XANES) in this system. The spectra can be modeled assuming an inhomogeneous electronic structure. As a complementary technique Resonant Inelastic X-ray Scattering (RIXS) yields additional information about the electronic structure as it probes both unoccupied states above the Fermi level, and occupied states below. We present calculations of RIXS spectra in FeSexTe1-x for x= 1,0.75, 0.50, 0, based on a one-electron approximation that expresses the cross section as a result of the convolution of the x-ray absorption spectra and x-ray emission spectra. These calculations show the same trends observed in experimental RIXS spectra.

**Keywords**: Fe-based calchogenide superconductors; X-Ray inelastic scattering; X-Ray absorption

# INVITED SPEAKERS

## Id-346

# Microscopic Characterization of 3D-Printed Metals with Data-Constrained Modelling and Quantitative X-ray CT

S. YANG[1], T. HUGHES[2], J. LI[3], H. WANG[3], A. B. MURPHY[4], L. PRENTICE[1]

[1]*CSIRO Manufacturing, Clayton, 3168, Australia;*

[2]*CSIRO Mineral Resources, Clayton, 3168, Australia;*

[3]*Shanxi University, Taiyuan, Shanxi, China ;*

[4]*CSIRO Manufacturing, Lindfield, 2070, Australia;*

Corresponding author: Sam.Yang@csiro.au

**Abstract:** The properties of 3D-printed metals are related to their internal microstructures. X-ray CT has been widely used for non-destructive 3D microstructure characterization. However, the mainstream image analysis techniques have limitations in resolving microscopic spatial features and material phases that are smaller than $10^{-3}$ times the sample size. By integrating statistical physics and quantitative X-ray CT imaging, the data-constrained modelling (DCM) approach has been able to overcome these limitations. It opens the possibility of non-destructive quality evaluation of parts produced by metal additive manufacturing (AM), for which the lack of an efficient and convenient technology for non-destructive quality evaluation is one of the major barriers to broader industrial adoption. The DCM can analyse data from synchrotron-based quantitative multi-energy X-ray CT imaging, or lab-based quasi-quantitative X-ray CT imaging. In additional to 3D-printed metals, DCM has also found applications in several other disciplines including metal corrosion protection, metal extraction from minerals, and microstructure characterization for unconventional oil and gas reservoir rocks, coal and soil clay.

**Keywords:** Data-constrained modelling; 3D-print; Metal; Microstructure.

# INVITED SPEAKERS

## Id-350

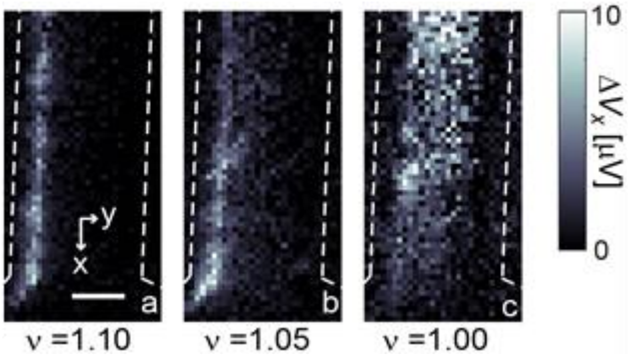
### Nuclear Resonance Spectroscopic Imaging of Hyperfine-Coupled Quantum Hall System

K. HASHIMOTO
Department of Physics, Tohoku University,Aoba-ku, Sendai 980-8579, Japan
Centre for Spintronics Research Network, Tohoku University, Aoba-ku, Sendai 980-8578, Japan
Corresponding author: hashi@tohoku.ac.jp

**Abstract:** Nuclear resonance (NR) spectroscopy is one of the powerful tools to probe both electron and nuclear spin polarization in conduction electron systems. In a low dimensional semiconcuctor systems in a high magnetic field such as quantum Hall (QH) systems, some spin charactaristics have been studied by, e.g., resistively-detected or optical Knight-shift measurements, implying an essential role of local variation of spin polarization. To directly access the local spin characteristics, we developed the scanning-probe NR technique and performed NR spectroscopic mappinp.

The scanning metallic tip was used to apply an RF electric field to nuclear spins in a 20-nm-wide GaAs quantum well located ~ 200 nm below the surface. The electric field at a NR frequency couples with the quadrupolar nuclear spins such as 75As nuclear spins with total spin I = 3/2. The resulting local NR was resistively detected by measuring the longitudinal voltage at constant source-drain current. All measurements were conducted with pumping dynamic nuclear polarization (DNP) in the nonequilibrium ν = 1 QH breakdown regime [2] at a sample temperature below 300 mK at magnetic fields of 7-8 T.

From the spectroscopic data, we constructed NR-intensity (Fig. 1) and Knight-sift (Fig. 2) images, which, respectively, revealed the spatial distribution of the DNP and electron spin polarization. This powerful technique can be utilised for direct microscopic examination of various hyperfine-coupled quantum systems, such as the QH Skyrmions, helical nuclear magnetism, and spin-helical topological surface states.



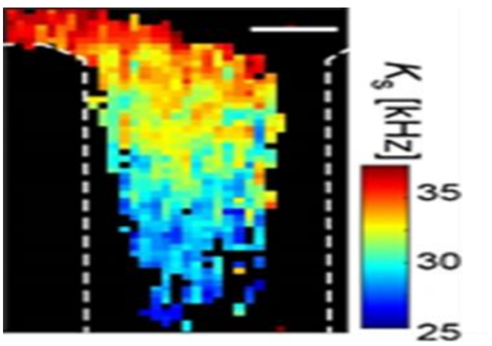


Fig 1. Fig.1 NR intensity images taken at different ν. Fig 2. NR Knight-shift image showing the spatial variation of the electron spin polarization.

**Keywords:** Scanning probe microscopy; Nuclear resonance; Knight shift; Quantum Hall effect.

# INVITED SPEAKERS

## Id-354

### A Micro-Combinatorial Approach for a Reveal of Composition Dependent Properties of Binary Films by TEM and Further Analytical Techniques

G. SÁFRÁN[1], T. LOHNER[1], M. SERÉNYI[1], P. PETRIK[1], B. KALAS[1], Z. ZOLNAI[1], M. FRIED[1],
J. GUBICZA[2], N. Q. CHINH[2]
[1]Institute for Technical Physiscs, Centre for Energy Research, HAS, H-1121 Konkoly Thege út 29-33,
Budapest. Hungary
[2]Roland Eötvös University, H-1117, Pázmány Péter sétány 1a, Budapest, Hungary
Corresponding author: safran@mfa.kfki.hu

**Abstract:** Combinatorial methods are widespread in chemistry and biology and have increasing importance in materials science. The traditional “multiple-sample concept” combinatorial technique produces numerous different samples, in one experiment, very effectively. Handling and measuring plenty of samples, however, is definitely not effective. We worked out "one-sample concept" micro-combinatory that provides extreme throughput in both preparation and measurement of composition dependent properties of binary films by TEM and further analytical techniques. A single micro-combinatorial sample includes the entire binary system of components $A_xB_{1-x}$ (where x=0...1) with a design that suits the measurement. For TEM, the sample is a 1x1.5 mm$^2$ layer stripe deposited on a microgrid with a concentration gradient typically 0.1%/µm. However, for RBS, XRD, ellipsometry and nanoindentation, an appropriate sample is of 12x25mm$^2$ size with a gradient of 0.5 %/µm. The method enables to collect a whole data library of the properties of a binary system within a single specimen and one measuring session.

For a demonstration of the method and due to technological importance SiGe and AlMg were studied by the above techniques for to reveal the correlations between composition, structure, optical and mechanical properties. Research of thin SiGe layers is motivated by widespread applications e.g. solar cells, thin film transistors, Schottky diodes, thermal sensors, bolometers. Due to enhanced non-linear properties of Ge to Si, layers of high Ge content are of interest in developing optoelectrics devices for the mid-infrared region. TEM, EDS and RBS showed amorphous structure of SiGe and a linear concentration change along the sample. The revealed linear concentration dependence of refractive index and absorption provides a potential of fine tuning of the optical properties, as well, the extension of the operational range up to 8 µm, the Si absorption wavelength, or to 14 um, the Ge transparency cut-off.

The efficiency of µ-combinatory was demonstrated also in case of AlMg binary layers. This study is motivated by technological applications and understanding of the mechanical properties. AlMg samples were studied by TEM and nanoindentation, especially, in the technologically important range of 0-30w% Mg. Nanohardness measurements were carried out with a Vickers diamond tip. Al layer showed fcc phase with grain sizes 60-120nm that decreased to 60-90nm at 1w% Mg associated with a remarkable hardness increase (~3x) compared to Al. The fcc Al(Mg) solid solution phase remains exclusive up to about 20% Mg, meanwhile the grain size decreases continuously and hardness increases. At 25% Mg and above, additional fine grained

$Al_3Mg_2$ phase of 10-20 nm grains appears, the hardness saturates and slightly decreases. 100% Mg layer shows cph Mg phase of 30-60nm grains and fine grained (1-2nm) cubic MgO, with hardness much lower than that of the Al-Mg alloys. Al-Mg films have fine grain-structures, showing remarkably higher strength compared to conventional bulk materials. This suggests potential application for coating against mechanical wear of Al or Mg. The new technique facilitates a revealing of phenomena that define growth mode, microstructure, physical- and chemical properties of binary films and anticipates the potential to perform extremely effective automated TEM measurements.

**Keywords:** Binary layers, One sample concept, Micro-combinatory, Gradient film, Library of properties.

# INVITED SPEAKERS

## Id-355

### Understanding the $LiMn_2O4$ Structure by Differential Phase Contrast

S. CALDERON V.[1], P.J. FERREIRA[1,2,3]

[1] INL - International Iberian Nanotechnology Laboratory, Av. Mestre José Veiga s/n, 4715-330 Braga, Portugal

[2] Materials Science and Engineering Program, The University of Texas at Austin, Austin, Texas 78712, USA

[3.] Mechanical Engineering Department and IDMEC, Instituto Superior Técnico, University of Lisbon, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

Corresponding author: sebastian.velasco@inl.int

**Abstract:** Lithium ion batteries are, nowadays, one of the most attractive technologies to store electrochemical energy. Among the large variety of cathode materials used in these Li-ion batteries, $LiMn_2O_4$ is a well-known candidate due to its capacity to exchange Li-ions in a three-dimensional spinel structure. However, this material has shown capacity loss, attributed to the dissolution of Mn to the electrolyte. Amos et. al [1] have demonstrated a restructured surface in this material, where a stable surface layer of $Mn_3O_4$, followed by a subsurface $Li_{1+x}Mn_2O_4$ with retention of bulk $LiMn_2O_4$ is formed.

In this context, we use recent concepts in differential phase contrast (DPC) coupled to scanning transmission electron microscopy (STEM) to reveal new insights about the structure of $LiMn_2O_4$, by measuring the projected electric field and charge distribution in pristine $LiMn2O_4$. The DPC-STEM technique is capable of measuring the projected electric field by determining the in-plane displacement of electrons after crossing the sample, while the projected charge distribution is obtained by calculating the divergence of the projected electric field. This approach allows us to acquire simultaneously the lithium, oxygen and manganese atomic columns, when observed along the <0 1 1> type direction, together with the determination of polarization and charge.

The DPC-STEM images were acquired using a four segmented annular detector, coupled to a double-corrected FEI Titan-Themis microscope operated at 200kV. Images from individual segments, together with (H/L) AADF images were acquired. Our results clearly show local regions depleted in Li and the existence of manganese atoms in tetrahedral sites occupying a typical Li atom position or occupying a free octahedral site in the same column, in agreement with Mn disproportionation reported for such compound. Simulated DPC-STEM images obtained by the multislice calculations were performed to confirm the experimental images. This finding contributes to a better understanding of the mechanisms of lithium and manganese exchange in $LiMn_2O_4$ spinel structures.

**Keywords:** DPC, $LiMn_2O_4$.

**References:**

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# INVITED SPEAKERS

## Id-359

### Phase Transformations in Nanoscale Metal-Semiconductor Couples: In Situ (S)TEM studies

A. KRYSHTAL[1], S. BOGATYRENKO[2], A. MINENKOV[2], P. FERREIRA[3,4,5]

[1]Faculty of Metals Engineering and Industrial Computer Science & International Centre of Electron Microscopy for Material Science, AGH University of Science and Technology, Poland,

[2]Department of Physics and Technology, Karazin National University, Kharkiv, Ukraine

[3]Iberian International Institute of Nanotechnology, Braga, Portugal

[4]Materials Science & Engineering Program, The University of Texas at Austin, Austin, USA

[5]Mechanical Engineering Department and IDMEC, Instituto Superior Técnico, University of Lisbon, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

Corresponding author: kryshtal@agh.edu.pl

**Abstract:** Despite previous studies, the fundamental mechanisms and kinetics of interphase interactions in two-component nanomaterials are still under debate. In this paper, we address this theme in a comprehensive experimental study of phase transformations in nanoscale Ag-Ge and Bi-Ge eutectic systems by advanced *in situ* TEM techniques.

First, the effect of size on the eutectic and crystallization temperatures for Ag/Ge, Ge/Ag/Ge, and Ge/Bi/Ge layered films will be discussed and related to the quest for understanding binary phase diagrams at the nanoscale. Self-supported Ag-Ge and Bi-Ge layered films with equal layer thicknesses and varying from 40 to 2 nm were used as a model, ensuring a true binary system free of substrate-induced stresses. Lowering in the eutectic temperature by several hundred degrees has been measured with the layer thickness reduction in both Ag-Ge and Bi-Ge film systems.

Furthermore, it was shown that the eutectic temperature is not a monotonic function of size in the Bi-Ge system. It decreases gradually as the size of the Ge/Bi/Ge system reduces and reaches its lowest value ≈190°C for the ≈5 nm thick Bi film. For a thinner Bi film, the liquid phase formation temperature quickly rises. The melting process in Ag/Ge, Ge/Ag/Ge films occurs in a range of temperatures. Our results reveal that the role of the Ge film is not limited only by supplying the substance to the eutectic alloy, but its thickness affects the morphology of the Ag layer via Ag-mediated crystallization process of amorphous Ge films.

Second, a new insight into the mechanism of metal-induced crystallization of amorphous germanium films in contact with Ag will be presented and discussed. We investigated the initial stages of the aforementioned reaction by aberration-corrected TEM. The nucleation and growth of crystalline Ge phase was observed at the atomic scale in real time by heating the system in a double corrected FEI Titan Themis operated at 200 keV. *In situ* monochromated electron energy loss spectroscopy STEM-EELS was performed in the 350-450°C temperature range, using a probe corrected FEI Titan operated at 80 keV and probe current of 8 pa. A surface plasmon resonance peak of Ag and a bulk Ge plasmon peak were used for mapping of the interphase boundaries and chemical elements in the same nanoparticle at different stages of the reaction without substantial radiation damage. It was shown that the formation of metastable intermetallic and liquid phases at Ag-Ge interface is the possible mechanism of metal-induced crystallization of the amorphous semiconductor films.

**Keywords:** Eutectic temperature; Size effect; In Situ TEM; Thin films; Phase diagram; Metal-induced crystallization of semiconductors; Nucleation phenomena.

**Acknowledgement:** Financial support from the National Science Centre, Poland under Grant No. 2016/23/B/ST8/00537 is gratefully acknowledged.

# INVITED SPEAKERS

## Id-360

### Characterization of Superhydrophobic Breakdown on Nanostructured Silicon Surfaces

X. XU[1], N. VRANCKEN[1,2]
[1]Imec, Kapeldreef 75, 3001 Leuven, Belgium
[2] Vrije Universiteit Brussel, Pleinlaan 2, 1050 Elsene, Belgium
Corresponding author: xiumei@imec.be

**Abstract:** Superhydrophobic surfaces are highly promising for self-cleaning, anti-fouling and anti-corrosion applications. However, accurate assessment of the lifetime and sustainability of super-hydrophobic materials is hindered by the lack of characterization techniques. We have demonstrated that superhydrophobic breakdown on nanosctructured silicon surfaces can be accurately measured by a novel application of FTIR. [1–3]

For local area characterizations (measurement spot size ~ 50-500 µm), optical reflection spectra are taken with a FTIR spectrometer (Bruker Vertex 80v) equipped with a microscope (Bruker Hyperion). When water starts to infiltrate in between nanostructures, the difference in refractive index can result in a big change in the optical reflectance spectra. There are some resonant modes due to the constructive interference of beams reflected at the top and bottom interfaces of the nanostructures. The resonant wavelengths are sensitive to any change in refractive index, and thus can be used to determine the water imbibition depths. [1,4]

For large area wetting characterizations (measurement area ~ cm$^2$), ATR-FTIR (Nicolet 6700) is used. When air is present in between the nanostructures, the relative intensities of the water bands in the absorption spectrum are changed due to the wavelength-dependent attenuation of the evanescent wave.[2] The high surface sensitivity of ATR-FTIR allows for accurate detection of local liquid infiltration. Using a customized liquid cell, spontaneous breakdown of the superhydrophobic state is triggered by in-situ modulation of the liquid surface tension. Breakdown kinetics is found to slow down dramatically when the liquid surface tension approaches the transition criterion.[3] Precise actuation of the superhydrophobic breakdown process is demonstrated through careful modulation of the liquid surface tension around the transition criterion. The developed ATR-FTIR method can be a promising technique to study wetting transitions and associated dynamics on various types of superhydrophobic surfaces.

**Keywords:** Wetting characterization; Superhydrophobic; FTIR.

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# INVITED SPEAKERS

## Id-371

## X-ray Spectroscopies Techniques Applied in Archaeometry at the Institute of Physics, University of Sao Paulo, Brazil

H. C. SANTOS*, C. L. RODRIGUES, T. F. SILVA, P. H. O. V. CAMPOS, M. A. RIZZUTTO, N. ADDED, M. H. TABACNIKS.

Institute of Physics, University of Sao Paulo. Rua do Matão 1371. CEP 05508-090 São Paulo, SP, Brazil.

Corresponding author: hsantos@if.usp.br

**Abstract:** Since 2003, the Group of Applied Physics with Accelerators (GFAA) of the Institute of Physics of the University of Sao Paulo has been working on the characterization of several types of samples using mainly IBA (Ion Beam Analysis) methods, such as PIXE (Particle Induced X-ray Emission), RBS (Rutherford Backscattering), NRA (Nuclear Reaction Analysis) and IBL (Ion Beam Luminescence). New analytical possibilities and applications were created after the implantation of an external sub-millimeter beam line, which allows simultaneous in-air analysis by all these IBA techniques. More recently in 2013, we developed a high precision and large range (60 cm) 3D motorized stage, totally developed in LAMFI-USP (Laboratory for Ion Beam Analysis of Materials), coupled to the external beam line to obtain elemental maps of large area samples. This is a unique instrument for multi-technique analysis of large area samples and elemental mapping. The group also operates a dedicated laboratory, well equipped with portable instruments, mostly used to study cultural, artistic or archaeological objects using various kinds of non-destructive analytical techniques, working together with many of the university's museums. This presentation will give a brief outlook on our last achievements using IBA techniques, examples of XRF applications, and some instruments developed to be used on the investigations in archaeometry.

**Keywords:** X-ray spectroscopy; Cultural heritage; IBA.

# INVITED SPEAKERS

## Id-375

### Investigation of the Local Chemistry Effects in Magneto-Electric Electrically Poled Multiferroic Thin Films

A. BARBIER[1,*], B. SARPI[2], P. L. NGUYEN[1], M. RIOULT[1,2], T. AGHAVNIAN[1,2], J.-B. MOUSSY[1], D. STANESCU[1], H. MAGNAN[1],
F. MACCHEROZZI[3], R. BELKHOU[2]
[1]Service de Physique de l'Etat Condensé, Gif-sur-Yvette, France
[2]Synchrotron SOLEIL, L'Orme des Merisiers Saint-Aubin, France
[3]Diamond Light Source, Oxforshire, United Kingdom
Corresponding author: antoine.barbier@cea.fr

**Abstract:** Multiferroic materials are of high technological interest because they are expected to become key components of genuine devices in important technological fields like spintronics, sensors, multiple state memory cells, energy harvesting etc. Within this technological context magneto-electric multiferroics having simultaneous ferromagnetic and ferroelectric long range orderings are particularly relevant. Artificial multiferroics obtained by combining ferroelectric and ferromagnetic materials are a promising route to overcome the lack of intrinsic single phase multiferroics. We considered nanometric combinations of epitaxial thin films of BaTiO3, which is the prototypical ferroelectric material, and ferrites over-layers (NiFe2O4, MnFe2O4 and CoFe2O4), which are respectively low, moderately and highly magnetostrictive as well as Fe3O4 and Fe2O3 iron oxides. These combinations are of practical interest since all ordering temperatures are above room temperature and the compounds are fully oxidized and highly stable. All films were single crystalline and were elaborated by atomic oxygen plasma assisted molecular beam epitaxy. The layers were electrically polarized by using local pattern writing with Piezo-Force Microscopy (PFM) and we studied the ferroelectric, chemical and magnetic responses of the individual layers by spectromicroscopy. For all systems electrical local polarization revealed a spectroscopic response. Our observations show that the chemical and ferroelectric contributions are highly entangled in these systems.

**Keywords:** Spectromicroscopy; X-PEEM; Absorption; Ferroelectric; PFM; Oxide.

# INVITED SPEAKERS

## Id-377

# Combined Diffraction and Spectroscopic Study of Temperature Dependent Magnetic Behaviour of Co(II)-Containing Layered Double Hydroxides

A. N. SALAK[1,*], D. E. L. VIEIRA[1], R. YU. BABKIN[2], YU. G. PASHKEVICH[2], I. M. LUKIENKO[3], A. V. FEDORCHENKO[3], V. A. DESNENKO[3], E. L. FERTMAN[3], J. L. RIBEIRO[4], L. G. VIEIRA[4]

[1]Department of Materials and Ceramics Engineering, CICECO – Aveiro Institute of Materials, University of Aveiro, Aveiro 3810-193, Portugal

[2]O. Galkin Donetsk Institute for Physics and Engineering of NASU, 46 Nauky av., Kyiv 03680, Ukraine

[3]B. Verkin Institute for Low Temperature Physics and Engineering of NASU, 47 Nauky av.,
Kharkov 61103, Ukraine

[4]Physics Centre, University of Minho, Braga 4710-057, Portugal

Corresponding author: salak@ua.pt

**Abstract:** Layered double hydroxides (LDHs) find a wide application mainly owing to their unique anion-exchange properties. LDHs are composed of the alternating positively-charged mixed metal $M^{II}$-$M^{III}$ hydroxide layers and interlayers occupied by anions and crystal water molecules. The metal cations in the layers are coordinated by six oxygen atoms forming 2-D structures of the edge-linked oxygen octahedra. Anions of different nature (either inorganic or organic), size, configuration and charge can be intercalated into the interlayer. As a result, the characteristic layer-interlayer scale in LDHs can be from about 0.7 nm to several nanometers. In terms of the anion exchange properties that determined by the layer charge, the $M^{II}/M^{III}$ cations ratio rather than the nature of the cations is important. At the same time, a use of cations of the particular nature can extent the conventional functionalities of LDHs and even induce new properties. LDHs with the hydroxide layers, where at least one of $M^{II}$ and $M^{III}$ is magnetic, are of fundamental interest as promising models to study magnetic interactions in 2-D systems. The magnetic characteristics of such LDHs are expected to be reversibly tuned by variation of the interlayer distance through anion exchanges and control of the crystal water content. The objectives of this work were to study temperature-induced structural changes in hydroxide layers of $Co^{II}$-containing LDHs and to correlate them with the unusual magnetic behaviour, particularly with an anomalous temperature dependence of their effective magnetic moment and the paramagnetic Curie temperature. Layered double hydroxides with the $Co^{II}/Al^{III}$ cation ratio = 2, 3, and 4 intercalated with different anions were prepared and investigated. Due to the significant size difference between cations $Co^{II}$ and $Al^{III}$, the non-equivalent locations of $Al^{III}$ in these LDHs depending on the cations ratio result in the appearance of a large number of additional distortions that lower the initial symmetry of the octahedra. In this work, by means of combination of *in situ* X-ray diffraction with optical, Raman and infrared spectroscopy, the actual distortion configurations were identified and used for the calculations of the temperature dependent magnetic susceptibility. It was shown that the presence of a shoulder-like anomaly on the magnetic susceptibility curve of the studied LDHs compounds in the middle-low temperature range is due to freezing of the lattice parameters and, as a consequence, freezing of the tilting fluctuations of the O–H bonds. The financial support of P2020 COMPETE and FCT-Portugal through project POCI-01-0145-FEDER-016686 - PTDC/CTM-NAN/2418/2014 (NANOCONCOR) is gratefully acknowledged.

