E·MRS

2014 Spring Meeting Lille, France – May 26th - 30th

SYMPOSIUM H

ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

Symposium Organizers:

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Published in pss(c) and pss(a) (Wiley). Guest editor: Andreas Nutsch, Physikalisch-Technische Bundesanstalt, Berlin Germany.

PROGRAM VIEW : 2014 Spring
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ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

26 May 2014 | 27 May 2014 | 28 May 2014 | 29 May 2014 | 30 May 2014

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09:00	**Welcome to ALTECH 2014 - opening addresses by current and former ALTECH organizers as well as by LNE : Burkhard Beckhoff, Bernd Kolbesen and Jean-Rémy Filtz**	
	Recent Developments in Instrumentation for Nano Characterisation 1 : Burkhard Beckhoff and Luca Boarino	
09:15	**Plasmonics for solid state lighting** **Authors :** Jaime Gomez Rivas, Gabriel Lozano, Said R.K. Rodriguez, Marc A. Verschuuren **Affiliations :** Center for Nanophotonics,FOM Institute AMOLF, c/o Philips Research, High-Tech campus 4, Eindhoven, The Netherlands and COBRA Institute, Eindhoven university of technology, Eindhoven, The Netherlands; Center for Nanophotonics,FOM Institute AMOLF, c/o Philips Research, High-Tech campus 4, Eindhoven, The Netherlands ; Center for Nanophotonics,FOM Institute AMOLF, c/o Philips Research, High-Tech campus 4, Eindhoven, The Netherlands; Philips Research, High-Tech campus 4, Eindhoven, The Netherlands **Resume :** During recent years, developments in light emitting materials have opened the door for white-light LEDs, in which several wavelengths are combined to mimic the solar spectrum. The most extended route to achieve white-light emitting LEDs consists in using a material, the so-called phosphor, which absorbs a fraction of the light emitted by a blue LED and re-emits at a longer wavelength. The mixing of the non-absorbed blue light with the emission of the phosphor provides a white spectrum. In order to develop efficient light sources based on LEDs, research efforts have mainly focused on both improving the intrinsic quantum efficiency (QE) and the stability of light emitters, and on light extraction mechanisms. We have recently demonstrated that metallic nanoparticles can significantly improve the performance of highly efficient dyes employed in SSL by measuring up to a 60-fold enhancement of the emission in certain directions [1]. Specifically, we made use of square arrays of nanoparticles that sustain localized surface plasmon polaritons. These localized resonances couple with each other through diffracted orders in the periodic array, leading to collective plasmonic modes. The collective resonances are responsible for large field enhancements that extend into the surroundings of the particles, where the phosphor is located, shaping the spectrum of the emitted light and beaming most of this emission into very small solid angles. [1] Lozano et al., LSA, 2, e66 (2013).	H1 1
	add to my program (close full abstract)	
09:45	**Recent achievement in the micro- and nano-materials characterization by scanning photoemission imaging and spectromicroscopy** **Authors :** Matteo Amati, Luca Gregoratti **Affiliations :** Elettra - Sincrotrone Trieste S.C.p.A. di interesse nazionale, Trieste, Italy **Resume :** Scanning PhotoEmission Microscopes (SPEMs) use a direct approach to chemically characterize surfaces at the submicron scale. They employ a small, focused X-ray probe and a photoelectron detection setup synchronized with the scanning system of the sample with respect to the photon beam. The X-ray beam can be downsized to a diameter of 120 nm which allows an effective imaging resolution of less than 50 nm with an overall energy resolution better than 200 meV. Recent achievements in the chemical, physical and electronic characterization at the micro- and nano- scale of materials will be presented	H1 2

providing an overview of the capabilities of this powerful technique. Examples will be focused on the characterization of Solid Oxide Fuel Cells showing how dynamic phenomena such as mass transport can be monitored by photoemission spectromicroscopy. In particular the authors have developed novel and cheap technical solutions capable to address one of the major limitation for photoemission techniques (XPS and AES), namely the need for low -vacuum environment (pressure gap), allowing detailed analysis of materials under working conditions. Feasibility test and first results of the technical approaches developed at Elettra for photoemission microscopes, but potentially able to address the "pressure gap" in any XPS system, will be presented and discussed.

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10:00 **Coffee break**

Recent Developments in Instrumentation for Nano Characterisation 2 : Miklos Fried and James Blakesley

10:30 **Transmission electron microscopy of bimetallic nanoparticles: In-situ heating and 3D tomography** H2 1

Authors : Ning Lu*, Jinguo Wang*, Shuifen Xie+, Younan Xia+, Moon Kim*
Affiliations : *Department of Materials Science and Engineering, The University of Texas at Dallas, Richardson, Texas 75080 USA; +The Wallace H. Coulter Department of Biomedical Engineering, Georgia Institute of Technology and Emory University, School of Chemistry and Biochemistry and School of Chemical and Biomolecular Engineering, Georgia Institute of Technology, Atlanta, GA 30332 USA

Resume : Nanosize metallic particles have long been recognized as important heterogeneous catalytical materials. Recent studies show that the atomic characteristics of metallic nanoparticles, including particle size, shape and surface composition, are critical to catalytic activity and selectivity. It has been proven that the catalytic activities of noble metals are highly dependent on their surface structures in many reactions. Given that the surface structures of nanocrystals have a strong correlation with their morphologies, morphology control of nanocrystals has become a central theme of research with an ultimate goal to tune the nanocrystal catalytic performance. Herein, we use advanced scanning/transmission electron microscopy (S/TEM) techniques, in-situ heating and tomography, to investigate the shape stability of nanocubes at elevated temperature and the 3D morphology of concave particles. Using in-situ heating S/TEM, we have demonstrated one effective approach to enhance thermal stability of Pd nanocubes at elevated temperature by coating high melting point metal Rh on corners and edges to form core-frame bimetallic nanocubes. The Pd -Rh core-frame cubes could maintain cubic shape after annealing 1 hour at 500 °C in contrast to the apparent truncation of pure Pd cube at 400°C for 8.5 min. Surface pre-melting of Pd is suppressed by the surface diffusion of Rh from corners and edges to {100} side surface. This strategy of shape stabilization can be extended to the development of other multi-metallic nanocrystals for broad high temperature applications. High angle angular dark field (HAADF) - scanning transmission electron microscopy (STEM) tomography allows a very precise 3D description of the particle size and morphology. Concave Pd-Pt bimetallic nanocrystals with ~1 nm Pt frame were investigated. Via 3D tomography, the morphology of the concave shape has been clearly confirmed. Combined with aberration (Cs) corrected STEM atomic and composition analysis with 3D results, the site-specific growth mechanism of Pt on Pd seed can be confirmed. These unique and effective site-specific analysis tools could be widely applied to other bimetallic or multi-metallic nanocrytals. This work was supported by AOARD-AFOSR (FA2385-10-1-4066). The synthesis work was supported in part by a grant from the NSF (DMR-1215034) and start-up funds from Georgia Institute of Technology.

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10:45 **Design and development of a new experimental set-up to study solid-gas reactions and processes in nanomaterials at isobaric and isothermal environment by synchrotron X-ray powder diffraction** H2 2

Authors : E. Salas-Colera 1-2, A. Mu?oz-Nova l1-2, C. Heyman 3, J. Rubio-Zuazo 1-2 and G.R. Castro 1-2
Affiliations : 1 Spanish CRG BM25 Beamline SpLine at the ESRF, Grenoble, France; 2

Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC, Madrid, Spain; 3 CAO-DAO Heyman 5, place de Gordes,38000 Grenoble, France.

Resume : A new experimental set-up has been designed and performed for synchrotron X-ray powder diffraction in transmission geometry (capillary) for in situ solid-gas reactions and processes at isobaric and isothermal environment. One of the novel elements of this set-up is the home designed rotor sample holder which permits spinning the capillary and keeping constant the gas atmosphere in the sample. The temperature and pressure of the sample can be set and kept stable in the ranges from 77 to 1000 K and from 1 to 103 mbar, respectively, from a vacuum condition < 10-3 mbar. The studies of gas induced structure deformation in a nanoporous material such as a zeolitic imidiazol framework (ZIF-8) by the gas adsorption at cryogenic temperature at isothermal/isobaric conditions have been carried out to test capacities of this new experimental setup. Direct evidences of structure deformation by the adsorption of Ar and N2 have been observed in situ, making this set-up suitable for direct structural analysis at in operando conditions. Therefore, the presented results demonstrate the feasibility of this novel experimental station for the characterization at real time of solid-gas reactions and other solid gas processes by high resolution powder diffraction. This set-up has been developed and installed in the Spanish CRG BM25-SpLine beamline at the European Synchrotron Radiation Facility (ESRF).

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11:00

In situ HVEM irradiation study of intrinsic point defect behavior in Si nanowire structures

Authors : J. Vanhellemont1, S. Anada2, T. Nagase2, H. Yasuda2, H. Bender3, R. Rooyackers3, and A. Vandooren3
Affiliations : 1Department of Solid State Sciences, Ghent University, Belgium; 2Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, Japan; 3IMEC, Leuven, Belgium

Resume : Si nanowire-based tunnel-Field Effect Transistor characteristics are intensively studied as function of nanowire diameter and doping. A significant reduction of B diffusion with decreasing nanowire diameter is e.g. observed and attributed to reduced transient enhanced diffusion close to the nanowire surface caused by the recombination and out-diffusion of excess self-interstitials. In an Ultra High Voltage Electron Microscope (UHVEM), the formation of self-interstitial clusters can be studied in situ while varying e-beam flux, irradiation temperature, impurity concentration and capping layers surrounding the nanowires. First results are presented on {113}-defect formation in Si nanowires with diameters between 40 and 500 nm. The Si nanowires on a heavily As doped Si substrate, are embedded in Si oxide and are etched into an epitaxial Si stack, of which the top layer is either in situ B doped or implanted. In situ UHVEM studies are performed on focused ion beam prepared cross-section samples, irradiating with different fluxes of 2 MeV electrons between room temperature and 375 ºC. A strong dependence on nanowire radius and doping is observed. The observations are compared with simulations based on quasi-chemical reaction rate theory.

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11:15

Laboratory based X-ray Emission Spectroscopy for the investigation of chemical states

Authors : L. Anklamm, W. Malzer, C. Schlesiger, S. Schuh, and B. Kanngießer
Affiliations : Technische Universität Berlin, Hardenbergstr. 36, 10623 Berlin, Germany

Resume : High resolution X-ray emission spectroscopy (XES) is an emerging method for the investigation of the electronic structure of materials. Measurement of the band structure is one of the applications. Nowadays, research relying on XES is dominated by experiments using synchrotron radiation. While current experimental setups allow for advanced experiments, the “high barrier” accessibility hampers the exploitation of the entire potential of XES. With this contribution, we present first experimental results and characteristics of a laboratory based XES spectrometer. It combines high efficiency with high spectral resolving power. In favorable situations, a complete spectrum of the Kβ multiplett of a transition metal can be aquired within half an hour and with a spectral resolution E/ΔE of around 2000. Nano-scaled layer require longer acquisition times. Taking an XES spectrum of a Fe-layer of 300 nm thickness takes around 10 hours in the current configuration. In future setups, the power of the X-ray tube, and thus the sensitivity of the instrument, could be scaled up by a factor of 10. We will present the principle of the XES laboratory spectrometer and results of demonstration and characterization experiments. The properties and peculiarities of the spectrometer and the

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spectra obtained will be discussed as well as possible future developments and improvements.

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Analytical Techniques for Determination of Physical and Chemical Surface Properties : Bernd Kolbesen and Cor Claeys

11:30 **Metrology for surface chemical analysis at the nanoscale: Status and challenges**

Authors : W. E. S. Unger
Affiliations : BAM Federal Institute for Materials Research and Testing Unter den Eichen 87, 12205 Berlin Germany

Resume : The International Bureau of Weights and Measures (BIPM) defines metrology, known also as the Art of Measurement, as "the science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology." [1] Besides the establishment of full uncertainty budgets traceability is another aspect of metrology. BIPM explains the concept of traceability as it follows. "Traceability means that the result of a measurement, no matter where it is made, can be related to a national or international measurement standard, and that this relationship is documented. In addition, the measuring instrument must be calibrated by a measurement standard that is itself traceable. Traceability is thus defined as the property of the result of a measurement or the value of a standard whereby it can be related to stated references … The concept of traceability is important because it makes possible the comparison of the accuracy of measurements worldwide according to a standardized procedure for estimating measurement uncertainty." [2] Surface chemical analysis is a much younger discipline in comparison to other branches in analytical chemistry as, e.g., electro-chemistry, inorganic and gas analysis, and lots of work has to be done to make XPS, AES and SIMS based quantitative analysis of surface chemistry a metrological one. Looking to the analytical methods established in surface chemical analysis we may differentiate classes: Primary methods measuring amount of substance as [atoms/cm2, …] Empirical methods measuring amount of substance after calibration as fractions of a nano scaled surface layer Primary and empirical methods measuring amount of substance expressed as the thickness of a thin film [nm]. In most cases we are using empirical methods when XPS, AES and SIMS are applied to deliver quantitative data. It follows also from that list that traceability to the mol or the meter can be established. Relevant initiatives to metrologically underpin surface chemical analysis have been launched under the umbrella of the Surface Analysis Working Group at CCQM/BIPM where the National Metrology Institutes are running world-wide inter-laboratory comparisons. In Europe we have the European Metrology Research Program (EMRP) where a number of projects directly address issues of surface chemical analysis (all EMRP Projects are presented on the EMRP website, cf. [3]) and most often also by individual websites. Another aspect is that there is a strong impact of metrology in surface chemical analysis on standardization under ISO TCs 201, 202 and 229. For example, ISO 14701 [4] has been prepared using outcome of a huge key comparison (K-32) organized under CCQM/BIPM. The talk will present basics of metrology in surface chemical analysis, results of successful key comparisons organized under CCQM/BIPM and a survey on the main scientific challenges to be addressed in quantitative surface chemical analysis in the next future. References [1] www.bipm.org/en/convention/wmd/2004/ [2] www.bipm.org/en/convention/wmd/2004/ traceability.html [3] www.emrponline.eu/ [4] ISO 14701:2011 – Surface chemical analysis – X-ray photoelectron spectroscopy – Measurement of silicone oxide thickness

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12:00 **Qualitative and quantitative analyses of functionalized diamond nanoparticle for precise characterization**

Authors : Naoki Komatsu, Li Zhao, Hongmei Qin, Takahide Kimura
Affiliations : Shiga University of Medical Science

Resume : Organic functionalization of nanoparticles has been attracting significant interest to impart multiple functions to the nanoparticles, in particular, for their biological and medicinal applications. Among them, diamond nanoparticle, so called nanodiamond (ND), has been recognized as one of the best platforms due to the non- or low-toxic property and the organic character enabling the covalent functionalization. In this context, we demonstrated multi-step organic transformations on the ND surface [1] to impart the requisite functions as ND-based drug carrier [2,3] and imaging probe [4]. The

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functionalities introduced onto the ND surface were well characterized qualitatively and quantitatively by solution-phase NMRs, IR and Raman spectroscopies, and thermogravimetric and elemental analyses. Although IR has been mainly used for this kind of characterization, we realized more precise characterization of functionalized nanoparticle by use of various kinds of analytical methods. [1] L. Zhao, N. Komatsu, Angew. Chem. Int. Ed., 50 (6), 1388-1392 (2011) [2] L. Zhao, N. Komatsu, X. Chen, submitted [3] L. Zhao, H. Kojima, N. Komatsu, submitted [4] L. Zhao, A. Shiino, N. Komatsu, J. Nanosci. Nanotechnol. in press

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12:15 **Highly reproducible substrate for Surface Enhanced Raman Spectroscopy exploiting block copolymer self-assembly**

Authors : (a) Claudia Diletto, (b) Pasquale Morvillo, (a) Auriemma Finizia, (a) Claudio De Rosa, (c) Antonio Sasso, (c) Giulia Rusciano, (c) Gianluigi Zito
Affiliations : (a) Department of Chemical Sciences University of Napoli "Federico II" (b) ENEA-Italian National Agency for New Technologies, Energy and Sustainable Development-Portici Research Center (c) Department of Physics University of Napoli "Federico II"

Resume : Block copolymers (BCPs) consist of covalently linked chemically distinct macromolecules that tend to segregate into different microdomains due to their mutual repulsion. This results in the spontaneous formation of nanostructures. These microdomains may act as hosts for sequestering nanofillers of appropriate chemical affinity and geometry producing long-range order in the positioning of nanoparticles. A poly(styrene-b-methylmethacrylate) (PS-b-PMMA) amorphous BCP was employed. Thin films of PS-b-PMMA was obtained by spin-coating on ITO (Indium Thin Oxide) substrate; by applying an external electric field these lamellae were perfectly aligned side by side realizing a periodic tridimensional structure with a long range order. Finally, an innovative procedure based on the selective deposition of gold nanoparticles (AuNPs) by evaporation and condensation was also used to obtain nanostructured hybrid systems. We demonstrate that metal atoms diffuse to the preferred vertically aligned domains forming nanoparticles with spatial selectivity on the surface of the film. These nanocomposites were investigated using surface-enhanced Raman Spectroscopy (SERS). Highly reproducible substrates for SERS applications were obtained exploiting self-assembly of BCP, selective inclusion of AuNPs in the PS domains via evaporation and condensation and successive alignment of lamellar domains by application of electric fields. These substrates hold premise for single molecule detection.

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12:30 **Doped tin oxide layers as sensitising layers in optical gas sensors: A study of optical properties using spectral ellipsometry**

Authors : D. Fischer, A. Nooke, A. Hertwig, M. Weise, U. Beck, M. Kormunda (1)
Affiliations : BAM Federal Institute for Materials Research and Testing, Division 6.7, Unter den Eichen 87, 12205 Berlin, Germany; (1)J.E. Purkyne University, Faculty of Science, Department of Physics, Ceske mladeze 8, 40096 Usti nad Labem, Czech Republic

Resume : During the last decades, ellipsometry has become more and more important, parallel with the development in surface science. By using it as a key technique for the analysis of thin films, it gives access to essential surface specific information like layer thicknesses. Additionally, with applying a proper model, the dielectric function of each layer can be extracted which allows a deeper understanding of its properties. In the presented work, new non-invasive gas sensors based on the SPR-effect with ellipsometric readout were build and characterized. The sensor consists of a gold layer (~40 nm) top-coated with a doped metal-oxide (Tin Oxide, ~5 nm). The coating was attached by sputtering with doped targets with different doping concentration and doping material. In the past, it could be shown that, without the top-coating, these type of sensors can detect various gases in sensitivities down to the ppm range (in air). With the help of the doped-metal oxide, the sensitivity increases dramatically by a factor of 100. Furthermore, a selectivity for specific gases was observed which depends on the doping of the coating. To find a relation between the layer properties and the sensitivity of the sensing process, the dielectric function is characterized by spectroscopic ellipsometry with respect to the deposition parameters and the dopent concentration.

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12:45 **Lunch break**

Advanced Optical Metrology : Omar El Gawhary, Bernd Güttler and Uwe Beck

14:15 **Optical metrology for photo-lithography**
Authors : Wim M.J. Coene
Affiliations : ASML Research, Veldhoven, The Netherlands.
Resume : The technology in photo-lithography is highly driven by the ITRS roadmap, leading to a continuous shrink of the features on ICs as used in the smart devices of today's connected society. Consequently, the lithographic scanner needs to operate as close as possible to its physical limits dictated by its wavelength and numerical aperture (which is known as low-k1 imaging); this implies increasingly tighter process windows, and thus an increased need for advanced lithographic process control and metrology. Optical metrology is well qualified as a valuable metrology technique for the photo-lithographic industry, since it is fast and non-destructive with respect to the patterns written in the photo-resist layer, and since it offers a high precision. Moreover, optical metrology applied on periodic gratings can be used both for the purpose of overlay metrology as well as for critical-dimension (CD) metrology. For the latter, a model-based approach is implemented, in which grating profile parameters like line-width and side-wall angle, are retrieved which relate to the relevant scanner control knobs, like scanner focus. In the presentation, apart from the core technology of optical scatterometry, a range of applications in photo-lithography will be addressed together with a number of new challenges, like reduction of target size for overlay metrology and metrology of scanner aberrations.

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14:45 **Multi-angle spectroscopic EUV reflectometry for analysis of thin films and interfaces**
Authors : Serhiy Danylyuk, Stefan Herbert, Peter Loosen; Larissa Juschkin; Rainer Lebert;
Affiliations : Chair for the Technology of Optical Systems, RWTH Aachen University and JARA - Fundamentals of Future Information Technology; Chair for the Experimental Physics of EUV, RWTH Aachen University and JARA – Fundamentals of Future Information Technology; Bruker ASC;
Resume : Modern nanotechnology is constantly raising demands to quality and purity of thin films and interlayer interfaces. As thicknesses of employed layers decrease to single nanometers, traditional characterization tools are no longer able to satisfy throughput, precision or non-destructibility requirements. Extreme ultraviolet (EUV) and soft X-ray reflectometry has not only demonstrated the ability to detect the sub-nm thickness variations but also was shown to be very sensitive to the chemical composition changes. Since the laboratory radiation sources in this wavelength range often emitting in a relatively broad spectral range, a spectroscopic EUV reflectometry has been developed with the added benefit of the rapid measuring time on the order of milliseconds to seconds. In this contribution the extension of the method to multi-angle measurements will be presented that allows to reduce a number of fit parameters in the analysis model, making the method suitable for complex samples of unknown composition. First experimental examples for Si-based layer systems measured under grazing incidence angles between 2 and 15° will be demonstrated and discussed.

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15:00 **Micro-Raman spectroscopy as a complementary technique to High Resolution X-Ray Diffraction for the SiGe thin layers characterization**
Authors : Aurèle DURAND1-2, Denis ROUCHON1, Delphine LE-CUNFF2, Patrice GERGAUD1
Affiliations : 1 CEA - LETI, MINATEC Campus, 17 rue des martyrs, 38054 Grenoble cedex 9, France; 2 STMicroelectronics, 850 Rue Jean Monnet, 38926 Crolles, France
Resume : In the advanced transistor technology, SiGe is being used as a replacement for Si channel to achieve higher mobility and to adjust the threshold voltage. µRaman spectroscopy with its good spatial resolution and low detection limit of mean is well adapted to characterize such structures. We have evaluated the technique for the measurement of the composition measurement and we have compared it to other metrology techniques. To evaluate the µRaman capability, we have first considered thin SiGe films with a thickness

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ranging from 4 nm to 12 nm. Assuming an epitaxial pseudomorphic structure, the Ge content has been extracted for all samples with an average stability of 0.5% and accuracy better than 1% as confirmed by complementary HRXRD and SIMS measurements. Then, we studied a more complex SiGe/FD-SOI structure which is critical for the development of advanced technological nodes. Since µRaman requires hypothesis on the structural state, we discuss the interest to combine µRaman measurements with X-rays Diffraction in order to extract the Ge composition on such stacks. µRaman shows a real potential for thin SiGe film characterization as, in contrast with HRXRD, this technique does not suffer from the lack of precision for very thin film. However, some developments are required before using µRaman as a metrology technique. Moreover its potential could be enhanced by coupling it with HRXRD. In particular, this approach could be used to control SiGe condensation process on FD-SOI.

