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INVITED SPEAKERS

Id-579

ELECTRICAL CHARACTERIZATION of SEMICONDUCTOR FILMS Through DEPTH PROFILES OBTAINED by DIFFERENTIAL HALL EFFECT METROLOGY (DHEM)

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Abstract: Carrier concentration, mobility and resistivity are critical electrical parameters that define the quality of a semiconductor layer and performance of an electronic device fabricated on it. The commonly used techniques to determine these parameters are 4-point probe and Hall effect measurements, which yield Hall mobility (μ_H) and carrier concentration (n) values for the whole film. These measured bulk values can be used effectively for correlating process conditions and film attributes only if the mobility and carrier concentration are uniform through the thickness of the measured film. If the mobility and/or the carrier concentration vary through the thickness of the film, then the bulk mobility measurement from Hall effect gives an effective mobility value, and an effective carrier concentration value may be derived using the 4-point probe data. Where, n(z) and μ(z) are the carrier concentration and mobility varying in "z" direction, which is perpendicular to the surface of the film. As can be seen from the equations above, depending on how the mobility and the carrier concentration values vary through the thickness of the film, the bulk effective values obtained from these measurements cannot define the true characteristics of the film and therefore can be very misleading. Recognition of the need for depth profiling electrical parameters gave rise to development of techniques such as Spreading Resistance Profiling (SRP), Scanning Spreading Resistance Microscopy (SSRM), Scanning Capacitance Microscopy (SCM), and Electrochemical Capacitance-Voltage (E-CV) techniques. However, one limitation of these approaches is the fact that they measure either the spreading resistance or the carrier concentration values, without providing mobility depth profiles. Furthermore, conversion of spreading resistance to resistivity is often problematic and requires extensive calibration for different materials. These techniques also have difficulty in meeting the sub-nm depth resolution required for characterizing Si and SiGe films employed in advanced node transistor structures. Differential Hall Effect Metrology (DHEM) has been commercialized recently, and it is the only technique that provides depth profiles of all the critical electrical parameters through semiconductor layers at nm or sub-nm level depth resolution. DHEM provides depth profiles of mobility, carrier concentration and resistivity through a semiconductor layer by making successive sheet resistance and Hall voltage measurements using Hall effect/Van der Pauw techniques, as the electrically active thickness of the layer is reduced through successive oxidation steps. Controlled oxidation is achieved by electrochemical anodization using electrolytes specially formulated for the material being characterized. In a DHEM measurement, first a cross-shaped Van der

Pauw test-pattern is prepared on the sample to be measured. The cross has a mesa structure isolating the top film to be characterized from the surrounding portion of the layer. Measurement is typically carried out on p-type films formed over n-type substrates, or n-type films formed over p-type substrates, so that the p-n junction underneath isolates the top film from the substrate. Four electrical contacts are formed at the ends of the four arms of the cross and the nozzle of a small process chamber comprising a cathode is sealed against a process area that includes a test region at the center of the test-pattern. The nozzle has the capability of delivering an electrolyte, DI water and nitrogen gas to the process area. Through electrochemical oxidation the electrically active thickness of the layer at the test region is reduced in a highly controlled stepwise manner and measurements of sheet resistance and Hall voltage are carried out after each thickness reduction step. Data collected can then be processed to yield depth profiles of resistivity, mobility, and carrier concentration. Through this technique a depth resolution of ~0.3nm can be achieved for Si. In this presentation we will present fundamentals and capabilities of the DHEM technique and the ALPro™ series depth profiling tools developed to apply this method. We will discuss examples demonstrating how DHEM data obtained from Si, SiGe, GaAs samples may be used in correlating electrical properties of these layers with processing conditions.

Keywords: Hall effect; Differential hall effect; Depth profiling; Van der Pauw; Dopant activation.

INVITED SPEAKERS

Id-583

STRUCTURE of SILICA-YTTRIA-ZIRCONIA ADVANCED BIOCERAMICS DOPED with METAL OXIDES

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Abstract: Zirconia-based ceramics with improved properties were promoted as high strength materials for medical and technological applications. Zirconia (zirconium dioxide) has three different patterns: monoclinic (at room temperature and remains stable up to 1170^{o}C), tetragonal (between 1170 and 2370^{o}C) and cubic (>2370^{o}C) crystalline phase. During cooling, the transitions between these phases back to monoclinic phase involve a large volume expansion (4-5%) which produces micro-cracks, inclusions and catastrophic failure. The main problem of the zirconia based ceramics is the stabilization of tetragonal and cubic ZrO_2 crystalline phases at room temperature after sintering. The addition of different oxides such as yttrium oxide (ytria) and magnesium oxide (magnesia) can be added to zirconia to stabilize it, allowing the tetragonal and/or cubic crystalline phase to exist at room temperature after sintering. The aging process still remains an open question. When a larger Y_2O_3 amount (over 6 and up to 12 mol%) is added, the cubic ZrO_2 phase can be formed and dental ceramics can be still desirable due to the improved translucency and the complete absence of hydrothermal degradation. Accordingly, the engineering properties and materials performances of zirconia ceramics are controlled by the crystallinity. The aim of the present study was to investigate the microstructure and spectroscopic properties of SiO_2, Fe_2O_3, Na_2O and MgO doped in 8 mol% ytria stabilized zirconia by: SEM and XRD analysis, FTIR, UV-Vis, EPR and PL spectrocopy, XANES and EXAFS.

Keywords: Fourier Transform Infrared Spectroscopy.

INVITED SPEAKERS

Id-586

IMPLEMENTATION of RAMAN SPECTROSCOPY to CIVIL ENGINEERING MATERIALS

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Abstract: For years, Raman spectroscopy was dedicated to laboratory investigations and to materials with given degrees of heterogeneities and complexity, from biological materials to semi-conductors or sophisticated metallic alloys. Nevertheless, materials dedicated to civil engineering have barely been investigated by means of Raman spectroscopy. It has been implemented since the late seventies, and encountered in many studies till then, although not all the time the cornerstone of the investigations. In this presentation, some results of the benefits of implementing Raman spectroscopy will be given. It consisted in first characterizing specific phases present in cementitious materials, up to a continuous monitoring of some alteration processes such as carbonation or sulphatic attack, or the characterization of an archeological sample, with the benefits of chemometrics.

Keywords: Raman spectroscopy; Civil engineering; Chemometrics.