**Keywords:** Octahedral distortion, O-H bond fluctuations, Effective magnetic moment.

# INVITED SPEAKERS

## Id-379

### Microscopy and Spectroscopy of Nanobioconjugates of Carbon Nanotubes

K. UMEMURA

Tokyo University of Science, Japan

Corresponding author: umemura@rs.tus.ac.jp

**Abstract:** Nanobioconjugates of carbon nanotubes (CNTs) are useful for various biological applications. To establish the nanobioconjugate applications, fundamental knowledge of the conjugates is necessary as well as practical researches. We have investigated structures and physicochemical properties of the bioconjugates of CNTs by microscopy and spectroscopy. From atomic force microscopy (AFM) in liquid, flexibility of biomolecules on CNT surfaces were well evaluated. Near infrared absorbance and photoluminescence spectroscopy revealed that wrapping CNT surfaces with biomolecules drastically change the spectroscopy profiles. For example, differences of DNA bases could be recognized by the difference of the near-infrared spectra. Antioxidant effects of catechin involved in Japanese tea also could be detected.

**Keywords:** Near-infrared spectroscopy; Scanning probe microscopy.

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# INVITED SPEAKERS

## Id-385

### Comprehensive Analysis of the Ferromagnetic Semiconductors in Terms of the Crystalline Structure and Magnetic Quality on the Example of (Ga,Mn)(Bi,As) Thin Epitaxial Layers

K. LEVCHENKO[1], T. PROKSCHA[2], J.Z. DOMAGAŁA[1], J. SADOWSKI[1,3], P. DŁUŻEWSKI[1], A. KALETA[1], R. JAKIEŁA[1], M. TRZYNA[4], T. ANDREARCZYK[1], T. WOSINSKI[1]

1 Institute of Physics, Polish Academy of Sciences, PL-02668 Warsaw, Poland
2 Paul Scherrer Institute, Laboratory for Muon Spin Spectroscopy, CH-5232 Villigen, Switzerland
3 MAX-IV Laboratory, LundUniversity, P.O. Box118, SE-221 00 Lund, Sweden
4 Faculty of Mathematics and Natural Sciences, University of Rzeszów, PL-35310 Rzeszów, Poland
Corresponding author: levchenko@ifpan.edu.pl

**Abstract:** Evaluation of the structure quality is a vital first step in any research of a new material, bringing it closer toward implementation and application. Even while using high-quality growth technique, like low-temperature molecular-beam epitaxy (LT-MBE), a further comprehensive analysis of the structure homogeneity and composition is important.

During our presentation we'd like to show versatile complementary spectroscopy and microscopy techniques that were used to describe structure parameters of (Ga,Mn)(Bi,As) (6% Mn, 1% Bi) dilute magnetic semiconductor (DMS) 50-nm-thick layers [1]. High-resolution X-ray diffractometry provided detailed insight into the structure quality and crystal lattice parameters along with the strain in the top investigated layer. Secondary ion mass spectroscopy (SIMS) determined elements distribution throughout the epitaxial layers, while high-resolution transmission electron microscopy (HR-TEM) visualized fine structure of the lattice.

Superconducting quantum interference device (SQUID) magnetometry was used as a primary tool for phase transition and magnetic anisotropy investigations, together with a unique muon spin relaxation (μSR) spectroscopy [2]. Special attention will be paid to the low-energy fully spin-polarized muons analysis, conducted in Paul Scherrer Institute, as a powerful technique for the evaluation of the spatially-resolved (with nm resolution) magnetization homogeneity of the DMS layers.

**Keywords:** Ferromagnets, Dilute magnetic semiconductors (DMS), Thin films, low-temperature molecular-beam epitaxy (LT-MBE), Secondary ion mass spectroscopy (SIMS), High-resolution transmission electron microscopy (HR-TEM), Superconducting quantum interference device (SQUID) magnetometry, Muon spin relaxation (μSR) spectroscopy.

**Acknowledgement:** This work was supported in part by National Science Centre (Poland) through grants UMO-2016/23/N/ST3/03501 and DEC-2012/07/B/ST5/02080.

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# INVITED SPEAKERS

## Id-387

# Impedance Spectroscopy of Advanced Materials: Study of Ionic Conduction and Degradation Processes

P. KNAUTH[1], M. L. DI VONA [2]
[1] Aix Marseille University, CNRS (UMR 7246), campus Etoile-St Jérôme, 13013 Marseille, France
[2] University of Rome Tor Vergata, Dept. Industrial Engineering, Via del Politecnico 1, 00133 Rome, Italy
Corresponding author: philippe.knauth@univ-amu.fr

**Abstract:** Impedance spectroscopy is an advanced technique for the study of various electrical properties of materials and devices, ranging from high frequency investigations of dielectric materials to the low frequency analysis of slow electrochemical processes at working electrodes.

In this invited talk, we will present applications of impedance spectroscopy especially concerning the analysis of ion conducting solids and their mechanistic details and the degradation of materials, including durability of ion exchange membranes and corrosion processes of materials for microelectronic devices.

The talk will address the following topics:

- *Ion conduction and diffusion in anion-conducting materials: analysis by Nernst-Einstein equation.* This study combines impedance spectroscopy for the measurement of the ionic conductivity of fluoride ion conducting polymers and $^{19}F$ NMR spectroscopy for the measurement of fluoride-ion diffusion coefficients [1].
- *Degradation analysis of anion exchange membranes in alkaline medium.* The alkaline attack of ammonium functional groups is a major degradation mechanism of anion exchange membranes in operating anion exchange membrane fuel cells. We use impedance spectroscopy to study the kinetics of the degradation reactions [2].
- *Corrosion study of Cu-Ni-Au multilayers for microelectronics.* Microelectronic chip cards use Cu-Ni-Au multilayers for the electrical contacts. The corrosion in air limits the lifetime and reliability of the contact layers. In this study, impedance spectroscopy is used to determine the interface resistance, which is inversely proportional to the corrosion current, and approaches of corrosion protection by electropolymerized protection layers [3].

**Keywords:** Ion exchange membranes; Alkaline stability; Microelectronic contacts; Corrosion.

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# INVITED SPEAKERS

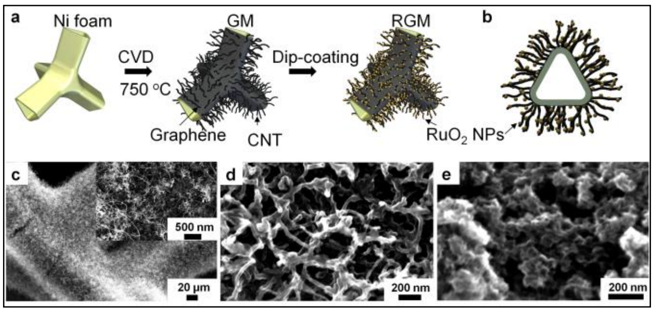
## Id-388

### Design of Materials for Advanced Energy Storage

C. S. OZKAN
Professor of Mechanical Engineering and Materials Science and Engineering
John J. Guarrera Engineering Educator of the Year
SRC STARNet Center for Spintronic Materials and Novel Architectures
University of California Riverside, CA, 92521 USA
Corresponding author: cozkan@engr.ucr.edu

**Abstract:** The global electrochemical energy storage market ranging from electric vehicles and personal electronics to physical grid storage and defense applications demands the development of new classes of materials for fabricating high performance batteries and supercapacitors. I will describe innovative approaches for the design and synthesis of nanostructured materials towards enhanced reversible capacity; superior rate performance and cycling stability; superior gravimetric capacitance; and enhanced energy density and power density. Hierarchical three dimensional (3D) graphene-carbon nanotube hybrid materials called pillared graphene nanostructures (PGN) grown by chemical vapor deposition possess ultra large surface area, tunability, mechanical durability and high conductivity which are appealing to diverse energy storage systems. Integration of nanostructured pseudocapacitive metal oxides to such 3D hierarchical templates provides superior electrochemical performance. Among the high performance capacitor systems developed includes MGM (graphene-MWNT-Manganese oxide) and RGM (graphene-MWNT-Ruthenium oxide) hybrid systems. High specific/areal capacitance and extended operational voltage window provides an exceptionally high energy density and power density. Similar three-dimensional templates are transformed into cone-shaped carbon nanotube clusters decorated with amorphous silicon for lithium ion battery anodes (SCCC), by depositing amorphous silicon onto the mesoporous nano-carbon templates via magnetron sputtering. The seamless connection between silicon decorated CNT cones and the graphene substrate facilitates charge transfer and provides a binder-free technique for preparing lithium ion battery anodes. Lithium ion batteries based on the SCCC architecture demonstrated ultra-fast charging, high reversible capacity and excellent cycling stability. Mildly reduced graphene oxide (mrGO) and silica coated Sulfur particles (SCSP) have been developed as new generation cathode materials, forming the basis for Li–S batteries. During cycling, SCSPs fracture and release active material, and mrGO helps to contain the ruptured particles, thereby reducing the polysulfide shuttling effects and improving the cycling stability.

**Keywords:** Hierarchical three dimensional (3D) graphene-carbon nanotube hybrid materials, MGM (graphene-MWNT-Manganese oxide), silica coated Sulfur particles, Li–S batteries



**Graphene – CNT – Ruthenium Oxide Hybrid Architecture for Supercapacitors**
(a-b) Schematic illustration of the preparation process for RGM architectures. SEM images of (c) as-grown GM foam (d) lightly loaded RGM, and (e) heavily loaded RGM.

# INVITED SPEAKERS

## Id-390

### To Catch a Moment in the Nematode Life

V. ORO

Nematology Laboratory, Institute of Plant Protection and Environment, Teodora Drajzera 9, 11000 Belgrade, Serbia, +381112660049

Corresponding author: viooro@yahoo.com

**Abstract:** "Life is a series of moments…" it is said, and if we do not capture such moments it is as they do not exist. Biology is the science of life and living organisms. Without a microscope and a camera, we would not be able to see the micro world of living organisms. Nematodes are generally small invertebrates, important in the food chain, with extremely varying dimensions between 100 micrometers and 10 meters. The vast amount is microscopic and with transparent bodies. They can be mycophagous, bacteriophagous, predators and also insect, animal, human and plant parasites. Morphological dimorphism may occur in different developmental stages as well as sexual dimorphism. The nematodes are mostly wormlike although some species are cysts or pouch-like. They inhabit terrestrial and marine areas and can be found even in the extreme environments like glaciers or hot springs.

This study demonstrates the applicability of bright field microscopy, dark field, phase contrast, Nomarsky contrast and scanning electron microscopy in nematological research particularly in morphological and anatomical studies.

Each technique examines nematodes from different perspectives. The bright field microscopy and low magnification reveal gross morphology or some critical moments in life cycle such as hatching, mating, ecdysis etc. The dark field microscopy emphasizes natural look and colour of specimens so their appearance is closest to the real one. The phase contrast microscopy focuses on anatomical features which can be obscured by three-dimensional techniques. Nomarsky or differential interference contrast visualises three-dimensional appearance of specimens enabling monitoring from different focal planes. In addition, Nomarsky interference contrast generates an image with "shadow" effect particularly useful in the nematode diagnostics. Scanning Electron Microscopy reveals minutious details that are not clearly discerned by any other technique. As some nematodes have diagnostic characters measured in nanometers, nanotechnology significantly improves visualisation of the "nano-morphology".

Each technique provides insight into both macro- and ultrastructural features of these microscopic organisms, enabling the deeper knowledge of nematodes as model organisms. The micrographs of nematodes are evidences of the real moments in their life. Let them be unique!

**Keywords:** Bright field microscopy; Dark field; Phase contrast; Nomarsky contrast; Scanning electron microscopy; nematodes.

# INVITED SPEAKERS

## Id-392

### Microstructural and Spectroscopic Changing on the $MgB_2$ Bulks after Laser Process

C. AKSOY
Karadeniz Technical University, Technology Faculty, Electronics and Communications Engineering,
61830, Of, Trabzon
Corresponding author: cananaksoy@ktu.edu.tr

**Abstract:** $MgB_2$ is a second type superconducting material, have a critical temperature at around 39 K. In this study, the nano size Ag at a certain wt % added to the $MgTi_{0.06}B_2$ bulk sample by using both solid state method and the regional doping method together and the IR laser were applied to the sample as a second local sintering process. The fabricated superconducting bulk samples were excited by 59.5 keV X-rays from a 241-Am annular radioactive source. The K X-rays emitted by the samples were counted by an Ultra-LEGe detector with a resolution of 150 eV at 5.9 keV. The fluorescence parameters such as fluorescence cross section and fluorescence intensity ratios of those samples were calculated theoretically and experimentally. The obtained results were compared with the pure $MgB_2$ and the previous studies. The results showed that there is improvement in a good way for all properties as microstructural, and spectroscopic properties. The results will be valuable for both superconductivity and spectroscopy researchers.

**Keywords:** EDXRF; $MgB_2$; Laser.

# INVITED SPEAKERS

## Id-394

### Photoluminescence of Diamonds Excitation with Far-Ultraviolet Light from a Synchrotron

B. M. CHENG
National Synchrotron Radiation Research Center, No. 101, Hsin-Ann Road, Hsinchu Science Park,
Hsinchu 30076, Taiwan
Corresponding author: bmcheng@nsrrc.org.tw

**Abstract:** Among many and diverse analytical methods established to determine qualitatively or quantitatively the impurities and defects in diamonds, both destructive and non-destructive techniques have been developed to characterize not only natural diamonds but also those from synthesis at great pressure and from deposition of a chemical vapour (CVD). In particular, photoluminescence (PL) emission upon excitation with visible or ultraviolet light provides a sensitive and non-destructive technique for the optical characterization of impurities and defects in diamonds. In this work, we applied the PL technique with excitation from a synchrotron light source in far-ultraviolet (FUV) region to identify sensitively the various defects in diamond materials. The PL excitation (PLE) spectra of natural diamond of type IaAB show four vibrational progressions -- A, B, B′ and N3 related to nitrogen defects in diamond. The vibrational progressions in PL excitation spectra of N2, N3, and N4 centers in diamond of type IaAB at 13 K are identified for the first time. Upon excitation with light at wavelength less than 200 nm, the distinct zero-phonon lines of N3 and N4 centers in diamond at temperature 13 K become prominent at 416.0 and 491.2 nm, respectively. We used PLE spectra to quantitatively analysis of the B center as a N4 nitrogen defect in diamonds; the least detectable concentration of the N4 nitrogen defect is about 13 ppb; the sensitivity of PLE is about 30 times than that practicable with infrared absorption spectra. Associated with nickel defect, prominent spectral PL features were detected at 484.6 and 489.0 nm. During our measurement of PLE spectra of Ni defect in diamond, we observed a distinct PLE line at 215 nm for the first time. In addition, we will present the first direct experimental evidence that diamond nanoparticles containing nitrogen-vacancy (NV) defects are capable of emitting red photons upon exposure to FUV radiation. The knowledge is crucial for the identification of NV as the carrier of extended red emission (ERE) bands detected in diverse astrophysical environments. Our results provide strong evidence that nanodiamonds are a major component of cosmic dust in the interstellar medium. Our model lends strong support to the notion that diamond nanoparticles are a major component of interstellar dust in space.

**Keywords:** Diamond.

# INVITED SPEAKERS

## Id-396

### Relativistic Femtosecond-Pulsed Electron Microscopy

J. YANG, Y. YOSHIDA
The Institute of Scientific and Industrial Research, Osaka University,
8-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan
Corresponding author: yang@sanken.osaka-u.ac.jp

**Abstract:** Ultrafast electron microscopy (UEM) with femtosecond temporal resolution is a very promising technique to observe directly ultrafast dynamic processes in materials science, chemistry and biology. In this paper, we report a relativistic UEM instrument [1, 2] using a compact radio-frequency (rf) photocathode electron gun, and present the results of high-quality electron pulse generation and electron microscopy imaging with the relativistic femtosecond electron pulses.

The relativistic UEM is constructed with an S-band (2.856 GHz) photocathode rf gun and a high-voltage TEM column including an electron illumination system and an imaging system. The rf gun is driven by the third harmonic of a Ti: Sapphire femtosecond laser (wavelength: 266 nm, pulse duration: 100 fs in full width at half maximum). A very fine copper photocathode is used to generate femtosecond electron pulses. The electron illumination system consists of two condenser lenses and a condenser aperture to generate a parallel electron beam after the rf gun, resulting in a small illumination convergence angle at the specimen. After the specimen, we used four relativistic-energy electron lenses in the imaging system: an objective lens, two intermediate lenses and a projector lens, in which the TEM image produced by the objective lens and to project it onto a viewing screen (scintillator). To achieve high sensitivity to MeV electron detection with a high damage threshold, we chose a Tl-doped CsI columnar crystal scintillator equipped with a fiber optic plate (Hamamatsu Photonics) to convert the relativistic-energy TEM images into optical images [3]. The optical images are detected with a digital CMOS camera of size of 2048 × 2048 pixels.

The rf gun has generated high-quality electron pulses, including the emittance of 0.1 mm-mrad, pulse duration of 100 fs, and pulse charge of 1 pC at the energy of 3 MeV. These pulses successfully facilitated single-shot imaging at the magnification of approximately 500 ×. The TEM images of polystyrene latex particles and gold nanoparticles were observed in the imaging operation mode. In the diffraction operation mode, we observed high-contrast diffraction patterns (DPs) from single-crystal materials with a single shot. The measurements were successful in facilitating the detection of higher-order DPs with a spatial resolution of 0.02 Å-1. The single-shot imaging with relativistic femtosecond electron pulses is expected for studying ultrafast structural dynamics in materials, i.e., phase transformations of crystalline materials, chemical reactions, and structural dynamics of biomolecules at the femtosecond time scale.

**Keywords:** Electron microscopy; Electron pulse; Ultrafast dynamics.

**Acknowledgement:** This work was supported by a Basic Research (A) (No. 22246127, No.26246026, and No. 17H01060) of Grant-in-Aid for Scientific Research from MEXT, Japan.

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# INVITED SPEAKERS

## Id-398

### Coal Tar Terahertz Spectrum Investigation

D. LI, Z. LI, W. ZHOU, B. JI, B. SONG

Key Laboratory of Terahertz Technology, Shandong University of Science and Technology, Qingdao, China
Corresponding author: jcbwl@sdust.edu.cn

**Abstract:** Terahertz time-domain spectroscopy (THz-TDS) is used to investigate organic molecule structure and spectral properties. Coal tar is a complex mixture of a large number of polycyclic aromatic hydrocarbons (PAHs) and heterocyclic compounds. The absorption coefficient, refractive index and relative permittivity of coal tar in the range of 0.1-2.5 THz were measured. Comparing with the pure cyclic hydrocarbons - benzene, quinolone, carbazole, etc. which is extracted from coal tar, coal tar was found to be more transparent to THz waves than its components.

**Keywords:** Terahertz; THz-TDS; Coal tar.

# INVITED SPEAKERS

## Id-400

### Interaction of Hesperidin with Phospholipid Bilayers Studied by Fourier Transform Infrared Spectroscopy

C. ALTUNAYAR-UNSALAN
Ege University Central Research Testing and Analysis Laboratory Research and Application Center,
35100, Bornova, Izmir, Turkey
Corresponding author: cisemaltunayar@gmail.com

**Abstract:** Hesperidin is the main flavonoid found considerably in sweet orange and lemon as well as in some other fruits and vegetables. Hesperidin can be correlated with potential benefits for the prevention of diseases, such as decreased capillary permeability, inflammation, microbial infection and carcinogenic effects (Bigoniya and Singh, 2014). Fourier transform infrared (FTIR) spectroscopy is a powerful spectroscopic technique for investigating the structure and organization of phospholipid bilayers. This technique gives information about all parts of the phospholipid molecule simultaneously (Lewis and McElhaney, 1998). In the current study, the effect of hesperidin on the structural and functional properties of phosholipid bilayers was studied for the first time by using Fourier transform infrared (FTIR) spectroscopy. FTIR studies reveal that in the gel phase all studied samples show the same position of $CH_2$ antisymmetric stretching band maximum, reflecting a similar trans/gauche ratio. In addition, hesperidin does not change the ester stretching band position significantly at temperature below the main phase transition.

**Keywords:** Hesperidin; Flavonoid; Phospholipid Bilayers; FTIR Spectroscopy.

**Acknowledgements**: This work was supported by Ege University Scientific Research Projects Coordination Unit. Project Number: 18-FEN-030.

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# INVITED SPEAKERS

## Id-401

### Fourier Transform Infrared Spectroscopy (FTIR) for Characterization of Ablative Materials in Defence Technology

P. NGAMSANTIVONGSA
Defence Technology Institute (Public Organization), Thailand
Corresponding author: phimraphas.n@dti.or.th, phimraphas@windowslive.com

**Abstract:** Fourier Transform-Infrared Spectroscopy (FTIR), which is an analytical technique used to identify organic (and in some cases inorganic) materials, is an experimental technique used initially for qualitative and quantitative analysis of organic compounds, providing specific information on molecular structure, chemical bonding and molecular environment. This technique measures the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands identify molecular components and structures. Ablative materials play an important role in the aerospace industry. Fiber-reinforced polymer (FRP) is a composite material made of a polymer matrix reinforced with fibers. High-strength, lightweight FRP composites have been widely used in defense and aerospace systems for many years. The ability of glass fiber-reinforced phenolic resins to withstand extremely high heat flux for short periods has made their use for re-entry nose cones and rocket nozzles possible. Phenolic resins are the most regularly used polymer matrix for ablatives. These thermal protection systems are exposed to a thermochemical flow and subjected to high temperatures in excess of 3,000°C with very high heating rates. In this study, the composites fiber glass were obtained with a phenolic resin (PR) and modified PR by the addition of epoxy resins and phenyltrimethoxysilane for improving the ablation resistance using the hot compression molding technique. Moreover, the physical as well as chemical properties of the matrix and the composites were determined by using standard experimental methods. The comparison between modified PR and traditional PR were analyzed by Fourier transform infrared spectroscopy (FTIR) and the ablative measurement on laminate specimens were tested by using oxyacetylene flame testing. The results showed that the modified PR composites significantly enhanced ablation resistance.