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15:15 **A geometry independent approach to the measurement of the piezoelectric coefficient of thin films**

Authors : M Stewart, S Lepadatu, M Cain
Affiliations : National Physical Laboratory

Resume : Piezoelectic transistors have the potential to increase the clock speed of chip processors tenfold to 30GHz, helping the IT industry to meet the need for continuously faster computing. The operation of these transistors depends on the generation of controlled strain in features of 10nm or less. Accurate measurement of these strains is being addressed in the pan European project Nanostrain, and here we will describe advances in understanding the measurement of strain in piezoelectric thin films using interferometry. Methods to determine the indirect piezoelectric coefficient of thin films use interferometric methods to measure the dilation of the film. The design of the experiment is crucial, most workers have found that results are highly dependent on conditions such as electrode size and sample mounting. Here we propose a new method of determining the piezoelectric effect in thin films based on a measurement of the step height at the region between the active and inactive film. This method uses the displacement profile across the electrode edge as a built-in reference to eliminate the effects of bending. Measurements and simulations show this step height is constant for a range of electrode sizes, where the traditional methods fail. This work shows, for the first time, a new method for characterizing the piezoelectric properties of thin films, which is geometry independent and allows for the rapid evaluation of new materials.

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15:30 **Coherent Fourier scatterometry as a metrology tool for periodic nanostructure characterization**

Authors : N. Kumar(a), P. Petrik(a,b), S. F. Pereira(a), H. P. Urbach(a)
Affiliations : a) Optics Research Group, Department of Imaging Physics, Faculty of Applied Sciences, Delft University of Technology, P. O. Box 5046, 2600GA Delft, The Netherlands b) Research Centre for Natural Sciences, Institute for Technical Physics and Materials Science, Hungarian Academy of Sciences, H-1121 Budapest, Konkoly Thege Miklós út 29-33, Hungary

Resume : Optical scatterometry is a well-established tool in semiconductor industry for the quality control of the lithographic manufacturing process, where gratings are used as the test target. It is fast, non-destructive, high resolution and highly accurate technique. But, with continual shrinkage in the feature size of the printed structures, there is a need of a more sensitive, robust and reliable metrology tool. Recent studies have shown that under suitable conditions Coherent Fourier scatterometry (CFS) is more sensitive to any change in the shape parameters of the grating as compared to the current metrology tools based on incoherent optical scatterometry. In CFS, a tightly focused spot from a coherent source interacts with the grating and the far field diffraction pattern is recorded for several scan positions within a period of the grating. The diffraction efficiency of the propagating order depends in a nonlinear way on the material and shape parameters of the grating. The shape parameters are then determined inversely from the far field intensity. Rigorous coupled wave analysis (RCWA) is used as the rigorous solver for the forward computation of the far field intensity maps to compare with the experimental far field. CFS performance in characterization of periodic nanostructure and the effect of surface over-layers on the structure will be discussed. Optical properties of the surface over-layers are measured by ellipsometry and the error, accuracy and sensitivity of grating parameters characterization in presence of the oxide layer will be presented. References: 1. O El Gawhary et al., Applied Physics B,105:775, 2011. 2. N Kumar et al., Proc. SPIE, 8324:83240Q, 2012.

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15:45

Challenges in using polarisation dependent Raman spectroscopy as a probe of molecular orientation in organic thin films

Authors : Alina Zoladek-Lemanczyk (1), Jong Soo Kim (2), Ji-Seon Kim (2), James Blakesley (1), Fernando A. Castro (1)
Affiliations : (1) - Materials Division, National Physical Laboratory, Hampton Road, Teddington TW11 0LW, United Kingdom; (2) - Department of Physics & Centre for Plastic Electronics, Imperial College London, London SW7 2AZ, United Kingdom

Resume : Orientation of molecules and bonds within matter is often a major factor determining its properties. Order in molecular orientation between and within domains plays a critical role in determining the optical and electronic properties of organic semiconductor films resulting in anisotropic energy levels and charge mobilities. For example, in polymers, if molecular packing is sufficient, electronic orbitals will delocalize along the polymeric backbone or couple strongly between molecules, allowing for efficient charge transport along specific directions in the material and making it suitable for use in devices such as light-emitting diodes, transistors or solar cells. Therefore understanding and quantifying the molecular alignment of functionalized materials and conjugated polymers within thin-film samples is essential for a complete picture of their optical and transport properties for the continuous development of optoelectronic device applications. Polarization-dependent Raman microscopy is a powerful technique to perform both structural and chemical analyses with submicron spatial resolution. Raman data can also be collected in situ, which makes this technique even more attractive as a non-invasive device probe. We report here on the practicality and limitations of Raman polarisation measurements as a probe of molecular orientation in organic thin films for transistor applications. Challenges in correcting for system calibration will be discussed and a simple model for the effect of molecular orientation on measurement results will be introduced in order to facilitate validation of results.

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16:00 Coffee break

Metrology for 'More than Moore' Technologies : Thierry Conard and Matthias Müller

16:30

A comparative analysis of different measurement techniques to monitor metal and organic contamination in silicon device processing

Authors : M.L. Polignano, D. Codegoni, S. Grasso, I. Mica, G. Borionetti, A. Nutsch
Affiliations : ST Microelectronics, via Olivetti, 2, 20864 Agrate Brianza (MB) Italy; MEMC Electronic Materials SpA, a Sunedison Company Viale Gherzi ,31, 28100 Novara Italy; Physikalisch-Technische Bundesanstalt, Abbestr.2-12, 10587 Berlin, Germany

Resume : A few key techniques for the analysis of contamination in silicon are compared for their ability to detect different impurities. Both metal and organic contamination is included in this study. In addition, common contaminants and elements recently introduced in the fabrication process are considered. For what concerns metal contamination, it is shown that different approaches are required depending on the in-depth distribution of the contaminant and hence on its diffusivity. Copper, iron, molybdenum and tellurium are chosen as the examples of contaminants with different diffusivity and solubility properties. TXRF, recombination and generation lifetime measurement techniques, DLTS and capacitance vs. voltage measurements are compared. The detection of slow diffusers is found to be very critical, because a very low dose may result in a non-negligible concentration in the device region. As a consequence, the sensitivity per unit area required for these elements is difficult to reach with surface techniques such as TXRF. On the other hand, very fast diffusers such as copper can be hardly revealed in the solid solution in silicon. Copper in silicon can be revealed at the oxide-silicon interface by TOF-SIMS measurements, or by surface generation velocity measurements with the Zerbst method. For what concerns organic contamination, surface recombination velocity, interface state density measurements by the conductance method, MOS-DLTS and gate oxide integrity tests were compared. The most relevant effects of organic contamination were observed by electrical stress of the oxide. Indeed, the fraction of capacitors with degraded breakdown voltage increased dramatically in wafers with intentional organic contamination.

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17:00 **Dimensional and defectivity nanometrology of directed self-assembly patterns**

Authors : C. Sim?o,a W. Khunsin,a A. Amann,b N. Kehagias,a M. A. Morris c,d and C. M. Sotomayor Torres a,e
Affiliations : Catalan Institute of Nanoscience and Nanotechnology ICN2, Campus de la UAB, Barcelona 08193, Spain; b School of Mathematics and the Tyndall National Institute, UCC, Cork Ireland; c School of Chemistry and the Tyndall National Institute, UCC, Cork Ireland; d Centre for Research on Adaptive Nanostructures and Nanodevices, TCD, Ireland; e Catalan Institute of Research and Advanced Studies, Barcelona 08010 , Spain;

Resume : One-dimensional nanostructures are a class of elements or components with key technological importance in many areas including electronic and lithography applications, self-assembly templates, x-ray gratings, photonic crystals, etc. Directed self-assembly (DSA) of block copolymers (BCP) appears in the ITRS roadmap as a potential bottom-up lithography solution for the 11 nm node. [1] The success of the implementation of block copolymer lithography in industry relies not only on the fabrication but also requires specialized nanometrology tools to control the quality of the fabricated structures.[2] The key for success of a production compatible nanometrology system is based on reliable, large-area, inline and robust inspection tools to assure the quality of the fabricated nanostructures. In our work on colloidal crystal structures and block copolymers hexagonal pattern image analysis, a positive correlation between the "opposite partner" concept and transmission spectroscopy confirmed our method to assess three-dimensional ordering.[3] Here we present the extension of this concept to self-assembled nanowires from BCPs, thus 1D features, with feature sizes below 20 nm. By analogy, our method can be used in nanowires samples of phase-separated BCPs as an inline technique. In the present work, high   BCP system polystyrene-b-polyethylene oxide (PS-PEO) line patterns in silicon substrates with different surface treatment were analysed with our full operational software. The output is time efficient (< 1 minute) morphology statystical data (length and number of lines), defect density quantifcation and alignment quantification. The defects are identified as dislocations, branching points, lone points and turning points and are depicted in histograms and analised statistically according to type. Furthermore, the pitch and linewidth variations are estimated. The morphology analysis, defect and alignment quantification of linear patterns presented in this work makes it probably unique and the fast response and simplicity of operation positions this technique as a highly promising nanometrological tool to standardize DSA characterization. The research leading to these results has received funding from the European Union Seventh Framework Program ([FP7/2007-2013] project LAMAND under grant agreement n? [245565]) and by the Spanish Ministry of Economics and Competitiveness under contract no. MAT2012-31392 (Plan Nacional de I D I (2008-2011). The contents of this work are the sole responsibility of the authors. References 1. M. Salaun et al, J. Mat. Chem. C 2013, 1, 3544?3550; D. Borah et al, Eur. Polym. J. 49 (11), 3512?3521. 2. H.N. Hansen, et al, CIRP Annals - Manufacturing Technology, 55 (2006) 721 3. W. Khunsin, et al, Adv. Funct. Mater., 22 (2012) 1812.

H5 2

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17:15 **Distinction between silicon and oxide traps in UTBOX devices using single-trap spectroscopy**

Authors : Wen Fang, Eddy Simoen, Marc Aoulaiche, Jun Luo, Chao Zhao, Cor Claeys
Affiliations : Wen Fang1,2,3; Eddy Simoen1; Marc Aoulaiche1,4;Jun Luo3;Chao Zhao3; Cor Claeys1,2 ; 1 Imec, Kapeldreef 75, B-3001 Leuven, Belgium; 2 E.E. Dept., KU Leuven, Leuven, Belgium; 3 Key Laboratory of Microelectronics Device & Integrated Technology, Chinese Academy of Sciences, Beijing 100029, China; 4 presently at Micron Technology Belgium, imec Campus, Belgium

Resume : As CMOS technology dimensions reach the nanoscale, both static and dynamic variability affect the device and circuit performance [1]. Dynamic variability better known as low-frequency (LF) noise generally scales with the inverse of the transistor area, so that large current fluctuations with time can occur in small transistors [2]. Ultimately, this results in so-called Random Telegraph Noise (RTN), caused by a single defect in the device. In that case, the current switches between a high and a low state with amplitude ID and average up and down time constants, which usually correspond with the capture and emission time [2]. The corresponding noise spectrum is a Lorentzian, characterized by a constant plateau amplitude and a corner frequency fc. While most RTNs are believed to originate from traps in the gate dielectric at some distance from the channel, it has recently been shown that also traps residing in the fully depleted film of an Ultra-thin Buried Oxide (UTBOX) Silicon-on-Insulator (SOI) nMOSFET can give rise to RTN [1]. In this work, methods to

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trace back the origin of Lorentzian-type Generation-Recombination (GR) noise will be discussed. For UTBOX devices, analysis of the LF noise at one interface can give information on the trap origin, by imposing the accumulation mode at the other interface, thereby screening the influence of the corresponding oxide traps from the other interface. Frequency-domain spectroscopy combined with time-domain measurements were taken for two typical UTBOX SOI nMOSFETs. A first essential step is to distinguish the RTN-related Lorentzians from the 1/f or flicker noise background, which can be achieved by dedicated methods. As will be shown, for a film-related RTN, fc is usually independent of the gate voltage, while a strong VGS dependence is found for an oxide RTN. This translates in the time domain in a different behavior of the capture and the emission time constants and their ratio: for an oxide RTN, an exponential gate voltage dependence is observed for the time constant ratio, from which the trap depth in the oxide can be derived. On the other hand, for a defect in the silicon film, the ratio remains constant around 1, indicating that the Fermi level is close to the trap level in that case. In conclusion, the proposed methods enable to identify the exact origin, i.e., semiconductor or oxide traps, of dynamic variability in advanced MOSFETs. References [1] E. Simoen, M. Aoulaiche, S. D. dos Santos, J.A. Martino, V. Strobel, B. Cretu, J.-M. Routoure, R. Carin, A. Luque Rodríguez, J.A. Jiménez Tejada, and C. Claeys, ECS J. of Solid St. Science and Technol., 2, Q205 (2013). [2] M.J. Kirton and M.J. Uren, Adv. in Phys., 38, 367 (1989).

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17:30 **Towards single-trap spectroscopy: Generation-recombination noise in UTBOX SOI nMOSFETs**

Authors : E. Simoen1,2, B. Cretu3, W. Fang1,4,5, M. Aoulaiche1,6, J.-M. Routoure7, R. Carin7, S. dos Santos7, J. Luo4, C. Zhao4, J.A. Martino8 and C. Claeys1,5

Affiliations : 1Imec, Kapeldreef 75, B-3001 Leuven, Belgium 2Depart. Of Solid-St. Physics, Ghent University, Gent, Belgium 3ENSICAEN, UMR 6072 GREYC, F-14050, Caen, France 4Key Laboratory of Microelectronic Devices & Integrated Technology, Institute of Microelectronics, Chinese Academy of Sciences, Beijing, 100029, China 5E.E. Dept., KU Leuven, Leuven, Belgium 6presently at Micron Technology Belgium, imec Campus, Belgium 7 University of Caen, UMR 6072 GREYC, F-14050, Caen, France 8LSI/ PSI/USP - University of S?o Paulo, SP, Brazil

H5 4

Resume : Performing defect spectroscopy in nano-scaled structures is a challenging endeavor. One technique that appears to be very suited for this task is low-frequency (LF) noise spectroscopy [1]. It is well established that in the ultimate limit, the LF noise spectrum is dominated by a single trap, giving rise to a so-called Random Telegraph Signal (RTS), whose up and down time constants can be studied to derive the defect level parameters [2]. At the other end of the scale, large MOSFETs may exhibit Generation-Recombination (GR) noise, which corresponds with a Lorentzian spectrum. The corner frequency f0 of such a Lorentzian can be investigated as a function of the temperature and analyzed in an Arrhenius diagram, yielding the activation energy and the capture cross section of the underlying GR centers in the depletion region of the semiconductor [3,4]. In this paper, the principles of GR noise spectroscopy will be outlined, based on n-MOSFETs fabricated in Ultra-Thin Buried Oxide (UTBOX) Silicon-on-Insulator (SOI) substrates. As will be shown, the fully depleted (FD) nature of the devices offers some unique opportunities to study deep levels in the thin silicon film [5]. In fact, it is demonstrated that the Lorentzians observed in the noise spectra correspond with only one or at most a few traps in the film, so that the corresponding RTS becomes visible in the time domain [6]. It will be shown that analyzing the RTS parameters (current amplitude ID; capture and emission time constant) as a function of the operation biases enables to determine the lateral and vertical position of the GR center in the silicon film [7]. References [1] N. Lukyanchikova, in Noise and Fluctuations Control in Electronic Devices, edited by A. Balandin, American Scientific, Riverside, CA, 2002. [2] M.J. Kirton and M.J. Uren, Adv. in Phys., 38, 367 (1989). [3] I. Lartigau, J.M. Routoure, W. Guo, B. Cretu, R. Carin, A. Mercha, C. Claeys, and E. Simoen, J. Appl. Phys., 101, 104511-1 (2007). [4] S. D. dos Santos, B. Cretu, V. Strobel, J.-M. Routoure, R. Carin, J.A. Martino, M. Aoulaiche, M. Jurczak, E. Simoen and C. Claeys, Solid-St. Electron. (accepted). [5] A. Luque Rodr?guez, J.A. Jim?nez Tejada, S. Rodriguez-Bol?var, M. Aoulaiche, C. Claeys and E. Simoen, IEEE Trans. Electron Devices, 59, 2780 (2012). [6] E. Simoen, M. Aoulaiche, S. D. dos Santos, J.A. Martino, V. Strobel, B. Cretu, J.-M. Routoure, R. Carin, A. Luque Rodr?guez, J.A. Jim?nez Tejada, and C. Claeys, ECS J. of Solid St. Science and Technol., 2, Q205 (2013). [7] W. Fang, M. Aoulaiche, E. Simoen, J. Luo, C. Zhao, and C. Claeys, submitted to IEEE Electron Device Lett.

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18:00 **Effect of the miniband formation on the carrier lifetime in silicon nanodisk array structure fabricated by using bio-templates and neutral beam etching techniques**

Authors : D. Ohori(1), Y. Murayama(1), K. Kondo(1), M. M. Rahman(2), M. E. Syazwan (2), T. Okada(2), S. Samukawa(2), A. Fukuyama(1), and T. Ikari(1)
Affiliations : (1) DEEE, University of Miyazaki, 1-1 Gakuen Kibanadai-nishi, Miyazaki, Japan (2) Institute of Fluid Science, Tohoku University, 2-1-1 Katahira, Aoba, Sendai, Japan

Resume : Semiconductor nanostructure has recently received broad attention for applying to photovoltaic cell with advanced performances. Miniband formation between the quantum dots is a key feature for materializing higher efficiency. It is then necessary to control precisely the sizes and their intervals. Although it is very difficult to grow well-arranged quantum dots by a conventional technique, such as Stranski–Krastanov growth mechanism, we succeeded for fabricating the exactly arranged Si nanodisk (Si-ND) of dimensions around sub-10-nm embedded in SiC matrix by our original top-down process with bio-templates and neutral beam etching [1]. Therefore, the carrier transport process in the Si-ND array structure could be investigated in terms of the lifetimes of photoexcited carriers by using a microwave photoconductivity decay method. Among the five types of Si-ND array structure samples, longest lifetime of 3.4 μsec was observed for the sample with the disc size of 4-nm-thick/6.4-nm-diameter and the spacing of 2 nm along the thickness direction. We considered that the observed long carrier lifetime is due to decrease of the carrier recombination thanks to the miniband formation, say coupling the wave functions, along the thickness direction. The confirmation of the miniband formation of well-arranged nanodisk structure indicates the solar cell efficiency might be further increased by our sample growth technique. [1] S. Samukawa, et al., Appl. Surf. Sci. 253 (2007) 6681

H5 5

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18:15 **Quantitative compositional analysis of BeMgZnO alloys using combined ion beam techniques**

Authors : Z. Zolnai(1), J. Volk(1,2), M. Toporkov(2), D. Demchenko(3), V. Avrutin(2), H. Morkoç(2,3), Ü. Özgür(2), and G. Battistig(1)
Affiliations : (1) Research Centre for Natural Sciences, Institute for Technical Physics and Materials Science, H.A.S., 1121 Budapest, Konkoly-Thege M. út 29-33, Hungary (2) Department of Electrical and Computer Engineering, Virginia Commonwealth University, 601 W Main St, Richmond, VA 23284, USA (3) Department of Physics, Virginia Commonwealth University, 701 W. Grace St., Richmond, VA 23284, USA

Resume : Recently ZnO and related BeMgZnO alloys have been achieved considerable attention due to their promising properties for optoelectronic applications. Although theoretical studies on bandgap variation for the BeMgZnO system were performed no considerable attention was paid to systematic quantitative comparison of measured and calculated properties vs. the Be and Mg concentrations. This is partly due to difficulties when looking for appropriate tools to measure Be and Mg contents with satisfactory accuracy. In this work quantitative compositional analysis of BeMgZnO alloys is performed with typical uncertainty of 1-2 atom % for the Be and Mg concentrations. Our concept is based on the non-Rutherford proton elastic backscattering reaction around 2.5 MeV proton energy where an enhancement factor of 60 in the cross section of Be can be achieved compared to the Rutherford value. For depth dependent concentration profiling proton beam analysis was combined with 1 MeV He+ Rutherford Backscattering Spectrometry (RBS) and multiple spectrum fit evaluation procedure. Based on the measured atomic composition of BeMgZnO layers, bandgap calculations were performed and compared to experimental optical bandgaps. Good agreement was found for Be concentrations in the range 7-19 atom %, and for Mg contents up to 52 atom %, respectively. In summary, we present accurate doping concentration analysis in order to achieve precise bandgap engineering and structure control of BeMgZnO alloys.

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Poster Session 1 - Analytical Techniques for Nanomaterials : Claudia Fleischmann, Bernd Kolbesen, Miroslav Valtr, Andreas Hertwig, Luca Boarino and Omar El Gawhary

18:30 **Multimodal imaging in nano-sciences**

Authors : U. Schmidt, A. Jauss, M. Tchaya, H. Fischer
Affiliations : WITec GmbH, Lise-Meitner Str. 6, Ulm, 89081, Germany

HP6 1

Resume : New developments and applications in the field of materials science and nanotechnology require characterization methods which provide as many physical properties from the same sample area on the micro-and nanoscale as possible. This is strongly facilitated by combining several measuring techniques in one instrument, which allows the differentiation of chemically different materials with high spatial resolution without surface damage, staining or preferential solvent washing. The combination of a confocal Raman microscope with atomic force microscopy (AFM) was a breakthrough in this field. By simply rotating a microscope turret, the user can link the molecular information obtained by confocal Raman microscopy with the ultra-high spatial and topographical information acquired by AFM. These measuring techniques provide complementary information of the analyzed sample: State of the art confocal Raman microscopes provide the distribution of various chemical species in heterogeneous samples with diffraction limited resolution within seconds by employing modern high speed, high sensitivity CCD cameras with high throughput spectrometers and highly optimized confocal microscopes. Such high speed Raman imaging methods also allow the acquisition of complete volume stacks in less than 30 minutes, leading to 3D reconstructions of the sample volume. The various AFM techniques enable the acquisition of real 3D topography images from samples with resolution far below the diffraction limit. In addition to the surface topography, various images resulting from the tip-sample interaction can be acquired. Such images can provide information about the viscoelastic properties of the sample, as in the case of AC/tapping mode imaging in the recorded phase image, or as adhesion, stiffness or viscosity images in the case of pulsed force mode imaging. In addition to Raman and AFM, further techniques can also be implemented into one microscope. These are for example scanning near field optical microscopy, true surface microscopy, time resolved microscopy or photoluminescence microscopy in the UV, visible and IR region.

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18:30 **Development of polymeric membranes for anion analyses in solutions with TXRF** HP6 2

Authors : N. Kallithrakas-Kontos, V. Hatzistavros
Affiliations : Analytical and Environmental Chemistry Laboratory, University Campus, Technical University of Crete, GR-73100 Chania, Greece

Resume : Total Reflection X-Ray Fluorescence (TXRF) is a relatively new analytical technique with many powerful possibilities (multi-elemental analysis, direct analysis in solid phase, no destructive, extremely small sample quantities/concentrations etc.). Although TXRF is used almost exclusively for the analysis of solid samples many of its applications is intended for (indirect) analysis of liquid samples by evaporation of small liquid droplets on suitable substrates (reflectors). Besides TXRF advantages there are two significant disadvantages: The minimum detection limits (for liquid analysis) usually are not adequate (compared to other modern analytical techniques) and the lack of possibility of speciation analysis. An alternative method is suggested in the present work in order to overcome these drawbacks. Various thin membranes with special ligands were prepared directly on TXRF reflectors surfaces. These membranes were used for ion-selective preconcentration from water solutions and they were analyzed by TXRF. With this process the two TXRF drawbacks were outreached: the minimum detection limits were improved by a factor of 10 -100 and speciation (selective preconcentration) was possible. Various membranes have been tested for different ions. In the present work the membrane creation is presented as well as the effect of the experimental conditions. Different applications are given with special interest in interference effects and detection limit improving.

add to my program **(close full abstract)**

18:30 **Combining SAXS and DLS for real-time analysis of nano-particle production** HP6 3

Authors : Alexander Schwamberger; Bert De Roo; David Jacob; Leander Dillemans; Lutz Bruegemann; Jin Won Seo; Jean-Pierre Locquet;
Affiliations : Bruker AXS, Karlsruhe, Germany; Department of Physics and Astronomy, KU Leuven, Belgium; Cordouan Technologies, Pessac, France; Department of Physics and Astronomy, KU Leuven, Belgium; Bruker AXS, Karlsruhe, Germany; Department of Metallurgy and Materials Engineering, KU Leuven, Belgium; Department of Physics and Astronomy, KU Leuven, Belgium;

Resume : Real-time characterization of nano-particles (NPs) while ongoing synthesis is a promising mean to produce NPs under control with targeted physical properties depending on their morphology (e.g. size, shape). We present here a NP production and characterization tool which combines a laboratory Small Angle X-ray Scattering (SAXS) instrument with a specially

designed Dynamic Light Scattering (DLS) device to be used simultaneously with SAXS. For the synthesis of NPs a micro-flow reactor is used. Owing to the novel liquid anode X-ray source monitoring of the synthesis process with a sampling rate of approx. 20s is achieved. SAXS and DLS measurements are performed directly inside a glass flow-through capillary located inside the SAXS vacuum chamber. In-situ SAXS and DLS measurements during the synthesis of silica NPs were performed.