INVITED SPEAKERS

Id-590

SIX DECADES of MOSSBAUER SPECTROSCOPY

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Abstract: Principles of the Mössbauer effect and achievements of the Mössbauer spectroscopy will be shortly reviewed. Interactions of electronic shells with nucleus, called hyperfine interactions, allow measurement of electron density, electric field gradient and magnetic field at the position of the nucleus. Orientations of the hyperfine fields can be determined by polarization analysis. Ides of synchrotron sources working at time domain broaden the application area in the context of beam intensity and collimation. In particular, measurements of atomic probe selective phonon density of states are available in Mössbauer synchrotron technique. Next generation of the synchrotron sources working at energy domain are installed and their quality is continuously improved. Efforts on the on the collimation of radiation of radioactive sources, particularly important for microscopy, are still continuing. Mössbauer spectrometers were also miniaturized and were used for Mars planet exploration. Among variety of materials investigated by Mössbauer spectroscopy, particularly important are multiferroics, materials that exhibit more than one of the primary ferroic properties in the same phase. One of them is $GaFeO_3$ orthoferrite. The unit cell contains two nonequivalent gallium (Ga_1, Ga_2), two iron (Fe_1, Fe_2) and six oxygen sites. The cation in Ga1 has tetrahedral coordination while in remaining Ga_2, Fe_1 and Fe_2 cations are octahedrally coordinated. Pronounced cation site disorder between Ga_2 and Fe_1, Fe_2 exists, resulting in the net magnetic moment about 0.7 μ_B/Fe at ground state. The ionic state of iron is Fe^{3+} with a half-filled $3d^5$ configuration, in which the orbital momentum vanishes. Despite of it magnetocrystalline anisotropy of the order of one meV per unit cell, iron atoms have relatively large orbital momentum caused by anisotropic O2p-Fe3d hybridization of distorted local octahedra. The mechanism of electrical polarization switching is still under debate. Local hyperfine structure investigated in Mössbauer experiments on polycrystalline samples, single crystals, measurements also in high magnetic field will be reviewed. Results of the Mössbauer measurements performed on the oriented single-crystal absorbers will be presented. The analysis of line intensities on the single crystal orientation indicate for possible magnetic component perpendicular to the magnetization easy axis. The orientations of hyperfine interactions, including their tensor properties, will be discussed. This work was supported by the National Science Centre, Poland, under grant OPUS no 2018/31/B/ST3/00279.

Keywords: Mössbauer Spectroscopy; Frerrites; Multiferroics.

INVITED SPEAKERS

Id-591

ON-SITE QUALITY CONTROL and PROTECTION against PRODUCT COUNTERFEITING by HANDHELD VIBRATIONAL SPECTROMETERS: ANYWHERE and ANYTIME

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Abstract: Recently, miniaturization of Raman, mid-infrared (MIR) and near infrared (NIR) spectrometers has made substantial progress, and marketing companies predict this segment of instrumentation will have a significant growth rate within the next few years. This increase will launch vibrational spectroscopy into a new era of quality control by in-the-field and on-site analysis. While the weight of the majority of handheld Raman and MIR spectrometers is still in the ~1 kg range, the miniaturization of NIR spectrometers has advanced down to the ~100 g level, and developments are under way to integrate them into mobile phones. Thus, based on high-volume manufacturability and significant reduction of costs, numerous companies target primarily with NIR instruments a non-expert user community for consumer applications. Especially from this last-mentioned development, a tremendous potential for everyday life can be expected ranging from food testing to detection of fraud and adulteration in a broad area of materials (pharmaceuticals, textiles, polymers, etc.). However, contrary to the exaggerated claims of many direct-to-consumer companies that advertise their “scanners of the future” by oversimplifying the measurements to “point-and-shoot” procedures with any deficiencies in sample presentation and sample heterogeneity being taken care of by “cloud evaluation of big data”, this presentation will provide an impression regarding the present user potential of these instruments by discussing in detail selected, realistic industrial, environmental and every-day-life applications. Notwithstanding the advantage of the NIR technique, industrial applications with handheld Raman and MIR spectrometers will also be addressed.

Keywords: Handheld Vibrational Spectroscopy; On-site analysis; Mid-infrared; Raman; Near-infrared.

INVITED SPEAKERS

Id-593

POSSIBILITIES and FEATURES of AUGER SPECTROSCOPY STUDIES of NANOSTRUCTURES and NANOINCLUSIONS in COMPOSITE CERAMIC BULK MATERIALS, COATINGS and THIN FILMS

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Abstract: The Auger spectroscopy method is a powerful method for studying the nanostructure of composite materials, in particular, those containing nanoscale inclusions of secondary phases, materials containing light elements. This method in combination with other methods for studying the structure, such as X-ray analysis with Rietveld refinement, scanning electron microscopy methods - obtaining images in the modes of secondary electrons (SEI) and back-scattering electrons (BEI), X-ray microprobe analysis, application of Kikuchi method, etc., provides unique opportunities for a comprehensive study of materials. Auger spectroscopy method is applicable to the study of bulk materials, coatings and thin films, allowing 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). While 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 superconducting MgB_2 –based bulk materials and thin films (140 nm thick), MAX phases bulk and coatings (4-8 μm thick), refractory bulk B_4C-SiC, α-AlB_{12}- and $AlB_{12}C_2$-based, 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-596

USING MICROSCOPY to ASSESS THE EFFECTS of SURFACE PROPERTIES and CULTURE MEDIUM DURING RECOMBINANT PROTEIN PRODUCTION in BIOFILMS

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Abstract: Biofilms are mostly known for their negative effects on human health and industrial productivity. However, they can be used for the production of valuable compounds such as solvents, organic acids, and enzymes. The effects of surface properties and culture medium composition on *Escherichia coli* biofilm formation and production of a model recombinant protein (eGFP) were assessed using Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) for surface analysis and Epifluorescence microscopy (EP) and Confocal Laser Scanning Microscopy (CLSM) to monitor the number of biofilm cells, eGFP expression, and for 3D visualisation of biofilm architecture. Biofilm assays were performed in 12-well plates for 9 days using culture media containing different sources of carbon and nitrogen (Lysogeny broth (LB), Terrific broth (TB), and M9ZB broth) and three surface materials: stainless steel (SST), polyvinyl chloride (PVC) and silicone (SIL). Surface image analysis by SEM revealed that SIL had protuberances, which contributed to the higher surface roughness parameters determined by AFM when compared to SST and PVC. PVC was the smoothest surface. eGFP expression was always higher for PVC, with the highest specific production attained with TB medium. Regardless of the culture medium tested, CLSM analysis showed that the biofilm formed on PVC had higher biovolume and thickness than those formed on SST and SIL. Looking at the percentage of biomass expressing the recombinant protein, it was also higher for the biofilm formed on PVC compared to the other surface materials, with a higher fraction of eGFP-expressing cells found in TB medium. Regardless of the culture media and surfaces tested, the spatial distribution of the biofilm showed that most eGFP-producing cells are located in the outermost layers of the biofilm. These results provide further insight into the use of *E. coli* biofilms for the production of added-value compounds such as recombinant proteins.

Keywords: Biofilms; Recombinant Protein Production.

INVITED SPEAKERS

Id-598

A NOVEL APPROACH to SAMPLE PREPARATION TECHNIQUES for BIOACTIVE COMPOUNDS

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Abstract: Vacuum, as used in the laboratory, is simply the pressure below atmospheric pressure. The most familiar laboratory vacuum applications are filtration and drying. In the classical filtration method, the process is usually very slow due to the wide spectrum of solvents and solids. To speed up the process, low pressure (vacuum), is created. However, some situations lead to more important problems than a waste of time. In particular, vacuum systems are used for air and/or oxygen sensitive molecules and preparation of biological samples or genome and proteome experiments. Obtaining these substances without being affected by the air is of vital importance both in terms of the amount of substance, its purity and facilitating the analysis method to be applied later. This study aims to show the applicability of some vacuum systems more frequently used in inorganic chemistry laboratories, by explaining their importance and rare use in analytical chemistry or preparation of biological samples. Among these vacuum systems, Glove box and Schlenk line can be used easily in many laboratories. However, their use for different purposes is rare except the synthesis of inorganic substances. In this sense, theses methods are not novel, but their use to prevent oxidation of biomolecules is rare. These methods have started to be used in areas such as the synthesis of RAFT polymers used in many biologically related fields such as drug delivery, biomedical imaging and tissue engineering, the production of Multifuctional Polymer-Nanoparticle Composites, the production of organometallic molecules, the production of anaerobic bacteria, production of plant-derived air sensitive compounds. In addition, they are used in the purification of phytochemicals or food chemicals. In such examples, vacuum systems prevent the oxidation of organic structures and their degradation by polymerization (browning). Normal filtration methods are used in the preparation and isolation of phytochemicals or biological samples, however their interaction with air reduces the efficiency. The use of vacuum methods in inert atmosphere is also extremely useful in terms of preventing interference with the spectroscopic methods applied later. The use of vacuum/inert gas manifold systems are ideal for isolating, protecting, and handling air-sensitive bioactive compounds. Their more widespread use reduces errors in sample preparation and increases the yield of the obtained compounds.