**Keywords**: Ablative materials; Fiber-reinforced polymer matrix composites; Phenolic resin (PR); Rocket motors; Fourier transform infrared spectroscopy (FTIR).

# INVITED SPEAKERS

## Id-406

### Analysis of Pathological Tissues by Vibrational Micro- and Nano-Spectroscopy Methods

C. PALUSZKIEWICZ[1], W. M. KWIATEK[1], M. CESTELLI GUIDI[2], N. PIERGIES[1], E. PIĘTA[1], M. WOŹNIAK[1],
M. MISIOŁEK[3], W. ŚCIERSKI[3]
[a]Institute of Nuclear Physics, Polish Academy of Science, 31-342 Krakow, Poland
[b]Laboratori Nazionali di Frascati INFN, Via E. Fermi 40, 00-044 Frascati, Italy
[c]Medical University of Silesia, Department of Otorhinolaryngology, 41-800 Zabrze, Poland
Corresponding author: czeslawa.paluszkiewicz@ifj.edu.pl

**Abstract:** The possibilities offered by Fourier Transform Infrared (FTIR) and Raman spectroscopy (RS) methods make them attractive tools to perform characterization of biological components of human tissues. The application of these two complementary spectroscopic approaches ensures complete vibrational characterization of the investigated biomolecules and biomaterials composition. However, the diffraction problem of FTIR is still limited which significantly reduces spatial resolution. Conventional infrared spectroscopy, even combined with optical microscope, limits spatial resolution to ~1 μm. This limit can be circumvented by employing atomic force microscope (AFM) along with IR. Application of AFM-IR technique provides IR characterization of biochemical composition of the samples with nanoscale resolution. Moreover, AFM gives information about the topography and the structure of investigated compounds. The presentation describes different applications of micro- and nano- spectroscopies studies. The general aim of this investigation is better understanding the structural changes in pathological tissues using the abovementioned techniques. The first aspect concerns the human lens tissue study. We present the images of the healthy and cataractous human lenses in micro- and nanoscale together with the amide I distribution maps. Based on the obtained results it can be stated that there is a significant correlation between the disease progression and the secondary structure of proteins and conformational variations of the amino acid residues. Moreover, the fragmentation of the tissue appearing upon cataract was observed. The second approach includes cancer tissues tests with the main emphasis on head and neck regions. These tumors are often diagnosed in salivary glands and paranasal sinuses. Additionally, non-malignant types of cancer can often transform to pathogenic forms what promotes the development of malignant tumors. The structural variations occurring due to the cancer development in cellular biomarkers, such as RNA, DNA, proteins, lipids, phosphates and carbohydrates, can be successfully detected in the collected spectra. In this work, we explore the salivary glands tissues derived from patients with cancer disease during routine surgical intervention. Additionally, the spectra collected for the studied tissues were compared with results obtained for saliva samples from the same patients.

**Keywords:** FT-IR; RS; AFM-IR; Pathological tissues; Cancer.

**Acknowledgements:** The research leading to this result has been supported by the project CALIPSOplus under the Grant Agreement730872 from the EU Framework Programme for Research and Innovation HORIZON 2020. The research was partially carried out using equipment purchased in the frame of the project co-funded by the Małopolska Regional Operational Program Measure 5.1. Krakow Metropolitan Area as an important hub of the European Research Area for 2007-2013, project No. MRPO.05.01.00-12-013/15.

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# INVITED SPEAKERS

## Id-408

### Calibration Problem and Solution in Transmission Line Pulse of Packaged Sample

T. SUZUKI
2-1844-2 Kozoji-cho, Kasugai, Aichi, 487-0013, Japan
Corresponding author: suzuki.teruo@socionext.com

**Abstract:** Electro-static Discharge (ESD) protection circuit should have enough current capability so that the voltage can be suppressed below the damage level of the protected circuit. For this purpose, both I-V performance of the ESD protection circuit and withstand voltage of the protected circuits should be defined. Recently, Transmission Line Pulse (TLP) test system is available to analyse the electrical performance of the ESD protection circuit as well as the withstand voltage of the protected circuit, and the ESD design cannot be done without it. The degree of leaning of the ESD wave form (hereinafter referred as RON) of ESD protection parameters is closely related to the internal core circuit damage. In particular, for the high-precision ESD design of the thin gate oxide transistor in the most advanced technology, the high accuracy RON measurement of TLP is required.

In many cases, TLP is measured using a wafer. However, when a failure is found in the packaged product, it is necessary to carry out TLP measurement using the packaged product. During short calibration, we found the contact resistance between the packaged sample and integrated circuit (IC) socket varied depending on the calibration current. If packaged Device Under Test (DUT) is loaded in the socket and I-V curve is measured by TLP test system, it was found that the resistance of the I-V curve decreased by increasing measurement current. By Scanning Electron Microscope (SEM) picture analysis, trace of the solder melting by the TLP calibration current was found. There is package level TLP measurements requirement for the packaged product, a few solutions to avoid the mismatch between short calibration and actual measurement should be provided.

In this paper, we clarified the problem of carrying out the package based short calibration using a packaged sample and IC socket and showed that kelvin measurement is effective.

**Keywords:** ESD; TLP; Calibration; Kelvin measurement.

# INVITED SPEAKERS

## Id-411

### Tailoring the Microstructure and Performance of Ceramic Composites with Graphene Nano-Platelets: Electron Microscopy as a Basic Tool

Á. GALLARDO-LÓPEZ[1]*, C. LÓPEZ-PERNÍA[1], C. MUÑOZ-FERREIRO[1], A. MORALES-RODRÍGUEZ[1], F. GUTIÉRREZ-MORA[1], R. POYATO[2]

[1] Universidad de Sevilla, Departamento de Física de la Materia Condensada, ICMS, CSIC-Universidad de Sevilla, apdo. 1065, 41080 Sevilla, Spain

[2] Instituto de Ciencia de Materiales de Sevilla, ICMS, CSIC-Universidad de Sevilla, Avda. Américo Vespucio 49. 41092 Sevilla, Spain

Corresponding author: angela@us.es

**Abstract:** Ceramic composites with graphene nano-platelets (GNP) exhibit an interesting combination of properties of the ceramic matrix and the carbon nanostructures. Some of these properties, such as the electrical conductivity and mechanical response, can be tuned by adjusting the ratio ceramic-filler, selecting the type of graphene nano-platelets and choosing adequate processing routines and sintering conditions. Achieving a high microstructural homogeneity, especially for high GNP contents, is one of the most difficult processing issues to overcome, due to the GNP's high tendency to agglomerate. The agglomerates decrease the percolation degree of the GNP network, affecting the mechanical and electrical performance of the composites, so the ceramic powder-GNP mixing stage during processing is critical. The sintering technique and conditions, especially the sintering temperature and the applied pressure, will determine the density, anisotropy degree and grain size of the composites.

The objective of this work is to assess the microstructural homogeneity and anisotropy of these ceramic composites, the structural integrity and percolation degree of the GNP and the interfacial characteristics of the graphene and ceramic phases using electron microscopy as a basic tool. The results of the microstructural evaluation will allow us to adjust the processing and sintering conditions in order to tailor composites with the desired electrical and mechanical performance. To that end, the microstructure of 3YTZP composites with 1-20 vol% graphene nano-platelets -with different GNP characteristics and different powder processing conditions and sintering methods- has been analyzed. The Young's modulus, hardness, fracture propagation, electrical conductivity and wear resistance of the composites have been also assessed and related to the microstructural features.

Transmission electron microscopy (TEM) analysis of spark plasma sintered (SPS) composites revealed the existence of an intermediate zirconia oxycarbide in the grain boundaries disregarding the number of layers of the filler, which confirms interfacial bonding between matrix and filler. The use of few-layer graphene (~3 graphene layers) -with a higher interplanar spacing revealed by HRTEM- increases the microstructural homogeneity and gives the best electrical conductivity results. Using thicker nano-platelets (~15-30 graphene layers) and planetary ball milling during processing also increases greatly microstructural homogeneity, electrical conductivity and isotropy as well. The composites sintered by SPS present a much higher structural anisotropy -due to a preferential orientation of the GNP perpendicular to the sintering pressing axis- than the composites consolidated by pressureless sintering (PLS). This anisotropy can be reduced by increasing GNP content or by incorporating high energy milling during the powder processing step.

The GNP-3YTZP composites show lower friction coefficients and higher wear resistance than the monolithic 3YTZP. Composites with the lowest GNP content showed an overall improved tribological behavior. Increasing the GNP content leads to lower densification - especially with pressureless sintering techniques- hardness and elastic modulus values, but in contrast it greatly enhances electrical conductivity. The electrical percolation threshold of the composites is situated between 2.5 and 5 vol% GNP content for both -SPS and PLS- sintering methods.

**Keywords**: 3YTZP; Graphene nano-platelets; Processing; Electron microscopy; Electrical conductivity.

# INVITED SPEAKERS

## Id-416

### EPR Spectroscopy of pH-Sensitive Spin Probes and Labels in Materials Characterization

KOVALEVA E.G.[1*], MOLOCHNIKOV L.S.[2], ANTONOV D.O.[1,2], TAMBASOVA D.P.[1]
[1]Ural Federal University, Yekaterinburg, Russia
[2]Ural State Forest Engineering University, Yekaterinburg, Russia
Corresponding author: e.g.kovaleva@urfu.ru

**Abstract:** Material Science is nowadays a wide research area which involves both some traditional materials formed from metals, ceramics, polymers and their various composites and novel ones including nanostructured, molecular, organic, hybride organo-inorganic and biological materials ad systems with unique functionalities. Electron paramagnetic resonance (EPR) spectroscopy is a versatile and exceptionally sensitive technique for detecting and studying molecular systems possessing unpaired electronic spins. The scope of EPR applications in material science can be expanded significantly by means of spin labels and spin probes – exogenous EPR-active species that are introduced solely into initially no-paramagnetic systems for the purpose of gaining information under study. These labels and probes are persistent free radicals or paramagnetic metal ions designed to be partitioned or attached to specific molecular sites [1]. Recently, pH-sensitive nitroxides have been found a great number of applications due to the exquisite sensitivity of their EPR spectra nitroxides (NR) to reversible protonation of functional groups adjacent to the nitroxide moiety [2], though the ionization state of more remote functional groups may also be ascertained [3-6]. Proton concentration measured as pH is of fundamental importance for many chemical and biochemical processes including inorganic catalysis and living systems. The aim of this study is to cover the applications of pH-sensitive NR ranging from inorganic (nanoporous and nanostructured oxides) through organic (ion-exchange resins) and organo-inorganic (hybride chitosan and cellulose-inorganic oxides) [5,6] to biomaterials (oxide systems with phospholipids and enzymes) [7]. It has been found that pH-sensitive NR can be useful for studying acid-base and electrosurface properties of various materials and systems and specific processes in which they occur to be involved including based on local pH, surface charge, electrical potential, polarity and functional groups pKa determination.

**Keywords:** EPR spectroscopy; pH-sensitive nitroxides; Surface Electrical Potential; Local pH values; Solid-phase inorganic and polymeric materials; Biomaterials.

**Acknowledgement:** This study was supported by the Program of the Ministry of Education of the Russian Federation (1.1.07 E3H, 2007-2011), RFBR Grants 14-03-00898, 17-03-00641 and 18-29-12129mk, and the State Task from the Ministry of the Education and Science of the Russian Federation nos. 4.9514.2017/8.9 (2017-2019).

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# INVITED SPEAKERS

## Id-420

### Application of Broadband Dielectric Spectroscopy to Study Molecular Dynamics and Kinetics of Crystallization Processes

M. JASIURKOWSKA-DELPORTE
The Henryk Niewodniczanski Institute of Nuclear Physics, Polish Academy of Sciences, Radzikowskiego 152,
31-342 Kraków, Poland
Corresponding author: Malgorzata.Jasiurkowska-Delaporte@ifj.edu.pl

**Abstract:** Broadband dielectric spectroscopy (BDS) is a versatile tool for investigation of the electrical properties of a various material such as polar liquids, polymers, liquid crystals, pharmaceutical compounds, ionic liquids, ceramics or even semiconductors. BDS finds a large number of applications in various fields of science and technology including monitoring of chemical processes and phase transitions. The principle idea of BDS is to probe the response of polar substances to the external, variable electric field over a wide frequency range up to 16 decades. This talk will present the application of dielectric spectroscopy in the investigations of molecular dynamics in glass-forming systems and liquid crystals in bulk and under geometrical restriction [1,2]. Our proposed combined broadband dielectric spectroscopy and Fourier-transform infrared spectroscopy analysis will be also discussed [3]. This approach lets us to trace how the different, also non-polar molecular units are influenced by temperature changes and confinement. Finally, the studies of isothermal crystallization kinetics in molecular systems by means of BDS will be presented [4].

**Keywords:** Braodband dielectric spectroscopy, Molecular dynamics, Crystallization kinetics.

**Acknowledgement:** Financial support from the National Science Centre (Grant SONATA11: UMO-2016/21/D/ST3/01299) is gratefully acknowledged.

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# INVITED SPEAKERS

## Id-421

### Near Infrared Spectroscopy in Biomedical Applications

H. A. RAHIM
Universiti Teknologi Malaysia, Malaysia
Corresponding Author: herlina@fke.utm.my

**Abstract**: Diabetes is a medical condition that can lead to various diseases such as stroke, heart disease, blindness and obesity. In clinical practice, the concern of the diabetic patients towards the blood glucose examination is rather alarming as some of the individual describing it as something painful with pinprick and pinch. As for some patient with high level of glucose level, pricking the fingers multiple times a day with the conventional glucose meter for close monitoring can be tiresome, time consuming and painful. With these concerns, several non-invasive techniques were used by researchers in measuring the glucose level in blood, including ultrasonic sensor implementation, multisensory systems, absorbance of transmittance, bio-impedance, voltage intensity, and thermography. This paper is discussing the application of the near-infrared (NIR) spectroscopy as a non-invasive method in measuring the glucose level and the implementation of the linear system identification model in predicting the output data for the NIR measurement. In this study, the wavelengths considered are at the 1450nm and 1950nm. Both of these wavelengths showed the most reliable information on the glucose presence in blood. Then, the linear Autoregressive Moving Average Exogenous model (ARMAX) model with both un-regularized and regularized methods were implemented in predicting the output result for the NIR measurement in order to investigate the practicality of the linear system in this study. However, the result showed only 50.11% accuracy obtained from the system which is far from the satisfying results that should be obtained.

**Keywords:** Diabetes, glucose level, linear, near-infrared (NIR), non-invasive, prediction system.

# INVITED SPEAKERS

## Id-422

### In Situ Environmental TEM/STEM Study of Oxidation and Reduction Processes in Metallic Nanoparticles and Thin Oxide Films

V. K. LAZAROV[1,2], L. LARI[1,2], E. BOYES[3], R. DOUTHWAITE[3], P. GAI[1,2,3], A. LAGROW[3], K. YOSHIDA[4]

[1] Department of Physics, University of York, UK

[2] Department of Chemistry, University of York, UK

[3] York-JEOL Nanocentre, University of York, UK

[4] Institute for Materials Research, Tohoku University, Japan

Corresponding author: vlado.lazarov@york.ac.uk

**Abstract:** Reduction and oxidation processes that involves metallic nanoparticles and thin film oxides are of importance for catalysis as well as the electronic and spintronic devices technology. In this talk we will present our recent in situ TEM/STEM results on oxidation Fe and Cu nanoparticles in vacuum in $O_2$ gas as function of temperature up to 1000°C. Structural changes during oxidation process as revealed by HRTEM/HAADF and nano-diffaction will be discussed in details. Similarly, reduction processes of oxidized particles in H atmosphere within the same temperature processes will be presented. We will present also the effect of the e-beam on the oxidation processes and effects such as Kirkendall effect. In addition to nanoparticles we will present also CO and $H_2O$ vapour interaction with NiO extended surfaces as well as $TiO_2$ nanoporous films formation. We will show the self-catalytic properties and atomic visualization of water-shift gas reaction on NiO (111) surface, as well as simterin processes of $TiO_2$ nanoparticles into nanoporous thin films for solar cells and Li ion batteries.

**Keywords:** Oxidation; Reduction; HAADF; HRTEM; In-situ; Environmental TEM/STEM.

# INVITED SPEAKERS

## Id-423

### Characterization of Aqueous Media through the Analysis of the O-H Stretching Band by Raman Spectroscopy

D. IVANA
71 rue de la Grande Haie, 54510 Tomblaine, France, Cerema, TEAM
Corresponding author: ivana.durickovic@cerema.fr

**Abstract:** The aim of this work is to show the possibility to use Raman spectroscopy (RS) to study aqueous solutions and chemical species dissolved in water. The application of RS on the study of aqueous media is very efficient if a proper signal treatment is applied.

In a Raman spectrum of water, a special attention is devoted to the study of the O─H stretching region, generally considered to be closely related to the structure of water. It is an indicator of the hydrogen-bonding network since, besides the intra-molecular O─H pairs, intermolecular O─H linked by hydrogen bonds contribute to the O─H stretching. The role of the hydrogen bonds is therefore of great importance for the understanding of this spectral range.

This region is of great interest because the designations of the symmetric and asymmetric O-H stretching normal modes are still not well elucidated. It is generally admitted that all the bands of water are made up from contributions from different components from water molecules in different hydrogen bonded environments. In order to analyze this region in a more detailed way, it is necessary to proceed to a deconvolution of the band corresponding to this complex contribution of the water spectrum into various peak components, to assign each vibrational mode and to get back to their molecular origins. However, when it comes to bands that are as large as the O─H stretching band, the deconvolution is rather complicated and numerous deconvolution models can be found in the literature. The Raman spectrum of water, and particularly the O-H stretching band, is sensitive to temperature and dissolved species. Through the presentation of the elaboration of calibration curves (based on a follow-up of specific peaks and an appropriate signal treatment), we will show that the study of this spectral region can be used for the detection and quantification of chemicals dissolved in water. The developped method is easy and quick to be employed, the deconvolution of Raman spectra not being necessary. It is particularly interesting for the detection and quantification of monoatomic ions that do not present a specific signature in the Raman spectrum. The application of this technique on the study of several aqueous solutions where the dissolved chemicals have different influences on the O-H stretching band will be presented. Another physical quantity that can be deduced from the analysis of the O-H stretching band in a Raman spectrum of water or of an aqueous solution is its phase. A specific analysis set to determine the solution solid-liquid phase transitions will be presented as well as the thus-constructed phase diagram. This information permits to determine the compound's eutectic concentration and thus, its hydration (compound/water ratio) underneath the eutectic temperature. The results obtained on several de-icers will be presented, as the knowledge of these product's phase diagram is of capital importance for the winter maintenance domain.

**Keywords:** O-H stretching band; Aqueous solution; Phase transition; Quantification.

# INVITED SPEAKERS

## Id-424

### Interesting Microstructural Features of Ultrahigh Strength Steel Characterized by SEM/EBSD and TEM

Y. SHEN
Northeastern University, China
Corresponding author: shenyf@smm.neu.edu.cn

**Abstract:** Nanostructures play important roles in determining mechanical properties of metallic materials. Here we show some interesting results associated with nanosized precipitates in ultrahigh strength steels. (1) TEM observations and atom probe topography (APT) studies presented an intriguing observation that the combination of ultra-high strength and good ductility can be achieved by changing the variety nature of precipitates (rod-like ε-$Fe_3C$ and spherical Cu) and the ratio of microstructural constituents in a low-manganese-medium-carbon steel. The ultra-high tensile strength of 2,800 MPa TEM observations and APT analysis showed a dissolution of nanosized $Fe_3C$ with increasing temperature and strain rate in an ultrafine-grained (UFG) low-alloy medium-carbon steel (D6AC), which had an average grain size of ~ 500 ± 30 nm, coexisting with nanosized $Fe_3C$ particles ($d$ = 80 ± 10 nm). (3) TEM results revealed that the softening behavior in a 20Mn-0.6C TWIP steel was related to significant strain localization within the bands and the adiabatic heating. The increasing twinning activity leads to the intersection between primary and secondary twin systems, resulting in a stress concentration that promotes pronounced shear bands. (4) TEM observations showed that deformation twinning and martensitic phase transformation are significantly retarded with increasing grain refinement in an ultrafine-grained 304 austenitic stainless steel with a grain size of ~270 nm. Uniaxial tensile testing at room temperature revealed an extremely high yield strength of 1890 ± 50 MPa and a tensile strength of 2050 ± 30 MPa. (5) Some interesting results in ultrahigh strength steels characterized using SEM/EBSD are expected to be presented.

**Keywords:** Ultrahigh strength steel; Nanosized precipitates; Nanoscale phase; Nano twin.

# INVITED SPEAKERS

## Id-425

### Characterization of CNTs and CNT Fibers

K. HATA
Director, Nanotube Research Center, National Institute of Advanced Industrial Science and Technology (AIST), Japan
Corresponding author: kenji-hata@aist.go.jp

**Abstract:** More than 25 years has pasted since the discovery of carbon nanotubes (CNT), and now many real applications are well recognized in the market such as Li ion batteries electrodes, CNT touch panel used in smartphones, etc, and much more are going to show up soon. AIST has established the CNT Application Research Center whose primary mission is to support the CNT industry. In general, the CNT users who are developing applications do not care what CNT (e.g., whether it is MWNT or SWNT) they are going to use, but would choose the best CNT suited for their purpose. Frequently, people who have started using CNT`s has asked me “There are many different CNTs in the market, and which one should I use?” The same question could be done for many types of CNT materials such as fibers, master batches, dispersions. The CNT Application Research Center has developed many new CNT characterization techniques specialized to evaluate industrial CNT materials. Examples of the new characterization techniques are using plasmon resonance to measure the length of CNTs and using CPS disc centrifuge to characterize the size of the CNT aggregate dispersions. In this talk, I would overview our efforts to characterize and classify many CNT materials with the help of these tools from which we have and found many intrinsic and fundamental trends.

**Keywords:** Carbon nanotubes, CNT

# REGULAR SESSIONS

## Id-220

## Transition Metal Chalcogenides Based Polymer Composites: Microscopic and Spectroscopic Characterization and Applications

*E. MİJOWSKA, K. MAŚLANA, K. SZYMAŃSKA, K. WENELSKA
Nanomaterials Physicochemistry Department, Faculty of Chemical Engineering, West Pomeranian University of Technology, Szczecin, Poland
Corresponding author: emijowska@zut.edu.pl

**Abstract:** In recent years, effective halogen-free flame-retarded systems became one of the most popular topics of relevant materials research due to the environment problems of halogencontaining flame retardants. Polyolefin - based products are used in cables, automobiles, electronic cases, interior decoration, packaging, and so on. However, because of their inflammable nature, there is a considerable risk of fire - related injury and property loss and so the flame retardancy must be improved. However, owing to emission of toxic chemicals during burning, their use on a large scale is not suitable and they are actually banned in some countries. Thus, an environment friendly flame retardant must be needed. The discovery of graphene and its superior properties have focused research interest on other two-dimensional (2D) groups of materials like hexagonal boron nitride (h-BN) and transition metal dichalcogenides (TMDs). Recently last group of materials has attracted increased attention in the field of nanocomposites fillers, due their graphene-like properties such as high thermal and mechanical properties. In this work, polyolefines composites are prepared using the melt - mixing method. The MoS2 and WS2 in the polymer composite serves as a flame retardant, and improves the efficiency of polymer carbonization. Additionaly, MoS2 or WS2 are functionalized by the metal oxide nanoparticles are the catalysts for carbonization of polymer matrix during combustion. The co - existence of layerd materials and metal oxide nanoparticles produces synergistic effects compared to a single component. The enhancement mechanism is proposed and discussed. Probably under the same conditions, the carbonization rate can be the critical factor to influence the flame retardancy performance. Among Fe, Co, and Ni, Ni has the fastest carbonation rate, which leads to the best flame retardancy performance.