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18:30 **Characterization of TiO2 antireflection coatings elaborated by APCVD for monocrystalline silicon solar cells**

Authors : D. Hocine1, M.S. Belkaid1, M. Pasquinelli2, L. Escoubas2, P. Torchio2, A. Moreau2

Affiliations : 1Laboratory of Advanced Technologies of Electrical Engineering (LATAGE). Mouloud Mammeri University (UMMTO), Algeria. 2Aix-Marseille University, Institut Matériaux Microélectronique Nanosciences de Provence – IM2NP CNRS UMR 7334, France.

Resume : In this work, high quality titanium dioxide thin films were grown by an efficient, less expensive and rapid method of Atmospheric Pressure Chemical Vapor Deposition (APCVD) from $TiCl_4$ precursor for application as antireflection coatings on monocrystalline silicon solar cells with the aim to reduce the front surface reflection losses. The photovoltaic electricity is obtained by the direct conversion of light into electricity by means of conventional solar cells which operate on the basis of the photovoltaic effect that exists at semiconductor junctions, performing two processes simultaneously: absorption of light, and the separation of electric charges. Now, antireflection coating (ARC) is one of the most investigated parts of the solar cell, since it reduces the optical losses leading to improve the efficiency of the photovoltaic device. The main parameters for fabrication and design of antireflection coatings are the refractive index and the film thickness of the layer. For this application, titanium dioxide (TiO_2) has been widely used in silicon solar cells due to its excellent electrical and optical properties such as a high refractive index, and excellent transmittance in the visible and near IR region of the solar spectrum. In this work, TiO_2 thin films were grown on glass and monocrystalline silicon substrates by APCVD method from the reaction of titanium tetrachloride $TiCl_4$ with oxygen. The microstructural, electrical and optical properties of the produced coatings were successfully characterized by Atomic Force Microscopy (AFM), Four Point Probe (FPP) and Spectroscopic Ellipsometry (SE) combined with Transmission Spectroscopy, respectively. In the light of these results, these properties were exploited for application of the TiO_2 coatings as single-layer antireflection coatings (SLARC) on monocrystalline silicon solar cells. According to Shui-Yang Lien [1], for achieving low reflectance in the case of monocrystalline silicon solar cells, the TiO_2 SLAR coating should have a refractive index of 2.2 at the wavelength $\lambda = 550$ nm and an optimal thickness of 56.8 nm. These calculated values can be achieved experimentally by optimization of the film preparation conditions. The AFM images reveal the polycrystalline structure and the homogenous surface of the prepared TiO_2 films. These films are dense, uniform and compact which is good in solar cell applications. As estimated by AFM, the thickness of the TiO_2 film on silicon substrate was found to be 56.2 nm which confirms the optical thickness obtained by ellipsometry measurements. The RMS surface roughness is determined from the area of 1.5 µm × 1.5 µm for all samples. The bare silicon is relatively smooth and its characteristic RMS roughness is 2.5 nm. An RMS roughness of 5.1 was estimated for the as-deposited TiO_2 films at 450°C. After annealing (at 450°C for 1 hr), we observed that the surface roughness increases to $R_{rms} = 7.97$ nm due to an increase in grain sizes. The measured average optical transmittance of the TiO_2 layers was about 85-90%. The experimental refractive index of our TiO_2 thin films was found to be $n = 2.25$ at the wavelength $\lambda = 550$ nm, with a thickness of 56.2 nm. Our results show the possibility to fabricate TiO_2 layers with the optimal optical qualities required for antireflection coating, using the APCVD technique. An excellent agreement is reached between our experimental results and calculated results for TiO_2 single-layer antireflection coating on monocrystalline silicon solar cells. The density of the deposited TiO_2 films is found to be $\rho' = 3.11$ g/cm3. The porosity φ (the volume of pores per volume of film) of the TiO_2 film depends on the refractivity of the film layer. The porosity of these films is estimated to $\varphi = 24$ %. The electrical resistivity of the deposited TiO_2 films at 450°C annealed at 450°C for 1 hr, was found to be $\rho = 1.7 \times 10^{-3}$ Ω.cm. The sheet resistance of our TiO_2 films was equal to $R_{\square} = 303$ Ω/□. The obtained results demonstrate the real opportunity of the APCVD technique to prepare

HP6 4

high quality antireflection coatings for high efficiency silicon solar cells. [1] S. Lien, Solar Energy Materials & Solar Cells 90 (2006) 2710 – 2719.

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18:30 **Optical characterization of MoO3 and ITO layers made by reactive magnetron sputtering**

Authors : C. Major1, Z. Labadi1, Z. E. Horvath1, Z. Zolnai1, M. Fried1, 2
Affiliations : 1. Institute for Technical Physics and Materials Science, Research Centre for Natural Sciences, H-1525 Budapest, POB 49, Hungary 2. Doctoral School of Molecular and Nanotechnologies, Faculty of Information Technology, University of Pannonia, Egyetem u.10, Veszprem, H-8200, Hungary

Resume : This presentation reports on the deposition conditions and optical properties of thin layers of molybdenum oxide (MoOx) and tin-doped indium oxide (ITO) on silicon and glass substrate made by DC reactive magnetron sputtering. Samples were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction (XRD) prior to spectroscopic ellipsometry (SE) analysis. SE measurements were carried out on a J. A. Woollam M2000DI ellipsometer in the range of 300-1700 nm. Evaluation of SE data was performed by least square fitting the Cauchy dispersion relation and Tauc-Lorentz model dielectric function. Complex refractive index, layer thickness and surface roughness data are determined by SE, as verified by comparison with SEM and TEM morphologies furthermore optical properties have been associated with microstructure and growth conditions. [This work was supported by the National Development Agency grant KMR-12-1-2012-0225 and T?T_10-1-2011-0305 project]

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18:30 **High-accuracy determination of X-ray fundamental parameters using reference materials certified by ion beam analysis**

Authors : J.L.Colaux, P.Hönicke, C.Jeynes, B.Beckhoff
Affiliations : University of Surrey Ion Beam Centre, Guildford GU2 7XH, England; Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany

Resume : Fundamental parameter (FP) based X-ray spectrometry (XRS) is very well suited for quantitative analysis of nanoscaled materials: this method is beneficial especially when no reference samples with sufficient quality are available or when the layer systems are complex. However, for quantitative investigations of elemental concentrations in unknown specimens by XRS, exact knowledge of the FPs is essential. These parameters include e.g. the fluorescence yields, the absorption cross sections or Coster-Kronig transition probabilities. For most chemical elements, only calculated data with unknown or estimated uncertainties are available. The experimental determination of such parameters is challenging, especially for elements where free standing thin foils are not available. Standard Ga-implanted Si wafers were prepared where the implanted dose was certified by Rutherford backscattering spectrometry (RBS) at 1% absolute accuracy [1]: these samples were used to determine the product of the photoionization cross-section and the fluorescence yield of the Ga-K edge. The reliability of this result was estimated by constructing a formal uncertainty budget. The XRS experiments were carried out using the calibrated instrumentation of the Physikalisch-Technische Bundesanstalt at the BESSY II laboratory [2]. [1] J. L. Colaux and C. Jeynes, Analytical Methods (2014) 6, 120-129. [2] M. Kolbe, P. Hönicke, M. Müller, B. Beckhoff, Phys. Rev. A (2012) 86, 042512.

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18:30 **Ultra-trace material analysis in conditions of the planar X-ray waveguide-resonator application**

Authors : 1 V.K. Egorov, E.V. Egorov, 2 E.M. Loukiantchenko
Affiliations : 1 IMT RAS, Chernogolovka, Moscow District, 142432 Russia 2 OOO “Poljus”, Saint-Petersburg, Russia

Resume : Historically, the mass-spectrometry methods form the basis for the ultra-trace analysis of materials. Its are characterized by multielements, high sensitivity and beautiful mass-resolution but the destructive procedures and show some problems at the element quantitative diagnostics. At the same time, there was elaborated the tools nondestructive method for ultra-trace multielemental material analysis, which shows the reassuming quantitative characteristics. It is the X-ray fluorescence analysis at external total reflection (TXRF) [1]. The efficiency of the method is defined primarily by the exciting beam radiation density. Because of this, the best efficiency of TXRF diagnostics was obtained in case of the exciting beam formation by X-ray waveguide-resonators [2]. These devices increase the radiation density in formed fluxes on four orders in comparison with ones formed by slit-cut flux formers. In result of

HP6 7

it, element detection limits decrease on two orders. The work presents construction of TXRF experimental cell built on base of the original design waveguide-resonator and results of different objects diagnostics obtained about this cell application. There are discussed ways for waveguide-resonators property improving and its possible influence on the TXRF efficiency upgrading. [1] R. Klockenkamper. Total reflection X-ray fluorescence analysis. Wiley: New York. 245 p. [2] V.K. Egorov, E.V. Egorov // Adv. X-ray Chem. Anal. Japan. v44. 2013. pp. 21-40.

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18:30 **Control current stress technique for the investigation of ultrathin gate dielectrics of MIS devices** HP6 8

Authors : V.V.Andreev1, G.G.Bondarenko2, V.M.Maslovsky3, A.A.Stolyarov1, D.V.Andreev1
Affiliations : 1) Bauman Moscow State Technical University, Kaluga Branch. 4, Bazhenov St., Kaluga, 248600, Russia 2) National Research University Higher School of Economics, 20, Myasnitskaya Ulitsa, Moscow 101000, Russia 3) The state unitary enterprise of a city of Moscow Research-and-production centre "SPURT", Zelenograd, West of the 1-st proezd 4, 124460, Russia
Resume : In this study, a new technique of control current stress to investigation thin and ultrathin gate dielectrics of MIS structures is proposed. This technique allows to research charge processes which take place in gate dielectric of MIS structures and its interface under high-field and another stress situations (irradiation, plasma, hot carriers, etc.). The technique also may be used for testing thin gate dielectric defects. Unlike simple techniques, for example constant current stress and J-Ramp current stress, the developed technique for MIS structures applies of current stress with a special algorithm. At the same time characteristics of gate dielectric monitored by voltage time dependence, taking into account charging capacitance and charge trapping. Charging of MIS structure from inversion to accumulation modes or back way lets to receive low capacitive-voltage characteristics. Account charging capacitance of MIS structure and charge trapping in gate dielectric at injective mode lets considerably increase metrological characteristics of this technique and reduce inaccuracies. The models describing the change in the charge state of MIS structures, both in the charge capacity, and in the mode of injection of charge carriers were developed. Using these models let to choose optimal algorithm of current stress and increase measurement accuracy. This technique was used for research SiO_2 and SiOxNy gate dielectrics with thick range 1-10 nm. The technique may be used for research of high-k and multilayer gate dielectrics.

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18:30 **Consideration of high inclusions concentration influence on absorption of thin nanocomposite films** HP6 9

Authors : Lozovski V. Z. 1, Mytiansky D. S. 2, Strilchuk G. M. 1
Affiliations : 1Institute of High Technologies, Taras Shevchenko National University of Kyiv, 64 Volodymyrska Str, 01603 Kyiv, Ukraine; 2Radiophysical Department, Taras Shevchenko National University of Kyiv, 64 Volodymyrska Str, 01603 Kyiv, Ukraine
Resume : The theoretical method for calculation of the dissipative function of thin nanocomposite film, in the case of high concentration of inclusions was proposed. The observed model takes into account interaction between film inclusions, when they are close to each other, including the case of distance less than size of inclusion particle. Nanocomposite thin films embedded with ellipsoidal inclusions and placed on the substrate was theoretically characterized. Calculations were carried out by effective susceptibility method, using Lippmann-Schwinger equation. The dependence of the absorption of such nanocomposite film on inclusions concentration was obtained. Analysis of the results was performed in the cases of low and high concentration. Was shown, that it is necessary to take into consideration interaction of particles, when distance between them is less than it's size. It was shown in the cases of different dielectric susceptibility and different film thickness.

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18:30 **Surface/interface characterization of semiconductors and solar cells via air Photoemission Spectroscopy and Surface Photovoltage Spectroscopy** HP6 10

Authors : Iain D Baikie, Angela C Grain, James Sutherland, Jamie Law
Affiliations : KP Technology Ltd, Burn Street, Wick, Caithness, KW1 5EH, UK
Resume : We have developed a dual-mode Kelvin probe featuring two novel detection modes comprising Air Photoemission Spectroscopy (APS), which yields information on the absolute work function (Φ) of a surface/thin film, and Surface Photovoltage Spectroscopy (SPS) which produces infor-mation relevant to

spectroscopic characterization of solar cells. These measurement modes allow a full characterization of the electronic energy band diagram including valance band energy, fermi-energy and surface potential (band-bending) under standard conditions. The traditional Scanning Kelvin probe (SKP) measures small changes (1-3 meV) in a non-contact fashion, using a vibrating tip. This system is extremely versatile, capable of automatic monitoring of changes in Φ or sample fermi-level under ambient, controlled atmosphere and UHV environments. Using a combination of Visible/IR and deep UV illumination (1.2 - 7.3 eV) we have characterized the work function of metallic and ionization potential of semiconducting thin films and TCO's utilised in device fabrication such as Au, Ag, Al, Cu, cSi, mcSi, aSi, ITO, CuO, ZnO, TiO2, Pedot, GaP, Graphene. Other examples include the Density of States (DOS) in Nickel-Phylocyanine (NiPc). The resulting tool is extremely useful for surface characterization of organic semiconductors and solar cells, allowing clarification of the energy band diagram. Further we show how Pulsed Light SPV Transients can be used to characterize interface trapping.

add to my program **(close full abstract)**

18:30 **Growth of dilute bismides GaAsBi/GaAs nanowires by metalorganic vapor phase epitaxy**

Authors : Y. Soda*, H. Fitouri, C. Bilel, A. Rebey, and B. El Jani
Affiliations : University of Monastir, Faculty of Sciences Unité de Recherche sur les Hétéro-Epitaxies et Applications (URHEA), 5019 Monastir, Tunisia E-mail: * benzaied.yethreb@yahoo.fr

Resume : Dilute bismide GaAsBi nanowires were grown on GaAs substrates using atmospheric pressure metalorganic vapor phase epitaxy (AP-MOVPE). The structure and morphology of the GaAsBi nanowires are investigated using high resolution X-ray diffraction (HRXRD) and scanning electron microscopy (SEM). Growth of the nanowires at low temperature results in round Bi-rich balls can be found at the tips of all nanostructures. The bases of the tapered structures can be quite large, but they decrease in diameter. The growth conditions affect the initial nucleation of GaAsBi structures, resulting in different numbers and shapes of nanowires. They also influence the orientation and growth quality of the nanowires. The optical properties are studied by photoreflectance (PR). Keywords: GaAsBi nanowires; AP-MOVPE; High resolution X-ray diffraction; scanning electron microscopy; photoreflectance.

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add to my program **(close full abstract)**

18:30 **Fundamental parameters for reference-free quantitative X-ray fluorescence analysis**

Authors : Michael Kolbe, Philipp Hönicke, Matthias Müller, Burkhard Beckhoff
Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany

Resume : The further development of more complex materials with distinct properties needs an analysis independent from any reference material such as X -ray fluorescence analysis (XRF). For a reliable quantitative XRF the exact knowledge of atomic fundamental parameters involved is inevitable. In this work, fundamental parameters [1] including mass absorption and photo ionization coefficients, fluorescence yields, Coster-Kronig transition probabilities for several chemical elements are experimentally determined using the calibrated instrumentation of the Physikalisch-Technische Bundesanstalt (PTB) [2]. The experiments were carried out in the PTB-laboratory as well as at the wavelength shifter beamline (BAMline) at the electron storage ring BESSY II, where monochromatized synchrotron radiation of high spectral purity up to about 100 keV is available. The knowledge of fundamental parameters with low uncertainties leads to significant improvements in quantitative XRF analysis in fact reference-based as well as reference-free. [1] M. Kolbe, P. Hönicke, M. Müller, B. Beckhoff, Phys. Rev. A 86 (2012), 042512 [2] B. Beckhoff, J. Anal. At. Spectrom. 23 (2008), 845-853

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add to my program **(close full abstract)**

18:30 **High-resolution atomic force microscopy of high-quality, solvent-free crystals of [6,6]-phenyl-C61-butyric acid methyl ester**

Authors : Giovanni Mattia Lazzerini, Giuseppe Paternò, Franco Cacialli, Andrew Yacoot
Affiliations : G.M. Lazzerini and A. Yacoot: National Physical Laboratory, Hampton Road Teddington Middlesex TW11 0LW, UK; G. Paternò and F. Cacialli: London Centre for Nanotechnology and Department of Physics and Astronomy, University College London, Gower Street, London WC1E 6BT, UK

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Resume : We present results from high-resolution traceable atomic force microscopy (AFM) of high-quality, solvent-free single crystals of [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) - the election electron-acceptor in organic

solar cells. We used an in-house constructed AFM working in closed-loop, non-contact mode. The AFM uses a fibre interferometer to detect bending of the cantilever and a custom feedback system to compensate for the bending. An NPL plane mirror differential optical interferometer, fibre-fed with light from a frequency stabilized laser (638 nm), is then employed to traceably measure the height of the features on the surface.1 We grew PCBM crystals by solvent casting a 20 mg/ml chlorobenzene solution on Indium Tin Oxide pre-patterned substrates. The samples were kept in a capped Petri dish during deposition, and dried in vacuum (~10-2 mbar) overnight at room temperature to remove residual solvent.2 Interestingly, we observe that the height of the single crystals, only takes specific values such as 49 nm ± 2 nm and 105 nm ± 3 nm or 72 nm ± 2 nm and 139 nm ± 3 nm, in the different crystals investigated. With the low uncertainty of the AFM in the z-direction (σ < 0.3 nm), we could also identify a step on the crystal surface whose height corresponds to one of the lattice constants of the single PCBM crystal (1.3 nm) as measured with X-ray diffraction.2 1 A. Yacoot, et al. Meas. Sci. Technol. 18, 350 (2007) 2 G. Paternò, et al. J. Mater. Chem. C 1, 5619 (2013)

add to my program **(close full abstract)**

18:30 **In-situ time-resolved GISAXS investigation during the RF-sputter deposition of Ag** HP6 14

Authors : G. Santoro, S. Yu, P. Zhang, Sarathlal K.V., J.F.H Risch, M. Hernández, C. Domingo, S.V. Roth
Affiliations : Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany;Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany;Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany;Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany;Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany;Institute of Structure of Matter, IEM-CSIC, Serrano 121-123, E-28006 Madrid, Spain;Institute of Structure of Matter, IEM-CSIC, Serrano 121-123, E-28006 Madrid, Spain;Photon Science, DESY, Notkestr. 85, D-22607 Hamburg, Germany
Resume : Metallic nanocoatings are of great scientific and engineering relevance since they are used in advanced optical, electrical and medical applications. In particular, Ag nanocoatings have been shown to exhibit excellent properties that can be exploited for applications such as antibacterial coatings, plasmonic devices or sensors. Nevertheless, in order to fully control the desired final properties of the nanocoatings, that are very sensitive to the morphology developed on the surface during the growth as well as to the arrangement of the metal nanoclusters on top of the substrate, it is mandatory to achieve a profound understanding of the growth kinetics. In this sense, micro Grazing Incidence Small Angle X-ray Scattering (microGISAXS) is a very powerful and valuable tool for the in-situ characterization of the growth kinetics, providing morphological information of the surfaces that are developing with time resolution in the order of the millisecond [1,2]. This work presents in-situ time-resolved microGISAXS results concerning the time evolution of the structures developed during the RF-sputtering of Ag. The Surface-Enhanced Raman Spectroscopy (SERS) activity of the prepared Ag nanocoatings for different film thicknesses is also presented and correlated to their nanostructure. [1] M. Schwartzkopf, et al. RSC Nanoscale 5, 5053 (2013). [2] S. Yu, et al., J. Phys. Chem. Lett. 4, 3170 (2013).

add to my program **(close full abstract)**

18:30 **Study on reinforcement of polyamide 1212 via graphene oxide** HP6 15

Authors : Ziqing Cai, Xiaoyu Meng*, Yingshuai Han, Qiong Zhou*, Lishan Cui
Affiliations : College of Science, China University of Petroleum, Beijing 102249, China. Beijing Key Laboratory of Failure, Corrosion and Protection of Oil/gas Facilities, Beijing 102249, China.
Resume : The graphene has attracted interest recently in the fabrication of graphene based polymer nanocomposites due to its excellent properties. In this paper, graphene oxide (GO) was synthesized from graphite power by the Hummers' method. Meanwhile, the GO was characterized by several techniques, such as XRD, SEM, TEM and AFM. The results indicated that the GO singer layer and multi layers were successfully synthesized. The polyamide 1212 (PA1212)/GO nanocomposites were prepared by melt-compounding method after the GO was pre-mixed with a small amount of ethanol soluble polyamide (ES-1) in ethanol solution. The mechanical properties of the PA1212/GO nanocomposites were improved efficiently after addition of 0.3% GO. The GO as the nucleation template showed obvious heterogeneous nucleation effect due to its large specific surface area according to DSC results and it induced α-form crystal formation in PA1212 matrix from WAXD results.

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18:30 **Boron doped cubic silicon probed by high resolution X-ray RSM**

Authors : T. Ulyanenkova1, M. Myronov2, A. Ulyanenkov1
Affiliations : 1Rigaku Europe SE, Am Hardtwald 11, Ettlingen, Germany 2Department of Physics, The University of Warwick, Coventry, UK

Resume : The Bragg peak position of a homogeneous solid solution epitaxial film is directly related to the solid solution concentration, film strain and, consequently, residual stress. The peak shape contains information about defects present in the sample. Here we report structural experiments performed at room temperature and atmospheric pressure on a set of BSi thin layers on Si substrate. We analyzed the thin film BSi using high resolution rocking curve and reflectivity measurements and high resolution reciprocal space mapping (HR-RSM) made on Rigaku SmartLab diffractometer. Highly Boron doped epitaxial Silicon, with Boron concentrations well above 1•1020 cm-3, is of great interest for applications in large variety of electronic and photonic devices where it is used as a low resistivity contact.