Keywords: Bioactive Compounds; Glove Box; Schlenk Line; Air Sensitive; Sample Preparation.

INVITED SPEAKERS

Id-611

LOW ENERGY ELECTRON EMISSION from SURFACES, 2D-MATERIALS FORMATION of 2-D ELECTRON CASCADE in THE STM-FIELD EMISSION REGIME

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Abstract: Emission of secondary electrons (SEs) upon electron impact is a phenomenon of overwhelming importance in many areas of science and technology, for example for visualisation of nanostructures the Secondary Electron Microscope (SEM), for charged particle detectors, plasma display panels, plasma stability in fusion reactors, They can critically affect charged particle storage rings, e.g. in the Large Hadron Collider (LHC) at CERN, are responsible for the proximity effect in electron beam lithography, etc. Low Energy Electrons (LEE) cause damage to bio materials leading to tumour formation, but can also be used for therapeutic purposes. The mechanism of SE-emission is far from understood presently. In my talk I will address the mechanism leading to emission emission of low energy electrons, which can be elucidated by means of spectroscopy with correlated electron pairs. With this technique one can identify the spectrum of emitted secondary electrons differential with respect to the energy loss suffered by the primary electrons, by measuring the two interaction partners in coincidence. It will be shown that bulk plasmon decay leads to emission of secondary electrons in a Markovian process while the invacuo-excitation of surface plasmons leads to SE emission from the very surface of a material. The second part of my lecture concerns 2-D van der Waals materials, multi-layer graphene (MLG). Here, plasmon decay breaks the D6h honeycomb-symmetry leading to a hybridisation of interlayer states with atom-like states, opening a gateway for characteristic electrons in graphitic materials to escape from the surface. The final part of my lecture concerns the formation of a 2-D electron cascade in the scanning tunnelling microscope, operated in the field emission regime. In view of the fact that a 3D-electron cascade has been a paradigm in electron microscopy over the past century, the prediction of a 2D-cascade, followed by its recent experimental verification opens novel avenues for electron microscopy and other electron beam techniques in nanotechnology. Here an STM-tip, operated in the field emission mode scans a surface and elastically backreflected electrons are forced back into the surface by the strong field in the tip-sample junction. Inside the surface, the momentum is again reversed in elastic scattering processes with the ionic subsystem of the solid. After repeatedly ”bouncing” off the surface, primary electrons reach a detector, carrying the spin signature of the sample, as evidenced by the hysteris of a ferromagnetic model system.

Keywords: Low Energy Electrons; Secondary Electrons; 2-D Materials; Correlated Electron Spectroscopy.

INVITED SPEAKERS

Id-612

X-RAY FLUORESCENCE SPECTROSCOPY and IMAGING ANALYSIS USING GASEOUS DETECTORS

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Abstract: EDXRF analysis techniques are known for its non-destructive and non-invasive character, which makes them suitable for the identification and quantification of elements in analytes with good detection efficiency and sensitivity. As such, EDXRF is widely used on a myriad of areas, including biomedical research, industry, art and cultural heritage studies. Most of these studies are conducted using conventional EDXRF systems, such as portable XRF spectrometers or imaging spectrometers, either scanning systems or full-field of view systems, usually based on solid state detectors, that either allow elemental identification/quantification and/or elemental distribution mapping of small regions of interest. Gaseous detectors, such as the Micropattern Gaseous Detectors have received increasing attention in the scientific community, during the last years, and can be a good alternative to the full-field detectors used in conventional EDXRF imaging systems, especially due to the accurate determination of the interaction position of each X-ray photon in the sensitive area of the detector, as well as the energy information, making possible to map the distribution of elements in large area samples with fair energy resolution and detection efficiency, at a reasonable cost. This type of systems has already been used for the elemental mapping of different samples, ranging from applications in the cultural heritage field (contemporary indian miniature; analysis of pigments in an illuminated parchment from the XV-XVI century; analysis of pottery items and tiles, to evaluate the deterioration of ceramic glazes and/or assess the restoration of these items), to the biomedical field (analysis of human teeth treated with metallic amalgam; heavy metal distribution in zebrafish; elemental mapping studies on healthy and cancerous human tissues). In this talk, a complete overview of the EDXRF imaging systems based on gaseous detectors, both apparatus and applications, will be presented. This work was partially supported by projects CERN/FIS-INS/0025/2017 and STRONG-2020 – 824093 through FCT and H2020 programs. It was also developed within the scope of the i3N (UIDB/50025/2020 & UIDP/50025/2020) financed by national funds through the FCT/MEC COMPETE, FEDER and POCI programs.

Keywords: X-ray Fluorescence Imaging; Elemental Mapping; Gaseous Detectors.

INVITED SPEAKERS

Id-614

COLD ION FRAGMENTATION SPECTROSCOPY-MASS SPECTROMETRY for IDENTIFICATION of ISOMERIC CARBOHYDRATES and LIPIDS

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Abstract: Lipids and carbohydrates are ubiquitous in living organisms but appear to be one of the most difficult type of biomolecules to study on atomic level. Lipids are very homogeneous in chemical composition and, compare with proteins and carbohydrates, have no well-classified structural units. Carbohydrates contain a variety of isomeric units, but different from peptide, they can be differently interconnected in branched chains. All these properties make identification of these two types of molecules extremely difficult. In addition, their absorption is tiny in UV and uncharacteristic in IR, making them, essentially, indistinguishable by spectroscopy. We make use of intermolecular hydrogen bonds to distinguish very similar isomeric glycans and isomeric lipids by fragmentation spectroscopy of cryogenically cooled complexes of the molecules with aromatic peptides. An aromatic reporter molecule changes its absorption differently in the complexes with different isomers of a host molecule. We detect these differences and use it for library-based quantifications of isoforms mixed together. The non-covalent complexes of the analyte molecules with a reporter aromatic molecule are readily formed in solution upon mixing. Soft mode of electrospray ionization allows for transfer of the complexes to the gas phase with little dissociation. The ions of interest are, first, m/z selected by a quadrupole mass filter and then transferred to an octupole ion trapped, where they are cooled to T=10 K in collisions with He buffer gas. Cooling suppresses inhomogeneous spectral broadening, making the spectra structured and more distinguishable. The cold complexes are then irradiated by a 5-ns pulse of UV or IR light, and the appearing fragments are detected by a quadrupole MS. Scanning wavelength of UV/IR laser light while detecting the appearing charged fragments generates a respective spectrum of the complex. We first measure such spectra for a set of isomeric molecules of interest and store them as a library. Next, the spectrum of a mixture of these isomers is measured and mathematically decomposed in the basis set of the library. This allows for quantitative identification of the mixed in solution isomers of the same mass. The typically achieved accuracy for small saccharides is 1-3%, increasing to 5% for large molecules and/or for sets of small isomers. We demonstrate that such analytical identifications can be done online with HPLC.