**Keywords:** Transition metal chalcogenides, polymer composites, metal nanoparticles, flame retardancy

**Acknowledgements:** The research was funded by The National Science Center, Poland (OPUS 10 UMO-2015/19/B/ST8/00648

# REGULAR SESSIONS

## Id-221

### On the Structural, Elastic, Electronic and Optical Properties of Cds/Zns Super Lattice Under High Pressure.

D. CHERRAD*, F. NEMLA

Laboratory for Developing New Materials and their Characterizations, University of Setif, Algeria.

Corresponding author: cherradphisic@yahoo.fr

**Abstract:** In this work, we study the structural, elastic, electronic and optical properties of the super lattices CdS/ZnS under hydrostatic pressure using the pseudo-potential plane wave method (PP-PW) scheme in the fram of generalized gradient approximation (GGA). Structural and elastic results are given for lattice constant (a,c), bulk modulus B their pressure dependence and elastic constants $C_{ij}$. The band structure calculation together with the density of states shows that this compound has a direct energy band gap (Γ→Γ). We have calculated the frequency dependent complex dielectric function ε(w) and its zero frequency limit $\varepsilon_1(0)$. We have found that the values of $\varepsilon_1(0)$ increases with decreasing the energy gap for radiation up to 25 eV.

**Keywords:** CdS/ZnS; PP-PW method; Elastic constants; Electronic properties; Optical properties.

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## Id-228

# Evidence of Large Hopping Polaron Conduction Process in Strontium Doped Calcium Copper Titanate Ceramics

S. AMHIL*, L. ESSALEH, E. CHOUKRI, A. ALIMOUSSA, R. BOUFERRA

[a] Laboratory of Condensed Matter and Nanostructures (LMCN) , Cadi-Ayyad University, Faculty of Sciences and Techhnology, Departement of Applied Physics, Marrakech, Morocco.

[b] Laboratoire Procédés, Métrologie, Matériaux pour l'Energie et Environnement (LP2M2E), Cadi-Ayyad University, Faculty of Sciences and Technology, Departement of Applied Physics, Marrakech, Morocco.

Corresponding author : amhilsamira@gmail.com

**Abstract:** Strontium (Sr) doped Calcium Copper Titanate $Ca_{1-x}Sr_xCu_3Ti_4O_{12}$ with two values $x$=0 and $x$=0.05 commonly known as CSCTO ceramics have been obtained by using the semi wet route synthesis method. Rietveld refinement shows that the powders crystallize in the cubic perovskite related structure with Im3 space group. Scanning Electron Micrograph (SEM) analysis shows that the average grain size of ceramics becomes larger when Sr doping is considered. Both DC and AC electrical conductivity are investigated throughly in the temperature and frequency ranges between [373-653K] and [20Hz-1MHz], respectively. The values of the average activation energies for CSCTO-0 ($x$=0) and CSCTO-5 ($x$=0.05) were found to be 612meV and 576meV, respectively. In Fact, Sr doping has an effect on the broadening of the impurity band that can lead to a dramatic decrease in the activation conduction energy of CSCTO ceramics. A systematic study of AC electrical conductivity reveals that the predominant conduction mechanism existing in these ceramics is generated by large polaron hopping process. This mechanism has also been identified by the modulus analysis and confirmed by comparing the hopping polaron size with the lattice parameter.

**Keyword:** CCTO, Semi wet route, Electrical properties, OLPT.

# REGULAR SESSIONS

## Id-234

### Films and Coatings Based on Polycarbonate-Based Polyurethanes and Polyurethane Nanocomposites

M. ŠPÍRKOVÁ*, J. HODAN, S. KREJČÍKOVÁ AND L. MACHOVÁ
Institute of Macromolecular Chemistry AS CR, Heyrovského nám. 2, 162 06 Prague 6, Czech Republic
Corresponding author: spirkova@imc.cas.cz

**Abstract:** The contribution is aimed on the preparation procedures of complex polycarbonate-based thermoplastic polyurethane (PU) and PU nanocomposite films and coatings. The products, being intended as materials for biomedical applications, are prepared either in bulk or from waterborne PU dispersions (WPUDs). The innovated procedures producing WPUDs either without any chain extender (short-chain diol or diamine) or WPUDs including D, L-lactide-based linker in PU backbone represent significant potentials for the preparation of stable or degradable 2D materials. The other potential consists in miscellaneous PU nanocomposites that can be prepared very simply: water is evaporated from the aqueous mixture of WBPU and appropriate nanofiller. Targeted properties are achieved by the optimized setting PU-to-nanofiller ratio. Multidisciplinary characterization of PU and PU nanocomposites is based on the comprehensive investigation of properties spanning from segmental up to macroscopic levels. For this purpose, the set of spectroscopic, microscopic, thermal and mechanic analyses was used. Swelling and stability/degradability properties were tested in different solvents (water, acetone, heptane, xylene) at room temperature, in phosphate buffer saline (PBS) or mixture of $H_2O_2$ + $CoCl_2$, both media at 37 ºC. Mechanical characteristic (tensile strength, elongation-at-break, Young´s modulus, toughness) were found to be the most sensitive quantities being significantly changed if the composition of starting material was modified only slightly. Therefore, tensile tests can be used for the preliminary comparison of potential suitability of novel PU products for practical applications. PU films and nanocomposites prepared in bulk have considerably better tensile properties than materials prepared from WPUDs.Surface morphology and internal arrangement of PU materials was checked by the set of microscopy (SEM, AFM and TEM) techniques. The films are flat or slightly wavy. If nanofiller are protruded on the surface, it is well built-in PU matrix. High degree of PU chain organization was detected by TEM and AFM (surface measurements of cryo-cut or freeze fractured samples). Spherulites of micrometer size occur even in films made from WPUDs consisting of particles lower than 100 nm in diameter. All films are thermally stable at least up to 200 ºC. Swelling and stability/degradability tests found out that either stable or degradable materials can be produced, depending on starting composition and procedure protocol. Films made from WPUDs are more susceptible to degradation compared to PU films made in bulk, as verified by accelerated *in vivo* tests in $H_2O_2$ + $CoCl_2$ mixture. All the new products, especially those prepared by the 'green process' should meet requirements for specific applications, e.g., as coatings in biomedicine.

**Keywords:** polyurethane, nanocomposite, film, coating, biomedical application, degradability

**Acknowledgement:** The authors thank to the Czech Science Foundation (project **18-03932S**) for financial support.

# REGULAR SESSIONS

## Id-240

# NMR Spectroscopy: a Tool for Chiral Recognition of Less Polar Classes of Organic Molecules

S. HAMEED
Department of Chemistry, Quaid-i-Azam University, Islamabad-45320, Pakistan
Corresponding author: shameed@qau.edu.pk

**Abstract:** The continuous interest in enantioselective syntheses has seen a rapid development in the techniques to measure the purity of enantiomers. Inspite of the fact that chromatographic methods, especially GC and HPLC, are being used more frequently, the NMR methods are still being prefered by most of the organic chemists for chiral recognition. Most extensively used reagents for the purpose are chiral lanthanide shift reagents (CLSRs). However, CLSRs fail to bind to certain classes of less polar compounds. Dirhodium *tetra*-(R)-$\alpha$-methoxy-$\alpha$-(trifluoromethyl)phenylacetate complex has been found suitable to bind to less polar compounds for NMR analysis. In the present work, the chiral recognition of compounds with low polarity (nitriles, selenides and iodides) was explored using dirhodium *tetra*-carboxylate complex. We synthesized a reasonable number of nitriles with varying structural features and recorded their $^{1}$H NMR spectra with and without dirhodium *tetra*-carboxylate complex. Almost all the proton signals were found shifted to low field and diastereotopic dispersions were observed for the signals making the enantiomeric ratio determination possible. The tentative statements regarding the binding mode of the nitrile group with dirhodium *tetra*-carboxylate complex are presented. CLSRs also fail to bind an other important class of compounds, namely selenides. We also studied the chiral recognition of some representative selenides using dirhodium *tetra*-carboxylate complex. The technique was also used to study the chiral recognition of iodides and bromides. The results revealed that although the signal shifts and dispersions were low in case of selenides and iodides, yet they were large enough for enantiomeric purity determination. However, the dirhodium *tetra*-cafboxylate complex failed to bind to bromides and effects similar to nitriles, selenides and iodides were not observed.

**Keywords:** Chiral recognition, HNMR, Less polar compounds

# REGULAR SESSIONS

## Id-250

### Imaging and Measurements of Cell Wall Nanomechanical Properties in Broadleaved Forest Tree Species Using Atomic Force Microscopy Peakforce Quantitative Nanomechanical Mapping

J. ĎURKOVIČ[1], R. LAGAŇA[2], M. KARDOŠOVÁ[1], S. BHAGIA[3]
[1] Department of Phytology, Technical University in Zvolen, 960 53 Zvolen, Slovak Republic
[2] Department of Wood Science, Technical University in Zvolen, 960 53 Zvolen, Slovak Republic
[3] Department of Chemical and Biomolecular Engineering, University of Tennessee, Knoxville, TN 37996, USA
Corresponding author: jaroslav.durkovic@gmail.com

**Abstract:** The atomic force microscopy (AFM) technique called PeakForce quantitative nanomechanical mapping (PeakForce QNM) was applied to reveal the nanomechanical properties and topography of both the fiber cell walls in wood and the tracheary element cell walls in the leaf primary xylem of broadleaved forest tree species. The objectives of this study were to determine the reduced Young's modulus of elasticity (MOE), adhesion force, deformation and dissipation energy of xylem cell walls from the various plant origins. All these nanomechanical traits were infered from the approaching and retracting parts of the force-versus-separation curves produced during tapping cycles. For the wood of *Populus trichocarpa*, we compared secondary cell wall nanomechanics of fibers between the low lignin natural variant (GW-11012 genotype) and the high lignin comparator (BESC-316 genotype). BESC-316 had a significantly higher tip-surface adhesion than GW-11012 possibly due to its higher lignin content. Differences in MOE, deformation and dissipation were non-significant. AFM and transmission electron microscopy observations suggested high heterogeneity in the cell walls of BESC-316. For the hybrid poplar *Populus tremula* × (*Populus* × *canescens*) leaves, we assessed nanomechanical properties of tracheary element cell walls within the midrib primary xylem comparing the plants propagated from in vitro tissue culture with those propagated from root cuttings. Tracheary element cell walls of the micropropagated plants showed higher values for dissipation energy than those of the plants propagated from root cuttings. Dissipation refers to the area in between approaching and retracting parts of the force-versus-separation curve, reflecting the energy dissipated between the tip and the sample during each tapping cycle. The observed result may indicate a higher nanoscale heterogeneity of cell wall constituents, and also a higher damping capacity within the midrib primary xylem tissue under abiotic stress conditions for the micropropagated plants. For Dutch elm hybrid 'Dodoens' (i.e., open pollinated *Ulmus glabra* 'Exoniensis' × *Ulmus wallichiana*) leaves, we compared cell wall nanomechanics of tracheary element cell walls within the primary xylem of the midrib for both the non-infected plants and the plants infected by the ascomycetous fungus *Ophiostoma novo-ulmi* ssp. *americana* × *novo-ulmi*. Three years following artificial inoculations, no significant differences in the above nanomechanical traits were found in the infected plants. Using PeakForce QNM imaging and measurements, our findings provided new insights into the xylem cell wall properties of various broadleaved forest tree species under different experimental conditions. This relatively new technique of imaging provided direct control of the maximum loading force and the deformation depth keeping indentations small, while eliminating damaging lateral forces in order to preserve both the tip and sample.

**Keywords:** Cell Wall Nanomechanics, Xylem, Populus, Ulmus, Peakforce QNM

**Acknowledgements:** This work was supported by the Slovak scientific grant agency VEGA (1/0450/19).

# REGULAR SESSIONS

## Id-267

## Detection of Autofluorescence Using Visible Wavelength: Induction of Cancer Stem Cell Sphere Explosion by UV Irradiation/Cold Shock or Therapeutic Chemicals

N.-S. CHANG
Molecular Medicine, National Cheng Kung University College of Medicine, Tainan, Taiwan
Corresponding author: wox1world@gmail.com

**Abstract:** In this study, cancer stem cell sphere explosion was imaged by enhanced visible wavelength. When cells are exposed to UV irradiation followed by incubation at 37$^{o}$C, they undergo typical apoptosis. However, when the UV-irradiated cells are incubated at room temperature or down to 4$^{o}$C, they generate nitric oxide (NO)-containing nuclear bubbles – one bubble per cell. We designated this novel type of cell death as “bubbling cell death (BCD)”. The bubbles contain. Unlike apoptosis, there are no caspase activation, flip-over of phosphatidylserine, and DNA fragmentation (Oncotarget 2015;6:8007-18). Here, by time-lapse microscopy, we demonstrated that when breast cancer 4T1 stem cell spheres were subjected to UV and a brief cold shock (5 min at 4$^{o}$C), followed by imaging at room temperature. The spheres initially shrank, then slowly expanded and finally exploded, and the cells died. Similar results were observed when the spheres were exposed to an aliquot of cocktail of protease inhibitors and an ERK inhibitor U0126, or an anti-lung cancer drug ceritinib. Notably, prior to sphere explosion, there was an emission of luminescent light under bright light stimulation. We developed a novel approach to measure visible wavelength-induced autofluorescence up to energy for DAPI imaging. In addition, a time-dependent initial increase and then reduction in redox activity and formation of stress fibers occurred. This correlates with time-related increased in fluorescence as starting “driving force”, followed by second raised fluorescence as “execution phase”. Loss of cellular lipids and nuclear uptake of DAPI first and then propidium iodide prior to death were also observed. The entire course lasts 24-48 hr at room temperature. However, sphere explosion is accelerated dramatically by blebbistatin, an inhibitor of myosin II, suggesting the participation of cytoskeletal proteins in the sphere explosion.

**Keywords:** Bubbling Cell Death; Cancer Stem Cell Sphere; Explosion; Autofluorescence

**Acknowledgement:** MOST and NHRI, Taiwan.

# REGULAR SESSIONS

## Id-294

### Genetically Encoded FRET Biosensors in Cell Migration Biomechanical Studies

Z. BASTER[1*], O. ADAMCZYK[1], L. HODGSON[2], Z. RAJFUR[1]

[1]Department of Molecular and Interfacial Biophysics, Institute of Physics, Jagiellonian University, 11 Lojasiewicza Street, Cracow, Poland

[3]Department of Anatomy and Structural Biology, Albert Einstein College of Medicine, Bronx, NY 10461; Gruss-Lipper Biophotonics Center, Albert Einstein College of Medicine, Bronx, NY 10461, USA

Corresponding author: zbigniew.baster@doctoral.uj.edu.pl

**Abstract:** Cell migration and protrusion underlie many physiological processes such as embryological development, immune response and wound healing, and pathophysiological like tumor metastasis. Therefore, understanding the molecular mechanisms behind these processes is a key goal in cell biology. Coordination of all of the stages of cellular movement requires a vast amount of signaling pathways that cross-over at many levels. However, by employing the latest optical microscopy techniques it is possible to reveal mechanisms underlying cell migration, what may contribute to discovery of targets and development of new therapies in the future. Our studies concentrate around small G protein of Rho family, responsible for regulation cell migration and adhesion. We are especially interested in two of them RhoA and Rac1. Activity of RhoA is connected with cell contractility, especially in the cells tail region, but also plays an important role in cell protrusion. Rac1, on the other hand, mediate cell spreading and adhesion stabilization. With using genetically encoded FRET (Förster Resonance Energy Transfer) we are able to monitor proteins activity within a living cell, what no other method can provide. Currently, most cellular research is conducted on stiff glass or plastic substrates. These kind of substrates do not represent well mechanic properties of a human body, as we are created neither from glass nor from plastic. In case of elasticity only bones have similar stiffness. To mimic natural cellular mechanical environment, during experiments we culture our cells on elastic polyacrylamide substrates. Our studies showed considerable differences in cell migration parameters, when they are plated on soft substrate. Our goal is to characterize biochemical differences within cells that drive those changes. The presented work concentrates on differences in spatiotemporal activity of small G protein that mediate cell migration. In our research, beside sophisticated tools of cellular and molecular biology, we employ the latest methods of modern optical microscopy. For example, Fluorescence Lifetime Imaging Microscopy (FLIM) allows us to simplify FRET experiments and gain better resolution, whereas radiometric FRET provides us with better signal-to-noise ratio. This presentation will demonstrate the methodology of conducting FRET experiments using intramolecular genetically encoded biosensors. Furthermore, we present differences RhoA and Rac1 family activity due to the impact of substrate elasticity.

**Keywords:** FRET, biosensors, cell migration, elastic substrates, RhoA, Rac1

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# REGULAR SESSIONS

## Id-306

### Applications of Infrared Microspectroscopy in Basic Science and Disease

S. KUMAR[1,2,3*] ,M. THAM[4] ,B. POPESCU[4] ,E. GOORMAGHTIGH [5] ,F. NIKOLAJEFF[2]
[1]All India Institute of Medical Sciences, New Delhi, India
[2]Department of Engineering Science, Uppsala University, Sweden
[3]Canadian light source, Saskatoon, Canada
[4]Cameco MS Neuroscience Research Centre, University of Saskatchewan, Saskatoon, Canada
[5] SFMB, Université Libre de Bruxelles, Belgium
Corresponding author: sarojgupta.k@gmail.com

**Abstract:** Fourier transform infrared (FTIR) spectra accounts not only for the chemical nature of cell molecules, but also for their conformations and are, in particular, very sensitive to protein secondary structure. FTIR microspectroscopy have a histo-chemical correlation of the disease lesion as it can detect chemical changes arising from morphological changes of proteins, lipids, nuclei acids and carbohydrates in biological samples. This technique has been used to diagnose and differentiate the molecular differences between normal and diseased tissues. Here I am presenting my work with the FTIR imaging technique for understanding the disease pathology (breast cancer, multiple sclerosis, Ischemic stroke). Formalin-fixed, paraffin-embedded 5 μm-thick tissue sections of Breast cancer biopsy tssue and Multiple sclerosis autopsy tissue were stained histochemically and unstained adjacent tissue was subjected to FTIR imaging. 5 μm-thick sections were collected on CaF2 slides and FT-IR spectra were acquired using a Hyperion 3000 FTIR imaging system at the Canadian LightSource Using infrared imaging together with multivariate statistical tools we attempted to in a first step to discriminate the reactive stroma in contact with the diseased epithelium on one side and the normal stroma on the other side. A subsequent analysis of the spectra assigned to collagen was then performed to identify any potential influence of the tumors. An initial work on 8 patients with different tumor grades has shown promising findings [1]. In a second step we collected infrared spectra of fibroblasts growing in culture on $CaF_2$ windows in order to evaluate the impact of the different shapes encountered in fibroblasts. This investigation shows a significant separation could though be observed that the fibroblasts stimulated by these cancer cell lines grouped together and remained distinctly separated from normal fibroblasts indicating a modified different chemical composition in the cancer-stimulated fibroblasts [2, 3]. Demyelinated active MS lesions had prominent lipid oxidation, protein aggregation, phosphate reduction, and lower levels of sulfur and phosphorous, compared to the non-demyelinated tissue. Initial observations suggest there is a profound pathological heterogeneity in active MS lesions and proposes that tissue injury through metal dysregulation and oxidative damage have an important role in the pathogenesis and evolution of lesions.

**Keywords:** Infrared Imaging, Breast Cancer, Multiple Sclerosis, Diagnosis

**Acknowledgement:** We are grateful to AIIMS Intramural, VINNOVA-VINNMER, Brain back to Brussels and Swedish research for postdoctoral fellowships.

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# REGULAR SESSIONS

## Id-336

### Clinical Implications of Microscopy and Computer Image Analysis of the Enteroendoctine Cells in Patients with Irritable Bowel Syndrome

M. EL-SALHY

Section of Gastroenterology, Department of Medicine, Stord Hospital, and Department of Clinical Medicine, University of Bergen, Norway

Corresponding author: Magdy.elsalhy@sklbb.no

**Abstract:** Irritable bowel syndrome (IBS) is a common gastrointestinal disorderwhere the pathophysiology of which is not completely known. The gastrointestinal tract contains endocrine cells, which secret at least 15 different hormones. These cells are spread among the epithelial cells of the mucosa and constitute about 1% of all epithelial cells the gut. The hormones secreted by these cells regulate several functions of the gastrointestinal tract such as gut sensation, motility, secretion, absorption, local immune defense and appetite. These cells interact and integrate with each other and with enteric nervous system. IBS patients exhibit visceral hypersensitivity, disturbed gastrointestinal motility, and abnormal gut secretion. The density of serotonin producing cells in all the gastrointestinal segments of IBS patients did not differ from that of healthy individuals. However, the densities of serotonin transport receptor (SERT) are increased in the ileum and reduced in the rectum of these patients. A reduction in SERT results in impaired intracellular uptake and degradation in the gut epithelial cells, consequently increasing the availability of serotonin within the gut mucosa. Considering that the serotonin cell density in IBS does not differ from that of a healthy subject, a decrease in SERT would markedly increase the amount of serotonin available at its receptors. An increase in serotonin at the 5-hydroxytryptamine $(5\text{-HT})_3$ receptors of the ENS sensory neurons would activate the sensory nerves, which would then transmit nociceptive information to the nervous system resulting in visceral hypersensitivity. The abnormalities in the densities of ghrelin, cholecystokinin (CCK), secretin, and peptide yy (PYY) cells can explain the dysmotility seen in the gastrointestinal tract of IBS patients. Furthermore, the abnormalities in the densities of CCK, secretin and PYY can explain the abnormal secretion observed in IBS patients. Computer image analysis of immunostained section of the gastrointestinal tract of patients with IBS provided evidence for an anatomic defect in IBS patients, namely the gastrointestinal endocrine cells. This defect is most likely to be one of the factors contributing to the pathophysiology of IBS.