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18:30 **C K-edge XANES-measurements using an off-axis reflection zone plate with a Laser-Produced Plasma Source**

Authors : Katharina Witte* (1), I. Mantouvalou (2), S. Martyanov (2), S. Günther (2), R. Jung (1), H. Stiel (1), B. Kanngießer (2)
Affiliations : (1) Max-Born-Institut, Berlin, Germany; (2) Technical University of Berlin, Berlin, Germany; Berlin Laboratory for innovative X-ray Technologies (BLiX)

Resume : In the last decades, tools and techniques have been developed to analyze the structural, electronical and chemical properties of materials on the molecular and macroscopic scale. With X-ray absorption spectroscopy (XAS) such kind of investigations are feasible. In the soft X-ray region especially the K-edges of C, N and O are of great interest, since they are the main components of biological samples. The required high spectral brightness and high average photon flux usually necessitates measurements to be performed at synchrotron radiation sources. Another possibility is to utilize X-ray sources based on laser-produced plasmas (LPP). With our compact LPP source, we have the ability to transfer soft X-ray spectroscopy in the range of 80 to 1200 eV into the laboratory. The radiation emission relies on the formation of hot localized plasma on a solid metal-target through the focusing of a short laser pulse [1]. We will present X-ray absorption near-edge structure (XANES)-measurements at the C K-edge with the LPP source and a spectrometer using a novel X-ray optical element, an off-axis reflection zone plate (ORZ) [2]. Investigations of different thin foils are realized and structural characteristics due to the chemical composition can be made visible. The presented measurements are realized on a reasonable time scale in the range of seconds. [1] I. Mantouvalou et al., Rev. Sci. Instrum. 82, 066103 (2011) [2] U. Vogt et al., Rev. Sci. Instrum. 75, No. 11, 4606-4609 (2004)

HP6 17

add to my program **(close full abstract)**

18:30 **Roughness effects in nanoindentation on thin films**

Authors : Anna Charvátová Campbell, Petr Klapetek, Jan Martinek
Affiliations : Czech Metrology Institute

Resume : Measurement of local mechanical properties is an important topic in the fields of nanoscale device fabrication, thin film deposition and composite material development. Nanoindentation instruments are commonly used to study hardness and related mechanical properties at the nanoscale. However, not all aspects are also fully understood from a metrological point of view. For the determination of mechanical properties of thin films the penetration depth must be as low as possible so that the deformation zone does not exceed the film. However, for small depths the results may be significantly distorted by the roughness of the thin film. The contact area is one of the crucial quantities involved and the most prone to errors. Since it cannot be measured directly in the experiment, it is usually determined independently either measuring directly the shape of the tip or using a known sample. However, there may be discrepancies between the contact areas for different setups. Instruments often just assume the sample to be perfectly flat and a perfect alignment of the sample and the indenter tip axis. Obviously roughness or a tilt of the surface can change the contact area and represent a significant source of uncertainty, especially for small depths. Experimental data will be used to simulate the change of contact area on a rough sample using the finite element method (FEM) and Monte Carlo methods. Results will be compared to theoretical models.

HP6 18

add to my program **(close full abstract)**

18:30 **Morphology of multiphase and functional thin films by Atomic Force Microscopy**

Authors : M. Perani (1), E. di Russo (1), N. Brinkmann (2), B. Terheiden (2), D. Cavalcoli (1)
Affiliations : (1) Department of Physics and Astronomy, University of Bologna, Italy; (2) Department of Physics, University of Konstanz, Germany

Resume : The Scanning Probe Microscopy is a very useful technique that provides a variety of information about the surface of materials. The Atomic Force Microscopy (AFM) can be used to measure the topography of a sample, the energy dissipation that occurs between the tip and surface, the local conductivity and many other features. Topography images were obtained with the AFM in tapping mode on composite multiphase materials interesting for their photovoltaic applications, such as nc-SiOxNy thin films. The knowledge of the structure of this kind of materials is highly relevant as the dimension and the distance between nano-crystals can influence the electrical properties of the layer and the active incorporation of dopants inside the nano-crystals. The Height – Height Correlation Function (HHCF) is used within this framework in order to evaluate different parameters of the surface under investigation, such as roughness, lateral correlation lengths and wavelengths. The latter two, in particular, are related to the size and distribution of the grains. A "watershed" algorithm is also used on the AFM images, allowing an efficient marking of the grains and the evaluation of different properties of the nano-crystals, such as the size, area and volume, providing a better knowledge of the characteristics of the analyzed surfaces. This work has demonstrated the capabilities of statistical analysis of AFM topography data for obtaining insight into the material properties.

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18:30 **Grazing-incidence X-ray fluorescence analysis of buried interfaces in nanostructured crystalline silicon thin-film solar cells**

Authors : D. Eisenhauer 1, B. Pollakowski 2, J. Baumann 3, V. Preidel 1, D. Amkreutz 4, B. Rech 4, F. Back 5, E. Rudigier-Voigt 5, B. Beckhoff 2, B. Kanngießer 3, C. Becker 1
Affiliations : 1: Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Young Investigator Group Nanostructured Silicon for photovoltaic and photonic implementations (Nano-SIPPE), Kekuléstr. 5, 12489 Berlin, Germany; 2: Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany; 3: Technische Universität Berlin, Institut für Optik und Atomare Physik, Analytische Röntgenphysik, Hardenbergstr. 36, 10623 Berlin, Germany; 4: Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Institut Silizium-Photovoltaik, Kekuléstr. 5, 12489 Berlin, Germany; 5: SCHOTT AG, Hattenbergstr. 10, 55122 Mainz, Germany

Resume : Crystalline silicon thin-film solar cells on glass fabricated by liquid-phase crystallization enable an excellent electronic material quality providing open-circuit voltages up to 600 mV. Periodic nanostructuring of the substrate by nanoimprint-lithography efficiently increases the absorption in the thin silicon layer. Due to the high temperatures applied during crystallization (T > 1414°C), the consistency of the nanopatterned interface between substrate and silicon, and the choice of appropriate interlayers is crucial for device performance. In this study, we performed grazing-incidence X-ray fluorescence (GIXRF) measurements at the electron-storage ring BESSY II for depth-dependent characterization of the buried substrate-silicon interface region. The interface of the silicon absorber was made accessible for GIXRF measurements by removing both the glass substrate and interlayers in concentrated hydrofluoric acid. GIXRF provides a non-destructive access to the depth distribution of the involved elements. In particular for nanopatterned structures the determination of these elemental profiles is challenging, as it is significantly influenced by the 3D interfacial structure. Calibrated instrumentation was used for the measurements allowing for a reference-free quantization. The contamination level in the absorber was found to be strongly influenced by the interlayers (silicon oxide, silicon carbide or a combination of both) - and correlates with respective solar cell results.

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18:30 **Structural properties of Pb(ZrxTi1-x)O3**

Authors : Anna V. Kimmel, Carlo Vecchini, Markys Cain
Affiliations : National Physical Laboratory, Teddington, TW11 0LW, UK.

Resume : The structure of Pb(ZrxTi1-x)O3 at the composition x=52%, where morphotropic phase boundaries are formed, presents significant fundamental and practical interest. It is believed that this composition (PZT52) exhibits two coexisting phases with space group symmetry Cm and P4mm. We used density function theory and structure searching algorithm to characterize the variety of energetically favorable phases that PZT52 adopts. Further performed structure-

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refining procedure based on experimental X-Ray data for ceramic samples of PZT52 at ambient conditions demonstrated that this material adopts a coexistence of crystallographic structural configurations with prevalent P4mm contribution and low symmetry configurations with smaller statistical weights.

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18:30 **Hard and soft condensed matter X-RAY Reflectometry and GISAXS studies uzsing the incoatec microfocus source IµS**

Authors : André Beerlink, Jürgen Graf, Bernd Hasse, Andreas Kleine, Carsten Michaelsen, Jörg Wiesmann

Affiliations : Incoatec GmbH, Max-Planck-Strasse 2, 21502 Geesthacht, Germany

Resume : The Incoatec microfocus source IµS is a low power air cooled X-ray source for diffractometry applications. It is available with Cr, Co, Cu, Mo, and Ag anodes. The source is equipped with a two dimensional beam shaping multilayer optics. Therefore, we can form either a highly collimated beam with a low divergence (below 0.5 mrad) or a focusing beam with higher divergence (up to 10 mrad) and very small focal spots (diameter below 100 µm). Equipped with a collimating optics it can be used for GISAXS, SAXS and X-ray reflectometry studies. When using focusing optics all those experiments can be carried out in transmission geometry, especially in powder diffraction applications. With the Mo-IµS highly absorbing and radiation-damage sensitive materials can be investigated. Consequently, this source is often used for single crystal diffractometry in the chemical crystallography and becomes more and more interesting for investigations of soft matter samples. In our presentation we will give an overview of representative experimental setups and results demonstrating the potential of our IµS in XRD studies. These take advantage of the brilliance and outstanding beam quality of this low-maintenance microfocus source. It is shown how the IµS can be used to achieve excellent results in both home-lab and synchrotron pre-characterization experiments, e.g. the investigation of in-situ thin film deposition in UHV chambers or the structure of oriented two-dimensional liquid crystalline samples.

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18:30 **Synthesis and utilization of LaVO4:Eu3+nanoparticles as fluorescent near-field optical sensors**

Authors : S.G. Nedilko (1), O.V. Chukova (1), Yu.A. Hizhnyi (1), S.A. Nedilko (1), T. Voitenko (1), L. Aigouy (2), L. Billot (2)

Affiliations : (1) Taras Shevchenko National University of Kyiv, 64 Volodymyrska St., 01601, Kyiv, Ukraine; (2) Laboratoire de Physique et d'Etude des Matériaux, ESPCI-CNRS UMR 8213, 10 rue Vauquelin, 75231 Paris France

Resume : Scanning near-field optical microscopy (SNOM) is a powerful tool for investigation of the materials properties at the nanoscale [1]. An effective way to detect the near-field in SNOM technique is to use a fluorescent nanoparticle located at the end of a sharp tip. Several kinds of fluorescent particles of rare-earth-doped inorganic materials have been successful as near-field probes (see e.g. [2]). In our work, we develop the near-field optical sensors usingthe Eu3+-doped LaVO4 submicron-sized particles as fluorescent probes. Single particles of La1-xEuxVO4 (with x ranging from 0.1 to 0.3) vanadate crystals were synthesized by two different methods. The particles were glued at the end of a sharp atomic force microscope tip and scanned on the surfaces of a sample made of thin metal film with sub-micron sized holes. Illumination of the samples was performed at 532 nm wavelength. The collection of the fluorescence light as a function of the tip position above the surface allowed to map the near-field optical distribution in the vicinity of the holes, at different heights above the surface showing the beam divergence in free space above the sample. Other possible applications of LaVO4:Eu3+ nanoparticles in SNOM technique, in particular as thermal sensors are analyzed. [1] L. Novotny, B. Hetch, in Principles of Nano-Optics, Cambridge University Press (2009). [2]L. Aigouy, Y. De Wilde, M.Mortier, Appl. Phys. Lett.83, 147 (2003).

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18:30 **Nano-grained thin film TiO2 characterized by ion, X-ray, and electron scattering**

Authors : K. A. Janik 1, B. Seger 2, M. B. Sillassen 3, C. D. Damsgaard 1, I. Chorkendorff 2, J. B. Wagner 1

Affiliations : 1 Center for Electron Nanoscopy, Technical University of Denmark, 2800 Kongens Lyngby, Denmark 2 Department of Physics, Technical University of Denmark, 2800 Kongens Lyngby, Denmark 3 Interdisciplinary Nanoscience Center and Department of Physics and Astronomy, Aarhus University, 8000 Aarhus, Denmark

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Resume : Titanium dioxide (TiO2) receives a lot of attention in the context of hydrogen production by photocatalytic water splitting, where TiO2 can be used as a layer protecting silicon photocathodes. For this application, the TiO2

material is synthesized by the physical vapor deposition (PVD) in form of thin films. Our work focuses on a comprehensive characterization and description of compositional, morphological, and crystallographic aspects of such TiOx deposits grown on Si <001> by reactive DC magnetron sputtering from a metal titanium (Ti) target in Ar/O2 gas mixture. Characterization of the TiOx thin film on a um-mm scale by means of Rutherford backscattering spectrometry (RBS) and grazing incidence X-ray diffraction (GIXRD) shows that the film is close to a stoichiometric compound, i.e. TiO_2, composed of two crystallographic phases, i.e. rutile and anatase. In order to follow the structure and the spatial distribution of the crystallographic phases on the sub-um level electron scattering, i.e. transmission electron microscopy (TEM) and electron diffraction (ED) is used. TEM and ED are performed on thin focused ion beam (FIB) milled lamellas prepared in the direction parallel and perpendicular to the direction of the TiO_2 film growth.

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18:30

Structures and transitions in vanadium dioxide

Authors : Kerrie Smith (1), Ana M Sanchez (1), David H Cobden (2) and Richard Beanland (1)
Affiliations : (1) Department of Physics, University of Warwick, Coventry CV4 7AL, UK (2) Department of Physics, University of Washington, Seattle WA98195, USA

Resume : Vanadium dioxide is a correlated electron material (CEM), and is one of the most promising thermochromic materials with a near room-temperature metal-insulator transition (MIT) at ~65°C. At low temperatures, VO_2 exhibits two monoclinic, insulating phases, M1 and M2, and in certain strain conditions a poorly-characterised triclinic phase, thought to be intermediate between M1 and M2. Upon heating, the material undergoes a phase transition to a metallic rutile (tetragonal) phase. Here, we investigate free-standing and strained PVD-grown single crystal nanobeams of this intriguing material through the use of transmission electron microscopy and novel forms of electron diffraction. We use computer control of the microscope to rapidly collect several thousand convergent beam electron diffraction (CBED) patterns, each with a different angle of incidence. This dataset can be employed to form 'digital' large-angle CBED patterns, which are very sensitive to material symmetry and structure. Data were obtained around the MIT of VO_2 in different strain states. We present evidence for a transitory non-centrosymmetric phase which persists for several minutes in a freestanding nanobeam after cooling through the MIT. As well have having applications in devices, achieving control of this well known phase transition could lead to improved understanding of CEM's in general.

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18:30

MARS (Modelling Angle Resolved Spectroscopy), a new software for GIXRF (Grazing Incidence X-ray Fluorescence Analysis) data analysis

Authors : Lars Luehl 1, Christian Herzog 2, Janis Eilbracht 3, Beatrix Pollakowski 3, Werner Jark 1, Markus Kraemer 4, Burkhard Beckhoff 3, Birgit Kanngiesser 2, Diane Eichert 1,
Affiliations : 1 Elettra - Sincrotrone Trieste, Italy; 2 Technische Universitaet Berlin (TUB), Germany; 3 Physikalisch Technische Bundesanstalt (PTB), Germany; 4 AXO DRESDEN, Germany

Resume : The use of functional nano-materials spreads rapidly in modern technology demanding a steady development of analysis tools at the nanoscale. GIXRF is a promising method for analyzing buried or near surface nanostructures but data handling is challenging and has to be adapted to the sample and experimental conditions. Different material structures were analyzed by now from various research groups not by employing common software tools, but by developing proprietary group software. Elettra presents MARS, a software developed for the new XRF beamline at Elettra in cooperation with TUB. Present features and planned extensions are described and their potential for experimental data interpretation emphasized. Synchrotron based GIXRF experiments on relevant thin film test structures are presented and the results interpreted with MARS simulations. Additionally, MARS is compared with the options implemented in MXSW (M. Kr?mer) and XSWini (PTB) and the commonly used program IMD. The comparison is done in terms of included physical aspects like geometry effects, absorption and beam divergence when applied to samples structured with gradients (i.e. layered systems, nano-particles on surfaces). Program accessibilities and their user-friendliness are also discussed. The aim of this study is to direct future GIXRF users to the most appropriate characterization tool for their specific material system, and intends to give a general insight into the challenges in GIXRF data evaluation.

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18:30 **Self-assembled growth of Ni nanoparticles in amorphous alumina matrix**
Authors : M. Jerčinović1, N. Radić1, M. Buljan1, J. Grenzer2, I. Delač Marion3, M. Kralj3, I. Bogdanović Radović1, R. Hübner2, P. Dubček1, K. Salamon3, S. Bernstorff4
Affiliations : 1 Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia; 2 Helmholtz-Zentrum Dresden-Rossendorf, P O Box 510119, 01314 Dresden, Germany; 3 Institute of Physics, Bijenička cesta 46, 10000 Zagreb, Croatia; 4 Elettra-Sincrotrone, SS 14 km163.5, 34149 Basovizza, Italy
Resume : We present the formation of ordered 3D lattice of Ni nanoparticles (NP) in amorphous alumina matrix achieved by a selfassembly process during a single-step magnetron sputtering deposition of Ni/Al2O3 multilayer at room temperature. The structure of the films was analyzed using Grazing Incidence Small and Wide Angle X-ray Scattering, Transmission Electron Microscopy, Atomic Force Microscopy, Grazing Incidence Wide Angle X-ray Scattering, and Time-of-Flight Elastic Recoil Detection Analysis measurements. The self-assembly is driven by surface morphology effects and it results in a body-centered tetragonal (BCT) lattice of Ni particles with crystalline face-centered cubic (FCC) internal structure in amorphous Al2O3 matrix. The size distribution of Ni NPs is narrow, and the material has good mechanical properties due to the alumina matrix. We show that the NP sizes and separations can be easily tuned by a suitable choice of the deposition conditions. The quality of the ordering achieved in the alumina matrix is found to be significantly better than the ordering of Ni particles in silica. The obtained results are important for the understanding of the self-assembly process of metallic particles in amorphous matrices and the applications of such materials. The prepared materials are potentially interesting for spintronic applications.

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18:30 **Magnetically enhanced batch electrosharpening of tungsten probes**
Authors : Richard Stone, Leon Bowen, Karl Coleman, Mike Petty, Dagou Zeze
Affiliations : Durham University School of Engineering, Department of Physics, Department of Chemistry
Resume : Tungsten probes are disposable tools utilised in a wide range of nanoscale applications and analytical techniques, such as scanning probe microscopy and dielectrophoretic manipulation. However, existing electrochemical methods can only fabricate individual probes one at a time to a fixed length. Here, we demonstrate an automated process capable of fabricating multiple probes simultaneously to a desired length whilst maintaining tip sharpness below 20 nm radius. The fabrication exploits the tungstate by-product formed during electrochemical etching to form a decreasing etch rate along the probe such that to a ~20 nm sharp tip is produced. However, etching can be prolonged to control the probe aspect ratio. Probe lengths are controlled by monitoring the current which is defined by the overall surface area of these probes. This process enables multiple probes to be fabricated simultaneously, albeit with varying lengths. To control the length of these probes, a divergent magnetic field is applied to induce a Lorentz force, decreasing from the tips down towards the base. This creates a differential etch rate which decreases from the tip to the base such that all probes tend to exhibit the same length (up to 5 mm) and conical shape. This offers a greater mechanical strength and resistance to vibration. Probe sharpness was analysed by scanning electron microscopy and confirmed with scanning tunnelling microscopy testing of highly ordered pyrolytic graphite.

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add to my program **(close full abstract)**

18:30 **Laboratory full-field transmission X-ray microscopy and applications**
Authors : C. Seim (1+), H. Legall (1+), A. Dehlinger (1+), H. Stiel (2+), F. Rancan (3), M. Meinke (3), S. Rehbein (4) and B. Kanngießer (1+)
Affiliations : (+) Berlin Laboratory for innovative X-ray technologies (BLiX); (1) Technische Universität Berlin, Institut für Optik und Atomare Physik, Hardenbergstr. 36, 10623 Berlin; (2) Max-Born-Institut, Max-Born-Str. 2A, 12489 Berlin; (3) Charité Berlin, Clinical Research Center for Hair and Skin Science, Charitéplatz 1, 10117 Berlin; (4) Helmholtz-Zentrum für Materialien und Energie, Albert-Einstein-Str. 15, 12489 Berlin
Resume : Water window X-ray microscopy has become a valuable imaging tool with a resolution in the nanometer regime. The development of highly brilliant laboratory X-ray sources has led to an upcoming of laboratory based transmission X-ray microscopes (LTXM), and thus opened up a technique, previously limited to large scale synchrotron facilities, to a broader community. We introduce the laser plasma driven laboratory full-field transmission X-ray microscope located at the Berlin Laboratory for innovative X-ray technologies [1]. The LTXM reaches a resolution of $\Delta x = 31$ nm ± 3 nm (half-pitch), which is comparable to resolutions achieved at synchrotron facilities. The microscope’s

HP6 29

wavelength at 2.478 nm lies within the water window. Herein, the low absorption of water, in comparison to carbon, offers high natural contrast for the investigation of cryo fixated biological specimens in their natural environment, without the need of extensive sample preparation. Working in the water window also enables the investigation of thick aqueous samples of up to 10 µm, making soft X-ray cryo tomography feasible. An overview of first applications, like measurements on cryo-frozen yeast cells and human primary keratinocytes, will be given. [1] H. Legall, G. Blobel, H. Stiel, C.Seim et al., "Compact x-ray microscope for the water window based on a high brightness laser plasma source," Opt.Express, 20(16), 18369-18369 (2012).

add to my program **(close full abstract)**

18:30 **Investigating embedded nanosystems with angle resolved fluorescence spectroscopy** HP6 30

Authors : J. Baumann[1], B. Pollakowski[2], K. Bethke[3], D. Eisenhauer[4], K. Rademann[3], B. Beckhoff[2], and B. Kanngießer[1]
Affiliations : [1] Institute for Optics and Atomic Physics, Technical University of Berlin, 10623 Berlin, Germany; [2] Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany; [3] Institute of Chemistry, Humboldt Universität zu Berlin, Brook -Taylor-Strasse 2, D-12489 Berlin, Germany; [4] Helmholtz-Zentrum Berlin für Materialien und Energie, Institut für Silizium-Photovoltaik, Kekuléstraße 5, 12489 Berlin, Germany

Resume : By changing the angle of incident X-radiation on a sample and detecting the fluorescence signal, elemental depth profiles of nanolayered systems can be obtained. In addition, with the analysis of the absorption edges of the probed atoms, depth resolved chemical information is available, too. This combination of grazing incidence X-ray fluorescence (GIXRF) and near edge X-ray absorption fine structure (NEXAFS) can be used in a reference free manner and thus is a valuable tool to get structural and chemical information about novel nanosystems without the need of usually rare reference materials[1]. The electrical bonding of embedded nanosystems is of mayor interest for a large variety of scientific areas, e.g. in semi-conductor industry or thin-film solar cell technology. In the frame of the School of Analytical Sciences Adlershof (SALSA), nanolayers of coinage metals deposited on top of soda-lime glass are investigated with regard to their electrical properties. Reference-free GIXRF-NEXAFS measurements of thin-film silver-copper-glass systems, which show promising behavior for electrical bonding, were performed at the PGM beamline of the Physikalisch-Technische Bundesanstalt (PTB) at the Synchrotron BESSY II in Berlin. First results concerning diffusion processes and chemical states will be presented. [1] Pollakowski, B. et al., Analytical Chemistry, 2013, 85, 193-200

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18:30 **Characterization of ceria nanoparticles through advanced modelling** HP6 31

Authors : Kersti Hermansson, jolla Kullgren, Adri van Duin, Peter Broqvist
Affiliations : Department of Chemistry, Ångström Laboratory, Uppsala University; BCCMS, University of Bremen; Mechanical and Nuclear Engineering, Pennsylvania State University; Department of Chemistry, Ångström Laboratory, Uppsala University

Resume : In this talk we will discuss ceria clusters and nanoparticles in reducing, oxidative and humid environments and the powerful analyses and predictions that can be achieved though computational materials modelling. Ceria (CeO_2) is an interesting material with a range of technical applications, for example in solid oxide fuel cells and for the purification of exhaust gases in vehicle emissions control. Behind these technically important applications lies one overriding feature, namely ceria's exceptional reduction-oxidation properties enabled by the duality of cerium, which easily toggles between Ce^{4+} and Ce^{3+} thanks to its 4f electron. High-quality calculations need to be able to mimic these intricate details and at the same time treat realistically large (and dynamic) nanoparticles.This is a challenge. We use a range of theoretical methods, including DFT, DFTB and force-field calculations with a newly parametrized reactive force-field - all within a multi-scale simulation environment. Based on the quantum-chemical calculations, we find that small ceria nanoparticles of certain shapes (such as perfect octahedra) can be stabilized through the adsorption of oxygen molecules in the form of superoxo species (in agreement with experimental studies), and water in the form of hydroxo species. Moreover, based-on force-field simulations we can predict the relative stabilities of very large ceria nanoparticles of different shapes.