Keywords: Cold Ion Spectroscopy; Mass Spectrometry; Lipids; Carbohydrates; Laser.

INVITED SPEAKERS

Id-616

TRI-MODAL (IR/RAMAN/FLUORESCENCE) OPTICAL-PHOTOTHERMAL INFRARED (O-PTIR) SPECTROSCOPY & IMAGING – ADVANCES in SPATIAL RESOLUTION & MEASUREMENTS in WATER

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Abstract: Optical Photothermal Infrared (O-PTIR) spectroscopy has established itself as a cutting edge vibrational microspectroscopy tool, offering significant advantages over the traditional FTIR/QCL & Raman spectroscopic tools, providing submicron simultaneous IR+Raman microscopy, in non-contact mode with high sensitivity. The ability to collect, for the first-time submicron IR spectroscopic data in an optical microscope has enabled new research outcomes across a range of application fields, such as life sciences (cells, tissues, bacteria), polymers, cultural heritage and microplastics. A new modality, “counter-propagating” has been engineered to provide for enhanced IR (and Raman) spatial resolution and sensitivity, through decoupling the need for a reflective objective. The IR pump beam can now be directed to the sample via the underside, thus allowing the collection objective for the visible probe (and Raman excitation beam) to be a high-NA refractive objective. This improves spatial resolution to ~300nm for both IR and Raman, whilst improving sensitivity, image quality and facilitating immersion objective studies with early results demonstrating near water-free infrared micro-spectroscopy of cells in water with a water-dipping objective for data collection To further integrate vibrational spectroscopic tools into life science workflows, we coupled widefield epifluorescence to facilitate a novel concept – fluorescence guided (or fluorescence co-located) O-PTIR microspectroscopy. Rather than, or in addition to the visible image, the fluorescence image can now be used to guide the user to the region of interest, thus combining the well-established specificity of fluorescence imaging with the broad macromolecular profiling capabilities of IR spectroscopy Several life sciences examples from bacteria, cells and tissues will be provided to demonstrate these new capabilities and how they can enable new experiments and research findings.

Keywords: O-PTIR; Raman; FTIR; Cells; Tissues; Spatial resolution; Fluorescence.

INVITED SPEAKERS

Id-618

DYNAMIC FORCE SPECTROSCOPY in SILICO: EXPLORING STRENGTH, DEFORMABILITY and TOUGHNESS of BIOLOGICAL PARTICLES

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Abstract: Mechanical testing experiments in silico provides high-resolution view of dynamic processes in soft biological matter. Two applications of dynamic force spectroscopy in silico will be presented to describe 1) the catch-slip non-covalent bonds in fibrin fibers; and 2) the dynamics of mechanical deformation biological particles. First, formation of fibrin network and its mechanical stability are essential in thrombosis and hemostasis. The strength of non-covalent A:a knob-hole bond stabilizing fibrin polymers first increases with tensile force ("catch bonds") and then decreases with force when the force exceeds a critical value ("slip bonds"). The biphasic "catch-slip" kinetics of knob-hole bond rupture is quantitatively explained using Fluctuating Bottleneck theory, formulated in terms of structural transitions in the binding pocket between the low-affinity (slip) and high-affinity (catch) states. General framework to understand the mechanical response of protein pairs capable of tension-induced remodeling of their association interface will be provided. Strengthening of the knob-hole bonds at 30-40 pN forces might favor formation of nascent fibrin clots subject to hydrodynamic shear in vivo. Second, Fluctuating Nonlinear Spring (FNS) model describing the dynamics of mechanical deformation of biological particles, such as virus capsids, will be presented. The model interprets the force-deformation spectra in terms of the “Hertzian stiffness”, elastic constant, and force range in which the particle’s fracture occurs. This model enables one to quantify the particles’ elasticity (Young’s moduli for Hertzian and bending deformations), limits of their strength (critical forces, fracture toughness) and deformability (critical deformations). The FNS model is applied to describe the protein capsids of bacteriophage P22, Human Adenovirus and Herpes Simplex virus. These nanoshells are soft (~1-10-GPa elastic modulus) with low ~50-480-kPa toughness – a regime of material behavior that is not well understood, and with the strength increasing while toughness decreasing with size. All particles are weakly ductile with deformations not exceeding 10-20% of their size. Larger particles are more resilient and more plastic compared to smaller particles.

Keywords: Dynamic Force Spectroscopy in Silico; Protein-Protein Interactions; Catch-Slip Bonds; Biological Nanoparticles; Fracture Toughness.

INVITED SPEAKERS

Id-619

ATOMIC FORCE MICROSCOPY for BIOMECHANICAL CHARACTERIZATION of CELLS and MEMBRANES

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Abstract: Due to its high sensitivity for force measurement under physiological conditions, atomic force microscopy (AFM) is a nondestructive quantitative technique ideal to investigate elastic and adhesive properties of biological cells and membranes at the nanoscale. In a typical AFM force-distance measurement, the tip is brought towards and retracted from the cell/membrane surface, where its interaction forces are monitored as a function of cantilever's displacement and recorded as force-displacement curves. As a result, a wealth of quantitative biomechanical information can be extracted from these curves. For example, our recent AFM investigations on mouse embryonic fibroblast (MEFs) have helped to elucidate the mechanical properties of their cytoplasmic and nuclear membranes, demonstrating the relative contribution of individual actin isoforms to cell surface and nuclear mechanics; and on circulating tumor cells (CTCs), demonstrating cytoskeletal alterations cancer cells undergo from early mutant to aggressive metastatic ones at the single cell levels. However, when CTCs are of concern, probing their elasticity and adhesiveness with AFM could be very challenging for two important reasons: cell scarcity (CTCs typically range from 1 to 10 cells per milliliter of blood) and dislocation of cells by the AFM tip if they are not firmly attached to the substrate surface. The first part of this talk will highlight our efforts in developing microfluidic CTC capture platforms that are compatible with AFM, namely the AFM-Chip and HB-MFP. The platforms are utilized to efficiently isolate CTCs from whole blood samples of prostate cancer patients and firmly capture them on a glass substrate for follow up AFM measurements. The second part of this talk will present in detail our findings related to measuring elastic and adhesive properties of CTCs with localized and metastatic origins, along with mechanical properties of cytoplasmic and nuclear membranes of MEFs with various beta-actin conditions.

Keywords: Atomic Force Microscopy; Cell; Membrane.