**Keywords:** Endocrine Cells, IBS, Immunohistochemistry, Computer Image Analysis, Gut

# REGULAR SESSIONS

## Id-356

### *In-Situ* Real-Time Study of Libr, Lioh and $Li_4Br(OH)_3$ at Microscale

P. LEGROS*[1], E. LEBRAUD[2], S. PECHEV[2], J. TOUTAIN[3], F. ACHCHAQ[4].
[1]CNRS / PLACAMAT, 87 Avenue du Docteur Schweitzer - 33608 Pessac Cedex France
[2]CNRS / ICMCB, 87 Avenue du Docteur Schweitzer - 33608 Pessac Cedex, France
[3]INP Bordeaux, I2M-site ENSCBP UMR CNRS 5295, 16 avenue Pey-Berland 33607 PESSAC Cedex, France
[4]Université de Bordeaux, I2M-site ENSAM UMR CNRS 5295, Esplanade des Arts et Métiers, 33 405 Talence, France
Corresponding author: philippe.legros@placamat.cnrs.fr

**Abstract.** This preliminary study is performed in the framework of Pc2TES project dealing with the development of new thermal energy storage materials for temperatures varying from 300 to 600°C. This temperature range allows covering a wide spectrum of significant applications such as thermal power plants integrating renewable power plants, concentrated solar plants or mobile applications in the aerospace and automotive fields for instance. $Li_4Br(OH)_3$ peritectic compound has been identified as one of the most promising heat storage material in terms of energy density and of working temperature (~800 J/g ; 304°C). However, the peritectic microstructure development is in general much less understood than single-phase or eutectic solidification processes. The level of comprehension drops more drastically about ionic alloys such as LiBr/LiOH binary systems. Besides, $Li_4Br(OH)_3$ performances depend strongly on a complex interplay between the initial composition, the imposed temperature gradients, the operating surrounding atmosphere and the solid fraction growth velocity. As a consequence, $Li_4Br(OH)_3$ heat energy storage potential may be strongly affected.The objective of this work is hence to participate to an in-depth understanding of $Li_4Br(OH)_3$ peritectic compound formation process by establishing the critical links between the processing conditions and the final microstructure formation.To do so, the influence of working parameters such as the nature of the surrounding atmosphere, the cooling rates and the thermal gradients is studied by using a hot stage placed inside the ESEM chamber. First, the compositions of the initial compounds LiBr and LiOH and of the peritectic one are checked by using the X-ray powder diffraction technique. The material morphologies are then observed for three levels of vacuum applied inside the ESEM chamber. Finally, their morphology evolution is monitored for different applied heating/cooling temperature protocols for each vacuum level.

The first results of LiBr/LiOH binary systems studied *in-situ* and real-time at microscale are presented and discussed. As expected, the melting/solidification temperature values of the studied materials are different from the theoretical ones assessed, at ambient pressure, in calculated LiBr/LiOH phase diagram as well from the experimental values obtained by using the differential scanning calorimeter.

**Keywords:** Microscopy; Ionic Alloy Microstructure; Peritectic; Energy Storage; Libr/Lioh

# REGULAR SESSIONS

## Id-357

### Application of Fourier Transfom Infrared (FTIR) Imaging to Depict Structural Alterations in Nerve Injury and Neuropathy

O. BOZKURT-GIRIT
Department of Biophysics, School of Medicine, Aydın Adnan Menderes University, Aydın, Turkey
Corresponding author: ozlem.bozkurt@adu.edu.tr

**Abstract:** Peripheral neuropathy, an outcome of damage to peripheral nerves, often causes weakness, numbness and pain in extremities. Peripheral neuropathy can be triggered by a number of conditions including chemotherapeutic drugs and diabetes. Diabetes results in hyperglycemia, which in turn leads to the production of reactive oxygen species and advanced glycation end products, and eventually to nerve damage. Spinal cord injury (SCI) results from injuries to the spinal cord by traumas and produces neurological deficits, and thus significantly affect the lifestyle of the subjects as well as their families. Although the pathology of neuropathy and nerve injuries have been described, little is known on the diabetes and trauma-induced structural alterations in biomolecules of nervous tissue. Accordingly, this study was conducted to identify the structural alterations in biomolecules of sciatic nerves and spinal cords in diabetic neuropathy and SCI, respectively. The structural alterations were depicted via Fourier transform infrared (FTIR) imaging, which allows the rapid and sensitive characterization of the sample based on macromolecular structure and composition (1-2). Adult male Wistar rats were used as models of SCI and diabetic neuropathy. Diabetes was induced by a single intraperitoneal injection of STZ (50mg/kg), and after injection rats were maintained for five weeks for the generation of diabetic neuropathy model. Spinal cord contusion injury was generated via a computer-controlled trauma instrument at T10 level. Dissected sciatic nerve and spinal cord sections were obtained by a cryotome and placed onto $BaF_2$ windows for FTIR imaging studies. FTIR images were analyzed using Isys 3.0 software and band area ratios were considered in data analysis. The results demonstrated that diabetic neuropathy led to an increase in ester containing lipids and lipids without phosphate groups. There was an increase in the carbonyl and ethyl group amount in lipids in diabetic neuropathy. Moreover, the amount of lipid degradation was decreased in diabetic neuropathy resulting in longer hydrocarbon chains in lipid structure. There was an increase in the unsaturated lipid content and an increase in unsaturated/saturated lipid ratio (unsaturation index) in diabetic neuropathy. On the contrary, SCI led to an increase in the amount of phosphate containing lipids with a decrease in ester containing lipids and resulted in an increase in the content of shorter lipid acyl chains in comparison to laminectomy group. There was a decrease of the ethyl group amount in lipids and a decrease in the unsaturation index of lipids in SCI. In conclusion, structural alterations in biomolecules, especially lipids, in diabetic neuropathy and SCI were unveiled by FTIR imaging. The ability of the biomolecules to perform complex biological tasks depends on their three-dimensional structure determining their functional properties. Therefore, in order to describe the underlying mechanisms of diseases, it is important to identify the macromolecular interactions and structure-function relationships.

**Keywords:** Diabetic neuropathy, sciatic nerves, spinal cord injury, FTIR imaging, infrared microspectroscopy, structural alterations

**Acknowledgement:**This study was funded by Scientific Research Council of Adnan Menderes University (TPF-17039, TPF-17041).

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# REGULAR SESSIONS

## Id-389

### Scalable Multifunctional Ultra-Thin Graphite Sponge

M. OZKAN
University of California, Riverside
Corresponding author: mihri@ece.ucr.edu

**Abstract:** Over the past decade, alarming consumption of earth's resources and subsequent environmental pollution has raised major concerns. Frequent oil spills and oil tanker incidents which resulted in catastrophic consequences for marine wild life and ecosystems are evident instances and whistleblowers. To prevent further damage to the environment, novel materials and methods for immediate decontamination and purification of water as well as sustaining the scarce natural resources are most favored. Graphitic sponge materials are developed with superporous and superhydrophobic properties. The hierarchical network material can be used to clean up different types of oil, organic solvents, toxic and corrosive contaminants. I will describe a flexible chemistry scheme capable of synthesizing porous carbon scaffolds embedded with metal particles. An analysis of the growth mechanism of porous carbon structures by esterification of simple polysaccharides at low pH will be discussed. The presence of Fe nitrate during the oxidative process nucleates metal particles when annealed under a reducing atmosphere and produces an embedded Fe nanoparticle within a porous carbon matrix encapsulated by a graphitic layer. The study of these mechanisms was then applied to other metal nitrate systems which produced corresponding metal particles embedded within a porous carbon matrix. A potential mechanism by which graphitic encapsulation takes is suggested by comparison to phase separation during the growth of carbon nanotubes from catalytic metal nanoparticles. This flexible method for synthesizing porous carbon scaffolds with embedding metal nanoparticles is shown to be achievable using cheap, environmentally benign precursors which can be used for future applications toward electrochemical capacitors, oil sorption and spill recovery, and bactericidal coatings. Brief Biography: Prof. Mihri Ozkan is a faculty member in the Department of Electrical and Computer Engineering at UC-Riverside. She received her Ph.D. degree in Electrical and Computer Engineering at UC-San Diego. She is “the Climate Action Champion Professor” of UC-Riverside. She believes that adaptation of electric vehicles by 2040 depends on improvements on affordability, battery storage capacity, and battery durability. In addition, she states that this requires breakthroughs in raw materials, new battery technologies, material processing and battery manufacturing. Ozkan aims to reduce the cost of anode and cathode raw materials, use new robust and safer battery architectures and achieve performance increase in Li-ion batteries by using renewable and recyclable resources. Her research is featured many times in popular news outlets such as Forbes, Guardian, New York Times, Time, BBC, TBS, Discovery News, Popular Science, Huffington Post and more. She published about 300 technical papers, 79 patent disclosures, and has 15 patents licensed by the industry. She received national and international honors including; “the National Medal for Engineering Science Award” from the Society of

Engineering Science, “the Frontiers of Engineering Honor” by the National Academy of Engineering, and “the Emerging Scholar Award” by the American Association of University Women.

**Keywords:** Environmental pollution, purification of water, superhydrophobic

# POSTER SESSIONS

## Id-230

### Radiation Induced Effects in Highly Stressed Nanocrystalline ZrC and ZrN Thin Films

O. R. VASILE[1], B. S. VASILE[1*], D. CRACIUN[2], G. DORCIOMAN[2], P. GAROI[2], V. CRACIUN[2]

[1]Faculty of Appl. Chemistry and Mater. Science, National Research Center for Micro and Nanomaterials Univ. Politehnica Bucharest, Romania,

[2]National Institute for Lasers, Plasma and Radiation Physics, Magurele, Romania

Corresponding author: bogdan.vasile@upb.ro

**Abstract:** ZrC and ZrN films have many important applications in advanced nuclear reactors, plasma fusion installations or space exploration, where there are strong radiation fields. It has been recently reported that nanostructured thin films, which have grain sizes usually smaller than 30 nm, behaved differently under irradiation than polycrystalline or single crystal films and materials. The high density of surfaces and grain borders allows for very short diffusion paths of irradiation generated vacancies and interstitials towards grain boundary regions that act as sinks. Therefore, it is expected that the structure and properties of such thin films are less affected by exposure to radiation than large grain films. Also, dislocations could easily move out of these small crystal grains, which will affect the mechanical properties of irradiated films. We investigated by high resolution transmission electron microscopy the radiation effects on the microstructure structure of Pulsed Laser Deposition (PLD) grown ZrC and ZrN thin films. Films were irradiated by 800 keV Ar and 1.0 MeV and 1.5 MeV Au ions up to fluences of $10^{15}$ /cm2. Complementary investigation techniques such as X-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectrometry, and X-ray diffraction techniques were used to obtain a more complete picture of the irradiation effects. The results confirmed that nanocrystalline films could withstand high irradiation fluences without degrading their crystalline microstructure or changing composition.

**Keywords:** ZrC, ZrN, nuclear reactors, PLD, XPS

# POSTER SESSIONS

## Id-251

### Polyhydroxyoctanoate (PHO) – biopolymer creating new opportunities for medicine and biomedical sciences

T. WITKO[1*], M. GUZIK[2], D. SOLARZ[1], Z. RAJFUR[1]

[1]Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science, Jagiellonian University, Łojasiewicza 11, 30-348, Kraków, Poland

[2]Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Niezapominajek 8, 30-239 Krakow, Poland

Corresponding author: tomasz.witko@gmail.com

**Abstract:** Biopolymers represent one of the leading sectors for bio-based products and their expected growth is foreseen to be significant within the next years. Polyhydroxyalkanoates (PHAs) represent a class of optically active biodegradable polyesters accumulated by numerous bacteria as discrete intracellular granules or as a net like extracellular structures. PHAs are non-toxic materials and biodegrade to harmless products in the environment. A research on physical and biological properties of polyhydroxyoctanoate (PHO) polymer and comparison with currently widely applied and studied polylactic acid (PLA) and glass was conducted. In order to characterize the influence of different solvents used for biomaterial preparations (solvent casting of PHO films) three solvents, namely ethyl acetate, acetone and chloroform, were used to prepare thin polymer films. PHO fermentation was thoroughly characterized by a range of physiochemical testes (AFM, IR, PAS, etc.). Mechanical tests included nanoindentation and scratch testing of the material, the obtained results were compared with AFM data. The Young Modulus values are consistent for both methods and comply with literature. Cells vitality and behavior on different substrates were assessed to confirm unambiguously the material's suitability in biomedical applications. Tests based on double staining with propidium iodide (PI) and fluorescent diacetate (FDA) were conducted for cytotoxicity assessment. Long-term microscopic studies revealed high biocompatibility level of polymer and no toxic impact on examined cells. For further cellular studies advanced microscopic methods like confocal and fluorescent techniques were employed. A detailed 3D reconstruction and image deconvolution of cell shape and cytoskeleton morphology was performed. PHO substrate enables healthy proliferation and migration of fibroblasts. The cell vitality rates are comparable with other substrates. The advanced scientific methods used to examine a new biomaterial's usefulness in medical applications can contribute both to significant advances in biopolymer research and bring benefits in medical field by enabling the use of previously unavailable or strongly restricted techniques whose application was limited by the properties of materials available in therapies.

**Keywords:** Polyhydroxyalkanoates, Polyhydroxyoctanoate, Cellular Studies, Microscopy, Biopolymers, Medical Applications

# POSTER SESSIONS

## Id-254

### Advanced Cellular Studies of Novel Biodegradable Biopolymers – Polyhydroxyalkanoates (Phas)

D. SOLARZ[1*], T. WITKO[1], M. GUZIK[2], Z. RAJFUR[1]

[1]Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science, Jagiellonian University, Łojasiewicza 11, 30-348, Kraków, Poland

[2]Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Niezapominajek 8, 30-239 Krakow, Poland

Corresponding author: daria.solarz93@gmail.com

**Abstract:** The main goal of conducting scientific research at the cellular level is to study the molecular mechanisms governing the wellbeing of entire organism. A new trend in science is to search for biodegradable and biocompatible biomaterials in order to create innovative biomedical products such as wound dressings, scaffolds or endoprotheses coatings with controlled drug release.

Biopolymers represent one of the leading sectors for bio-based products and their expected growth is foreseen to be significant within the next years. Polyhydroxyalkanoates (PHAs) represent a class of optically active biodegradable polyesters accumulated by numerous bacteria as discrete intracellular granules or as a net like extracellular structures. PHAs are non-toxic materials and biodegrade to harmless products in the environment. The conducted research has shown that biopolymers such as polylactide (PLA) and poly (3-hydroxyoctanate) (PHO) favor cells growth and are not harmful for them, which can play an important role in medical purpose products. These biomaterials not only have favorable effect on the cells but their surface can be easily functionalized with different biomolecules and drugs. This important feature of the material allows the supplementation of dressings with antibiotics or other substances supporting wound healing or tissue regeneration. During research the cells grown on a substrate with high Young modulus value like glass or plastic versus cells grown on plastic biopolymers like PHAs was compared. Cytotoxicity assessment performed on cells cultured on polyhydroxyalkanoate substrate indicate that the survival rate is similar to that of cultures carried out on glass substrates. Cells grown on glass are characterized by a well-organized actin and microtubular filament network, as well as numerous, well defined focal adhesion points. Cells grown on polyhydroxyalkanoates show much more extensive, denser network of cytoskeleton filaments, which simultaneously are much thinner than those observed in cells grown on glass. An analysis of morphology changes including cytoskeleton dynamics was performed using confocal and fluorescent microscopy techniques. Application of advanced microscopic techniques allows imaging of the material in three dimensions, which gives additional information about the spatial structure of the sample. The combination of advanced research methods with new unexamined materials gives the opportunity to achieve revolutionary results and can contribute to significant advances in research on biopolymers.

**Keywords:** Phas, Polyhydroxyalkanoates, Biopolymers, Microscopy, Confocal Microscopy, Mouse Ebryonic Cells, MEF Cells

# POSTER SESSIONS

## Id-257

### Study on Effect Of Zr Doping on The Electrical Properties of Batio$_3$ Thin Film Resistive Switching Devices

N. SAMARDZIC[1], M. NEDELJKOVIC[1], J. VUKMIROVIC[2],
V. SRDIC[2], G. STOJANOVIC[2]
[1]Faculty of Technical Sciences, University of Novi Sad, Novi Sad, Serbia
[2]Faculty of Technology, University of Novi Sad, Novi Sad, Serbia
Corresponding author: nsamardzic@uns.ac.rs.

**Abstract:** Resistive switching devices (memristors) are basic nonlinear passive electronic component with both ability to preserve and change electronic state depending on electrical, temperature or photonic stimuli. These devices are extensively studied in the last 10 years due to intensive research in the field of nanoelectronic, nanomaterials and neuromorphic computing. Typical structure of memristive device is Metal-Insulator-Metal (MIM) where active nanomaterial (insulator or semiconductor) placed between two electrodes highly influences the properties of overall device. Here, we present electrical properties of thin film memristors with active $BaTiO_3$ nanomateral, deposited using spin-coating technique on platinized Si wafer. In order to improve OFF-to-ON state ratio and device dissipation we introduce Zr dopants in the pure $BaTiO_3$ film obtaining $BaZr_{0.1}Ti_{0.9}O_3$ and $BaZr_{0.2}Ti_{0.8}O_3$ structures. We compared current-voltage characteristics, retention time, repeatability, bipolar and unipolar response as well as switching dynamics of these devices in order to find optimal fabrication processing conditions to realize reliable device performance. Transport parameter measurement and impedance spectroscopy measurement of all samples were also performed, revealing the major carrier type and concentration, mobility as well as grains and grain boundary influence on electrical properties of devices. Conduction mechanisms were connected with thermionic emission and Fowler–Nordheim tunnelling and interplay between ionic and electronic conduction in the case of doped samples. All samples exhibit reproducible and stable electronic response, while metal-$BaZr_{0.2}Ti_{0.8}O_3$-metal devices show improved OFF-to-ON state ratio (~20) and good symmetry of hysteresis loop indicating low dissipation.

**Keywords:** Resistive Switching Devices, Memristors, Electrical Characteristics, Impedance Spectroscopy

**Acknowledgments:** This research was supported by the Ministry of Education, Science and Technological Development, Republic of Serbia under Project III-45021 and by the Provincial Secretariat for Science and Technological Development of the Government of Vojvodina (Project No 114-451-1745/2016-3).

# POSTER SESSIONS

## Id-260

### Application of the Energy Dispersive X-Ray Spectroscopy to the Studies of Materials of Cultural Heritage from The Collection of the State Historical Museum

O.B. LANTRATOVA[1]L. D. ISKHAKOVA[2*], A.V. SHULININA[1]
[1]Moscow, Red square 1, 109012, Russia, State Historical Museum,
[2]Moscow, Vavilov str. 38, 119333, Russia, Fiber Optics Research Center of Russian Academy of Science,
Corresponding author: Ldisk@fo.gpi.ru

**Abstract:** The State Historical Museum performs systematic studies of materials of cultural heritage from it`s collections by using energy dispersive X-ray spectroscopy (EDXS). This method is non-destructive, fast, universal, multi-elemental and sensitive. The objects of study are paintings (icons, oil painting, watercolors, etc.), metal artifacts, textiles, glass, ceramics, wood, bone, documents on paper basis. The purpose of research is to determine the chemical composition, which is often necessary to establish the provenance of the objects, the choice of restoration technique, for the reconstruction of the author's intention. Gilt threads microstructure and speciation characteristics clarification allowed us to reveal the technological features of medieval golden threads on an organic basis (paper, leather, intestinal membrane) manufacturing in Chinese silk fabrics. The study of gold threads from brocade of three XVII century chasubla gave us the opportunity to identify their threads types and to find out some of the features of textile production. The obtained data on the ratio of gold, silver and copper in threads composition can be used in the attribution of tissues and determining their place of origin (Eastern and European fabrics). The composition of natural and artificial mineral pigments (cinnabar, red lead, ocher, whitewash, Berlin blue, etc.) in paintings have been determined on the basis of elemental analysis for the content of Cu, Fe, Cr, Co, Zn, Pb, Hg, Ni, Ti, Ca, P, Hg, S, Se, C, N, O, etc, for example: The study of the paint layer on the Peter I intravital repousse copper work during restoration process allowed to reconstruct the authors color scheme. By the presence of traces of silver on most paint samples, it can be assumed that the copper relief has been silver plated. Currently, the silver coating is almost lost. It was assumed that the black filling in the grooves of the bone box-shoe thread is contamination. However, an elemental analysis of its speciation showed that it is a mixture of pigment (bone-black) with wax. Thanks to the obtained results, this handicraft was saved black “graphic” decoration from removal. Archaeological finds from excavations Gnezdovo were studied. The analysis of the composition of metallic thread figured ribbon of gold-plated silver wire is important for culture and trade relations of the Slavs, the Baltic and Scandinavian peoples study. A fragment of a wooden shield with a white powder on it`s surface was found in the same grave. According to EDXS and x-ray phase analysis, this powder is lead phosphate $Pb_3(PO_4)_2$. Its origin and destination is to be determined.

**Keywords:** EDXS, Gilt Threads, Pigment, Archaeological Find

# Id-261

## Determination of Chemical and Phase Composition of (Co, Mg)-Substituted Strontium Hexaferrites Single Crystals by Using Scanning Electron Microscopy, Energy Dispersive X-Ray Spectroscopy and X-Ray Powder Diffraction

L. D. ISKHAKOVA[1*], S. V. LAVRISHCHEV[1], A. M. BALBASHOV[2]
[1]Vavilov str. 38, Moscow 119333, Russia, Fiber Optics Research Center of Russian Academy of Sciences, ,
[2]Krasnokazarmennaya 14, Moscow 111250, Russia, Moscow power engineering institute,
Corresponding author: Ldisk@fo.gpi.ru

**Abstract:** M-type ferrite $SrFe_{12}O_{19}$ and substituted materials are widely used as permanent magnets. Until now $SrCo_xFe_{12-x}O_{19}$ were prepared as polycrystalline samples by different methods of synthesis. The aim of this work was attempt to grow single crystals of (Co, Mg)-codoped ferrites $Sr(Co,Mg)_xFe_{12-x}O_{19}$ ($0<x<1$). In the studied series of samples, designated as I, II, IV, V and VI, the value of **x** was equal to 0.1, 0.2, 0.3, 0.4, 0.8 and 1.0 respectively. The single crystals have been grown by crucibles floating zone (FZ) melting method with light heating at a rate 2-5 mm/h under 50 atm $O_2$ partial pressure. Microstructure of the samples was investigated using scanning electron microscope (SEM) in Z-contrast back-scattered electrons mode. Determination of the chemical composition was carried out with the help of the energy-dispersive X-ray spectroscopy (EDXS). X-ray analysis (XRD) of the samples was conducted with CuKα radiation; step time 6s; step size 0.02°; 10-125° 2θ. According to XRD and SEM data single-phase M-type crystals can be grown in the range of **x** values from 0.1 to 0.3 by varying the crystal growth rate in the range of 2-5 mm/h. When growing crystals with $x \geq 0.4$ at a rate of 5 mm/h, a mixed-phase area appeared in the crystals, in which, in addition to the M-type phases, the W-type $SrCo_2Fe_{16}O_{27}$ and X-type $Sr_2Co_2Fe_{28}O_{46}$ phases were detected using both the EDXS and XRD. The refined parameters of their unit cells were **a**=5.895(1) Å; **c**=32.746(5) Å (sp.gr. $P6_3/mmc$, W-type) and **a**=5.905(2); **c**=83.695(18) Å (sp.gr. R-3m, X-type). In this connection experiments on the growth of crystals with a value of **x** from ≥0.4 (series IV, V, and VI, respectively) were performed at a rate of 2 mm/h. Thus, single crystals of M-type were grown in IV series of experiment at a given growth rate. The refined parameters of their unit cells were **a**=5.851(1) Å; **c**=23.60(1)) Å, sp.gr. $P6_3/mmc$ and its composition corresponds to stoichiometry $Sr_{1-y}Fe_{12-x}Co_xMg_{0.1}O_{19\pm\delta}$, where $x = 0.041$ and $y = 0.05$. It was established in experiments series V and IV that increase of cobalt content in initial charges leads to the growing of W-type ferrites with lattice parameters for series VI **crystal a**=5.9026(9) Å; **c**=32.791(4) Å (). The changing crystals stoichiometry from M-type to W-type was the result of emergence second phase $Sr_4Fe_6O_{13}$. This phase arises both on the crystal surface as a layer in 50-70 μm thick, and mainly on the end of the crystal. There are no data on phase relations in the system $CoO-SrO-Fe_2O_3$ at high oxygen pressures in the literature. Perhaps in this diagram, W-type ferrite has a wider area of existence in comparison with that in pseudo-binary system $SrO-Fe_2O_3$ [1]. Thereby FZ melting method allow to grow M- and W-type of (Co, Mg)- substituted strontium hexaferrites and the combination of SEM, EDXS and XRD allowed to describe in detail elemental and phase composition the single crystals.