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18:30 **Ccmparative studies of isomerization reactions of undoped polyacetylene(PA) by 'R.M.N 13C – F.T.I.R – RAMAN et D.S.C '** HP6 32

Authors : Z. Skanderi1*, F. Mechachti1, S. Bitam2, A. Djebaili1

Affiliations : 1 Laboratory of chemistry and environmental chemistry L.C.C.E - University of Batna- Algeria 2 Laboratory of Physical chemistry- University of Media- Algeria

Resume : In order to find an interpretation to the isomerization reaction of undoped 'cis ' polyacetylene PA, we have resumed the recent work and analyzed them using the most advanced methods. We used the line intensities attributed to deformation vibrations of C-H bonds out-of-plane at 740 cm-1 for cis and 1015 cm-1 for trans to calculate the ratio cis / trans. The relative percentages of the cis and trans are calculated by: % cis = 100 x 1,3 . A cis (740 cm-1) / 1,3 . Acis (740 cm-1) + Atrans(1015 cm-1) % trans = 100 x A trans (1015 cm-1) / 1,3 . Acis (740 cm-1) + Atrans(1015 cm-1) These two bands are close to each other, we used a program based on deconvolution, in order to resolve these peaks: LineSim simulation program written by P.F. Barron from Brisbane NMR Centre, Griffith University, Natman Q 4111, Australia. The results of the rate of cis and trans isomers obtained by NMR were compared with those given by FTIR. We observed that for high levels of cis, values calculated by NMR are always higher than those given by infrared, while this trend is reversed at high levels in trans. The results of this study are presented in the following table: Ito1 F.T.I.R Gibson2 R.M.N Montaner3 F.T.I.R Tabacik4-5 Raman - DSC Energy Ea (cal / mole) 17181.295 12840.102 30524.790 31000.000 Collision factor A (s-1) 4.28 • 108 3.94 • 103 8.86 • 1012 2.27*1013 Temperature field (°C) 75 -- 115 90 -- 110 115 -- 160 120 -- 160 Regression coefficient r - 0.991113 - 0.946320 + 0.958370 + 0.999985

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18:30 **Characterization at the nano-scale with Small Angle X-Ray Scattering**

HP6 33

Authors : Sergio Rodrigues, Sandra Desvergne-Blenau, Frédéric Bossan, Manuel Fernandez-Martinez, Blandine Lantz, Ronan Mahe, Pierre Panine, Peter Høghøj,
Affiliations : Xenocs SA, Sassenage, France

Resume : Small Angle X-Ray Scattering (SAXS)1 is a technique well suited for investigating the structure of materials in the range from 1 to beyond 100 nm. In combination with Wide Angle X-ray Scattering (WAXS), the technique can provide information on structure with length-scales down to 0.1 nm, thus allowing measuring both crystalline and nano-structure properties. The technique gives information on sample structure parameters such as shape or size, size distribution, orientation and anisotropy, surface to volume ratio. The x -ray beam penetrates the sample and typically probes a sample volume from 0.1-1 mm3. The information on structure is therefore obtained from inside the sample and is statistically representative of the sample volume probed. This is a major difference to local probe techniques such as microscopy and makes the technique a good candidate for quality control. Furthermore, little sample preparation is needed and the technique is non-destructive. It is also possible to observe changes in the sample as a function of parameters such as (but not limited to) temperature. The capability of laboratory based solutions for characterization at the nano-scale is recognized in the ISO standard currently being drafted2 on measurement of particle size. We will show different examples of particle sizing and size-distribution measurements with SAXS. Nanostructure of advanced materials such as polymers is also an area of use for SAXS. We will show how the method can be used for mapping of local nanostructure and orientation of polymers and how this correlates with process parameters The technique can also be used in grazing incidence mode (GISAXS), making it sensitive to surface nano-structure. We will present SAXS results from a broad range of polymer samples with the aim to discuss how this technique can be a complement to other analytical methods. [1] Glatter O, Kratky O, ed. (1982). Small Angle X-ray Scattering. Academic Press., (downloadable from http://physchem.kfunigraz.ac.at/sm/Software.htm) [2] DIS ISO/NP 17867 prepared by committee ISO/TC24/SC4/WG10

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18:30 **Molecular level investigation of chain conformation for contact printed poly (dimethylsiloxane) by surface sensitive nonlinear optical measurement**

HP6 34

Authors : Soo Sang Chae, Woo Soon Jang, YOUNG Bum Yoo, Jin Young oh, Jee Ho Park, Keun Ho Lee, Sun Woong Han, Jee Hoon Lee, Kwang Hyun Kim, Hong Koo Baik
Affiliations : Yonsei University, Seoul, Korea

Resume : Sum-frequency vibrational spectroscopy and second-harmonic generation have been used to measure the orientational distribution of the oligomer chains and adsorbed 5CB liquid crystal molecules on a ultra-thin Polydimethylsiloxane (PDMS) films prepared by means of inkless contact printing. Results show that the methyl functional groups in the films to be heading toward to the air and also, the main chain to be well aligned by the

detaching of PDMS slabs after contact printing. The adsorbed liquid crystal molecules are aligned, in turn, by the surface oligomer chains. Strong correalation exists between the orientational distribution of the oligomer chain and the liquid crystal molecules, implied that surface-induced bulk alignment of a liquid crystal medium by the aligned oligomer on the surface is via an orientational epitaxy-like mechanism.

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PROGRAM VIEW : 2014 Spring
MY PROGRAM : 2014 Spring

Symposium : H

ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

26 May 2014	**27 May 2014**	28 May 2014	29 May 2014	30 May 2014

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start at	Subject	Num.
	Characterization of Nano Materials with Advanced X-Ray Technologies 1 : Birgit Kanngiesser and Diane Eichert	
08:30	**Nano-scale feature analysis: Achieving high effective lateral resolution with micro-scale material characterization techniques** **Authors :** T. Conard **Affiliations :** IMEC, MCACSA Kapeldreef 75, 3001 Leuven Belgium **Resume :** Achieving higher performances of electronic devices was first realized by decreasing feature size followed by the introduction of new materials and the switch from planar devices to non-planar ones. Unfortunately many characterization techniques do not reach lateral resolution compatible with device sizes. This is the case for instance for XPS or (TOF-)SIMS. We will present examples where, using specially designed samples and/or simple mathematics, the effective lateral resolution can be increased by several order of magnitude. In back-end, trenches are formed by RIE (CF-based chemistries) to be filled with metal lines. This process leaves residue on the surfaces that need to be cleaned. XPS allows to analyses the chemical composition of (remaining) surface components but does not have the resolution needed to investigate single sidewall. By using periodic structures combined with a mathematical model, the composition of the top, sidewall and bottom of the trenches can be separated and identified. In the most advanced processor, finfet transistors are used, with dimension down to several nm. These are fabricated from epitaxially grown trenches from the active material. A good control of the composition of the grown material is critical to the device performance. In this example, we show that retrieving vertical composition profile from trenches with width down to a few 10's of nm is possible using Dynamic SIMS or TOFSIMS. Results will also be compared with Auger analysis.	H7 1
	add to my program (close full abstract)	
09:00	**High-spatial resolution synchrotron X-ray scattering of 3D colloidal nanocrystals assemblies dried on superhydrophobic surfaces** **Authors :** Angelo Accardo, Francesco Di Stasio, Manfred Burghammer, Christian Riekel, Roman Krahne **Affiliations :** Istituto Italiano di Tecnologia, Via Morego 30, 16163, Genova, Italy; European Synchrotron Radiation Facility, B.P. 220, 38043, Grenoble, France **Resume :** Superhydrophobic surfaces (SHSs) are a versatile platform for the investigation and study of conformational changes and crystallization phenonomena in nanomaterials [1]. The slow and homogenous evaporation of a droplet containing molecules or nanoparticles on a SHS led to the formation of highly ordered aggregates, which have been investigated by Small and Wide Angle X-ray Scattering (SAXS/WAXS) at the ESRF ID13 Microfocus beamline. This spatially resolved X-ray diffraction technique is a powerful tool to give deeper insight into the 3D arrangement of nanomaterial deposits. In this work we investigate three-dimensional dome-shaped aggregates formed by core-shell CdSe/CdS nanorods that were dissolved in aqueous solution. Such core-shell nanorods are a very attractive material for optical applications due to their bright and tunable emission, gain and lasing properties [2]. The spatial X-ray diffraction mapping with few micron resolution revealed that the 3D dome-like residuals consisted of crystalline salt deposits (borax) and highly oriented nanorod assemblies. Our technique of slow evaporation of highly luminescent nanorods in a salty buffer solution on SHSs could lead to the development of	H7 2

solid 3D microstructures that can function as optical components, such as polarized light emitters. [1] Accardo et al., Langmuir, 2010, 26(18), 15057-15064; [2] Zavelani-Rossi et al., Laser & Photonics Reviews, 2012, 6(5), 678-683.

add to my program **(close full abstract)**

09:15 **Surface and sub-surface thermal oxidation of Ru and O diffusion in RuO_2 thin films**

Authors : R. Coloma Ribera, R. W. E. van de Kruijs, S. Kokke, E. Zoethout, A. E. Yakshin, F. Bijkerk
Affiliations : FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands. MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands; FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands. MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands; FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands; FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands; FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands. MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands; FOM Dutch Institute for Fundamental Energy Research (DIFFER), P.O. Box 1207, 3430 BE Nieuwegein, The Netherlands. MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands;

Resume : Ru and RuO_2 present attractive properties such as low resistivity, high thermal stability and good diffusion capability that can make them suitable candidates for many applications such as catalysis, electronics and optical coatings. In general, surface and near-surface oxidation of Ru is well characterized, but deeper sub-surface oxygen diffusion and subsequent oxidation is often not addressed. We present hard x-ray reflectivity (XRR) measurements as an accurate method for in situ monitoring of thermal oxidation of Ru. This method allows precise determination of the in-depth electron density distribution, providing information about densities, thicknesses and intermixing/roughnesses of formed RuO_x and remaining Ru layers. Combining x-ray reflectivity, Auger electron spectroscopy, angular-resolved x-ray photoelectron spectroscopy, atomic force microscopy and x-ray diffraction data, we present a detailed description of surface and sub-surface oxidation of ruthenium thin films. We establish a correlation between the oxide agglomerates appearing on the surface and the formation of sub-surface oxide layers during annealing. Furthermore, a reduction of the diffusion constant for O in RuO_2 is observed during RuO_2 growth, suggesting densification of the RuO_2. Finally, the activation energy for O diffusion in RuO_2 is linked to the initial density of the Ru layers, with low Ru density resulting in lower activation energy.

H7 3

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09:30 **Reference-free, depth dependent characterization of nanoscaled systems with advanced grazing incidence X-ray fluorescence analysis**

Authors : Philipp Hönicke, Matthias Müller, Burkhard Beckhoff
Affiliations : Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany

Resume : The accurate characterization of nanoscaled systems is an essential topic for today's developments in many fields of materials research. Thin high-κ layers and stacks as well as ultra-shallow dopant profiles are technologically relevant for current and future electronic devices. But the metrological challenges to characterize such systems require a further development of the current analytical techniques. When performing Grazing Incidence X-ray Fluorescence (GIXRF) analysis in combination with X-Ray Reflectometry (XRR), the characterization reliability for such nanoscaled systems can be improved. GIXRF is based on the incident angle induced changes of the X-ray Standing Wave (XSW) field intensity profile. The combination with XRR allows for a more reliable modeling of the XSW. Employing in-house built instrumentation [1] and radiometrically calibrated detectors at the Physikalisch-Technische Bundesanstalt the combined method allows for reference-free quantitative in depth analysis [2,3]. The capabilities of the XRR enhanced GIXRF method are demonstrated by means of several nanoscaled layer systems, ultra-shallow dopant profiles and self-assembled molecular samples. The results are validated by complementary investigation. [1] J. Lubeck et al., Rev. Sci. Instrum. (2013) 84, 045106. [2] P. Hönicke et al., J. Anal. At. Spectrom. (2012) 27, 1432-1438. [3] P. Hönicke, M. Müller, B. Beckhoff, Solid State Phenomena (2013) 195, 274-276.

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09:45 **In-situ SAXS study of phase segregation in thermoplastic eleastomers**
Authors : H. Hristov1, O. Thomas1, B. Hsiao2
Affiliations : 1-Kimberly Clark Corporation 2-SUNY Stony Brook
Resume : The thermoplastic elastomers are block copolymers comprised of ? hard? and ?soft? block chain segments. The hard blocks segregate into domains with typical dimensions in the range of 10nm-30nm. The mechanical response of the materials is strongly dependent on the final morphology, which in turn depends on the processing conditions. The equilibrium morphologies of the thermoplastic elastomers have been studied extensively; however the structural developments in dynamic regime are virtually unknown. X-ray diffraction measurements can provide detailed information regarding the phase segregation kinetics. To this end, series of in-situ Small Angle X-ray Scattering measurement were performed at the synchrotron line in Brookhaven National Laboratory. Mono-disperse SEBS and SEPS block copolymers were the materials of choice in our study. The polymers were extruded at high shear and deformation rates in a filament form, in the temperature range from 200oC to 250oC. The X-ray beam was positioned appropriately to record the structural changes in dynamic regime. The collected experimental data were compared to several model structures. It was found that the molten polymers start from the disordered state (Random Phase Approximation model) at temperatures higher than 230oC. On cooling to 200oC, rapid phase segregation with significant phase fluctuations occurs. The hard block segments segregated into hexagonally packed cylinders before leaving the spinneret of the extruder. The rapid segregation at higher temperatures is followed by a slower process of 3-D spatial ordering, which could take up to two weeks depending on the annealing conditions. The results of this study and different instrumental details will be presented and discussed.

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10:00 **Coffee break**

Characterization of Nano Materials with Advanced X-Ray Technologies 2 : Gerhard Ulm and Blanka Detlefs

10:30 **Characterization of Ni rods arrays distributed in SiO2 matrix by means of synchrotron radiation spectroscopic and microscopic techniques**
Authors : E.V. Parinova 1, S.Yu. Turishchev 1, R. Ovsyannikov 2, F. Kronast 2, D.E. Spirin 1, D.A. Koyuda 1, D.N. Nesterov 1, A.V. Mazanik 3, E.A. Streltsov 3, N.V. Malaschenok 3, A.K. Fedotov 3
Affiliations : 1 - Voronezh State University, Universitetskaya pl. 1, 394006, Voronezh, Russia. 2 - Helmholtz-Zentrum Berlin, Albert-Einstein-Str. 15, 12489, Berlin, Germany. 3 - Belarusian State University, pr. Nezavisimosti 4, 220030, Minsk, Belarus.
Resume : Magnetic materials rods distributed over the dielectric matrix is interesting for researchers because of the giant magnetoresistance property. We want to present results of the atomic and electronic structure as well as the remanence characterization of Ni rods arrays (~ 200-500 nm diameter at top) in SiO2 matrix by means of multiple surface sensitive techniques applied with the use of highly brilliant synchrotron and X-ray radiation. X-ray and electron spectroscopy and microscopy (XANES, USXES, XPS and PEEM respectively) were used as the combination of macroscopic and microscopic approaches. PEEM technique were applied for remanence study as well. BESSY II (Germany) and SRC (USA) storage rings synchrotron radiation was used for all studies except USXES investigations. The comprehensive work done allowed us to fully characterize the local atomic surrounding and phase composition of rods arrays surface at whole and possible rod-matrix and rod-rod interfaces. Relatively low surface contamination of residual Ni oxides is shown and nickel silicide phase contribution is barely noticeable (detected only by macroscopic O K XANES study) showing the absence of Ni rod atoms interphase interaction with ambient SiO2 matrix. It is shown that almost every rod are connected to another by ~ 50-150 nm Ni bridge. Remanence distribution is detected from at least 3 microns field of view for small rods array without and with applied magnetic field.

H8 1

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10:45 **Focused X-ray Diffraction measurements combined X-ray Emission Optical Light Spectroscopy on membrane structures**

H8 2

Authors : Grifone R. * 1, Kriegner D. 1 , Martin Sanchez J. 1, Trotta R. 1, Rastelli A. 1,

Stangl J. 1 , Schülli T. *
Affiliations : * European Synchrotron Radiation Facility, 6 rue Jules Horowitz, BP 220, 38043 Grenoble, France; 1 Institute of Semiconductor and Solid State Physics, JKU Linz, Altenbergerstr. 69, 4040 Linz, Austria

Resume : We present investigations on strain anisotropy in embedded quantum dot (QD) structures. The strain has been resolved locally using focused X-ray diffraction (XRD) and has been linked to micro-photoluminescence (μ-PL) characterisations. To render and modify the electronic structure of a QD as a source of entangled photon pairs, anisotropic strain can be used.[1] Due to fabrication inhomogeneities, post growth tuning of emission wavelength is essential.[2] For this purpose, QDs embedded into a (In,Al,Ga)As membrane matrix have tunable optical properties through applying an electric field and at the same time a strain field. The latter is achieved by bonding the micron-sized membrane onto a piezoelectric substrate. We characterize the in-plane strain distribution in individual membranes. At the same time X-ray excited optical luminescence (XEOL) is recorded from the specimen. This permits us probing a few μm^3 sized volume of the sample, while measuring the strain state of the very same spot. Both spectra, XEOL as well μ-PL can be correlated. These results are used to optimize the growth method and target a better understanding of the system. In particular how anisotropic strain evolves as a function of applied voltage and consequently can be tuned in the future. These measurements using focused XRD along with XEOL show the potential of combining both techniques. Ref: [1] Trotta R. et al., Adv. Mater. 24, 2668 (2012) [2] Rastelli A. et al., phys. stat. sol. (c), 249, No 4. 687–696 (2012)

add to my program **(close full abstract)**

11:00 **The X-Ray Fluorescence beamline at elettra: opening new opportunities for characterization of nanomaterials**

Authors : Diane Eichert, Werner Jark, Lars Luehl, Alessandro Gambitta, Andreas Germanos Karydas, Alessandro Migliori, Juan Jose Leani, Mladen Bogovac, Halim Sghaier, Ralf Bernd Kaiser
Affiliations : Diane Eichert - X-Ray Fluorescence Beamline - ELETTRA - Sincrotrone Trieste, Area Science Park, 34149 Basovizza, Trieste, Italy; Werner Jark -X-Ray Fluorescence Beamline - ELETTRA - Sincrotrone Trieste, Area Science Park, 34149 Basovizza, Trieste, Italy; Lars Luehl - X-Ray Fluorescence Beamline - ELETTRA - Sincrotrone Trieste, Area Science Park, 34149 Basovizza, Trieste, Italy; Alessandro Gambitta - X-Ray Fluorescence Beamline - ELETTRA - Sincrotrone Trieste, Area Science Park, 34149 Basovizza, Trieste, Italy; Andreas Germanos Karydas - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria; Alessandro Migliori - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria; Juan Jose Leani - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria; Mladen Bogovac - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria; Halim Sghaier - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria; Ralf Bernd Kaiser - International Atomic Energy Agency, Nuclear Science and Instrumentation Laboratory, Seibersdorf, Austria

H8 3

Resume : The XRF beamline is conceived as a multi-purpose beamline designed to accommodate a variety of end-stations dedicated to e.g. microscopy, Total Reflection XRF (TXRF) or spectroscopy. The XRF beamline is located at a bending magnet source. Its excitation energy range is 2-14 keV, its resolving power 1.4 10-4. The source is re-imaged to a 250 X 50 mm beamsize (hor X vert) in an exit slit, with an angular divergence of 0.15 mrad and a transmitted flux of about 5 109 ph/s (5.5 keV, 2GeV). The XRF beamline is presently hosting the IAEA Ultra-High-Vacuum Chamber (UHVC), based on a prototype built by PTB [1], currently under commissioning. The aim is using tunable synchrotron X -rays with 50 m beamsize for various X-Ray Spectrometry techniques: TXRF, Grazing Incidence/Exit XRF (GI-XRF/GE-XRF), X-Ray Reflectometry (XRR) or X-ray Absorption Spectroscopy (XAS). GIXRF is an established tool for the elemental analysis of nanoscaled materials surfaces or interfaces. Combined with XRR it provides relevant insights into nanoscaled layered systems, and correlated with XAS can probe elemental speciation. This addresses well characterization issues in the nanometer range of energy-related nano-scaled materials. A description of the beamline, analytical developments, commissioning results and pilot research experiments will be presented, and highlights put on the manifold possibilities that the setup offers to analyse nanoscale samples. [1] J. Lubeck et al (2013) Rev. Sci. Instrum. 84: 045106

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Innovative Ion Beam Surface Analysis : Thierry Conard and Bonnie Tyler

11:30 **Characterizations of the oxide semiconductor and OLED materials using combination of surface analysis methods**

Authors : Jae Cheol Lee, Dong Jin Yun, Jae Gwan Chung, Eun Ha Lee, Yong Koo Kyoung, Sung Heo, Gyeong-Su Park
Affiliations : Analytical Engineering Group, Platform Technology Lab, Samsung Advanced Institute of Technology, Samsung Electronics Co Ltd

Resume : This presentation is composed of two parts. The part one is the investigation of the interface reactions between SiO_2 or SiN_x passivation layer and HfInZnOx oxide semiconductor using XPS, TOF-SIMS, HEXPS in-situ PES and TEM. The other is the effects of Ar ion and Ar gas cluster ion beam (GCIB) sputtering processes on the core-level structure, valence band structure and work function of poly (3,4-ethylenedioxythiophene) polymerized with poly (4-styrenesulfonate) (PEDOT:PSS) and multi wall carbon nanotube (MWNT)/PEDOT:PSS films which were characterized by XPS. In the part one, we observed metallic indium and silicate states at the passivation layer/HIZO interfaces, and low binding energy shift of Zn 2p and In 3d as well. The metallic indium is believed to cause a conduction channel at the interface and thus originate the Vth negative shift. In addition, we found that indium diffused from HfInZnOx layers into passivation layers and silicate states were formed at the passivation layer/HIZO interfaces. HEXPS analyses revealed that in-gap states near 1.6eV and defect states near 0.2eV were present at the SiO_2/HIZO interface. In the part two, by PES combined with Ar GCIB sputtering, the core-level/valence band structures of widely applied organic semiconducting/conducting films are characterized in the damage minimized condition.

H9 1

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12:00 **Ultralow energy SIMS depth profiling of patterned III-V heterostructures**

Authors : V. Gorbenko(1,2), F. Bassani(2), T. Baron(2), R. Cipro(2), M. Martin(2) , A. Grenier(1) , Y. Bogumilowicz(1), G. Audoit(1), X.Y. Bao(3), Z. Ye(3), JB. Pin(3), E. Sanchez(3), J.-P. Barnes(1)
Affiliations : (1) CEA, LETI, MINATEC Campus, 17 rue des Martyrs, 38054 Grenoble Cedex 9, France; (2) LTM/CNRS, 17 rue des Martyrs, 38054 Grenoble Cedex 9, France; (3) AMAT, 3050 Bowers Avenue, Santa Clara, CA 95054, USA;

Resume : Secondary ion mass spectrometry (SIMS) allows thin multilayer systems to be studied with excellent depth resolution and sensitivity. SIMS is widely used in microelectronics for characterizing the quality of interfaces and chemical composition in two-dimensional layers. We have developed specific SIMS protocols in order to analyze III-V heterostructures grown by MOCVD on 300 mm Si wafers with an oxide pattern. These structures may find applications in future III-V FinFET devices monolithically integrated on Si. As-based heterostructures were deposited by selective epitaxial growth in SiO_2 windows of different widths. This method allows extended-defect-free materials to be obtained at the top of cavities as misfit dislocations are blocked by the oxide walls. The patterns are smaller than the resolution limit of most magnetic sector SIMS instruments. Nevertheless we have shown that it is possible to obtain depth profiles of InGaAs quantum wells a few nanometers thick in a non-planar sample. These high-depth resolution SIMS profiles from filled cavities of a few hundred nanometers in width are compared with FIB-STEM observations on the same wafers. TOF-SIMS with lateral resolution of less than 100 nm was used for 3D reconstruction of multilayers in patterned sample. Atom probe tomography was used to obtain 3D reconstructions of the layers from a single cavity. This confirms the SIMS analysis and allows the effect of single defects to be investigated.