INVITED SPEAKERS

Id-620

OPTICAL SPECTROSCOPY of NEUTRAL EXCITATIONS in LAUGHLIN LIQUIDS

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Abstract: Two-dimensional electron systems in a quantizing magnetic field are of exceptional interest from the point of view of involving quasiparticles with non-Bose and non-Fermi statistics (anyons) in the applied physics. Fractional FQHE states are currently the only experimentally realized candidates for the role of a system of quasiparticles with anyon statistics. To elucidate the statistical and thermodynamic properties of anyon systems, as well as the suitability of anyons for solving technological problems, macroscopic nonequilibrium ensembles of neutral excitations with unit spin in FQHE 1/3 were created using the technique of resonant photoluminescence. The essence of this technique was as follows. Upon resonant optical excitation of an electron system confined in a GaAs/AlGaAs quantum well, a photon with a certain energy above the band gap in GaAs is absorbed. As a result, an electron from the valence band passes to the upper spin sublevel of the zero Landau level of the conduction band. A photoexcited hole relaxes quickly to the lowest spin sublevel of the heavy hole subband due to the strong spin–orbit interaction in the valence band of GaAs. Since only the lowest spin sublevel in the conductance band of GaAs is filled in equilibrium at FQHE 1/3, the recombination of a photoexcited hole with an equilibrium electron leads to the formation of a neutral excitation with spin one in the conductance band. In all optical processes, the longitudinal momentum of a photon was chosen to be equal to zero, so two types of spin one neutral excitations are possible - a spin exciton and a spin one "magneto-graviton", of which only the spin one "magneto-graviton" is a long-lived excitation. It turned out that non-equilibrium ensemble of spin one "magneto-gravitons" has such long relaxation time to the ground state (more than 10 seconds) that it can be considered as new quasi-equilibrium state of anyon matter. An optical technique for measuring the amount of spin "magneto-gravitons" in a single quantum state has been developed. Surprisingly, these neutral excitations (made of anyon quasiparticles) are proved to be "good" bosons. The number of "magneto-gravitons" in a single quantum state exceeds 10 at a temperature of 0.5 K. The low-temperature properties (up to 0.1 K) of spin one "magneto-gravitons" have been investigated.

Keywords: Photoluminescence; Electron system; Anyones; Neutral excitations.

INVITED SPEAKERS

Id-621

The STUDY of CELL DEATH MECHANISMS by RAMAN and TRANSPORT of INTENSITY PHASE-IMAGING TECHNIQUES

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Abstract: Non-invasive methods of tracking morphological cell changes are based on measurements of phase, which is proportional to the cell thickness and can be extended to measure the optical path length, and, therefore, cell volume. Additionally, Raman micro-spectroscopy is widely used for the mapping of chemical composition within live biological samples, such as cells, organoids, and tissues. It permits non-invasive and non-destructive measurements that do not require special sample preparation processes, such as dye labelling or staining. We have previously reported that we used Raman spectroscopy and Digital Holography Microscopy (DHM) to study cells undergoing apoptosis. Another form of programmed cell death, ferroptosis, is the main cause of tissue damage driven by iron overload and lipid peroxidation. Currently, only invasive cell biological assays are used to monitor the expression level and subcellular location of proteins that are known to bind iron or be involved in ferroptosis. Our group has previously reported a Raman spectroscopic method to visualize and quantify the distribution of multiple iron-binding proteins in intact cells and tissues. Here, we have replaced DHM with another method that is capable of real-time high resolution phase reconstruction. Assembling or altering a system to make the measurements required to solve the Transport-of-Intensity Equation (TIE) is cheaper and easier than implementing the associated DHM setup and measurements. For the full phase retrieval TIE requires only the data collected in the focal plane and in two symmetrically separated planes about the focus. Furthermore, TIE is robust to reduced spatial and temporal coherence, and therefore can potentially achieve a higher resolution than DHM. Since TIE can utilize incoherent sources of illumination, we implemented the TIE setup within an existing Raman microscope, which provided near simultaneous chemical composition and morphological data about the cells.

Keywords: Quantitative phase; Phase imaging; Digital holography; Transport of intensity.

INVITED SPEAKERS

Id-622

SEM/EDS that is out of THIS WORLD! ENHANCING LOW VOLTAGE QUANTIFICATION for EXTRA-TERRESTRIAL SAMPLES

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Abstract: Scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM/EDS) is commonly used to perform analysis at high accelerating voltages (15 - 25 kV), at micrometre spatial resolution. Analysis has since been extended by using low accelerating voltages to analyse compositions at sub-micrometre spatial resolution (< 100 nm) in bulk samples. Our research project demonstrates the use of a high sensitivity, annular, four channel, silicon drift detector (SDD) within an SEM operating at 6 kV. By using ultra-low beam currents (10 – 100 pA), under high vacuum, it is now possible to resolve texture and compositions of topographic complex, non-conductive samples, without the need for carbon coating. An example of non-destructive, non-invasive, qualitative analysis using the annular SDD is shown. However, the application of low voltage EDS in collecting quantitative data presents new challenges. This research focusses on the development of low voltage quantitative SEM/EDS driven by the need to compositionally quantify sub-micrometre sized platinum group element (PGE) metal nuggets present within extra-terrestrial samples. Heterogenous, sub-micrometre PGE metal nuggets from Archean meteorite impact deposits of the Barberton Greenstone Belt, South Africa, were analysed at 6 kV and quantified using pure element references. The data indicate that the PGE L- and M-line families, which occur in the intermediate energy range at 2 - 3 keV, can be accurately deconvoluted and quantified. The contribution of the transition elements (e.g., Fe and Ni) in non-pure PGE phases resulted in high totals (~ 105 mass%). The data confirm the results on Cr-Ni steel. These studies have shown that low voltage quantification with pure element references result in higher errors for both EDS and wavelength-dispersive X-ray spectrometry (WDS). This affect is attributed to the absorption edges of the Bremsstrahlung background and the uncertainties of absorption effects in the low energy range (< 1 keV) for Fe-L and Ni-L. Our aim is to improve low voltage quantification, with the use of in-house, multi-element references, which mimic the PGE metal nugget matrix. Potential in-house references have been selected from the Natural History Museum's Mertie collection; a unique collection of predominantly PGE grains extracted from mining samples taken along the Cordilleran Belt on the western seaboard of America. Classification of over 150 grains from the Mertie collection involved EDS

automated feature analysis at 25 kV. Several potential grains were chosen for WDS analysis. Quantification and elemental mapping at 25 kV were utilised to identify homogenous grains and their corresponding composition. The selected homogenous grains will be used as reference samples to test the effect on low voltage quantification of both Mertie collection grains and the PGE metal nuggets within chondrite meteorites. These results will be compared to low voltage quantification using pure element references and presented as part of this research project. The ability to analyse samples using low voltage EDS resulting in sub-micron spatial resolution quantitative and non-invasive, non-destructive qualitative data is invaluable not only to the research at the Natural History Museum, but the wider microanalysis and cosmochemistry community. The findings in this research will also be relevant to the OSIRIS-REx (NASA) return mission, who are expected to return with samples of primitive asteroid that are likely to contain PGE nuggets. The research presented is part of the EUROPLANET project on accurately quantifying PGEs ‘nuggets’ in extra-terrestrial samples using SEM/EDS and has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 871149.

INVITED SPEAKERS

Id-623

RECENT ADVANCES in the STUDY of MOLECULAR and 2D SYSTEMS USING NON-CONTACT ATOMIC FORCE MICROSCOPY

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Abstract: The last decade has seen a dramatic improvement in our ability to characterize surfaces at the atomic scale via scanning probe microscope (SPM) techniques. In particular, non-contact atomic force microscopy (NC-AFM) is now routinely capable of obtaining sub-molecular resolution, readily resolving the carbon backbone structure of planar organic molecules adsorbed on metal substrates in real space. These developments have arisen in part due to the now routine combination of scanning tunneling microscopy (STM) and NC-AFM into the same instrument, operation at cryogenic temperatures, and the controlled functionalization of the scanning probe tip by a carbon monoxide (CO) molecule or similar known passivating moiety. In this talk, I will outline the recent developments made in our group in the field of high-resolution NC-AFM of single molecules, and show how sub-molecular force mapping of single molecules can be extended to a much wider range of conditions than previously thought, and discuss the ability of NC-AFM to probe changes in molecular reactivity induced by surface-molecule charge transfer.