**Keywords:** SEM, EDXS, hexaferrite, crystal growth, M-type, W-type

**Rerefences**

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# POSTER SESSIONS

## Id-270

### Spectroscopic Characterization of Yttrium Orthoferrite Nanoparticles Prepared by Mechanochemical Assisted Synthesis

DALIBOR L. SEKULIC[1*], ZORICA Z. LAZAREVIC[2], NEBOJSA Z. ROMCEVIC[2]
[1]Faculty of Technical Sciences, University of Novi Sad, Novi Sad, Serbia
[2]Institute of Physics, University of Belgrade, Belgrade, Serbia
Corresponding author: dalsek@uns.ac.rs.

**Abstract:** Yttrium orthoferrite ($YFeO_3$) nanoparticles with an average size of about 12 nm were successfully synthesized by a mechanochemical treatment of high–purity yttrium(II)–oxide ($Y_2O_3$) and hematite ($\alpha$–$Fe_2O_3$) as initial precursors. The particle size and morphology of powders were studied using X–ray diffraction (XRD) and scanning electron microscopy (SEM). The formation of pure orthorhombic crystal structure was confirmed by the powder XRD analysis of prepared $YFeO_3$. 11 first–order Raman active modes are observed in Raman spectrum at room temperature, while infrared reflectivity spectrum is fitted by 10 modes. Over the frequency range of 100 Hz to 10 MHz, complex impedance spectroscopy was used to characterize the electrical properties of synthesized yttrium orthoferrite nanoparticles at and above room temperature. The obtained impedance spectra indicated that $YFeO_3$ sample exhibit semiconducting nature (NTCR–type behavior) and non–Debye type of relaxation phenomena. Further, analysis of impedance spectra by means of an equivalent circuit model revealed the presence of a single temperature dependent relaxation. It was found that the relaxation time decreases with temperature and follows Arrhenius dependence. Additionally, complex impedance analysis indicates the dominance of grain effects which control the overall electrical behavior of studied ferrite material.

**Keywords:** Orthoferrite Nanoparticles; Mechanochemical Synthesis; Impedance Spectroscopy; Semiconductor

**Acknowledgments:** This research was financially supported by Ministry of Education, Science and Technological Development of the Republic of Serbia through Projects No. III43008 and III45003.

# POSTER SESSIONS

## Id-271

### Quantitative Analysis of ROS Production Following Drought and Heat Co-Stress in Wheat (Triticum Aestivum L.) Stigmas

A. FÁBIÁN*, E. SÁFRÁN, G. SZABÓ-EITEL, B. BARNABÁS, K. JÄGER
Plant Cell Biology Department, Agricultural Institute, Centre for Agricultural Research, Hungarian Academy of Sciences, Hungary
Corresponding author: fabian.attila@agrar.mta.hu

**Abstract:** Heat and drought stress during reproductive development may seriously affect crop yields, which can be attributed especially to the high sensitivity to stress shown by pollen development. Compared to pollen dysfunction, the significance of the damage sustained by the physically better protected female reproductive cells and organs, which are less vulnerable to abiotic stress and resilient to water stress, is generally considered as a minor factor in yield loss, therefore less attention has been paid to the heat and drought sensitivity of their development. Both extreme high temperatures and water shortage lead to the excessive generation of reactive oxygen species (ROS) and reactive nitrogen species (RNS), which function as signal transduction molecules, but can also cause extensive cellular damage. As generative processes show significant vulnerability to heat and drought stress, it can be hypothesized that ROS and RNS play an important role in the reduction in fertility and in consequent yield loss. Our study confirmed that the stigmatic papillae of a monocotyledonous wheat generate high amounts of ROS ($O_2^{\bullet -}$, $OH^-$, $H_2O_2$) and RNS ($ONOO^-$, NO) at anthesis. A tolerant (Plainsman V) and a sensitive (Cappelle Desprez) variety was studied. The assessment of general cellular oxidative stress using $H_2$DCFDA indicated a good correlation with fertility loss, implying that the high level of oxidants detected in the stigmatic papillae was located in the top half of the spikes of the sensitive Cappelle Desprez variety. Mitochondrial and cytoplasmic $O_2^{\bullet -}$ generation were determined using MitoSOX Red and dihydroethydium, respectively. While amount of mitochondrial $O_2^{\bullet -}$ increased selectively in the top half of Cappelle Desprez, cytoplasmic concentration of the compound rose in both varieties. Hydrogen peroxide content of papilla cells determined by Ampliflu Red and DHR 123 elevated significantly only in the top half of Cappelle Desprez spikes. The amount of highly reactive oxygen species ($OH^-$ and $ONOO^-$, both assessed using APF) increased exclusively in treated Cappelle Desprez spikes. The amount of oxidants in the tolerant Plainsman V rose to a smaller extent, which did not lead to fertility loss. The amount of nitric oxide in stigma papilla cells decreased after combined heat and drought stress in the sensitive variety, suggesting that this compound promotes successful fertilization in wheat.

**Keywords:** CLSM, drought and heat co-stress, ROS, stigma, wheat

**Acknowledgement:** The work was financed by a grant from the Hungarian Scientific Research Fund (NKTH-OTKA K108644) and by grants from the Hungarian Academy of Sciences (GENPROF IF-18/2012, KEP-5/2016 and KEP-5/2017).

## Id-272

# Stigma Receptivity and Fertility Are Reduced by Heat and Drought Co-Stress in Wheat (Triticum Aestivum L.)

K. JÄGER*, E. SÁFRÁN, G. SZABÓ-EITEL, B. BARNABÁS, A. FÁBIÁN
Plant Cell Biology Department, Agricultural Institute, Centre for Agricultural Research, Hungarian Academy of Sciences, Hungary
Corresponding author: jager.katalin@agrar.mta.hu

**Abstract:** High temperature and drought often occur simultaneously during plant development causing severe yield loss in most wheat-growing areas. The isochronal effect of these two stresses on wheat may be unique, differing from that of the individual stresses, but there is a dearth of information on this topic. Structural and functional anomalies occurring as a consequence of environmental stress during reproductive processes have a serious influence on the success of fertilization and thus on yield production. Reproductive sterility is a major threat to global food security in a world subject to climate change. Pollen sterility has long been known as a major determinant of fertility loss under high temperature and water scarcity, however, the sensitivity of female reproductive tissues to simultaneous heat and drought (HD) stress is not well understood. Changes in the phenology, morphology and anatomy of female reproductive cells and organs, in the ROS and RNS generation of stigmatic papilla cells, and in fertility and yield components in response to simultaneous high temperature and drought at gametogenesis were studied in the drought-tolerant Plainsman V and the drought-sensitive Cappelle Desprez winter wheat varieties. The combination of high temperature (32/22°C) and total water withdrawal for 5 days under controlled conditions at gametogenesis altered the phenology and physiology of the wheat plants, reduced pollen viability, modified the morphology and the anatomy of the pistils, enhanced the generation of ROS and RNS, intensified lipid peroxidation and decreased the NO production of stigmatic papilla cells, all leading to reduced fertility and to yield loss in the sensitive Cappelle Desprez genotype, depending on the position of the floret on the spike. Reduced functionality of female and male reproductive parts accounted for 34% and 65%, respectively, of the total generative cell- and organ-triggered fertility loss. Our study demonstrates that damage to female reproductive organs also contributes to losses of fertility and yield. Addressing the morphological, anatomical, physiological and molecular mechanisms conferring sensitivity and tolerance to HD co-stress will help to develop wheat genotypes capable of adapting to a changing climate.

**Keywords:** Anatomy, Fertility, Heat and Drought Co-Stress, Morphology, RNS, ROS, Stigma, Wheat

**Acknowledgement:** The work was financed by a grant from the Hungarian Scientific Research Fund (NKTH-OTKA K108644) and by grants from the Hungarian Academy of Sciences (GENPROF IF-18/2012, KEP-5/2016 and KEP-5/2017).

# POSTER SESSIONS

## Id-275

### Microscopical Evaluation of KIT Receptor Expression in Canine Cutaneous Mast Cell Tumors (Cmcts) Without C-Kit Mutation

V. IVANA[1], N. SLAĐAN[1], A.-K. SANJA[2*]

Faculty of Veterinary Medicine University of Belgrade, Bulevar oslobodjenja 18, 11000 Belgrade, Serbia

Corresponding author: skovacevic@vet.bg.ac.rs

**Abstract:** Cutaneous mast cell tumors are one of the most common neoplasms in dogs. They show a highly variable biologic behavior and histopathological examination sometimes is not enough to predict the prognosis and to select a treatment plan. The presence of aberrant CD117 expression and mutations of the c-kit proto-oncogene could be an indicative parameter for final histological grading of cutaneous mast cell tumors in dogs. In addition to the histopathological examination, grading, immunohistochemical staining and molecular genetic examinations are the proposed criteria that should be used for classification. Determination of the connection between the localization of KIT receptor expression and the histological grade of CMCTs without c-kit proto-oncogene mutations was the main goal of this study. The study included 36 CMCTs and six control skin samples from 42 dogs of different ages, breed and sex. Formalin-fixed and paraffin-embedded tissue samples were stained with hematoxylin-eosin and toluidine blue and immunohistochemically tested for CD117 expression. DNA was extracted from the same paraffin blocks and subsequent polymerase chain reaction amplification was performed using PE1 and PE2 primers. The degree of malignancy was determined based on the presence of mitotic figures, multinucleated cells and bizarre nuclei in 10 high power fields. Based on histological features, twenty-six of 36 CMCTs were of a high histological grade, while ten were classified as a low-grade malignancy. CD117 cytoplasmic expression was observed in fourteen of 26 high-grade malignancy CMCTs, which confirms the link between the aberrant CD117 expression and increased cell proliferation.

**Keywords:** Dogs, Mast Cell Tumor, Classification, CD117

## Id-278

# Cellular Mechanotransduction Processes Studied with Optical Techniques

ADAMCZYK O*., BASTER Z., RAJFUR Z.
Prof. Stanisława Łojasiewicza Street, 30-348 Cracow, Poland
Department of Molecular and Interfacial Biophysics, Smoluchowski Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science, Jagiellonian University
Corresponding author: oliadamczyk@wp.pl

**Abstract:** Cell migration is a major process taking place in every living multicellular organism. Nowadays, molecular mechanisms, responsible for the regulation of migration are being intensively studied. Cultured adherent cells, are the most frequently employed model biological organisms used during these studies. Understanding the mechanisms responsible for the regulation of the cell migration process can help find new therapies which, for example accelerate wound healing process or prevent metastasis of cancer. Optical microscopy, especially confocal and wide field fluorescent microscopy, is one of the commonly used method in these studies. We employed another advanced optical microscopy technique- optical tweezers – in our studies. Thanks to the possibility of manipulating small objects, could be observed the response of cell to a mechanical factor and cell machanotransduction. Latex beads, functionalized with specific protein, can interact with proteins located in the cell membrane. The result of such interaction can significantly affect the structure of the cellular cytoskeleton. The response could depend on the type of cells or the length of time of interaction between the cell and a functionalized bead. The combination of two complementary methods, confocal microscopy and optical tweezers allows us to observe possible changes in the cell cytoskeleton when acting on it with a mechanical stimulus provided by the bead and optical tweezers.

**Keywords:** Confocal Microscopy, Optical Tweezers, Cytoskeleton

**Acknowledgement:** This work was partially funded by diCella Digital Image Cell Analysis sp. z o.o., Kraków, Poland

# POSTER SESSIONS

## Id-279

### Detection of Cellulose in *Acanthamoeba* Cyst Wall by Various Microscopic Techniques

M. MRVA[1*], M. M. GARAJOVÁ[1], N. VAŠKOVICOVÁ[2], A. VALIGUROVÁ[3]
[1]Department of Zoology, Faculty of Natural Sciences, Comenius University in Bratislava, Mlynská dolina Ilkovičova 6, 842 15 Bratislava, Slovak Republic;
[2]Institute of Scientific Instruments, CAS, Královopolská 147, 612 64 Brno, Czech Republic;
[3]Department of Botany and Zoology, Faculty of Science, Masaryk University, Kotlářská 2, 611 37 Brno, Czech
Corresponding author: martin.mrva@uniba.sk

**Abstract:** Pathogenic strains of free-living acanthamoebae are causative agents of serious diseases in humans and animals: granulomatous amoebic encephalitis (GAE), disseminated infections, and *Acanthamoeba* keratitis (AK). The life cycle of *Acanthamoeba* spp. comprises two stages: an active trophozoite and metabolically almost inactive cyst. A serious problem in the treatment of diseases represent *Acanthamoeba* cysts which are highly resistant to biocides thanks to the cyst wall organization. With the use of ultrastructural analysis, we acquired new and unique data on cysts and encystment in *Acanthamoeba* clinical isolate of T4 genotype and Group II. Interference contrast microscopy visualised the double-layered cyst wall and a high content of cellulose was detected by fluorescence microscopy. The freeze-etching technique demonstrated cellulose in both layers of the cyst wall and in the intercystic space as well. The ectocyst structure was heterogenous with scattered cellulose fibrils. The endocyst was characteristic with extensive quantity of crossed cellulose fibrils which represented the main structural material. In association with the cellulose synthesis, the clustering of intramembranous particles (IMP) was detected in the cytoplasmic membrane of enycysting cells. With the use of confocal laser scanning microscopy, we detected active microtubular networks in encysting cells for the first time and the activity of filamentous actin even in the late phases of encystment. This activity is probably linked with the organisation of the cyst wall. The development of new strategies against acanthamoebae focuses on the cyst wall and especially on the cellulose biosynthesis pathway. Its disruption would prevent the most important part of encystment which is the synthesis of the cyst wall. Our results were acquired with the use of various methods of light, confocal, and electron microscopy which in combination brought new and highly comprehensive data on the encystment process and cell biology of acanthamoebae.

**Keywords:** Acanthamoebae; Cyst Wall; Cellulose; Intramembranous Particles; Encystment; Cytoskeleton

**Acknowledgement:** This work was supported by the Slovak Research and Development Agency under the contract No. APVV-15-0123. Further, it was supported with grants VEGA 1/0365/16, VEGA 1/0389/19, and KEGA 039UK-4/2019. AV was funded by the Czech Science Foundation project No. GBP505/12/G112 (ECIP), and by the project 7AMB14SK008 from the MEYS CR. NV was supported by MEYS CR (LO1212), MEYS CR and EC (CZ.1.05/2.1.00/01.0017), and by CAS (RVO:68081731).

# POSTER SESSIONS

## Id-282

### Application of Polymer Substrates in Cell Migration Research

T. KOLODZIEJ, D. DZIOB, P. CYZIO, J. RACZKOWSKA, Z. RAJFUR.
[1]Jagiellonian University, Faculty of Physics, Astronomy and Applied Computer Science,
Lojasiewicza 11, 30-348 Krakow, Poland
Corresponding author: tomasz.kolodziej@doctoral.uj.edu.pl

**Abstract:** Cell migration is one of the most fundamental biological phenomena. It is inherently connected with the physiological processes in our body, like wound healing, embryonic development, and functioning of the immune system. It also plays an important role in pathological processes, such as tumor metastasis, arthritis and osteoporosis. Migration of cells strongly depends on signals from extracellular stimuli. Many different factors, such as chemicals in medium (chemotaxis), physical interactions – for example voltage (galvanotaxis); gradient of adhesiveness (haptotaxis), or elasticity of the substrata (durotaxis), may influence the cell movement. Adherent migrating cells exert forces on the substrate. Application of different materials as substrates for cell migration can modify basic biophysical parameters of migration, such as cell velocity and directional persistence. Presumably, the elasticity of the substrate can drive the cellular response. In this work we compared four different substrates: glass, polystyrene (PS), polydimethylsiloxane (PDMS) and polyacrylamide (PA) in cell migration assay. To study cell migration phenomena, epithelial fish keratocytes were used as model migrating cells. They were obtained from single Molly fish scales, according to the standard procedures. Fish keratocytes has been cultured on four different substrates: glass, plastic (polystyrene), polidimethylosiloxane (PDMS) and polyacrylamide (PA) of different elasticities. In both types of cells, time lapse of single migrating cell was recorded, and migration parameters were calculated from images. We observed differences in velocity and directional persistence distribution of migrating cells on different substrates. Keratocytes were migrating faster on a glass substrate, slightly slower on the PDMS, while the slowest cells were observed on plastic substrates. In the similar order, the width of directional persistence distribution was increasing. In case of polyacrylamide substrates, basic parameters of migration were dependent on substrate elasticity. It shows that the substrate used for observation and cell culture have significant impact on cell behaviour. It also presents that the material of well-defined chemical composition, but different elasticities (PA) may change cell behaviour, which shows the role of mechanical signals in cell homeostasis.

**Keywords:** Biopolymers, Cell Migration, Elastic Substrate, Time-Lapse Microscopy

**Acknowledgement:** This work was partially funded by diCella Digital Image Cell Analysis sp. z o.o., Kraków, Poland

## Id-296

# Surface Enhanced Raman Spectroscopy (SERS) Based on Zno Nanorods for Detecting Nanometer Sized Bio-Markers

S. LEE[1], Y. KİM[1], B. PAULSON[1], M. JUE [1], J. Kİ KİM[1,2*]

[1] Biomedical Engineering Research Center, Asan Medical Center, 88, Olympic-ro 43-gil, Songpa-gu, Seoul, 138-736, Republic of Korea

[2] Department of Convergence Medicine, University of Ulsan College of Medicine, 88, Olympic-ro 43-gil, Songpa-gu, Seoul, 138-736, Republic of Korea

Corresponding author: kim@amc.seoul.kr

**Abstract:** Raman spectroscopy has been studied for the verification of various biological samples, such as tissue, cells, bacteria and proteins. Among them, detection of high performance of nanometer-sized biomarkers is attracting much research interest due to its application for the early diagnosis of disease. For highly sensitive optical biopsy, we introduce an approach to Raman signal enhancement on biosensing chips based on surface enhancement Raman spectroscopy (SERS) diagnostics. Significant signal enhancement was realized by introducing a porous ZnO nanostructure for the bio-liquid sample. Furthermore, the coffee ring effect produced by the liquid sample disappeared. SERS occurs well on rough metal surfaces such as gold, and when gold is sputtered on the ZnO nanostructure, a unique characteristic is occured that the nanorod heads first grow to form clusters. This clustering was the main factor for enhancement of local surface plasmon resonance (LSPR), which could be verified by analysis of finite element method (FEM). Biocompatibility tests were performed for the Raman application of living cells such as the measurement of cell secretion using this assay chip, and the cells were well alive on nanorods and clusters of various sizes. ZnO nanowire substrate may be used as a biomedical chip with excellent potential for detecting nanometric biomarkers.

**Keywords:** Surface Enhanced Raman Spectroscopy, Zno Nanostructure, Gold Clustering, Finite Element Method (FEM), Cell Viability

## POSTER SESSIONS

## Id-297

# Application of Simple Method Utilizing Polarized Light and Histopathological Staining for the Quantitative Microidentification of Lignin and Cellulose in Wood Cell Walls During Advanced Decaying Process Caused by Fungi

V. RAČKO[a*], O. MIŠÍKOVÁ[b]

[a]Technical University Zvolen, Department of Wood Science, Zvolen, Slovakia,

[b]Technical University Zvolen, Department of Wood Science, Zvolen, Slovakia,

Correspondig author: racko@tuzvo.sk

**Abstract:** At present, there are many known approaches to detect the changes of the main chemical compounds of wooden tissues investigated at micro scale levels. However, the complexity of many procedures is very high. The objective of this work was to test a fast method their identification in different advanced stages of fungal degradation. The method is based on known principles; the loss birefringence of polarized light between two crossed filters, in the case of identification the cellulose losses with simultaneous use of multichromatic histological staining by Toluidine Blue O, in the case of the losses and changes of lignin content. The results confirmed that the method is suitable for localization of infection in tissues, identification of degraded cellulose and lignin in individual cell types and determination of strategies for decomposition of wood with different types of attacking pathogens (Brown-rot and white rot fungi). However, the method it is not suitable for a more detailed evaluation of cellulose in cell walls, due to the high image blur of polarized light in magnifications over 400x.