H9 2

add to my program (close full abstract)

12:15 **Correlative microscopy using SIMS for high-sensitivity high-resolution elemental mapping**

Authors : T. Wirtz, D. Dowsett, S. Eswara, Y. Fleming, P. Philipp
Affiliations : Department “Science and Analysis of Materials” (SAM), Centre de Recherche Public – Gabriel Lippmann, 41 rue du Brill, L-4422 Belvaux, Luxembourg

H9 3

Resume : Nano-analytical techniques and instruments providing both excellent spatial resolution and high-sensitivity chemical information are of extreme importance in materials science and life sciences for investigations at the nanoscale. Transmission Electron Microscopy (TEM), Helium Ion Microscopy (HIM) and Scanning Probe Microscopy (SPM) are commonly used for high-

resolution imaging. However, these techniques have all the same important drawback: they provide no or only very limited chemical information. By contrast, Secondary Ion Mass Spectrometry (SIMS) is an extremely powerful technique for analysing surfaces owing in particular to its excellent sensitivity, high dynamic range, very high mass resolution and ability to differentiate between isotopes. In order to get chemical information with a highest sensitivity and highest lateral resolution, we have developed three prototype instruments combining TEM, HIM and SPM with in-situ SIMS. The results are very encouraging and show in particular that excellent detection limits are reached by using reactive gas flooding techniques. The combination of high-resolution microscopy and high-sensitivity chemical mapping on a single instrument represents a new level of correlative microscopy. In this talk, we will present the instruments that we have developed, give an overview of the obtained performances, present typical examples of applications and make a comparison between ex-situ and in-situ combination of these techniques.

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12:30 **Laterally resolved analyses of thin film electrodes by complimentary ion beam surface analysis techniques**

Authors : John Druce, Helena Tellez, Young-Wan Ju, John Kilner, Tatsumi Ishihara
Affiliations : wpi-I2CNER, Kyushu University, Fukuoka, JAPAN, 819-0395; wpi-I2CNER, Kyushu University, Fukuoka, JAPAN, 819-0395; wpi-I2CNER, Kyushu University, Fukuoka, JAPAN, 819-0395; wpi-I2CNER, Kyushu University, Fukuoka, JAPAN, 819-0395 and Department of Materials, Imperial College London, London UK, SW7 2BP; wpi-I2CNER, Kyushu University, Fukuoka, JAPAN, 819-0395;

Resume : The surface composition and chemistry of the oxygen electrode strongly influences both the performance (i.e. oxygen exchange rate) and degradation (e.g. segregation and poisoning) of electrochemical energy conversion devices such as Solid Oxide Fuel Cells (SOFC's) and Solid Oxide Electrolysers (SOEC's). However, little is known about the precise mechanisms and active sites involved in the surface exchange reaction on these solid oxide electrodes. Whilst Secondary Ion Mass Spectrometry (SIMS) approaches are well established for the analysis of these solid oxide electrode materials, the complementary, yet less well-known, technique of Low Energy Ion Scattering (LEIS) is recently attracting more interest. By combining the excellent elemental sensitivity and lateral resolution of Focussed Ion Beam (FIB)-based SIMS techniques with the quantitative analysis of the very outer atomic monolayers by LEIS, ion beam techniques can provide much-needed insight into the surfaces of the devices of interest. In this work, we exploit the increased lateral resolution (a few 10s of nm for SIMS, a few microns for LEIS) provided by recent developments in SIMS and LEIS instrumentation to analyse the surface composition of thin-film electrode structures fabricated by Pulsed Laser Deposition (PLD). The combination of these surface characterisation techniques with electrochemical measurements will be a powerful tool in understanding the performance and degradation of solid oxide electrochemical dev

H9 4

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12:45 **Lunch break**

Advances in thermal characterisation of thin films and nanomaterials : Bruno Hay and Petr Klapetek

14:15 **Thermal nanometrology and scanning thermal microscopy**

Authors : S. Gomés, S. Lefévre, P.-O. Chapuis
Affiliations : Université de Lyon, CNRS INSA-Lyon, CETHIL, UMR5008, F- 69621, Villeurbanne, France Université Lyon 1, CETHIL, UMR5008, F-69621 Villeurbanne cedex, France

Resume : Although significant progress has been made for managing heat transfer at small scales, much remains to be understood about heat flow in nanostructures. At the extreme length scales under consideration, the macroscopic physical laws and models fail. The required investigations will not be possible without the development of new thermal measurement techniques since classical thermal metrology methods are limited in resolutions. Various methods with high spatial and temporal resolutions have been developed in the last twenty years. The more confirmed ones are based on optical or photothermal techniques. While lateral spatial resolution of these optical techniques is limited by diffraction, Scanning Thermal Microscopy (SThM) is

H10 1

promising since its spatial resolution depends on the characteristic length of the predominant physical mechanism operating at the nanometric contact between the probe and the sample. After a review of the possibilities of the techniques proposed by various groups, an overview of the results achieved by our group since more than 10 years will be presented. The discussion will mainly focus on application of scanning thermal microscopy for the characterization of thermophysical properties of nanostructured materials from various application areas such as microelectronics and polymer sciences.This will highlight the challenges to be tackled to succeed in realizing Nanoscale Quantitative Thermal Analyses and Thermal Measurements by SThM.

add to my program **(close full abstract)**

14:45 **Microsecond-pulse heating nanocalorimetry: quasi-static method**

Authors : A.F. Lopeandia#&; M. Molina-Ruiz#; G.García#; O.Bourgeouis&; J.Rodriguez-Viejo#;
Affiliations : # Departament de Física, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain. & Institut NEEL CNRS/UJF, 38042 Grenoble cedex 9, France

H10 2

Resume : The use of membrane-based chip calorimeters has opened the way of studying size dependence of thermodynamic properties in nanomaterials. Among the different calorimetric methods implemented for chip, quasi-adiabatic nanocalorimetry [1] reports the better sensitivity per unit area, but do not offers the possibility of measuring heat capacity at constant temperatures as function other variables (time, magnetic field…) like ac-calorimetry [2]. We present a new operational method combining the better characteristics of both methods previously mentioned. In this method, the calorimetric cell, consisting of a silicon nitride membrane (~180nm thick) and a thin film metallic sensor, is heated by joule effect with train of current pulses (few us width, ms separated) promoting local temperature scans that span few K over the base temperature. The possibility of multiple scan averaging and the huge heating rates accessible (up to 10^6 K/s) permits to reach exceptional heat capacity resolution of 100 pJ/mm•K•√Hz. The method is demonstrated characterizing the antiferromagnetic transition in CoO thin film samples of 5 and 10 nm thick. [1] S.L.Lai et al., Appl.Phys.Lett. 67 (9), p1229 (1995) [2] P.F. Sullivan et al., Phys. Rev., 173 (3), p679 (1968)

add to my program **(close full abstract)**

15:00 **A novel high resolution contactless technique for thermal field mapping and thermal conductivity determination: Two-Laser Raman Thermometry**

Authors : J. S. Reparaz, E. Chavez-Angel, M. R. Wagner, B. Graczykowski, J. Gomis-Bresco, F. Alzina, and C. M. Sotomayor Torres
Affiliations : J. S. Reparaz^{1}; E. Chavez-Angel^{1,2}; M. R. Wagner^{1}; B. Graczykowski^{1}; J. Gomis-Bresco^{1}; F. Alzina^{1}; and C. M. Sotomayor Torres^{1,3} ^{1} - ICN2 - Institut Catala de Nanociencia i Nanotecnologia, Campus UAB, 08193 Bellaterra (Barcelona), Spain ^{2} - Dept. of Physics, UAB, 08193 Bellaterra (Barcelona), Spain ^{3} - ICREA, Passeig Llu ś Companys 23, 08010 Barcelona, Spain

H10 3

Resume : We present a novel high resolution contactless technique for thermal conductivity determination and thermal field mapping based on creating a thermal distribution of phonons using a heating laser, while a second laser probes the local temperature through the spectral position of a Raman active mode. The spatial resolution can be as small as 300 nm, whereas its temperature accuracy is ±2 K. We validate this technique investigating the thermal properties of three free-standing single crystalline Si membranes with thickness of 250, 1000, and 2000 nm. We show that for 2-dimensional materials such as free-standing membranes or thin films, and for small temperature gradients, the thermal field decays as $T(r) \propto \ln(r)$ in the diffusive limit. The case of large temperature gradients within the membranes leads to an exponential decay of the thermal field, $T \propto \exp[-A \cdot \ln(r)]$. The results demonstrate the full potential of this new contactless method for quantitative determination of thermal properties. The range of materials to which this method is applicable reaches far beyond the here demonstrated case of Si, as the only requirement is the presence of a Raman active mode.

add to my program **(close full abstract)**

15:15 **Measurements of thermal conductivity of phase-change chalcogenide thin films at high temperature by modulated photothermal radiometry**

Authors : Nolwenn Fleurence, Bruno Hay, Guillaume Davée, Andréa Cappella
Affiliations : Laboratoire national de métrologie et d'essais

H10 4

Resume : Industrials are constantly innovating in the field of thin films due to the continued drive towards miniaturization. As an example, to replace old floating gate technology producers are working on a new generation of non-volatile memory based on phase change materials, typically chalcogenide

materials. The phase change temperatures (amorphous and crystalline phases) of these type of alloys may reach several hundred degrees. These specific operating environments (nanoscale and high temperature) require accurate knowledge of the thermal characteristics of the materials under conditions of use. The understanding of the thermal behaviour of thin films is essential to improve for example the performances of these new generations of phase change memories (PCMs), by increasing storage density, access speed and reliability. This paper presents the thermal conductivity measurements of phase change chalcogenide thin films versus temperature and crystalline structure. These measurements were performed by using a modulated photothermal radiometry apparatus developed at LNE for accurate knowledge of thermal properties of thin films under their specific operating conditions. At the same time, the thermal resistance at the thin film / substrate interface was also evaluated. This work was funded through the European Metrology Research Programme (EMRP) Project IND07 Thin Films. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

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15:30 **Coffee break**

Latest Progress of Thin Film Characterization : Emmanuel Nolot, Uwe Beck and Carla Vogt

16:00 **Polarization dependent measurements of nanostructured surfaces**

Authors : Poul-Erik Hansen and Morten Hannibal Madsen
Affiliations : DFM A/S, Danish National Metrology Institute, Matematiktorvet 307, 2800 Kgs. Lyngby, Denmark

Resume : Nano and micro-structured surfaces can be characterized with a Muller Polarimeter, that analyzes the polarization parameters of light. Information about structural shape, i.e. height and width, and material composition can be obtained from a single measurement. Furthermore, the measurements are self-normalized, making calibration of the instrument much easier. The additional information gathered by measuring the complete polarization state of the diffracted or scattered light is quantitatively and qualitatively a substantial extension of the information gathered with conventional scatterometry commonly used for measuring structures. The measurements capability of scatterometry and Mueller Polarimetry are investigated with respect to pitch of the surface structure, the orientation of the surface structure and the feature size of the structure. The series of grating structures has a depth range of 40 nm to 8000 nm and a pitch range from 200 nm to 20000 nm. It is shown that the measurement of the full polarization matrix with the Mueller Polarimeter make it much more sensitive to periodic structure with a small pitch than scatterometry. The sensitivity of the Mueller matrix elements to anisotropy make it possible to determine the orientation of hidden structures such as grating that cannot be observed by eye and to observe process dependent strain/stress induced in an isotropic sample during fabrication.

H11 1

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16:30 **Epitaxial ferromagnetic MnSb on GaAs, InGaAs and Ge substrates: polymorphism and unconventional early stage growth**

Authors : C. W. Burrows, T. P. A. Hase, J. Aldous, S. Hatfield, M. J. Ashwin, G. R. Bell
Affiliations : University of Warwick

Resume : Half-metallic ferromagnets (HMFs) have huge potential for semiconductor spintronics as they exhibit near 100 % spin polarisation at the Fermi level. If they can be stabilised in thin film form they could transform the spin injection/detection efficiency within heterostructures. High quality epitaxial systems are then a prerequisite for such devices. The cubic polymorph of MnSb is a possible candidate for HMF electrodes at room temperature. MnSb is usually hexagonal, but TEM studies of our epitaxial thin films reveal the presence of the cubic polymorph although their structural origin remains unknown. Understanding how the different polymorphs are stabilised is crucial if MnSb is to be a usable HMF. In this study, we explore the structural precursors affecting polymorph content in epitaxial MnSb layers. We present an analytic structural study on films grown as a function of the Sb:Mn flux ratio by molecular beam epitaxy (MBE) on GaAs, Ge and InGaAs substrates. By exploiting symmetric and asymmetric triple axis x-ray diffraction, in conjunction with depth-dependent in-

H11 2

plane scattering, the strain of each polymorph has been measured as a function of layer thickness. The early stage pseudomorphic growth of MnSb on GaAs does not behave like conventional III-V high-strain epitaxy and the observed strain relaxation is remarkably independent of epitaxial mismatch over the range 0.3 % (InGaAs) to 3 % (GaAs, Ge). Epitaxial mismatch appears to have no effect on the amount of polymorph.

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16:45 **Direct evidence of Tungsten clustering in W0.02V0.98O2 thin films and its effect on the metal-to-insulator transition**

Authors : Xiaoyan Li 1,2, Alexandre Gloter 2, Alberto Zobelli 2, Hui Gu 1, Xun Cao 1, Ping Jin 1, Christian Colliex 2
Affiliations : 1 State Key Laboratory of High Performance Ceramics and Superfine Microstructures, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, China ; 2 Laboratoire de Physique des Solides, CNRS UMR 8502, Université Paris Sud 11, Orsay 91405, France

Resume : Substitutional tungsten doping of VO2 thin films and its effect on the metal-to-insulator transition are investigated by means of X-ray diffraction, Cs-corrected scanning transmission electron microscope (STEM), and ab-initio simulations. The W0.02V0.98O2 thin films deposited on (001) sapphire are studied in both planar and transverse geometries. Tungsten atoms are distinguishable from the V atoms in the Z-sensitive high angle annular dark field (HAADF) STEM image and their nature is confirmed by electron energy loss spectroscopy (EELS). The W dopants are found to substitute in the V sites and form local clusters. Ab-initio modeling for this 2 at.% W doped VO2 confirm the experimentally found W clustering mechanism to be the most stable substitutional configuration and they demonstrate that the binding energy of such cluster is 0.18 eV. Simulations indicate that the clustering helps in stabilizing the tetragonal structure, while a diluted W dopant induces more structural distortion and V-V pairing. This suggests that the clustering mechanism plays a critical role in the transition temperature evolution with the W dopants.

H11 3

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17:00 **3D determination of facets of nanoparticles by transimission electron microscopy**

Authors : Zhanbing He
Affiliations : State Key Laboratory for Advanced Metals and Materials University of Science and Technology Beijing No. 30 Xueyuan Road, Haidian District Beijing 100083, China

Resume : The physical/chemical properties, especially catalytic properties of nanoparticles are closely related with their enclosed facets and inspecting facet indexes of nanoparticles has attracted more and more attention. However, the experimental determination of the growth facets of nanoparticle catalysts in three dimentional (3D) space is very limited. Herein we developed a versatile method to determine the 3D growth facets of nanoparticle catalyst for the growth of carbon nanotubes (CNTs). By combining electron diffraction patterns, images, and electron tomography from the same nanoparticle, the indexes of the leading edges of the faceted Fe/Ni nanoparticle are well determined. This method can also be used to determine the facets in a 3D space of any faceted materials, even those with twin defects.

H11 4

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17:15 **Real time observation of metal oxide nanowire transformation through cationic exchange for RRAM application**

Authors : Chun-Wei Huang1, Jui-Yuan Chen1, Chung-Hua Chiu1,Cheng-Lun Hsin2,Wen-Wei Wu1
Affiliations : 1 Department of Materials Science and Engineering, National Chiao Tung University, No. 1001, University Rd, East Dist., Hsinchu City, 300, Taiwan; 2 Department of Electrical Engineering, National Central University, Taoyuan, 320, Taiwan;

Resume : One dimensional metal oxide nanostructures have attracted much attention owing to their fascinating functional properties. Among them, piezoelectricity and photocatalyst along with their related materials have stirred significant interests and widespread studies in recent years. In this work, we successfully transformed piezoelectric ZnO into photocatalytic TiO2 and formed TiO2/ZnO axial heterostructure nanowire with flat interface by solid state cationic exchange reactions in high vacuum transmission electron microscope (TEM). Kinetic behavior of the single crystalline TiO2 was systematic analyzed. The nanoscale growth rate of TiO2 has been measured using in-situ TEM videos. On the basis of the rate, we can control the dimensions of the axial-nanoheterostructure. In addition, the unique Pt/TiO2/ZnO/TiO2/Pt

H11 5

heterostructures with complementary resistive switching (CRS) characteristic were designed to solve the important issue of sneak-peak current. The resistive switching behavior was attributed to the migration of oxygen and TiO2 layer served as reservoir, which was confirm by EDS analysis. This study not only supplied a distinct method to explore the transformation mechanisms, but also exhibited the potential application of ZnO/TiO2 heterostructure in nano-scale cross-bar array resistive random access memory.

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17:30 **Properties of Pt/Pb(Zr0.5Ti0.5)O3 interfaces** H11 6

Authors : Anna V. Kimmel†,‡, Markys G. Cain‡
Affiliations : ‡ National Physical Laboratory, Teddington, TW11 0LW, UK. † University College London, Gower Street, London, UK WC1E 6BT, UK.

Resume : Capacitors based on nanoscale ferroelectric perovskites found a large variety of applications in nano-electronics. Use of nano-geometries increases the efficiency of nanodevices, however, brings certain disadvantages such as size effects, rapid polarization relaxation, interfacial capacitances. An insight into interfacial capacitance and its dependence on the combination of ferroelectric and electrode materials is important for device design. In this work we study structural and electronic properties of the interface between Pt (100) and PZT (001) using density functional theory. We performed an analysis of relaxed structure, electronic properties with respect to thickness of PZT. Considering two types of interfaces related to different termination of PZT (PbO and TiZrO2) we found a remarkable asymmetry of interfacial properties in terms of local chemical and electrostatic environment.

add to my program **(close full abstract)**

17:45 **Local electronic structures and the polarity dependent measurement of ZnO nanorods growth on GaN substrate** H11 7

Authors : Yen-Shuo Liu, Yen-Ju Wu, Cheng-Yi Liu
Affiliations : Department of Chemical and Materials Engineering National Central University, Jhongli, Taiwan

Resume : In this study, the ZnO nanorods (NRs) were prepared and grown on the GaN substrates by hydrothermal method to investigate the correlation of polarity effect. It showed that the feature of X-ray absorption spectroscopy (XAS) and the trend of the scanning electron microscope (SEM) of the ZnO NRs on the surface of p-GaN and n-GaN substrates are opposite. It may due to that the inverse polar-dominated of growing the ZnO NRs on the surface of the p-GaN and the n-GaN substrates. We also demonstrated that the growth and dissolution of nanostructure of the ZnO is correlated to the polarity of the ZnO c -plane surface, surface termination, and surface activity. The relationship between the polar of the GaN substrates and the growth trends of the ZnO implies the morphologies and the local electronic structures of the deposited ZnO films can be modified by controlling their growth and dissolution processes.

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18:00 **The effect of mutual order of B-cations on electronic and optical properties of strained Pb(ZrxTi1-x)O3** H11 8

Authors : Alex Bogdanov1,2, Anna V. Kimmel3
Affiliations : 1 A.P. Vinogradov Institute of Geochemistry SB RAS, Irkutsk, Russia; 2 Irkutsk State Technical University, Irkutsk, Russia; 3 National Physical Laboratory, UK

Resume : Pb(ZrxTi1-x)O3 x=52% is widely used material for industrial applications due to its exceptional piezo- and ferroelectric properties. We use density functional theory to provide an insight into the effect of mutual arrangement of Ti and Zr cations on structural, electronic, optical and ferroelectric properties of PZT. We have studied the response of electronic structure, symmetry of d-functions and optical properties to epitaxial strain. We found that optical band gap and the shape of optical absorption spectra change dramatically under stress.

add to my program **(close full abstract)**

18:15 **Temperature and frequency dependence of electric field induced phase transitions in PMN-xPT** H11 9

Authors : J. Wooldridge, M. Stewart, C. Vecchini, M. G. Cain, M. Gutmann, M. Reece
Affiliations : National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK;1National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK;1National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK;1National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK;ISIS Facility, Rutherford Appleton Laboratory, Didcot, Oxon OX11 0QX, UK;Materials Department, Queen Mary, University of London, Mile End Road, London E1 4NS, UK

Resume : Single crystal (1 − x)Pb(Mg1/3Nb2/3)O3-xPbTiO3 [PMN-xPT] is a relaxor-ferroelectric material known to exhibit 'giant' piezoelectric behaviour, with achievable strains in excess of 1% for samples of certain particular crystallographic orientations and chemical compositions close to the morphotropic phase boundary (MPB). In this work, we investigate the electric field-induced structural phase transitions in single crystal PMN-xPT with time-of-flight neutron diffraction and macroscopic electrical polarisation measurements. We show that the critical fields at which the electric field-induced phase transitions occur are strongly dependent on the frequency of the applied field, and are hysteretic in both electric field and temperature. We demonstrate that not only the previous E field loading conditions affect the state of the material, but so does the thermal history and the previous frequency cycling of the E field.

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Poster session 2 - Analytical Techniques for Nanomaterials : Claudia Fleischmann, Bernd Kolbesen, Petr Klapetek, Peter Petrik, Luca Boarino and Omar El Gawhary

18:30 **Reliable tip-enhanced Raman imaging of nanomaterials** HP12 1

Authors : Emmanuel Leroy, Renata Lewandowska, Ophelie Lancry, Andrey Krayev, Sergey Saunin
Affiliations : HORIBA Scientific, HORIBA Scientific, HORIBA Scientific, AIST-NT, AIST-NT

Resume : Tip Enhanced Raman has long been researched, yet to this day most publications are spectral data acquired in single points. This is due to the challenge of combination of slow scanning speeds of Raman Spectroscopy with nanometer scale resolution. In this presentation we will show how TERS can be performed at high speed, greatly reducing drift and noise issues in the process of imaging at the nanoscale, and how reliable probes are now available to perform routine imaging of carbon based nano-materials like Graphene and Carbon Nanotubes. We will introduce the latest technological developments for signal collection with high throughput, and high speed data acquisition that make TERS imaging now accessible with great ease-of-use.