Keywords: STM; NC-AFM; Force mapping; SPM; Single molecule.

INVITED SPEAKERS

Id-627

SPECTRAL ANALYSIS of the IMAGE of RANDOM MONOLAYER

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Abstract: Power spectral density of the image of random monolayer is one of its fundamental characteristics that we can use for determining key parameters of the monolayer. First, we will discuss a general equation for this function and demonstrate its similarity to those applied in the theory of scattering. Specifically, it is a linear function of the squared form factor and static structure factor. The form factor is a Fourier transform of single particle or cavity brightness function and the structure factor is given by the product of particle number density and Fourier transformed pair-correlation function, increased by one. The relationship suggests that roughness measurements of particle monolayers can be considered as a type of spectroscopy, in a wider sense of the term. In case of random monolayers, the function simplifies to a partially closed form. Next, we will show how to practically use the analytical equation to determine the structure factor and pair-correlation function of the monolayer. Finally, on the examples of monolayer images published in the literature, we will demonstrate a practical application of the approach to determine important parameters of the monolayers: the particle radius, surface coverage, and dimensions of analyzed area. Our results provide a framework for a quantitative, cheap, and easy method, based on statistical analysis of the digital monolayer image, for parametrization of particle or cavity monolayers. Unlike in the methods currently used in image analysis, relying usually on individual particle identification, we do not have to localize each of the particles here. Instead, we just need to calculate the Fourier transform of the monolayer image and to least-squares fit the monolayer parameters. Therefore, the novel approach provides means for simple, efficient, and accurate measurements of particle or cavity monolayers.

Keywords: Imaging Particle Analysis; Random Monolayer; Form Factor; Static Structure Factor; Power Spectral Density; Radial Distribution Function.

INVITED SPEAKERS

Id-632

NEW ADVANCES in IMAGE PROCESSING

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Abstract: Image processing is the corner stone in many biomedical analysis chains. Today, many advances have been made in this field, especially in image restoration. After a quick review, the talk will focus on the most versatile image restoration techniques, especially those inspired from physical laws, namely quantum interactions and other quantum concepts.

Some illustrations are provided to underline the robustness of the approaches.

Keywords: Image processing; Restoration; Enhancement.

INVITED SPEAKERS

Id-633

IN-SITU TEM for BATTERY MATERIALS

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Abstract: This talk gives an overview of the PI's research program on the opportunities and challenges of in situ transmission electron microscopy (TEM) to study novel two dimensional materials and metal oxides for Li-ion and Na-ion battery applications. Various anode materials including SnO2, ZnSb, MnO2 , phosphorene were subjected to lithiation process and the transport of Li ions was visualized within their atomic structure. For SnO2 nanowires, it was observed that the Li ion transport results in local strain development preferably along (200) or (020) plans and [001] crystallographic directions. Extremely fast ionic transport was also observed along [100] directions of phosphorene and in ZnSb crystalline materials indicating that atomic structure engineering of electrode materials can provide an efficient path for the movement of larger ions such as sodium ions overcoming their sluggish kinetics. In one-dimensional materials the structural engineering of tunnels and the tunnel stabilizers can be very effective in allowing the ions to be transported at fast rates. Overall a summary will be given on new science obtained from the in situ TEM studies and how this new information can help to further grow the opportunities in materials designed for electrochemistry.

Keywords: In situ TEM; Batteries.

INVITED SPEAKERS

Id-635

APPLICATIONS of INTERNAL REFLECTION SPECTROSCOPY in FORENSIC ANALYSIS

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Abstract: Internal reflection spectroscopy is a mid-infrared technique that has become an important tool in forensic laboratories. It is rapid, easy to use, non-destructive, and inexpensive. This paper presents a comprehensive summary of the theory and applications of the technique, which is also known as attenuated total reflection (ATR) spectroscopy, in forensic analysis.

Keywords: ATR; Forensic.

INVITED SPEAKERS

Id-636

ATOMIC FORCE MICROSCOPY CIRCULAR MODE: APPLICATIONS in MATERIAL SCIENCE and BIOLOGY

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Abstract: The atomic force microscopy circular mode was developed by O.Noël and P-E Mazeran in 2008. Its principle consists in applying a circular displacement of the *AFM* probe in the plane of the sample. In these conditions, the lateral force measurements are done in a stationary regime, without stop periods, and with a constant sliding velocity, whose value can reach more than 1 mm/s (depending on the piezo-ceramic scanner resonance). I will show that this mode can be a new powerful tool for investigating friction or wear mechanisms of materials such as polymers, composites, and for probing mechanical properties of complex biological objects such as cell membranes or skin at a local scale.

Keywords: Atomic Force Microscopy; Surface science; Friction; Wear; Physics of biological membranes.

INVITED SPEAKERS

Id-637

IN SITU GRAPHENE LIQUID CELL INVESTIGATION of METAL ION MODIFIERS on CALCIUM OXALATE

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Abstract: Calcium oxalate is a salt with ubiquitous presence in nature, including plants and as a part of the nephrolithiasis pathology in humans. Calcium oxalate and calcium oxalate mixed stones constitute approximately 70% of all urinary stones, thus making it a subject of research interest from both the pathological and materials science perspectives. Precipitation of calcium oxalate is possible due to the excretion pathway of oxalate and calcium, and crystallization is greatest when the two are in a 1:1 ratio. Although supersaturation of calcium oxalate often occurs *in vivo*, it does not always lead to crystallization due to the naturally occurring inhibitors. Recently, metal ions became of interest as calcium oxalate modifiers due to their ability to affect supersaturation and bind to calcium oxalate *in vivo*. To elucidate the mechanisms of crystallization, our group has previously identified the classical and non-classical nucleation pathways using *in situ* graphene liquid cell (GLC) TEM. Moreover, the addition of citrate, a known inhibitor, led to the formation of a metastable form, calcium oxalate dihydrate, suggesting an increased hydration state of calcium oxalate. The aim of the present study is to discern the effects of metal ions such as iron, nickel, and copper and their ability to modify calcium oxalate crystallization. Preliminary *in situ* GLC STEM studies using control calcium oxalate indicate the presence of classical and non-classical mechanisms at atomic level resolution, with evidence of specific non-classical aggregation mechanisms such as coalescence and Ostwald's ripening. *Ex situ* TEM work provides further supporting evidence of an amorphous to crystalline transition. In addition, both stable and metastable forms of calcium oxalate were observed. We anticipate that all three metal ions will have a modifying effect on calcium oxalate crystallization. The degree of modification is to be determined.

Keywords: TEM; Liquid Electron Microscopy; Calcium Oxalate, Biomineralization.