**Keywords:** Polarised light, Toluidine blue O, Cellulose, Lignin, Wood decay, Fungi

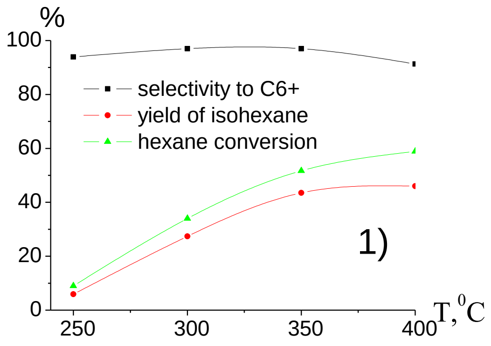
# POSTER SESSIONS

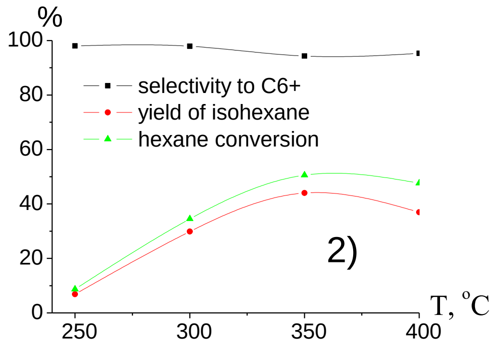
## Id-298

# Isomerization of N-Hexane on Pd- Catalysts on Pillared Aluminum-Zirconium Montmorillonite

L. DJUMABAYEVA*[2], N. ZAKARINA[1].
[1]D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Kunaev St. 142, 050010, Almaty, Republic of Kazakhstan, [2]The Kazakh National Research Technical University after K.I. Satpaev,
Satpaeva st. 22a, 050013, Almaty, Republic of Kazakhstan
Corresponding author: djumabaevals@mail.ru

**Abstract:** Catalysts based on pillared clays due to their high acidity show high activity and selectivity in the process of isomerization of n-paraffins [1, 2]. The strength of the acid centers can control by using different oxide phases to crosslink the layered aluminosilicates.In this work we synthesized Pd-catalysts, supported on columnar aluminum-zirconium Tagan (Kazakstan) montmorillonite (MM), were investigated their texture and catalytic properties in the isomerization of n-hexane, depending on content of the active metal and the temperature.In the synthesis of aluminum-zirconium pillared MM as fixing agents the hydroxocomplexes of aluminum and zirconium were used, the introduction of which into the interlayer spaces of montmorillonite was carried out according to known methods [3, 4]. In synthesized samples the $Zr^{4+}$ / clay and $Al^{3+}$/ clay ratio was 2.5 mmol / g clay. Palladium (0.35 wt%) and (0.1 wt%) were added to the pillared Al-Zr-montmorillonite by impregnation with a solution of $PdCl_2$ and a solution of Pd-sol which was prepared by the reduction of Pd (II) polyhydroxo complexes containing polyoxoanions Mo (VI). The texture characteristics of the samples were determined by the method of low-temperature adsorption of nitrogen (BET) using the ACCUSORB instrument. Acidity of the catalysts was determined by termodesorption of $NH_3$.The determination of Pd-particles sizes was carried out by high-resolution transmission electron microscopy (HRTEM) on a JEM 2100 instrument (JEOL).The activity of the samples in the isomerization of n-hexane was studied in a flow reactor with varying the temperature in the range 250-400°C.Analysis of products of the isomerization reaction was by gas chromatography.According to the BET analysis, the formation of the columnar Al-Zr-MM structure is characterized by the redistribution of pores in size, while the specific surface area of the samples and the total pore volume are practically do not change. The calculation of pore size distribution showed that





when Pd content is reduced from 0.35 to 0.1%, the amount of mesopores decreases from 77.3 to 72.7%, and the number of micropores increases from 22.6 to 27.2%. It was shown that the activity and selectivity of Pd catalysts in the isomerization of n-hexane change little with decreasing of palladium content at different temperatures (figure). Fig. Dependence of the yields of isohexanes, the selectivity to $C_{6+}$-isomers and the conversion of n-hexane on 0.35% Pd / AlZrNaHMM (1); 0.1% Pd / AlZrNaHMM (2) - catalysts at the temperatures 250-400$^0$C. The conversion of n-hexane increases with increasing temperature and reaches 58.9% at 400$^0$C on 0.35% Pd- catalyst and 50.6% on 0.1% Pd -catalyst at 350$^o$C. The selectivity for $C_{6+}$ isomers at 250-400° C remains high. The yield of dimethylbutane at 400$^o$C on 0.35% Pd- contact is 26.0%. When the palladium content is reduced to 0.1%, the yield of $C_6$ disubstituted isomers is 24.4%, at a temperature of 350$^0$C. At the same time, the yield of hydrocracking products does not exceed 0.3%. On a 0.35% Pd-catalyst at 400°C, the content of $C_6$ isomers is 46.0%, with a decrease of the palladium content to 0.1%, the isohexanes yield is 44.0% at a temperature of 350°C. It was found that the activity of the studied catalysts increases with an increase of the content of acid centers of medium strength, according to the thermodesorption of ammonia. The textural, acidic, and isomerizing properties of Pd- catalysts deposited on AlZr pillared montmorillonite from $PdCl_2$ and Pd- sol solutions were compared.

**Keywords:** Electron Microscopy, Isomerization, Acid Centers, Montmorillonite

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## POSTER SESSIONS

## Id-302

# Electron Microscopy: a Useful Tool for the Characterization of Novel Nanostructures with New Architectures

Bogdan S. Vasile[1*], Otilia R. Vasile[1], Roxana Trusca[1], Marin Cernea[2]
[1] Faculty of Applied Chemistry and Materials Science, National Research Center for Micro and Nanomaterials
University Politehnica of Bucharest, Romania,
[2] National Institute of Materials Physics, Ilfov, Romania,
Corresponding author: bogdan.vasile@upb.ro

**Abstract:** The composite materials consisting of components with different properties have attracted much attention in recent years due of their potential multifunctional applications. BNT-$BT_{0.08}$ and $CoFe_2O_4$ species can form composites which should combine their piezoelectric and magnetic properties and should lead to performance surpassing their individual components. New architectures as multilayer heterostructures, core-shell composites, nanowires and tubes are already used for some materials with multiple functionalities. We present composite materials based on BNT-$BT_{0.08}$ and $CoFe_2O_4$, prepared as core-shell (BNT-$BT_{0.08\ (core)}$ and $CoFe_2O_{4\ (shell)}$), composite thin film Si-Pt/BNT-$BT_{0.08}$/$CoFe_2O_4$ and BNT-$BT_{0.08}$/$CoFe_2O_4$ and coaxial composite nanotubes. The purpose of the paper is to study the phase composition using XRD, microstructure using SEM and TEM microscopies. These composites with new architectures are prepared by sol-gel technique along with spin coating and using a polycarbonate membrane template. In this paper, SEM and TEM microscopies have highlighted the granular morphology of films, powders and tubes, their dimensions, the crystalline phases, chemical composition and have proven the composite structure of these materials. In summary, novel ordered arrays of coaxial BNT-$BT_{0.08}$/$CoFe_2O_4$ core–shell nanotubes structure, core-shell and thin films were successfully fabricated based on sol-gel method. Composites of piezoelectric (BNT-$BT_{0.08}$) and ferromagnetic ($CoFe_2O_4$) nanotubes were fabricated and investigated in order to envisage new applications for such architectured materials.

**Keywords:** Electron Microscopy, Nanostructures

## POSTER SESSIONS

## Id-315

# A Novel Amplitude Control Algorithm for Frequency-Modulation Atomic Force Microscope Based on Kalman Filter

Z. SONG[1,3], Y. LI[1,3*], Y. ZHANG[2,3], R. LIN[1,3], J. QIAN[1,3]
[1]School of Physics, Beihang University, Xueyuan Road *No*.37, Haidian District, Beijing, 100191, China
[2]School of Instrumentation and Optoelectronic Engineering, Beihang University, Xueyuan Road *No*.37, Haidian District, Beijing, 100191, China
[3]Key Laboratory of Micro-nano Measurement-manipulation and Physics (Ministry of Education), Beihang University, Xueyuan Road *No*.37, Haidian District, Beijing, 100191, China
Corresponding author: liyingzi@buaa.edu.cn

**Abstract:** Frequency-modulation atomic force microscope (FM-AFM) has been a widely-used instrument to achieve atomic and subatomic resolution. However, the low bandwidth and low amplitude tracking accuracy of automatic gain control based on root-mean-square (RMS) converter has been a big stumbling block to increasing the scanning speed and accuracy of FM-AFM. To improve the performance of amplitude control loop, a linear time-varying Kalman filtering approach is proposed to take place of RMS converter, which is capable to accurately demodulate the amplitude of the frequency-varying signal of the cantilever in shorter time without losing noise immunity. Thus the settling time of the cantilever oscillation amplitude can be remarkably reduced. Through the proposed method the topography tracking accuracy of FM-AFM can be improved, and the scan speed can be increased without loss of precision.

**Keywords:** Frequency Modulation Atomic Force Microscopy, Frequency-Varying Signal, Automatic Gain Control, Kalman Filter

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# POSTER SESSIONS

## Id-328

## Spatially Resolved Photocurrent Mapping of Nanoscale Grating

W. S. CHANG
Nano-mechatronics department, University of Science and Technology (UST), 217 Gajeong-ro Yuseong-gu, Daejeon 34113, Korea
Nano-Convergence Mechanical Systems Research Division, Korea Institute of Machinery & Materials (KIMM), 156 Gajungbukno, Yuseong-gu, Daejeon 34103, Korea
Corresponding author: paul@kimm.re.kr

**Abstract:** Spatially resolved photocurrent images with continuously increased intensities of light source on the grating structural heterogeneous ZnO shows the geometrical light trapping effect and the influence of peculiar material properties. The photocurrent on the space between ZnO nanobeams increased more than the top of ZnO nanobeams with increased light intensity (≥393 μW/cm^2), which is attributed to increased scattering mostly reflected from substrate due to the geometric structure. Furthermore, ratio of photocurrent between interspace and top of ZnO nanobeam on the scanning photocurrent microscopy (SPCM) images allows to know intuitionally the grating structure is directly related with average photocurrent within the measured ZnO area. In this context, our further study of structural influence for light trapping shows the grating structure has significant reduction of reflection and 1.64 times higher absorbed volume than flat film. In the lower intensity (≤82 μW/cm^2), SPCM images show the subtle photocurrent related with peculiar material property, namely ZnO surface layer existing concentrated deep levels. Additionally, with control of light intensity and bias voltage, we found out that conducting path of drift current exists in the inner side (bulk) of heterogeneous ZnO nanobeams. Experimental methods controlling intensity of light source and bias voltage on the metal-semiconductor-metal type device help us to know realistic phenomena of geometrical light trapping, influence of peculiar material properties and location of the drift conducting path, which could be useful novel techniques for analyzing other textured photovoltaic devices.

**Keywords:** Light trapping; grating structure, scanning photocurrent microscopy (SPCM); ZnO; conducting path.

## POSTER SESSIONS

## Id-339

# Spider venom molecules impair tumor cells migration and morphology through RhoA-ROCK pathway: potential application as anti-cancer drug

N. BARRETO[1,2*], M. CABALLERO[1,2], A. P. BONFANTI[1,2], T. A.A. DA ROCHA - E - SILVA[3], R. SUTTI[4], L. VERINAUD[2], C. RAPÔSO[1,2]

[1]Faculdade de Ciências Farmacêuticas, Universidade Estadual de Campinas (UNICAMP), Campinas, SP, Brazil; [2]Departamento de Biologia Estrutural e Funcional, Instituto de Biologia, UNICAMP; [3]Faculdade Israelita de Ciências da Saúde Albert Einstein, São Paulo, SP, Brazil; [4]Faculdade de Ciências Médicas, Santa Casa de São Paulo, São Paulo, SP, Brazil.

Corresponding author: nataliabarreto.jdi@gmail.com

**Abstract:** Malignant gliomas are brain tumors originated from glial cells, mainly astrocytes. It is a devastating human cancer, whose incidence and mortality are expected to increase in the coming years. The RhoA/ROCK pathway regulates the cytoskeleton, cell morphology and motility, and mutations in this pathway are related to invasion and metastasis of tumor cells. Finding molecules that regulate not only survival and proliferation but also the migration and invasion of tumor cells is one of the major challenges in anticancer pharmacology. Multitarget molecules, such as components of animal venoms, are therefore a potential strategy for the treating of tumors. It has recently been demonstrated by our group that molecules present in the venom of the South American spider *Phoneutria nigrivent*er (PnV) (Ctenidae, Araneomorpha) affected astrocytes, inducing profound morphological alterations; these findings suggested that PnV molecules could target glioma cells. The objective of this study was therefore to screen the in vitro effects of PnV on migration/invasion, cellular adhesion and morphology of glioblastoma (grade IV glioma) cells, and to select the PnV-isolated molecule(s) with the highest antitumor effect, also contributing to clarify its mechanisms. Cultured human glioblastoma (NG97) cells were divided in groups, as following: treated with PnV (14 µg/ml); PnV-isolated (HPLC) toxins (called F1-F12; 0.1 and 1 µg/ml) for 12, 24, 48 and/or 72 h; control without any treatment. Cells were analyzed by migration assays (wound healing and transwell); the adhesion capacity was verified through a commercial colorimetric kit, in which the cells were seeded in 24-well plates coated with fibronectin; cell morphology was demonstrated by GFAP immunofluorescence and phalloidin labeling. During the experiments, some cells were pretreated for 30 minutes with Y-27632 (10 µM), an inhibitor of RhoA-ROCK, to evaluate the involvement of this signaling pathway in the mechanisms of PnV and its isolated components. The wound healing test showed that the untreated cells (control) completely filled the scratch after 48 and 72 h, however, cells treated with venom and toxins (specifically F1 and F11; 1 µg/ml) failed to fill the wound, even after 72 h. The exposure to PnV and its isolated toxins significantly decreased the number of cells migrating through the transwell membrane, and also reduced cell adhesion, compared to the control. In addition, cells treated with PnV and toxins exhibited morphological alteration, with shorter processes, when compared to untreated groups. The groups incubated with Y-27632 showed that the effects of venom and its toxins were abolished or modified, suggesting that RhoA-ROCK signaling pathway may be involved in the mechanism of action of PnV and its

toxins concerning to migration, adhesion and morphological parameters. Taken together, the results of the present study suggest that PnV can be composed of highly specific molecules (mainly F1 and F11) for targets involved in the migration, adhesion and morphology of glioblastoma cells; ongoing experiments will categorize in depth the mechanisms involved and will determine the chemical nature of the molecules.

**Keywords:** Glioma, Phoneutria Nigriventer, Migration, Metastasis, Rhoa/ROCK

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# POSTER SESSIONS

## Id-340

### Identification of Macrophages Modulators Molecules from Spider Venom: New Perspectives for the Treatment of Cancer

J. MUNHOZ[1,2*], G. PERON[2], A. P. BONFANTI[1,2], J. OLIVEIRA[2], T. A.A. DA ROCHA - E - SILVA[3], R. SUTTI[4], R. THOMÉ[5], N. BARRETO[1,2], L. VERINAUD[2], C. RAPÔSO[1,2]

[1]Faculdade de Ciências Farmacêuticas, Universidade Estadual de Campinas (UNICAMP), Campinas, SP, Brazil; [2]Instituto de Biologia, UNICAMP; [3]Faculdade Israelita de Ciências da Saúde Albert Einstein, São Paulo, SP, Brazil; [4]Faculdade de Ciências Médicas, Santa Casa de São Paulo, São Paulo, SP, Brazil; [5]Department of Neurology, Thomas Jefferson University, Philadelphia, PA, USA.

Corresponding author: jlima.munhoz@gmail.com

**Abstract:** In the last decade, the immune response evasion has been recognized as an important feature of tumor development. Among the major cells involved in the innate immune system activation are macrophages. They present a complex relation with the prognosis of tumors; treatments involving macrophage activation, deletion or reprogramming are being investigated. Recent studies in our group have demonstrated that *Phoneutria nigriventer* spider venom (PnV) kills tumor cells *in vitro* and reduces or eradicates xenographic glioblastomas developed in immunodeficient mice (RAG$^{-/-}$). However, the venom reduced the viability of cultured cells by about 20%, while mice treated with venom developed tumors 90% smaller than control animals. These results led to the hypothesis that PnV could be fighting the tumor *in vivo* through immunomodulation. The aim of this study was therefore to investigate the *in vitro* effect of PnV on the activation of macrophages, and to identify the molecule isolated from the venom responsible for such effects. Macrophages were differentiated from bone marrow cells of C57BL6 mice and cultured in IMDM with 30% of L929 supernatant (source of M-CSF). These cells were pre-activated with IFN-γ (20 ng/mL, 48 h) and divided in six groups: control - without any treatment; venom - treated with PnV (14 µg/ml, 24 h); Fractions - treated with PnV-fractions (called F1, F2, F3; obtained using Amicon® filters; 1 µg/mL, 24 h); or LPS - treated with lipopolysaccharide (1µg/mL, 24 h). The supernatant was collated for ELISA assays. In order to evaluate phagocytic and killing activity, after treatments, macrophages were incubated with *Paracoccidioides brasiliensis* (Pb) for 4h and 48 h, respectively. After the incubation, macrophages lysates were cultured for 7 days BHI medium supplemented. Analyses were performed by determining the number of Colony Forming Unit (CFU). In order to analyze cellular morphology, Iba-1 immunofluorescence was done; transwell assay was performed to investigate if the venom stimulates cell migration. Finally, treated macrophages were cocultivated with glioblastoma (NG97) cells for 24 h, and cells viability was analyzed by incubating the live cells with DAPI. Results showed that PnV-macrophages were significantly more efficient in phagocytizing and killilng Pb compared with control group. In addition, they presented a morphologically activated profile with increased extensions. Significantly more PnV-modulated macrophages were able to migrate inside transwell membrane, comparing to control cells. Finally, DAPI showed that PnV-macrophages strongly decreased tumor cells viability, comparing to control. Macrophages modulated by F2 and F3 were more efficient in all evaluated parameters, in comparison to

that stimulated with PnV or control. The efficiency of PnV- and fractions-macrophages in phagocyte and killing cells was similar to LPS-macrophages, however, LPS induced the release of TNF-α, IL-6 and IL-10 cytokines, while PnV and fractions did not change them. Taken together, these results suggest that PnV can modulate macrophages. The next steps are to identify the pure components from the fractions responsible for immunomodulator effects. This work contributed to the elucidation of relevant issues, which may result in the development of a new anti-cancer therapy using venom isolated components as immunoadjuvant.

**Keywords:** Macrophages, Spider Venom, Immunotherapy, Cell Activation, Migration, Glioma
**Acknowledgement:** FAPESP #2015/04194-0, #2018/03051-9; CNPq #431465/2016-9.

# POSTER SESSIONS

## Id-381

# Investigation of allotropic ß→α-Sn transition in high tin content solder alloys with different microscopy and spectroscopy techniques

B. ILLÉS[1], A. SKWAREK[2], T. HURTONY[1], PIOTR ZACHARIASZ[2], G. HARSÁNYI[1]

[1]Budapest University of Technology and Economics, Department of Electronics Technology, Egry József u. 18, Budapest, Hungary

[2]Institute of Electron Technology, Kraków Division, Zabłocie 39, 30-701 Kraków, Poland

Corresponding author: billes@ett.bme.hu

**Abstract:** In the microelectronics, tin (Sn) is the base material of the solder alloys and surface finishes. Sn has two main allotropes, the metallic ß-Sn (applied in microelectronics) and the mechanically brittle semiconductor α-Sn. ß-Sn is stable between 13.2°C and 231.9°C while α-Sn below 13.2°C. The "tin pest" is a spontaneous allotropic transition of ß-Sn to α-Sn below 13.2°C. The transition has 3 phases: nucleation, growth and saturation. The sign of the transition is the occurrence of discolored spots which later change into characteristic warts, thus the name "tin pest" is given to the phenomenon. The transition causes volume increases, which can lead to the deterioration of the samples. Alloying elements (like Pb, Bi, Sb), usually suppress the transition however, the transition can occur not only in pure Sn but in high tin content alloys as well. Nowadays, form the economic reasons, there is a new tendency in microelectronics to reduce Ag content in the SnAgCu (SAC) solder alloys. In these low Ag content SAC alloys, the tin content can reach 98wt% which could increase the possibility of different reliability problems including tin pest phenomenon. Therefore, the identification and characterization of tin pest in Sn-rich solders and surface finishes is crucial for electronic devices working in sub-zero temperatures in aeronautical, aerospace and automobile applications. Tin pest is an autocatalytic reaction, the appearance of α-Sn speeds up the transition. At natural conditions, the transition is very slow (takes years) due to a high activation energy. However, the nucleation time is significantly reduced at the presence of InSn and CdTe (substances with the same crystallographic parameters as α-Sn) or α-Sn itself, which can act as natural inoculators.

In our work, different analytical methods were applied in order to characterize the tin pest phenomenon in the case of different solder alloys and inoculator materials. Since the transition leaves significant marks on the surface of the tin objects, the tin pest phenomenon has been traditionally observed with simple optical imaging techniques on the surface of the tin object. One step forward is the application of metallurgical cross-sections and metal microscope in order to study the internal behavior of the phenomenon. More sophisticated method is Scanning Electron Microscopy and Focused Ion Beam - Scanning Ionic Microscopy (FIB-SIM) which can even provide information about the grain sliding and □-Sn expansion during the transition. With Mössbauer spectroscopy it is possible to get the information about the ratio of the transitioned tin and about the duration of the transition process. Our results have shown that the transition phases can considerably differ at the different alloys and inoculators, like different nucleation, growth and the saturation stages. However, the accurate characterization of the transition in the given cases is possible only with the combined application of the mentioned analytical methods.

**Keywords:** Tin Pest; SAC; Solder; Mösbauer Spectroscopy; Allotropic transition

## POSTER SESSIONS

## Id-393

# Effect of Phase Composition and Local Crystal Structure on Transport Properties of Solid Solutions $Zro_2$-$Gd_2O_3$

F.O. MILOVICH[A]*, T.V. VOLKOVA[B], E.A. AGARKOVA[C]

[a] National University of Science and Technology (MISIS), Leninskiy prospekt 4, 119049 Moscow, Russia

[b] Ogarev Mordovia State University, 68 Bolshevistskaya Str., Saransk 430005, Republic of Mordovia, Russia

[c] Institute of Solid-State Physics, RAS, Academician Osip'yan str. 2, Chernogolovk142432, Russia

Corresponding author: philippmilovich@gmail.com

**Abstract:** Currently, the use of materials based on zirconia as a solid electrolyte for solid oxide fuel cells (SOFC) is associated with the need to solve a number of scientific and technological problems. The production of materials with high conductivity in the range of average temperatures (500-700 ºC) and the increase in the stability of the electrophysical characteristics of solid electrolyte at operating temperatures for a long time are some of these issues. In accordance with this, the identifying of various factors, such as phase composition and local crystal structure, affecting the magnitude of oxygen-ionic conductivity of zirconia-based solid solutions, is a relevant research topic.Comprehensive studies of the phase composition and local structure of zirconium dioxide crystals stabilized by gadolinium oxide in a wide range of compositions from 2.7 to 38 mol.% were performed and the effect of $Gd_2O_3$ on the transport characteristics of crystals of these solid solutions was established. The presence of transformable t and untransformable t` tetragonal phases was established in crystals of zirconia partially stabilized by gadolinium oxide, with a concentration of 2.7 and 3.6 mol.%, and their crystal structure parameters were determined. The presence of a twin structure formed during the transition of the high-temperature cubic phase to the tetragonal phase is shown in $ZrO_2$ crystals with a content of 8 mol.% $Gd_2O_3$ by transmission electron microscopy and Raman spectroscopy. It was established that the twin structure is absent with the same content of yttrium oxide in $ZrO_2$-8 mol% $Y_2O_3$ crystals, and the structure of the crystals corresponds to the structure of the t''-phase. Studies of the features of local crystal structure $ZrO_2$-$Gd_2O_3$ were carried out using optical spectroscopy methods. $Eu_2O_3$ was used as a spectroscopic probe. It has been established that the local structure of the $ZrO_2$-$Gd_2O_3$ solid solutions is determined mainly by the concentration of stabilizing oxides $Gd_2O_3$. It was shown that the decrease in ionic conductivity in the concentration range of stabilizing oxides above 12 mol.% $Gd_2O_3$ is due to the increase in the relative fraction of $Gd^{3+}$ cations with oxygen vacancy in the nearest crystalline surroundings, and in the concentration range above 20 mol.% $Gd_2O_3$ the formation of two anionic ions vacancies in the nearest crystalline surroundings of $Gd^{3+}$ ions.