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18:30 **Effect of Cr doping on the structural, magnetic and magnetocaloric effect of La0.7Ca0.2Sr0.1 Mn1-xCrxO3 polycrystalline** HP12 2

Authors : Ah.Dhahri. E.Dhahri and E. K. Hlil
Affiliations : Ah Dhahri, E Dhahri : Applied Physics Lab, Faculty of Sciences Sfax, BP 1171, University of Sfax, 3000, Tunisia. E K Hlil : Institut Néel, CNRS, MCBT Department, BP 166, 38042 Grenoble Cedex 9, France

Resume : We have studied the effect of Cr substitution on structural, magnetic and magnetocaloric properties in La0.7Sr0.16Ca0.17Mn1-xCrxO3 (x = 0, 0.05, 0.1, 0.15 and 0.2) manganite. The X-ray diffraction studies show that all samples crystallize with the orthorhombic symmetry within the space group Pnma (No 62). Rietveld refinement shows that the chromium modify the structural parameters such as the volume, the Mn-O-Mn angles and the Mn-O bond length. All samples undergo a transition from paramagnetic(PM) to ferromagnetic(FM) phase at the Curie temperature, Tc which decreases from 295K down to 225K with increase in the Cr doping level from x= 0 to x= 0.2. Around TC, the magnetic entropy change (- Sm) was estimated from isothermal magnetization curves and it decreases with increase of Cr content from 6.20J.kg -1.K-1 at 295K(x =0) to 2.45 J.kg-1.K-1 at 225K(x=0.2) under µ0H=5T and the relative cooling power (RCP) approached 240 and 178.115 J.kg-1for Cr doped materials in the magnetic change of 5T. The obtained results suggest that Mn-site Cr dopant actually disfavors the enhancement of the magnetocaloric effect in some perovskite manganites due to a weakening of the ferromagnetic double-exchange interaction between Mn3+ and Mn4+ ions of the original atomic structure.

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18:30 **Ultra fast depth profile analysis of nanostructured materials by plasma profiling Time of Flight Mass Spectrometry** HP12 3

Authors : Agnès Tempez,Emmanuel Nolot,Sébastien Legendre,Jean-Paul Barnes,Fabrice Nemouchi, Cécile Maunoury
Affiliations : Horiba Jobin Yvon, CEA LETI

Resume : Plasma Profiling Time of Flight Mass Spectrometry (PP-TOFMS) provides direct measurement of the elemental composition of materials as a function of depth, with nanometre resolution and the capability to measure both thin and thick layers [1]. It consists in a pulsed radio frequency glow discharge

plasma source fed with pure Ar and created under a pulsed RF potential coupled to a time of flight mass spectrometer (TOFMS). The ultra-fast detection and quasi-simultaneous acquisition of all mass ions of the TOFMS fits well with the fast erosion rate of the high density and low energy plasma source. Furthermore the separation between sputtering and ionisation processes makes this technique much less matrix dependent compared to SIMS. In addition, the orthogonal TOFMS configuration allows for temporal monitoring of the transient signals generated in the pulsed plasma. This is all the more important as signals are largely enhanced in the plasma extinction phase (once RF is turned off) in the so-called afterglow region. Ion signals are then generated through Penning Ionisation by Ar metastables. Various examples in microelectronics and nanotechnology will be presented such as magnetic layers for 3D sensors, Pt-doped Ni-silicides for advanced contacts, and TiN layers for CMOS. It will be shown that PP-TOFMS allows for determining composition, detecting contamination, measuring doping level, and characterising diffusion mechanisms. Results will be compared to other techniques (XRF, SIMS, and XRR) and aspects of analytical performance with regards to sensitivity, quantification, repeatability and sample throughput will be discussed. [1] R. Valledor et al, Analytical and Bioanalytical Chemistry, 396, 2881-2887 (2010).

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18:30

High accuracy elemental composition in 3D by MeV ion beam analysis (IBA)

Authors : J.L.Colaux, C.Jeynes
Affiliations : University of Surrey Ion Beam Centre, Guildford, England

Resume : MeV IBA uses light ion scanning microbeams to non-destructively probe materials in 3D down to 10 microns depth and more, exciting both (atomic) particle-induced X-ray emission (PIXE) and (nuclear) elastic backscattering (EBS) a special case of which is Rutherford backscattering spectrometry (RBS). The high depth resolution of IBA comes from the inelastic energy loss processes that dominate nuclear spectrometry and are responsible for PIXE. Advances recently reviewed [Nucl. Instr. Methods B, 271 (2012) 107] have allowed spectra from both atomic and nuclear excitations to be handled self-consistently, meaning that X-ray data from layered materials whose layer structure is unknown can be interpreted fully quantitatively. PIXE and EBS are always both available, and are strictly complementary: where one is strong the other is weak (PIXE: mass and trace sensitivity; EBS: accuracy and depth). Fully quantitative analysis of completely blind samples is now feasible [Nature Geoscience 6, (2013) 1018]. Recently, we have demonstrated a class of samples for which the quantity of material can be determined by RBS with a traceable absolute accuracy of 1% [Anal.Chem.84 (2012) 6061; Anal.Meth.6 (2014) 120], an accuracy unprecedented for standard-less and model-free non-destructive analysis of thin film samples. We will discuss the applicability of these methods for wider classes of samples, and also discuss how PIXE inherits the accuracy of backscattering spectrometry.

HP12
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18:30

Combined SIMS-SPM instrument for high sensitivity and high resolution elemental 3D analysis

Authors : Y. Fleming, T. Wirtz
Affiliations : Department “Science and Analysis of Materials” (SAM), Centre de Recherche Public – Gabriel Lippmann, 41 rue du Brill, L-4422 Belvaux, Luxembourg

Resume : Owing to its excellent sensitivity, its high dynamic range and its good depth resolution, Secondary Ion Mass Spectrometry (SIMS) constitutes an extremely powerful technique for analysing surfaces and thin films. In recent years, considerable efforts have been spent to further improve the spatial resolution of SIMS instruments. As a consequence, new fields of application for SIMS, e.g. nanotechnologies, biology and medicine in particular, are emerging. State-of-the-art SIMS instruments allow producing 3D chemical mappings with excellent sensitivity and spatial resolution. However, several important artefacts and limitations arise from the fact that the 3D mappings do not take into account the sample’s surface topography. Therefore, we developed an integrated SIMS-SPM instrument, which is based on the Cameca NanoSIMS 50. This instrument, an in-situ combination of sequential high resolution Scanning Probe Microscopy (SPM) and high sensitivity SIMS, allows topographical images of the sample surface to be recorded in-situ before, in between and after SIMS analysis. Hence, high-sensitivity high-resolution chemical 3D reconstructions of samples are possible with this extremely powerful analytical tool. In addition, this integrated instrument allows a combination of SIMS images with valuable KPFM (Kelvin Probe Force Microscopy) data recorded in-situ in order to provide

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5

an extended picture of the sample under study, enabling new multi-channel nanoanalytical experiments.

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18:30

Towards SIMS on the Helium Ion Microscope: detection limits and experimental results on the ORION

Authors : D. Dowsett, P. Philipp, T. Wirtz, S. Sijbrandij, J. Notte
Affiliations : CRP - Gabriel Lippmann, 41 rue du Brill, L-4422 Belvaux, Luxembourg; CRP - Gabriel Lippmann, 41 rue du Brill, L-4422 Belvaux, Luxembourg;CRP - Gabriel Lippmann, 41 rue du Brill, L-4422 Belvaux, Luxembourg; Carl Zeiss NTS LLC, One Corporation Way, Peabody, MA, 01960; Carl Zeiss NTS LLC, One Corporation Way, Peabody, MA, 01960

Resume : The ORION Helium Ion Microscope (HIM) has become a well-established tool for high resolution microscopy and nanofabrication. To add capabilities for elemental analysis, we have investigated the feasibility of implementing secondary ion mass spectrometry (SIMS) on the HIM by adding a secondary ion extraction system and by focussing on detection limits and achievable lateral resolution. Secondary ion yields under helium and neon bombardment have been determined for a range of metal and semiconductor materials. Under pure helium and neon bombardment, secondary ion yields are low, but they can be enhanced by several orders of magnitude by oxygen and caesium flooding for positive and negative secondary ions, respectively. This optimisation of secondary ion yields leads to detection limits varying from 1E-3 to 1E-6 for a lateral resolution of 10 nm and 100 nm, respectively. The installation of the secondary ion extraction system on an ORION has proven that secondary ion imaging with helium and neon bombardment is possible. The obtained results are very encouraging and the prospects of performing SIMS on the ORION are very interesting. In this paper we will present an overview of our results to date and first experimental results of secondary ion detection on the HIM.

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18:30

A systematic study of Ga+ implantation in a PZT film during Focused Ion Beam micro-machining

Authors : Nicole Wollschläger, Werner Österle, and Mark Stewart
Affiliations : Nicole Wollschläger, Werner Österle: BAM Federal Institute for Materials Research and Testing, Berlin, Germany; Mark Stewart: NPL National Physical Laboratory, Teddington, UK

Resume : Piezoelectronic transistors are a candidate for the next generation of microprocessors. Here a piezoelectric material, with features less than 11nm, generates a strain in a piezoresistive material to form the switch. Therefore it is important to measure and control strain on the nanometer scale. One of the methods applied within the EMRP project "Nanostrain" is cutting out extremely small pieces of material from critical sites with a focused ion beam [FIB] and determining strain maps with a special technique called dark field electron holography [DFEH]. The major problem with this approach is that FIB machining induces defects at the specimen surface which may impair the results of strain measurements. The objective of the present work was to study the impact of FIB machining parameters on the thickness of the damaged layer. Therefore, different ion doses and ion energies were applied to a standard PZT film (80/20 lead zirconium titanate) under two beam incidence angles (90° and 1°). The thicknesses of the corresponding Ga+-implanted layers were then determined by cross-sectional TEM in combination with EDS line-scans and correlated with polarization hysteresis loops. The results show a decrease of Ga+-implanted layer thickness with decreasing inclination angle, ion energy and ion dose. Whereas under the most unfavourable conditions the depth of the affected zone was 26 nm, it was only 2 nm for the most favourable conditions.

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18:30

Reliable Raman Spectrometry

Authors : S. Zakel, S. Wundrack, C. Frank, P. Hinze, T. Weimann, T. Dziomba, B. Güttler, R. Stosch
Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany

Resume : Collaborative research and development is carried out at PTB to cover the metrological needs of Raman spectrometry. Current results achieving 2D/3D dimensional traceability of spatial mappings and depth-profiling as well as for amount-of substance measurements are presented. To underpin industrial and bio-medical applications of Raman spectrometry, this work is funded through the European Metrology Research Programme. While traceability of the chemical composition of a sample is achieved via high purity reference compounds, reference samples to provide traceability of the area distribution

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are of qualified acceptance. PTB has designed and fabricated a new reference sample that allows step size calibration and determination of the optical resolution. Measurement and calibration protocols are provided. The link to the meter as the SI unit of length was accomplished by a metrological AFM. Traceability of amount-of-substance measurements to the mole has been achieved at PTB by the adaption of the isotope-dilution (ID) technique. The method has been validated by successful participation in two international ring trials on clinical chemistry. The IDSERS approach was recently accepted as a higher order reference procedure for the determination of biomarkers in human serum and admitted to a publicly accessible database maintained by the International Bureau of Weights and Measures.

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18:30 **Characterization and simulation of optical absorption in Si nanocrystals**

Authors : Xuguang Jia, Lingfeng Wu, Ziyun Lin, Tian Zhang, Terry Yang, Binesh Puthen-Veettil,Ivan Perez-Wurfl,Gavin Conibeer

Affiliations : School of Photovoltaic and Renewable Energy Engineering, University of New South Wales, Sydney

Resume : The application of silicon quantum dot (Si QDs) based materials is regarded as a promising approach for the realization of high efficiency solar cells. When silicon nanocrystals are made very small, they behave as quantum dots due to the three-dimensional quantum confinement, which could cause the material’s effective optical band gap to increase. The optical band gap can be deduced from the absorption coefficient. In this paper, we analyze optical absorption and emission processes in Si QDs with a size distribution and attempt to simulate the band-edge absorption features based on the photoluminescence spectrum. We also investigate the application of ellipsometry in the study of optical properties of Si QDs thin film. Based on WVASE32 modeling tool, several models are developed to extract the absorption coefficient of these materials. From these results, we extract the effective optical band gap and analyze optical properties of Si QDs materials.

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18:30 **Ellipsometric measurements on monolayers of nanoparticles and other model systems for nanostructured optoelectronic systems**

Authors : Andreas Hertwig, Dana Rosu, Uwe Beck, Luca Croin, Giulia Aprile, Luca Boarino

Affiliations : BAM - Federal Institute for Materials Research, Germany; BAM - Federal Institute for Materials Research, Germany; BAM - Federal Institute for Materials Research, Germany; INRIM - Istituto Nazionale di Ricerca Metrologica, Italy; INRIM - Istituto Nazionale di Ricerca Metrologica, Italy; INRIM - Istituto Nazionale di Ricerca Metrologica, Italy

Resume : Ellipsometry is a valuable non-destructive tool for measuring the properties of thin films in optoelectronic systems. Apart from simple stratified transparent layer systems, micro and nanocomposite systems have been investigated in recent years following important improvements in ellipsometric analysis algorithms, layer models, and software tools. In this work, a study on polystyrene nanospheres and other nanoscale structures distributed on a silicon surface is presented. These systems are intended to be models in a strategy to develop analysis methods for composite organic materials. These materials are widely used in optoelectronic applications such as OLEDs and bulk organic photovoltaic systems. A combination of microscopic techniques and ellipsometry over a wide spectral range was used in this work to gain wide insight into the optical properties of the samples. The possible advantages as well as the limits of the ellipsometric measurement technique are discussed in this presentation.

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18:30 **Fast and robust characterization of polymers with a nano-textured surface**

Authors : Morten Hannibal Madsen, Poul-Erik Hansen, Maksim Zalkovski, Nikolaj Feidenhans’l, Jørgen Garnæs

Affiliations : DFM A/S, Danish National Metrology Institute, Matematiktorvet 307, 2800 Kgs. Lyngby, Denmark; DFM A/S, Danish National Metrology Institute, Matematiktorvet 307, 2800 Kgs. Lyngby, Denmark; NIL Technology ApS, Diplomvej 381, 2800 Lyngby, Denmark; DFM A/S, Danish National Metrology Institute, Matematiktorvet 307, 2800 Kgs. Lyngby, Denmark; DFM A/S, Danish National Metrology Institute, Matematiktorvet 307, 2800 Kgs. Lyngby, Denmark

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Resume : The market for industrial fabrication of micro- and nano-textured surfaces is currently evolving fast. This includes microchannel systems for cell analysis and nano-textured plastic surfaces for glimmering color effects. However, the production technologies are currently evolving much faster than measurement methods for characterization of such surfaces. We have

demonstrated that by adapting a commercial microscope to detect scattered light, we can obtain fast and robust optical measurements with a resolution in the nanometer range. With easy change of objective one can focus the beam to a small area (<100 µm), and with the possibility to use the instrument as a normal light microscope, finding specific areas are made much easier. Analyses of the recorded spectra are solved using an inverse modeling approach. Both rigorous coupled-wave analysis (RCWA) and finite element method (FEM) computer simulations are used for generating a library of nano-textured surfaces. The simulations require a priori information of the sample, but once generated, it is a very fast method for characterization. The microscope's measuring capability is compared to a metrology atomic force microscope using both high quality calibration grids and lower quality injection molded plastic samples (e.g. ABS). It is furthermore demonstrated how such an optical setup can be used outside a research environment, for instance at a production facility.

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18:30 **Combined XPS- and XRF-surface analysis in one instrument** HP12 12

Authors : Erik Darlatt, Michael Kolbe, Rolf Fliegauf, Philipp Hönicke, Ina Holfelder
Affiliations : all Authors: PTB – Physikalisch-Technische Bundesanstalt, Abbestraße 2 – 12, 10587 Berlin, Germany

Resume : Complex materials with defined surface functionalities need an analysis which goes beyond the possibilities of one single method. Thus the combination of complementary methods enables investigations of the complex correlations between materials composition and their properties. For that purpose PTB designs and constructs an experimental setup, which allows for the simultaneous investigation by X-ray photoelectron spectroscopy (XPS) and X-ray fluorescence spectroscopy (XRF) using laboratory X-ray sources as well as synchrotron radiation. Using PTB's calibrated instrumentation it is aimed for the investigation of metrological aspects like absolute mass deposition of various elements as well as their chemical speciation of functional material surfaces. One major task of the new instrumentation will be the characterization of the surface of a 94 mm diameter monocrystalline silicon sphere, which is basis for the new definition of the SI-base unit of the mass: the kilogram. The exact definition of Avogadro's number enables the redefinition of the kilogram which is achieved by "counting" Si atoms of the one kilogram Si sphere. We will present an overview of the scheduled setup of this instrumentation. It will be shown how the handling of the Si sphere in the UHV chamber is achieved. Further we will exhibit XPS and XRF test measurements of Si reference samples recorded by other instrumentations to demonstrate which information can be obtained by the combination of both methodologies.

add to my program **(close full abstract)**

18:30 **Applying Gold nanoparticle decorating silicon nanostructure for high sensitivity Surface Enhanced Raman Scattering substrate** HP12 13

Authors : Bi-Shen Lee, Ding-Zheng Lin, Ta-Jen Yen
Affiliations : National Tsing Hua University, Guangfu Rd.Sec.2 No. 101,Hsinchu, Taiwan

Resume : In this work, we provided a three-dimensional (3D) hybrid silicon-gold nanowires as a SERS substrate. Firstly, we utilized the statistical electroless metallic deposition (SEMD) method to fabricate silicon nanowires. Then, the glancing angle deposition was utilized to deposit gold nanoparticles (NPs) and thus gold NPs were successfully decorated on the sidewall of the silicon nanostructure. Therefore, our substrate possessed many small gaps between the gold NPs on the sidewalls of the silicon nanowires and generated a high density of the hot spots within a detection volume. As a result, leading to a strong local field. Here, we chose a commercialized substrate, Klarite, as a control group. After Raman measurement by the 785 nm He-Ne laser, the Raman signal of the thiophenol, a standard RS target, showed that our SERS substrate with Raman signal 2 orders larger than Klarite. Furthermore, the standard error deviation of our SERS substrate is less than 10%, revealing the good uniformity of our SERS substrate. To the best of our knowledge, this SERS substrate offers lots of benefits including the higher hot spot density, the signal uniformity, and the low-cost large area nanofabrication process compared to others SERS substrates reported up to now, suggesting our SERS substrate is very competitive in practical application.

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18:30 **Microscopy with nanoscale resolution studied by high-resolution RBS and micro-PIXE** HP12 14

Authors : Lagutin A.

Affiliations : Belarusian State Agrarian Technical University, Physics, Nezavisimosti Av., 220023, Minsk, Belarus

Resume : Biofortification by breeding staples with increased micronutrient content is a sustainable approach towards improving dietary quality. The direct analysis of trace metals in single cells and biological tissues is a challenging task that requires sophisticated analytical developments. The aim of this abstract is to present some recent achievements in the field of chemical element imaging of biological samples using ion beam micro-analysis. High spatial resolution nuclear microprobes (<1 μm) enable the investigation of subcellular chemotypes, i.e. the specific accumulation of trace elements in cellular organelles [1]. Moreover, accurate quantification of trace element distributions can be obtained using three complementary ion beam techniques, PIXE (Particle-Induced X-ray Emission), RBS (Rutherford Backscattering Spectrometry), and STIM (Scanning Transmission Ion Microscopy) which is a strength compared to other micro-analytical methods. The nuclear microprobe will be compared to other micro-analytical techniques such as the synchrotron radiation X-ray fluorescence microprobe. Micronutrient deficiencies affect a large proportion of the world population. Information about the distribution of micronutrients within seeds tissues can assist with the localization of sites of selective accumulation and barriers to the movement of micronutrients, in establishing how these elements are stored within the seed, and how they affect the nutritional status of the growing seedlings [2]. Knowledge of the distribution of micronutrients within mature seeds is limited. This study compares the distribution and concentrations of micro- and macronutrients in different seeds with the aim of optimizing the biofortification, a sustainable approach towards improving dietary quality. Rutherford backscattering spectrometry (RBS) is a versatile tool for analysis of composition and thickness of nm-films [3]. Other ion beam based techniques (e.g. SIMS, PIXE) have been successfully applied also for investigations of organic materials [4]. In the present work the potential of high-resolution RBS for detection of trace elements in an organic compound was tested. The main advantage of using RBS is that relative and absolute concentrations of present elements can be determined with very high accuracy. This study revealed the accumulation sites of micro- and macronutrients in seeds. Two selected examples of PIXE microanalysis in ecophysiology are presented. Studies of heavy metal distributions in seeds showed different filtration mechanisms of Zn/Pb and Fe/Mn, both enabling plants to cope with metals present in the environment. This information is crucial for future studies of the molecular mechanisms responsible for the micronutrients accumulation in seeds. In addition, this work provides fundamental information that will assist the development of biofortified crops and the study of micronutrient bioavailability.
References [1] Lagutin A. Proceedings of RuPAC-2010, abstract THPSC028. [2] Lagutin A., Gorodecka H., HRDP6 2011, abstracts P9, P. 82. [3] Lagutin A.E., XXXV CIOSTA & CIGR V Conference 2013, abstract R4_RHT1_P4. http://ciosta.org [4] Lagutin A., Gorodecka H., Bio-PIXE 7 2011, http://biopixe7.qse.tohoku.ac.jp

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18:30

Study on unique electronic properties of Pt@Ag core@shell nanoparticles via X-ray Photoelectron Spectroscopy

HP12 15

Authors : Anh T.N. Dao, Derrick M. Mott, Shinya Maenosono
Affiliations : School of Materials Science, Japan Advanced Institute of Science and Technology

Resume : Plasmonic-based sensing probes consisting of nanoparticles (NPs) have become highly desirable because of their enhanced sensitivity, low cost, and easy to use nature. Silver is the most common type of metal studied for NP-based sensors because of their strong surface plasmon resonance (SPR) properties and especially intriguing because it has the highest optical cross section for any metal, but still suffers from oxidation and an inability of its plasmonic properties to be tuned for a desired application. Electronic charge transfer effect in multi-metallic system has been found to have ability to modify characteristic of metals such as chemical stability, plasmonic property, etc. In our research, Pt@Ag core@shell NPs with controllable size and shell thickness are synthesized and characterized by UV-Vis, XRD, TEM, HR-TEM, EDS, HAADF-STEM, and Raman spectroscopy. Especially, systematical and precise analysis using X-ray photoelectron spectroscopy reveal that these NPs display unique electronic properties where the silver shell gains electron density from the platinum cores which can enhance stability of Ag site. In addition, the plasmonic properties and unique electronic structure of this system can give us a wide avenue for future catalytic, SERS and other applications.

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18:30 **X-ray focusing and imaging with multilayer Laue lenses**

Authors : A. Kubec1;2, S. Niese3;4, S. Braun1, K. Melzer1;2 , P. Krüger4, J. Patommel5 and A. Leson1
Affiliations : 1 Fraunhofer Institute for Material and Beam Technology, 01277 Dresden, Germany 2 Institute for Materials Science and Max Bergmann Center of Biomaterials, Technische Universität Dresden, 01062 Dresden, Germany 3 Dresden Center for Nanoanalysis, Technische Universität Dresden, 01187 Dresden, Germany 4 Fraunhofer Institute for Ceramic Technologies and Systems IKTS Materials Diagnostics Branch, 01109 Dresden, Germany 5 Institute of Structural Physics, Technische Universität Dresden, 01069 Dresden, Germany

Resume : X-rays offer another way to analyze a sample at high resolutions with a virtually nondestructive approach compared to electron microscopy. X-ray microscopy multilayer Laue lenses (MLL), for example, are a most promising approach to achieve nanometer resolutions in imaging and focusing applications. The manufacturing process involves the deposition of thousands of double layers with a precision in the sub-nanometer scale, whereas the total layer thickness reaches even more than 50 µm, which is a difficult task to solve. From this deposition a bar is cut and by using focused ion beam milling, a lamella with a thickness of only several µm is prepared. Using two of these lamellas arranged perpendicularly generates a full-fledged point focusing optical device. It can be used for focusing experiments with collimated light or for full-field imaging applications using a condensed beam. We have designed and manufactured pairs of crossed MLLs made of the combinations Si and WSi_2 or $MoSi_2$. These materials have proved to show good deposition characteristics and optical properties. MLLs with a focal length of 20 mm and 12.5 mm at 20 keV x-ray energy have been manufactured and used for synchrotron measurements. We have shown focal sizes of less than 50 nm determined using the technique of Ptychography [1] and first experiments with wedged MLLs have shown an increased efficiency. At an Xradia NanoXCT 100, full field imaging, using MLLs, was shown for the very first time.