REGULAR SESSIONS

Id-568

PRECIPITATION in Al-Li-BASED ALLOY STUDIED by IN-SITU TEM

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Abstract: Al-Li-based alloys belong to a group of age-hardenable structural materials used in stiffness-critical structures in aircraft and aerospace applications. Compared with commercial alloys of the AA 7XXX series, they possess better mechanical properties (high specific strength and low density). A new generation of Al-Li alloys contains lower Li content suppressing thus the formation of unfavorable metastable Al_3Li phase. This phase precipitating in the form of coherent spherical nanoparticles is responsible for a strain localization during loading and failure of structural materials manufactured from alloys belonging to the previous generation. In-situ electron microscopy with unique heating holders was established as a suitable and fast method providing first information about microstructural changes on a micro- and nano-scale level. A factor limiting the relevancy of observed processes is the thickness of specimens transparent for electrons. The thickness of aluminum foils generally used in TEM is in the order of 100 – 200 nm, a dimension comparable with the size of strengthening particles in Al-Li alloys in peak-aged conditions. Therefore, the precipitation of these particles in thin foils is significantly affected by a constraint volume and free surfaces imposed by the shape of the specimen. Precipitation and growth of strengthening particles in twin-roll cast Al-Li-Mg-Cu-Zr alloy were studied by in-situ TEM during isothermal heating at 160 and 180 °C. Results were compared with observations performed on annealed bulk specimens. The high strength of new alloys is induced by fine dispersions of precipitates of three main strengthening phases. Peak-aged materials contain plate-like or needle-shaped precipitates rich in Cu, Li, and Mg, namely T(Al_2CuLi), S' (Al_2CuMg), and θ' (Al_2Cu) phases. Significant differences in their morphology and distribution between bulk specimens and in-situ annealed ones were observed.

Keywords: In-Situ TEM; Aluminum alloy; Precipitation; Twin-Roll Casting; Age-Hardening.

REGULAR SESSIONS

Id-569

HIGH-TEMPERATURE TRANSFORMATIONS in Al-Li-BASED ALLOY STUDIED by ACOM-TEM during IN-SITU HEATING

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Abstract: The solidification range of aluminum alloys under metastable cooling conditions varies from 10 K for Al99.5% to more than 160 K for high-alloyed Al-Zn-Mg alloys. This feature achieves a homogeneous distribution of alloying elements difficult in high-alloyed aluminum manufactured by means of conventional casting processes, such as DC or mold casting. Twin-roll casting with cooling rates close to 10^3 K/s could partially suppress the formation of a significant macroscopic gradient of alloying elements – a feature typical for DC casting. The high solidification rate results in a formation of a fine dendritic microstructure with relatively small intermetallic particles. Therefore, the relatively high content of alloying elements in high-alloyed alloys can be almost homogeneously distributed in the strip. However, in materials containing iron as an impurity coarser eutectic Cu and Fe-bearing particles could form on boundaries of eutectic cells and they should be dissolved or transformed during high-temperature homogenization annealing. In-situ heating in TEM could provide fast information about temperature ranges of phase transformations. However, a parallel identification of newly formed phases based on commonly used chemical EDS analysis is restricted only to ambient temperatures due to a radiation saturation of the detector at higher temperatures. This problem could be partially suppressed in microscopes equipped with automated crystal orientation and phase mapping in TEM (ACOM-TEM). Al-Li-based alloy of a third generation containing 3.71% Cu, 0.91% Li, 0.31% Mg, 0.15% Zr, and 0.05% Fe was prepared by twin-roll and mold casting. Thin specimens were in-situ annealed in TEM up to 540 °C and the transformation of boundary eutectic particles was monitored. The combination of TEM images with signals from several detectors including conventional STEM and SEM detectors and ACOM-TEM system provided complex information about the decomposition of primary phase particles and their transformation into new ones. Dissolution of complex Al-Cu-Fe primary particles occurs above 500ºC and it is accompanied by a formation of binary phases Al-Fe and Al-Cu.

Keywords: In-Situ TEM; ACOM-TEM; Homogenization; Al-Li alloy; Twin-Roll Casting.

POSTER SESSIONS

Id-573

TIME-RESOLVED SOFT X-RAY EXCITED LUMINESCENCE SPECTROSCOPY of SOLIDS STATE SCINTILLATORS

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Abstract: We present the measurement of luminescence kinetics of scintillators, excited by a table-top soft X-ray source – laser-produced plasma. Plasma is induced by Q-switched Nd:YAG laser (λ = 1064 nm, 600 mJ, 7 ns pulse) focused into argon gas-puff target. Scintillators as photon energy converters constitute the foremost X-ray detection component in a wide variety of medical X-ray imaging systems. Thus, their properties are determinative for performance of such devices. However, practical use of scintillators is often limited by their luminescence kinetics, i.e., by the speed of radiative deexcitation of luminescent centers. Thus, the investigation of luminescence kinetics plays one of the major roles in the development of new scintillation materials. Our contribution to this research consists in development and application of a new spectroscopic technique, which would allow us to measure the luminescence decay within a very wide temporal range (ten ns to > ms), with nanosecond temporal resolution and very high signal-to-noise ratio ($> 10^5$). We meet these requirements by exploiting a combination of an intense nanosecond soft X-ray excitation pulse, a very short absorption length of the soft X-rays, and a fast photomultiplier-based detection. Our instrumentation allows us to measure different types of samples, such as powders, single crystals, and ceramics. Here we present a demonstration of measured luminescence response of rare-earth-doped lutetium aluminum garnet (LuAG:Ce) with/out Mg. The temporal instrumental response function (IRF) was detected by a fast ZnO:Gd scintillator (τ_i = 0.8 ns).

Keywords: Time-Resolved Spectroscopy.

POSTER SESSIONS

Id-625

ELECTRODEPOSITION of Ag COMPOSITES with CARBON NANOTUBES USING DIRECT and PULSE PLATING in CHOLINE CHLORIDE BASED IONIC LIQUIDS with APPLICATIONS in SURFACE-ENHANCED RAMAN SPECTROSCOPY (SERS)

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Abstract: Silver layers have been widely used in microelectronics, aerospace, automotive, jewelleries, catalysis, bio-sensing, and Surface-enhanced Raman spectroscopy due to their excellent physio-chemical properties, good corrosion resistance, high bulk conductivity, and decorative features. Electrodeposition is an easy and economical way to prepare metals, alloys, and composite coatings. However, Ag deposits are electrochemically prepared from cyanide-based electrolytes which exhibit higher toxic effect to the environment and human health. An environmentally friendly electrolyte for the synthesis of Ag deposits is „deep eutectic solvent "(DES). These solvents consist in a eutectic mixture of a hydrogen bond acceptor (HBA) with a hydrogen bond donor (HBD), in the right proportion, and exhibit high biodegradability and recyclability, low volatility, and good conductivity. In addition, they show high viscosity allowing them to act as good dispersants of carbon-based materials. Among carbon-based nanomaterials, carbon nanotubes present high electrical conductivity and high strength. Therefore, the incorporation of multi wall carbon nanotubes (MWCNTs) into the metallic matrix of silver can lead to an improvement of the properties of the deposits. Under these circumstances, the paper presents some experimental results regarding the electrodeposition and characterization of Ag composites with carbon nanotubes (denoted Ag-MWCNT) involving deep eutectic solvents. A novel DES electrolyte was proposed, consisting in choline chloride-triethanolamine (IL-TEA) eutectic mixture (1:1 molar ratio). Dispersion stability of multi-walled carbon nanotubes (MWCNTs) was found to be excellent in the used eutectic solvent. The influence of applied current form, respectively of direct current *vs.* pulse current on the deposit appearance and carbon nanotubes incorporation is also discussed. According to SEM and Raman analysis, the carbon nanotubes were successfully inserted into the metallic matrix, being favoured by the use of pulsed current. Metallic light grey coatings, with a good adherence and uniformity onto the Cu substrate have been obtained at a working temperature of 70-80^{o}. XRD pattern suggested a good crystallinity of the Ag layer. The Ag-MWCNTs composites obtained by electrodeposition under different plating parameters were tested as Surface-enhanced Raman spectroscopy substrates for methylene blue (MB) detection. The intensity of the characteristic methylene blue peaks is enhanced using Ag-MWCNT composite as substrate, even at quite low concentrations of MB dye. This work has received funding from the European Union's Horizon 2020 research and innovation program under the Marie Skłodowska-Curie grant agreement No764977 and from the ECSEL Joint Undertaking (JU) under grant agreement No. 875999 (IT2). The JU receives support from the European Union's Horizon 2020 research and innovation programme and Netherlands, Belgium, Germany, France, Austria, Hungary, United Kingdom, Romania, Israel.' ME acknowledge the ECSEL Joint Undertaking (JU) under grant agreement No. 826589 (MADEin4). The JU receives support from the European Union's Horizon 2020 research and innovation programme and Netherlands, Belgium, Germany, France, Italy, Austria, Hungary, Romania, Sweden and Israel,'