**Keywords:** Solid solutions ZrO2-Gd2O3, Skull melting, Ionic conducting materials, Solid oxide fuel cells

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## POSTER SESSIONS

## Id-402

# Signal Amplification Using Deuterated Water-Based Buffers in Capillary Electrophoresis Coupled to Laser Induced Fluorescence Detection

J. KUČERA[1*], Š. KÁŇA[1], A. CELÁ[2], O. PEŠ[3], I. VORÁČOVÁ[4], P. TÁBORSKÝ[1]
[1]Department of Chemistry, Faculty of Science, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic
[2]Department of Biochemistry, Faculty of Science, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic
[3]Department of Biochemistry, Faculty of Medicine, Masaryk University, Kamenice 5, 62500 Brno, Czech Republic
[4]Department of Bioanalytical Instrumentation, Institute of Analytical Chemistry, Czech Academy of Sciences, Veveří 967/97, 60200 Brno, Czech Republic
Corresponding author: 408285@mail.muni.cz

**Abstract:** Many fluorescent molecules can be quenched by OH oscillators present in water. The Increase of fluorescence intensity or lifetime can be obtained by replacing the light water for deuterium oxide. We can take advantage of this phenomena in some analytical techniques coupled to the fluorescence detection (e.g. capillary electrophoresis, high performance liquid chromatography, flow cytometry, fluorescence microscopy, etc.). The capillary electrophoresis coupled with laser-induced fluorescence detection can be used for the determination of anthracyclines and related compounds which are widely used drugs, especially in cancer treatment. The steady-state and time-resolved fluorescence measurements of chosen anthracyclines in light and heavy water were carried out on the spectrofluorometer. Those experiments have proved that the emission signal and lifetimes of anthracyclines can be increased up to 4.5 times using deuterium water as a solvent. Therefore, the deuterated water-based buffers can be used for the detection of anthracyclines by the capillary electrophoresis with LIF.The CE-LIF separation of chosen molecules was successfully done in borate buffers prepared in light and heavy water. The fluorescence signal in $D_2O$ based buffer increased up to 4.4 times for rhein. Around 2 times higher fluorescence emission signal was recorded also for other studied anthracyclines (doxorubicin, daunorubicin, epirubicin). The calculated limit of detection for rhein was $5.1 \cdot 10^{-8}$ mol. $l^{-1}$ and $5.0 \cdot 10^{-9}$ mol. $l^{-1}$ in light and heavy water buffer respectively. Over one order better limit of detection is not caused only by reducing of the dynamic quenching but also lower noise level achieved in the analysis in $D_2O$ buffer. The main advantage of capillary electrophoresis is the high sensitivity combined with the lower consumption of sample and background electrolyte (about hundreds of microliters) which means that only small volume of heavy water is needed for the analysis.

**Keywords:** Signal Enhancement, Increase of Fluorescence Emission, Deuterated Water, Capillary Electrophoresis, CE-LIF, Anthracyclines, Lower Limit of Detection

## POSTER SESSIONS

## Id-403

# Benzo[C]Phenanthridine Alkaloids and Their Interaction to Non-Canonical Dna Structures

P. JAROŠOVÁ[1*], R. GARGALLO[2], O. PEŠ[3], P. TÁBORSKÝ[1]

[1]Department of Chemistry, Faculty of Science, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic

[2]Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Martí i Franquès 1-11, 08028 Barcelona, Spain

[3]Department of Biochemistry, Faculty of Medicine, Masaryk University, Kamenice 5, 62500 Brno, Czech Republic

Corresponding author: 408846@mail.muni.cz

**Abstract:** Non-canonical DNA secondary structures are perspective target of research in recent years. The G-quadruplex (GQ) which is one of these structures is present in several protooncogenic-DNA promoters. It is important in biological processes such as replication, transcription and translation. Complementary cytosine-rich DNA strand can also form a four-stranded quadruplex structure called i-motif. The cytosine base pairs in the i-motif structure form an intercalated and anti-parallel tetramer structure. Quaternary benzo[c]phenathridine alkaloids, which are supposed to selectively bind these structures, have been tested. Influence of selected alkaloids on stability of double-stranded DNA and noncanonical form of DNA was observed by determining melting temperature of these DNA structures without alkaloid and in complex DNA: alkaloid. Competitive dialysis was performed for affinity comparison of certain alkaloids to different DNA structures including G-quadruplexes.

**Keywords:** G-Quadruplex, I-Motif, Alkaloids, CD Spectrometry

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## POSTER SESSIONS

## Id-404

# TEM Structure Study of Low-Temperature Thermoelectric Materials Obtained by Spark Plasma Sintering

D.I. BOGOMOLOV[A], M.V. VORONOV[B], M.G. LAVRENT`EV[A], V.P. PANCHENKO[A, B], N.YU. TABACHKOVA[a]*
[a] National University of Science and Technology (MISIS), Leninskiy prospekt 4, 119049 Moscow, Russia
[b] State Scientific-Research and Design Institute of Rare-Metal Industry "Giredmet" JSC, B. Tolmachevsky lane, Building 5-1, Moscow,119017 Russia
Corresponding author: ntabachkova@gmail.com

**Abstract:** Semiconductor thermoelectric materials are widely used in generators, refrigerators, thermostats, conditioners and other devices. The basic energy characteristics of the thermoelectric devices are defined by thermoelectric efficiency. The creation of nanostructured materials is one way to increase thermoelectric efficiency. However, the causes of increase in the thermoelectric efficiency ZT in materials, obtained by pressing of nanopowders, are still unclear. There is no clear understanding of whether the ZT growth is associated with effects within the grains due to a large number of defects generated during grinding and pressing nanopowder, or with the influence of grain boundaries. The most common point of view is the phonon scattering at the grain boundaries in nanostructured samples occurs stronger than the scattering of electrons, due to the thermal conductivity at nanostructuring reduce quickly then electrical conductivity. However, the comparison of the means free path of electrons and phonons with grain sizes do not confirm this point of view. In this work we used the method of spark plasma sintering powders for obtaining bulk nanostructured thermoelectric material based on $Bi_{0.4}Sb_{1.6}Te_3$. Milling was carried out in high-energy ball mill. The research of the material structure was carried out by X-ray diffraction and transmission electron microscopy. We showed that recrystallize of the initial grains occurred in the process of sintering. The crystallite growth and reduction of surface defects occurred with increasing of sintering temperature up to 400 °C. The grain size reached to several microns at the sintering temperature of 400 °C. A large number of nano-sized grains appeared in the structure at 450 °C. The composition of the nano-grains matched the composition of the sample. For samples annealed at 400 °C the mobility of charge carriers at 15 K was higher than the mobility in samples annealed at 450 °C. The number of new grains significantly increased at 550 °C, and they increased in size. This process did not accompany by changes in the concentration of holes that means the nano-grains appeared due to healing of some own neutral defects, and not necessarily point defects and located within or on the large grain's boundaries. These results suggest that there are other (high temperature) self-organizing process of formation of nanostructures based on redistribution nonequilibrium intrinsic point defects besides well-known low temperature. The contribution of grain boundary scattering of phonons in the increase of the figure of merit (Z) at room temperature is not dominant because it should be considered an essential role of point defects in the bulk of the grains.

**Keywords:** Spark Plasma Sintering; Solid Solutions; Thermoelectric Properties; (Bi, Sb)2Te3; Structural Defects; Figure of Merit.

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POSTER SESSIONS

## Id-407

# Characterization of Composite Anion Exchange Membranes with Layered Double Hydroxides by NMR Spectroscopy

M.L. DI VONA[A,C] , R. A. BECERRA[A,B,C*],R. NARDUCCI[A,C] , P. KNAUTH[B,c]
[(a)]University of Rome Tor Vergata, Dep. Industrial Engineering, 00133 Rome, Italy
[(b)]Aix Marseille Univ, CNRS, MADIREL (UMR 7246), Campus St Jérôme, 13013 Marseille, France
[(c)]International Associated Laboratory (L.I.A.), Ionomer Materials for Energy (Aix Marseille Univ, University of Rome Tor Vergata)
Corresponding Author: divona@uniroma2.it

**Abstract:** Anion Exchange Membranes (AEM) are used in alkaline fuel cells, electrolysers and redox-flow batteries. The main disadvantage of AEM is the degradation of the polymer matrix in basic conditions and high temperature. The formation of composite offers several advantages, such as enhanced hydrolytic stability and improved mechanical properties. The composite anion-conducting membranes were prepared by dispersion of Zn/Al or Mg/Al Layered Double Hydroxide (LDH) in the polymeric matrix made of Poly(sulfone) (PSU) [1,2] and poly(2,6-dimethyl-1,4-phenylene) oxide (PPO) grafted with 1,4-diazabicyclo [2.2.2] octane (DABCO). They present a reduced swelling and the mechanical properties in fully humidified conditions are enhanced. Furthermore, the membranes can be treated in alkaline conditions at 60 °C without losing their properties. The Zn/Al and Mg/Al LDH were also modified by adding ionic liquids (IL) in the inter-lamellar space. These lamellar materials show an improved ionic conductivity especially at low humidity. The structure and stability of composite materials was studied via $^{1}H$ and $^{13}C$ NMR spectroscopy.

**Keywords:** Anion exchange membrane, NMR spettroscopy

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## POSTER SESSIONS

## Id-410

# On the Composition of Crystalline Body and Reddish Globule in the Eustigmatophyte – *Vacuoliviride crystalliferum* NIES-2860

J. PILÁTOVÁ[1*], P. MOJZEŠ[2], K. HŮRKOVÁ[3], K. SCHWARZEROVÁ[1]

[1]Deparment of Experimental Plant Biology, Faculty of Science, Charles University; address: Viničná 5, Praha 2, 12843, Czech Republic;

[2]Institute of Physics, Faculty of Mathematics and Physics, Charles University; address: Ke Karlovu 5, Praha 2, 12116, Czech Republic;

[3]Department of Chemistry and Analysis, Institute of Chemistry and Technology, Technická 3, address: Praha 6, 166 28, Czech Republic;

Corresponding author: jana.pilatova@natur.cuni.cz

**Abstract:** The eustigmatophyte algae are widely used in biotechnology, while being also a fascinating object of basic research at the same time. Compared to other Stramenopiles, eustygmatophyte algae possess many distinct ultrastructural features. For example, extraplastidial stigma in zoospores, reddish globule and lamellate vesicle in vegetative cells were described in 1972 (Hibber and Leedale, 1972). Another example is newly described structure called the crystalline body in *Vacuoliviride crystalliferum* (Nakayama et al. 2015). The composition, physiology, and biogenesis of these specific structures and organelles is currently unknown. In our work we studied the composition of the **crystalline body** and the **reddish globule** of the species *Vacuoliviride crystalliferum.* By means of confocal Raman microspectroscopy *in vivo* and *in situ* we show that both organelles have lipophilic character. The crystalline body is composed of elementary fatty acids; as only simple spectra were obtained using Raman microspectroscopy. The reddish globule is way much more complex and is composed of fatty acids and sitosterols. Interestingly, no carotenoids were detected in the reddish globule spectra. Moreover, some of the compounds exhibit autofluorescence. For further and more detailed confirmation of our results we are proceeding with the isolation of above-mentioned organelles by gradient ultracentrifugation followed by LC MS.

**Keywords:** Microalgae, lipids, cell inclusions, Raman spectroscopy, LC MC

**Acknowledgement:** This work was supported by the Charles University Grant Agency (grant No. 796217), the Ministry of Education (grant NPUI No. LO1417) and the Czech Science Foundation (grant No. 17-06264S).

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# POSTER SESSIONS

## Id-412

## Confocal Laser Scanning Microscopy for Examination of the Parasitic Nematodes Larvae of the Genus *Trichinella*

G. INESE, R. ILZE, M. LIGITA, K. ELENA, K. MUZA

Daugavpils Univeristy, Institute of Life Sciences and Technology, Parādes street 1A, Daugavpils, Latvia, LV-5404

Corresponding author: inese.gavarane@du.lv

**Abstract:** Parasitic nematodes are roundworms that infect humans, animals and plants cause serious diseases that have socio-economic consequences. *Trichinella britovi* larva samples were used for this study. Parasitic foodborne pathogens of *Trichinella* genus are known as etiological agents of human trichinellosis, a disease that is a public health hazard. In many countries is the control system on *Trichinella* to avoid the contamination of food chain for human use by this parasite. During this study were use novel luminophore probes AZM and P13 and the staining technology were developed to visualise internal and external organs of parasite. Stained larvae were analysed utilising confocal laser scanning microscopy. Examined larvae were fixed in Bouin's solution. Staining of *Trichinella* larvae by developed technique is crucial for deeper understanding of parasite structure and varieties. Synthesized AZM and P13 luminophores are appropriate to visualise the external and internal structure of *T. britovi* larvae stored under different conditions and for different time periods. The developed method is suitable for accurate sex determination of larvae. Sex ratio was significantly biased toward females ($P < 0.05$). Additionally, accumulation of knowledge and the development of techniques for sex ratio manipulation of parasite population could offer new opportunities for controlling parasitic reproductive strategies.

**Keywords:** Trichinella, confocal laser scanning microscopy, luminophore

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## POSTER SESSIONS

## Id-413

# The New Identification Method of Toxigenic Airborne Microbes Based on Hyperspectral Imaging

R. MIKKOLA[1,2*], E. CASTAGNOLI[1,3], T. ALAPIETI[1,4], M. A. ANDERSSON[1,5] AND H. SALONEN[1,6]

[1]Aalto University, Department of Civil Engineering, Rakentajanaukio 4 A, 02150 Espoo, Finland.

Corresponding author: raimo.mikkola@aalto.fi

**Abstract:** The presence of toxigenic microbes (e.g. mycotoxin producers) in the indoor environment put the health of its residents at stake. For example, the *Trichoderma* spp., colonizer of wet wooden material, are well-known producer of toxic secondary metabolite –peptaibols– like the channel-forming trilongins. Currently, rapid detection and discrimination tools to distinguish between non-toxic and toxic microbes in indoor environment are lacking. In this study, the aim was to create a hyperspectral database of 1) known toxigenic fungi isolated from indoor environment and 2) perform hyperspectral imaging of on-field isolated unknown fungi collected with RCS microbial sampler. The hyperspectral database of known toxigenic fungi was obtained from toxin-producing fungal strains previously isolated from indoor environments. The selected strains and their identified toxins (by LC-MS) were: *Aspergillus westerdijkiae* PP2 producing avrainvillamide and stephacidin B; *Aspergillus versicolor* SL/3 producing sterigmatocystin and 5-methoxy-sterigmatocystin; *Penicillium chrysogenum* RUK 2/1 producing meleagrin, *Penicillium expansum* L121 producing chaetoglobosin and communesin, *Chaetomium globosum* MTAV35 producing chaetoglobosins and chaetoviridins and the *Trichoderma atroviride* 14/AM producing trichorzianines.Firstly, reference hyperspectral imaging were obtained from the selected strains cultivated on malt extract agar (MEA) plates and different HYCON® agar strips used for RCS microbial sampler: TC (total count, tryptic soy agar), SDX (yeasts and molds, Sabouraud dextrose agar), YM (yeasts and molds, rose bengal agar with streptomycin). Secondly, on-field microbial indoor air samples (1000 L at 100L/min) were collected with an RCS microbial sampler (RCS High Flow Touch, Merck) on the aforementioned HYCON® agar strips. Hyperspectral imaging of the reference and unknown fungal strains were obtained one week after cultivation. The snap shot hyperspectral camera (Senop, Finland) with spectral range in visible and near-infrared (500-900 nm) range was used to obtain hyperspectral images. The halogen lamp was used as light source and the white Teflon bar as white reference. Additional spectral data were obtained from the fungal colonies illuminated together with UV (4W/366 nm) and halogen lamps. Each of the selected toxin-producing reference fungal strains exhibited different hyperspectral spectra. The hyperspectral spectra were also medium-dependent. The unknown airborne-isolated fungi had different hyperspectral spectra when they were exposed to UV before hyperspectral analysis. The UV illumination effect on the fungus spectrum was also clearly seen when the exudates of *Trichoderma atroviride* 14/AM were collected (MEA agar plates) and were deposited onto filter paper before hyperspectral analyses. The hyperspectral imaging was used to create preliminary hyperspectral database of known toxigenic fungal isolates and several unidentified isolates.

The use of UV light provided additional spectral information on some of the fungal strains. The new developed method based on hyperspectral imaging and RCS microbial sampling is a promising technique for the fast detection of different microbes. In the future, automation of the sampling handling and data processing would be developed, and larger hyperspectral database would be created. This would enable fast identification of fungal indoor air isolates and help discriminate between toxic and nontoxic indoor air microorganisms using the developed hyperspectral method.

**Keywords:** Fungi, hyperspectral imaging, indoor air, microbe

# POSTER SESSIONS

## Id-414

# Confocal Laser Scanning Microscopy for Visualisation of Adult Trematoda Stained by Synthesised Luminophore

K. MUZA*, G. INESE, M. LIGITA, R. ILZE, K. ELENA

Daugavpils University, Institute of Life Sciences and Technology, Parādes street 1A, Daugavpils, Latvia, LV-5404

Corresponding author: muza.kirjusina@du.lv;

**Abstract:** The family Fasciolidae includes six genera (*Protofasciola, Fasciolopsis, Parafasciolopsis, Fascioloides, Fasciola, Teniufasciola*) and species that are common liver fluke of wild and domestic mammals, and birds. Parasites like *Fasciola gigantica*, *F. hepatica* and *Fasciolopsis buski* are infecting humans. For this study fasciolid parasite *Parafasciolopsis fasciolaemorpha* of veterinary importance was selected, which typically infects elk (*Alces alces*) and other Cervidae. However, the fluke may find new host species within of parasite intermediate host, such as European bison and distribution area of parasite can expand. Nowadays, human activities are associated with export of animals outside of their natural areas that have an influence on the formation of helminth fauna of wild animals including Cervidae. In last years, confocal laser scanning microscopy has become an important tool to examine various parasites due the visualisation of more detailed anatomical structure of parasites. A benzanthrone luminophore AZP5 was synthesized to visualise external and internal structure of trematoda. The samples were kept in 96% ethanol, stained in AZP5, washed with 96% ethanol and then with ethanol-xylene and 100% xylene was added. All specimens were mounted with Canada balsam. In this study the method was used to investigate musculature, morphology of oral and abdominal sucker, digestive, excretory and reproductive systems. The developed staining technique show sufficient results for trematoda *P. fasciolaemorpha* morphology investigation. The synthesis of new luminophores expands opportunities of confocal laser scanning microscopy.

**Keywords:** luminophore, Trematoda, CLSM, visualisation

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## POSTER SESSIONS

## Id-415

# Synthesis and Spectroscopy of New Luminescent Benzanthrone Dyes

E. KIRILOVA*, A. PUCKINS, T. GRIGORJEVA, E. ROMANOVSKA, A. BULANOVS

Daugavpils University, Institute of Life Sciences and Technology, Parades street 1A, Daugavpils, Latvia, LV-5404

Corresponding author: jelena.kirilova@du.lv

**Abstract:** Organic fluorescent dyes have received a considerable attention for their potential applications to biochemical and medical investigations. Benzo[de]anthracen-7-one dyes are a special class of environmentally sensitive luminophores that emit in the spectral region from green to red, depending on the molecular structure. As a consequence of their strong emission and good photostability benzanthrone derivatives have found application in a number of areas including coloration of many materials, laser active media, fluorescent sensors in biology, etc. Recently we realized the synthesis of new N-containing derivatives of benzanthrone with various substituted amine, amidine and amide groups. The main objective of present work is design of new benzanthrone dyes which contain various nitrogen containing heterocyclic moieties at 2 and 3-positions of benzanthrone core as well as analysis of spectroscopic features of obtained compounds. During our investigation new preparation methods were developed for synthesis of emissive heterocyclic derivatives of benzanthrone. The structure of obtained compounds was confirmed by NMR and FT-IR spectroscopy and mass spectra data. The photophysical properties of the novel compounds were investigated in various media. It is shown that newly prepared compounds emit with a high quantum yield in the spectral region 530-680 nm in organic solvents and some of them in solid state also. Interesting results about substituents effects on reactivity and photo physical properties of prepared substancies are obtained and discussed in present research. It was found that spectroscopic parameters of synthesized compounds are quite sensitive to the surrounding environments and are potential fluorescent probes for visualization of tissues and living cell structures.

**Keywords:** Benzanthrone; heterocyclic dyes; synthesis; fluorescence spectra

**Acknowledgement:** The study funded by the project No. 1.1.1.1/16/A/211.

## POSTER SESSIONS

## Id-417

# AFM Analisys of Multicompanent Photocatalitic System films stucture

V. A. TIMOFEEVA*, A. V. PAVLOVA, O. V. KHASANOVA, A. B. SOLOVIEVA

N.N. Semenov Institute of Chemical Physics Russian Academy of Sciences, Moscow, Russia

Corresponding author: vik.timofeeva@gmail.com

**Abstract:** In recent years for wounds' treatment the photodynamic therapy (PDT) is applying. It is effective in treatment of infection agents which are resistant to antibiotics. It based on the oxidative destruction of pathogenic microorganisms with laser irradiation of affected tissues, preliminarily treated with a photosensitizer. The efficient photosensitizers are porphyrins, which can to generate of singlet oxygen that is strong oxidizer. One way to increase of the porphyrin photosensitizer functional activity may be achieved by mixing the photosensitizers with surfactants, which are efficient dispersers preventing aggregation processes in porphyrin solutions. It is known that the aggregation usually decreases the photosensitizing activity of porphyrins. In our group we develop some new porphyrin-based polymer photosensitizing systems for PDT. We previously showed that use of the porphyrin complexes with amphiphilic polymers (AP) - the pluronics and polysaccharides (PS) – chitosan with different molecular weights increased photosensitizing porphyrin activity in the photogeneration of singlet oxygen in aqueous media, as compared to pure porphyrins and increases the efficiency of PDT. Atomic force microscopy was involved for such systems analysis. We attribute the observed alterations in the Pluronic structure to the formation of complexes between the polymer and porphyrin in dual system which account for enhanced photocatalytic activity of such systems in the generation of singlet oxygen. Photocatalytic activity of triple systems (porphyrin- AP-PS) depended on the molecular weight of chitosan. This effect can be bound up with the unique surface structure features of chitosan-based systems. In this work was developed new systems with polymer components amphiphilic polymer – polyvinylpyrrolidone (PVP) and polysaccharides - sodium alginate (AN) and porphyrine photosensitizer - photoditazine (dimethylglucamine salt of chlorin e6, PD). AN had its own bactericidal activity. Finding the characteristic features of such systems components interactions is important in development of novel pharmaceutical formulations. The addition of the PVP to the porphyrin-polysaccharide system leads to a further increasing of the photocatalitic activity. Such increase of effective rate constant is believed to be linked with the formation of double (porphyrin-PVP) and triple (porphyrin-polysaccharide-PVP) complexes with the photosensitizer being in disaggregated state. To confirm the obtained data about formation of complexes PD-PVP and PD-polysaccharide-PVP, was obtained (was held) AFM visualisation. In this study, we used atomic force microscopy to assess the details of the structures grown from water solutions of porphyrin- polymers systems on mica substrates. The interpenetration character of polymer components in the forming complex systems was established of the site of surface pellicle forming by evaporation of double (PD-PVP and PD-PS) and triple systems (PD-PVP-PS). There was determined the dependence of the surface structure of films of double and triple porphyrin-

containing polymer systems from the concentration of components. It is shown that the structure of the surface of the AN significantly is changed when porphyrin, porphyrin with an PVP is introduced into the system, and depends on their concentrations. In the presence of porphyrin, the structure of the AN is disturbed to form islet inclusions. The introduction of PVP increases the size of island formations by about 10 times. This is probably due to the partial destruction of porphyrin associates. Thus, obtained structural features confirm explain the ability to increase the photocatalytic activity of PD in the singlet oxygen generation of such systems.

**Keywords:** Atomic force microscopy, porphyrin photosensitizer, photodynamic therapy, Multicompanent Photocatalitic System

**Acknowledgement:**The work was performed in the framework of the state order, topic V. 46.14, № 0082-2014-0006, AAAA-A17 -117-117032750202-6

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