Keywords: Silver; MWCNTs; Deep eutectic solvents; SERS.

POSTER SESSIONS

Id-626

STRESS ANALYSIS by XRD and RAMAN on DIFFERENT SEMICONDUCTOR SUBSTRATES

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Abstract: Nowadays, semiconductors can be found nearly in every electronic device such as in our computers, phones, cars, and household appliances. Their impact in the global economy is huge, as a recent report from Oxford Economics pointed out, the U.S semiconductor industry contribute with 246.4 billion dollars to U.S. GDP. The most common intrinsic semiconductor elements are silicon (Si) and germanium (Ge), having an energy gap of 1.12 and 0.67 eV respectively. In order to improve the conductivity of semiconductors, impurities can be added which can either contribute with free electrons, named n-type semiconductors, such as P, As, Sb, or creating holes, p-type semiconductors, where elements as Ga, B, In and Al relies in this category. Apart from elemental semiconductors, compound semiconductors, result from the combination of two or more elements from Group III and Group V, such as GaAs, GaN, GaP, InAs, InSb, InN and others, or from Groups II and VI (CdSe, ZnS, ZnO) and even elements from the same group such as SiC. A compound semiconductor could also be dopped by slightly variating in the stoichiometry. The first wafer commercially available was produced in 1960s and was only 0.75-inch (20 mm) in diameter and made out of silicon. Half a century of trials and errors later, it finally became possible to manufacture 300 mm (12-inch) wafers. However, during the wafer manufacturing process, minor variations in the processing parameters can produce changes in the crystal lattice of the wafer. Therefore, stress control in semiconductor devices is of primary importance. Micro-Raman and X-ray diffraction spectroscopies can be used to investigate the possibility of existence of stress in semiconductors. Typically, it is possible to detect and observe stress by analyzing the Raman shift in the band position, and the band shape, broadening and deformation of peaks. The changed caused in the inter planar spacing, d, when a material is strained can be evaluated by X-ray diffraction where a precise measurement of this shift is determined. From the lattice strain, the stress can be determined considering the Young's modulus of the material. To determine the stress of different semiconductor materials, the Center for Surface Science and Nanotechnology (CSSNT) implemented the RAMAN and XRD analysis techniques. Measurements were made to characterize the stress on different semiconductors wafers with dimensions ranging from 150, 200 and 300 nm. For the measurements, CSSNT has developed a special stage for XRD and RAMAN, thus allowing a mapping

stress on the entire wafer surface from cm to cm. This project has received funding from the ECSEL Joint Undertaking (JU) under grant agreement No. 876124 (BEYOND5). The JU receives support from the European Union's Horizon 2020 research and innovation programme and France, Germany, Sweden, Turkey, Belgium, Poland, Israel, Netherlands, Switzerland, Romania. This work was supported by Romanian Ministry of Research Innovation and Digitalization, Romania, under ECSEL-JU POC 2014-2020 project: BEYOND5 Contract no. 12/1.1.3H/31.07.2020 Code MySMIS 136877. Also, from the ECSEL Joint Undertaking (JU) under grant agreement No. 783127 (OCEAN12). The JU receives support from the European Union's Horizon 2020 research and innovation programme and France, Germany, Austria, Portugal, Greece, Spain, Poland, Romania'. M.E acknowledge the support from the ECSEL Joint Undertaking (JU) under grant agreement No. 783158. The JU receives support from the European Union's Horizon 2020 research and innovation programme and Italy, Switzerland, Germany, Belgium, Sweden, Austria, Romania, Slovakia, France, Poland, Spain, Ireland, Israel.'

Keywords: Semiconductors; Wafer; X-ray diffraction; Raman.

ALL SUBMISSIONS & TOPICS

Topics	Submissions
Applications of Microscopy &Spectroscopy in the Biological Sciences	Id 596 - Using Microscopy to Assess the Effects of Surface Properties and Culture Medium during Recombinant Protein Production in Biofilms
	Id 614 - Cold Ion Fragmentation Spectroscopy-Mass Spectrometry for Identification of Isomeric Carbohydrates and Lipids
	Id 616 - Tri-modal (IR/Raman/Fluorescence) Optical-Photothermal Infrared (O-PTIR) Spectroscopy & Imaging – Advances in Spatial Resolution & Measurements in Water
	Id 618 - Dynamic Force Spectroscopy in Silico: Exploring Strength, Deformability and Toughness of Biological Particles
	Id 619 - Atomic Force Microscopy for Biomechanical Characterization of Cells and Membranes
	Id 621 - The Study of Cell Death Mechanisms by Raman and Transport of Intensity Phase-Imaging Techniques
	Id 637 - In situ Graphene Liquid Cell Investigation of Metal Ion Modifiers on Calcium Oxalate
Applications s of Microscopy & Spectroscopy in the Physical/Chemical Sciences, at All Dimensional Scales	Id 593 - Possibilities and Features of Auger Spectroscopy Studies of Nanostructures and Nanoinclusions in Composite Ceramic Bulk Materials, Coatings and Thin Films
	Id 633 - In-situ TEM for Battery Materials
Semiconductor Materials and Devices	Id 579 - Electrical Characterization of Semiconductor Films through Depth Profiles Obtained by Differential Hall Effect Metrology (DHEM)
	Id 626 - Stress Analysis by XRD and Raman on Different Semiconductor Substrates
Surfaces/Films/Coatings	Id 625 - Electrodeposition of Ag Composites with Carbon Nanotubes Using Direct and Pulse Plating in Choline Chloride Based Ionic

	Liquids with Applications in Surface-Enhanced Raman Spectroscopy (SERS)
Atomic Force Microscopy	Id 636 - Atomic Force Microscopy Circular Mode: Applications in Material Sciences and Biology
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Image Simulation and Image Processing Techniques	Id 632 - New Advances in Image Processing
In-Situ Microscopy Techniques	Id 568 - Precipitation in Al-Li-Based Alloy Studied by In-Situ TEM
	Id 569 - High-Temperature Transformations in Al-Li-Based Alloy Studied by ACOM-TEM during In-Situ Heating
Low Energy Electron Microscopy	Id 611 - Low Energy Electron Emission from Surfaces, 2D-Materials Formation of 2-D Electron Cascade in the STM-Field Emission Regime
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