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18:30 **Focusing of soft X-ray radiation with a single bounce monocapillary enabling X-ray emission spectrometry of nanoscaled materials**

Authors : R. Unterumsberger*1, M. Müller1 and B. Beckhoff1
Affiliations : 1 Physikalisch-Technische Bundesanstalt, Abbestrasse 2-12, 10587 Berlin, Germany

Resume : The chemical speciation of buried nanolayers is an important part of the material analysis. Here, this was achieved by efficient soft X-ray Emission Spectrometry (XES). An increased sensitivity was achieved by focusing monochromatized soft X-ray undulator radiation down to the micrometer range using a high quality single bounce monocapillary [1]. For validation purposes, the beam profile has been well characterized by two complementary methods, the knife-edge method and the so-called wire method. The effective focusing highly increases the sensitivity of a Wavelength Dispersive Spectrometer (WDS) [2] allowing for the detection and analysis of nanoscaled materials by XES. The lower limits of detection could be reduced below 1 nm for titanium L alpha- and boron K alpha-fluorescence radiation. Due to the increased sensitivity of the WDS, the chemical speciation of different nanoscaled titanium compounds was achieved. The measurements were carried out at the plane-grating monochromator beamline in the laboratory of the Physikalisch-Technische Bundesanstalt at the synchrotron radiation facility BESSY II using monochromatized undulator radiation and calibrated instrumentation [3,4].
References [1] R. Unterumsberger et al., Spectrochimica Acta Part B 78 (2012) 37–41 [2] M. Müller et al., Phys. Rev. A 79, 032503 (2009) [3] B. Beckhoff et al., Anal. Chem. 79, 7873 (2007) [4] B. Beckhoff, J. Anal. At. Spectrom. 23, 845 (2008) *corresponding author: rainer.unterumsberger@ptb.de

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18:30 **Combination TOF-SIMS and SFM measurements for characterization of inorganic and organic surfaces**

Authors : Adam Sears, Rudolf Moellers, Felix Kollmer, Ewald Niehuis
Affiliations : Laetita Bernard; Hans Josef Hug; Sasa Vranjkovic; Tim Ashworth; Raphaelle Dianoux; Adi Scheidemann;

Resume : Information concerning the chemical composition, physical properties and the three dimensional structure of materials and devices at the nanometer scale is of major importance for new developments in nanoscience and nanotechnology. Time-of-flight secondary ion mass spectrometry (TOF-SIMS) is a very sensitive surface analytical technique that provides detailed elemental

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and molecular information about surfaces, thin layers, interfaces, and full three-dimensional analysis of the sample. Scanning Force Microscopy (SFM) provides the required complementary information on the surface topography on the sub-nanometer level. Beyond that SFM can provide valuable information about the physical properties of the sample material if the cantilever is operated in the different dynamic operation modes. The combination of TOF-SIMS and SFM technique allows information concerning physical topography to provide accurate depth calibration of heterogeneous samples. This in conjunction with chemical information greatly enhances the 3D characterisation of a material in the near surface region. In this paper we will present first measurements illustrating the strength of this novel instrument and its potential for a wide range of applications including sputter induced effects on the surface morphology of inorganic as well as organic solid surfaces.

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18:30 **Topography artifacts compensation in scanning thermal microscopy on rough surfaces** HP12 19

Authors : J. Martinek, P. Klapetek, A. Campbellová, M. Valtr, R. Cimrman
Affiliations : Czech Metrology Institute, Okruzni 31, 638 00 Brno, Department of Physics, Faculty of Civil Engineering, BUT, Zizkova 17, Brno, 602 00, Czech Republic; Czech Metrology Institute, Okruzni 31, 638 00 Brno; Czech Metrology Institute, Okruzni 31, 638 00 Brno; Czech Metrology Institute, Okruzni 31, 638 00 Brno; New Technologies Research Centre, University of West Bohemia, Univerzitni 8, 306 14 Plzen, Czech Republic

Resume : Scanning thermal microscopy (SThM) is a technique suitable for high resolution mapping of thermal phenomena on surfaces of solid materials. Even if it is still more qualitative than quantitative, it has a large potential in the field of local thermal conductivity measurements and local temperature measurements, that are important in analysis of different nanostructures, like integrated circuits, thermal protection materials, nanocomposites, etc. As in all the SPM techniques, the measurement process is affected by finite size of the probe. SThM probe is often a bit larger than the probe used in atomic force microscopy, so the effects are even more pronounced. These topography related artifacts can significantly complicate quantitative data evaluation. In this contribution we will compare different techniques for processing the topography artefacts. First, we will use neural network approach based on learning the tip response while measuring on a thermally homogeneous material and then applying it to inhomogeneous one. Second, we will compare the results to thermal image calculated using Finite Element Method (FEM) taking into account the heat conduction between the probe and realistic surface. FEM is much slower but physically more accurate technique. The performance of both algorithms will be tested on different microelectronic and solar cell samples and an estimate of uncertainty components related to topography artifacts will be given.

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18:30 **Design of a large scale digital imaging spectrophotometer** HP12 20

Authors : Miroslav Valtr1, David Nečas2, Petr Klapetek1
Affiliations : 1 Department of Nanometrology, Czech Metrology Institute, Okružní 31, 638 00 Brno, Czech Republic; 2 Department of Physical Electronics, Faculty of Science, Masaryk University, Kotlářská 31, 611 37 Brno, Czech Republic

Resume : Thin films play a key role in our life. They can be used for example as wear-resistant protective coatings on cutting tools. Digital Imaging Spectrophotometry (also known as Imaging Reflectometry) is a fast non-contact technique that is often used for characterization of thin films. Although this technique is very fast, characterization of larger samples (> 10 cm2) would take enormous amount of time if it is possible at all. We designed a system with novel sample illumination set-up and a 2D stage for sample movement. The spectrophotometer can evaluate film thickness on area up to 113 x 136mm. It works in spectral range from 395 to 830nm and the lateral resolution in both axes is 18 um. The performance of the system will be demonstrated on a set of SiO2 thin films deposited on silicon substrate.

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18:30 **Post-growth annealing of GaAs/Ge layers studied by photoreflectance spectroscopy** HP12 21

Authors : S. Soltani, I. Zaied, Z. Chine, A. Rebey, B. El Jani
Affiliations : Unité de Recherche sur les Hétéroépitaxies et Applications, Faculté des Sciences de Monastir, 5019 Monastir, Tunisie

Resume : We have investigated the effects of post-growth annealing on optical and structural proprieties of GaAs thin films grown on Ge substrates by Metal Organic Vapor Phase Epitaxy (MOVPE). The GaAs/Ge samples were annealed at

650 °C for 20 min in N2 ambient. Photoreflectance (PR) and photoluminescence (PL) techniques were used in addition to high resolution x-ray diffraction (HRXRD) measurements to characterize GaAs epitaxial layers before and after annealing. PR spectra of as grown GaAs films did not show any Franz-Keldysh Oscillation (FKO) generated by the electric field inside these films. We believe that this is due to the highly non uniform electric field and the extremely narrow surface depletion region which are the results of high density of defects and traps in GaAs films. Unlike the as grown layers, PR spectra of the annealed ones exhibit FKO features. This appearance of FKO's after thermal treatment at 650 °C may be related to an improvement in surface quality of GaAs films.

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18:30 **On the metrology of amorphous transparent oxides by spectrometry techniques** HP12 22

Authors : E. Axente1, J. Hermann2, G. Socol1, A. C. Galca3, D. Pantelica4, P. Ionescu4 and V. Craciun1*

Affiliations : 1Laser-Surface-Plasma Interactions Laboratory, Lasers Department, National Institute for Lasers, Plasma and Radiation Physics, RO - 077125, Măgurele-Bucharest, Romania 2LP3, CNRS - Aix-Marseille University, Luminy, Marseille, France 3Laboratory of Multifunctional Materials and Structures, National Institute of Materials Physics, RO - 077125, Măgurele-Bucharest, Romania 4 National Institute of Physics and Nuclear Engineering Horia Hulubei, RO - 077125, Măgurele- Bucharest, Romania

Resume : The use of amorphous and transparent semiconductor oxides (ASOs) is key for the development of new thin film transistors (TFTs), solar cells electrodes and displays. By controlling the stoichiometry ASOs can be used as TFT channel (semiconductive behavior) or as transparent electrode (conductive behavior). Recently, room temperature deposited indium zinc oxide (IZO) and indium gallium zinc oxide (IGZO) was shown to exhibit a very good transparency in the visible range, low resistivity, and high mobility. Since the optical and electrical properties of these films depend on the In/(In+Zn) and Ga/(Ga+Zn) values, the measurement of this ratios is important for future developments and applications. Here we focused on the relationship composition - properties of IZO and IGZO thin films synthesized using advanced Pulsed Laser Deposition technique. An accurate monitoring of the thin films elemental composition was performed by Laser-Induced Breakdown Spectroscopy (LIBS) based on plasma modeling in view of further in-situ and real-time technological developments and process control in case of ASOs fabrication. The cation fractions measured by LIBS were compared to values obtained by complementary measurements using Rutherford backscattering spectrometry. The optical properties (thickness profile and refractive index determination) of the thin films were inferred from spectroscopic ellipsometry. Complementary investigations have been performed by fitting the measured X-ray reflectivity curves with simulated ones using a dedicated model to obtain the thickness and density of the deposited films. The room temperature electrical properties were investigated using typical four-point probe geometry and Hall measurements.

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18:30 **Synchrotron Radiation - based FIR/THz spectroscopy for studying membrane-targeting interactions of signal peptides** HP12 23

Authors : Andrea Hornemann, Arne Hoehl, Michael Andersch, Michael Vollmer, Gerhard Ulm, Burkhard Beckhoff

Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; University of Applied Sciences in Brandenburg, Magdeburger Str. 50, 14770 Brandenburg, Germany; University of Applied Sciences in Brandenburg, Magdeburger Str. 50, 14770 Brandenburg, Germany; Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany;

Resume : The development of new infrared radiation sources has initiated opportunities for exploring the molecular structure of many (bio-) materials in the far-infrared/terahertz (FIR/THz) spectral region. The identification of thin organic films derived from peptide layers at polymer/organic interfaces was performed by Synchrotron Radiation (SR) based spectroscopy at PTB's Metrology Light Source (MLS). For bioanalytical applications, the THz technique offers a unique tool for a distinct identification of biochemical components by their vibrational signatures. The excitation of molecular resonances of peptide films at flat surface layer-substrate interfaces is induced by broadband SR at the MLS, enabling the detection of molecular-specific low frequency modes in the spectral region between 400 cm-1 and 1 cm-1. The FIR/THz spectral region complements the mid-infrared range, where molecules such as proteins deliver characteristic modes, and entails additional molecular information on rotational

modes of the carbon backbone and H-bonds of biomolecules. We discuss the signatures of membrane-targeting model peptides that display defined secondary-structure motifs. The signatures exhibit NH bending, CN torsional and CO bending modes along with a most pronounced Amide VII indicator band that is suited for the study of secondary structure-function relationships among membrane-targeting peptides. Our approach entails cryogenic conditions between 300 K and 10 K.

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18:30 **Accurate measurement of segregation to grain boundaries or planar faults by analytical transmission electron microscopy** HP12 24

Authors : T. Walther 1, M. Hopkinson 1, N. Daneu 2, A. Recnik 2, Y. Ohno 3, K. Inoue 3 and I. Yonenaga 3

Affiliations : 1 Dept. Electronic and Electrical Engineering, University of Sheffield, Mappin Building, Mappin Street, Sheffield S1 3JD, UK 2 Department for Nanostructured Materials, Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia 3 Institute for Materials Research, Tohoku University, Katahira 2-1-1, Aoba-ku, Sendai 980-8577, Japan

Resume : We describe a method of analytical transmission electron micrsocopy has been successfully applied to study dopant segregation to inversion domain boundaries in ZnO, to quantify the thicknesses of sub-nm thin layers during epitaxial growth by molecular beam epitaxy of (In)GaAs and Si(Ge) and proved the absence of gettering of dopants at Sigma=3{111} grain boundaries in Si, with a precision < 1 atom/nm2 in all these cases

add to my program **(close full abstract)**

18:30 **Reference-free total reflection X-ray fluorescence analysis for quantification of functional groups on surfaces for bioanalytical applications** HP12 25

Authors : C. Streeck 1, A. Nutsch 1, J. Weser 1, T. Fischer 2, P. Dietrich 2, K. Rurack 2, W. Unger 2 and B. Beckhoff 1

Affiliations : 1 Physikalisch-Technische Bundesanstalt (PTB), Abbestr.2-12, 10587 Berlin, Germany 2 Bundesanstalt für Materialforschung und -prüfung (BAM), Unter den Eichen 87, 12205 Berlin, Germany

Resume : Functionalized surfaces are essential in biotechnology, e.g., for the development of biosensors and microarrays. Knowledge of the quantity of primary reactive groups on such surfaces is indispensable for a defined secondary modification with commonly biochemical entities, eventually resulting in more reliably tailored surfaces with better controlled properties. Here, we investigated aminated glass surfaces with varying densities of amino groups prepared from binary mixtures of silanes. Subsequent labeling of the amino groups with a fluorophore containing a high number of fluorine atoms allows complementary quantification of the organic groups by optical fluorescence spectroscopy, X-ray Photoelectron Spectroscopy (XPS) and traceable Total-Reflection X-ray Fluorescence analysis (TXRF). Reference-free TXRF with soft X-ray excitation determines the mass deposition of elements such as carbon, nitrogen and fluorine, yielding the areal density of primary functional groups and fluorophore on the surface. The TXRF-measurements were performed at the PTB beamline for undulator radiation at the electron storage ring BESSY II which provides monochromatic soft X-rays with high spectral purity and photon flux. This approach allows the calibration of optical fluorescence spectroscopy and XPS to deliver traceable quantitative data.

add to my program **(close full abstract)**

18:30 **An original method for evaluation of the isomeric rate of undoped polyacetylene by multichanel Raman** HP12 26

Authors : K. BENOUMSAAD1*; ILHEM. R. KRIBA1; A. DJEBAILI2

Affiliations : 1 Plasma Laboratory - Faculty of Sciences – Department of Physics-University of Batna- Algeria 2 Laboratory of chemistry and environmental chemistry L.C.C.E - University of Batna- Algeria,

Resume : The first originality of the experiments we carried out consists of the use of a laser beam as a double agent: simultaneously as activation agent inducing the isomerization reaction of the PA, and for the Raman diffusion. The laser beam power P (λ) is equivalent to the temperature T of isotherm i of isomerization reaction. The second originality consists of use of multichannel spectroscopy which enables the simultaneous observation of both reactants (PAcis) and product (PAtrans) in the same time, since PAcis absorption band and PAtrans absorption band are clearly shifted on a band of 512 diodes Then we have a double simultaneity (i) In one hand the heating and the diffusion of the laser beam, (ii) On the other hand the steady measurement of the concentrations. We elaborate a numerical model reproducing the Raman experiment within 5 % error. The rate constants, activation energy values, Arrhenius factors and linear regression coefficients are obtain with a small error.

The kinetic results obtained, such as reaction orders values obtained, varying from 1/2 to 2/3, showed clearly that, the isomerization reaction of undoped P.A. remains a complex process. The reaction order of 2/3 seems to be the most appropriate value in this case, since it refers to a solid state reaction propagation, where the reaction rate is controlled by a three dimensional development of active centers, in agreement with Sestak and Berggren theory

add to my program **(close full abstract)**

18:30 **An advanced X-ray Spectrometry facility for Materials Research** HP12 27

Authors : J.J. Leani(1), B. Beckhoff(2), M. Bogovac(1), D. Eichert(3), R. Fliegauf(2), A. Gambitta(3), D. Grötzsch(4), W. Jark(3), R. B. Kaiser(1), B. Kanngießer(4), A.G. Karydas (1), L. Lühl(3), M. Kiskinova(3), J. Lubeck(2), W. Malzer(4), A. Migliori(1), H. Sghaier (1,5), M. Spanier(4), N. Vakula(1), J. Weser(2)
Affiliations : 1)Nuclear Science and Instrumentation Laboratory (NSIL), IAEA Laboratories, A-2444 Seibersdorf, Austria, A.Karydas@iaea.org; 2)Physikalisch-Technische Bundesanstalt (PTB), 10587 Berlin, Germany; 3)Elettra - Sincrotrone Trieste (EST) S.C.p.A., 34149 Basovizza, Trieste, Italy; 4)Technische Universität Berlin (TUB), Institut für Optik und Atomare Physik, 10623 Berlin, Germany; 5)Institut Superieur d'Informatique et de Mathematiques de Monastir (ISIMM), Departement de technologie, 5000, Monastir, Tunisia
Resume : A novel Ultra High Vacuum Chamber (UHVC) end-station, developed by NSIL-IAEA and based on a prototype designed by PTB and TUB, is currently under commissioning at the new XRF beamline of EST. The UHVC end-station includes a motorized 7-axis sample manipulator. An air-cooled Mo anode X-ray tube is integrated allowing for a standalone operation. Different Silicon Drift Detectors and several Si/GaAsP photodiodes are employed for spectroscopy and monitoring purposes, whereas an Intuitive Graphical User Interface based on LabView software, controls all UHVC devices/detectors. The UHVC facility allows the application of several X-ray spectrometry techniques under different configurations: Grazing Incidence/Exit X-Ray Fluorescence analysis simultaneously with X-Ray Reflectometry measurements and X-ray Absorption Spectroscopy within the energy interval 2-14 keV. The purpose of this development is to strengthen synchrotron radiation research opportunities through on-the-experiment training and establishing coordinated research activities. Targeted analytical applications refer to the characterization of advanced nanostructured materials, environmental and biological samples, but also for the measurement/evaluation of X-ray fundamental parameters which are essential for standardless quantification of modern materials. First commissioning results with the UHVC facility will be presented and discussed demonstrating its analytical capabilities and performance on materials research.

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18:30 **Sorption capacity of cadmium- and cadmium sulfide containing zeolite thin films : an FTIR study** HP12 28

Authors : M. Bryckaert(1), I. De Waele(1), S. Thomas(2), S. Mintova(2), V. De Waele(1)
Affiliations : 1 -LASIR, UMR8516 CNRS-Universit? de Lille 1, Cit? scientifique, F-59655 Villeneuve d?Ascq, France 2- Laboratoire Catalyse et Spectrochimie, ENSICAEN - Universit? de Caen ? CNRS, 6, Boulevard du Mar?chal Juin, 14050 Caen, France
Resume : Microporous thin flims containing metal or semi-conductors nanoparticles are promising materials for applications in sensing, plasmonic catalysis, or energy conversion. Toward this end, the quantitative characterization of the microporosity, notably in terms of accessible volume, of sorption capacity, and of dispersion of the nanoparticles through the porous volume is fundamental. However, thin films with sub-micrometer thickness are difficult to be studied by using conventional analytical chemistry methods (TG, DTG, sorption porosimetry). Alternatively, we investigated the potential of using transmission FTIR spectrophotometry and imaging to characterize quantitatively the sorption capacity of zeolite thin films. The approach is illustrated for Cd- and CdS-containing colloidal zeolite.Thin films of different thickness were prepared by spin-coating and adsorption isotherms were determined by FT-IR for water, methanol, ethanol, propanol, acetonitrile. The changes in sorption capacity associated with the incorporation of CdS nanoparticle inside the microporous volume of zeolite are discussed. Finally FTIR microscopy measurement are performed as a non destructive method to control the thin film homogeneity and to determine the film density.

add to my program **(close full abstract)**

18:30 **Advanced X-ray spectrometry instrumentation based on synchrotron radiation for nanomaterials characterization and a design study of an analytical platform for 450 mm Si wafer** HP12 29

Authors : J. Lubeck1, I. Holfelder1, B. Beckhoff1, R. Fliegauf1, P. Hönicke1, M. Müller1,

A. Nutsch1, P. Petrik2, F. Reinhardt3, G. Roeder4, B. Pollakowski1, J. Weser1
Affiliations : 1 Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany 2 Institute for Technical Physics & Materials Science (MFA), Research Centre for Natural Sciences, Konkoly Thege u. 29-33, 1121 Budapest, Hungary 3 Bruker Nano GmbH, Am Studio 2D, 12489 Berlin, Germany 4 Fraunhofer IISB, Schottkystraße 10, 91058 Erlangen, Germany
Resume : A versatile UHV instrument [1] enabling synchrotron radiation based X-Ray Spectrometry (XRS) related techniques for nanoanalytics and X-Ray Reflectometry (XRR) was developed by PTB. An integrated 9-axis manipulator allows for a reproducible sample alignment in all degrees of freedom. A translational and rotational movement of several photodiodes as well as a translational movement of a rigid aperture system which enables reference-free X-Ray Fluorescence (XRF) analysis is possible. By means of a flexible beam geometry from total reflection (TXRF), grazing incidence (GIXRF) to conventional XRS, including access to polarization-dependent detection channels as well as simultaneous XRR measurements, reliable information about layer thicknesses, elemental or spatial compositions, elemental depth profiles, chemical bonding states and the molecular orientation of bonds can be determined. In addition, the steadily increasing demand for the validation, assurance, and support involving various analytical methods is driving the integration of multiple methods into one tool for 450 mm Si wafers. Therefore, a design study for a 450 mm analytical platform [2] was performed, integrating several complementary methods into one tool, X-ray analytical methods such as TXRF, GIXRF, XRF, XRR, XRD and GISAXS as well as ellipsometry and vacuum UV reflectometry. References: [1] J. Lubeck et al., Rev. Sci. Instrum. 84, 045106 (2013) [2] I. Holfelder et al., J. Anal. At. Spectrom. 28(4), 549-557 (2013)

add to my program **(close full abstract)**

18:30 **Analytical calculation and comparison of numerical solutions obtained by the technique of the transfer matrix and FDTD of the transmission and reflection of metamaterials**

Authors : C.Chettah, A.Chaabi
Affiliations : Laboratory of Hyperfrequency and Semi Conductor, Electronics Department, University Constantine 1, Algeria
Resume : We investigated the spectral properties of a new class of nanostructured artificial composite materials with tailored electromagnetic response, negative refractive index materials, also known as "left-handed" metamaterials. We analyzed structures incorporating both ordinary positive index media and negative refractive index metamaterials where the interface may be graded to an arbitrary degree. Utilizing a modified version of the Rosen-Morse function, we derived analytical expressions for the field intensity and spectral reflection and transmission through a graded interface between positive and negative index materials. We compared our results to numerical solutions obtained using the transfer matrix and Finite-difference time-domain method FDTD.

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18:30 **XPS study of oxide surface modification by low-energy ion bombardment**

Authors : Nikolai Alov
Affiliations : Moscow State University, Faculty of Chemistry
Resume : The irradiation of oxides by low-energy ion beams induces a change in the surface composition and structure. We have investigated the influence of low-energy Ar^+ ion bombardment on the surface composition of higher oxides MoO_3, WO_3, Nb_2O_5 and Ta_2O_5 by X-ray Photoelectron Spectroscopy (XPS). The Ar^+ ion bombardment of pressed oxide pellets was carried out with energy of 3 keV in wide range of fluence and direction normal to the oxide surface in high-vacuum preparation chamber of Leybold LHS-10 electron spectrometer. The subsequent determination of the surface composition by XPS was performed in situ in high-vacuum analysis chamber of the electron spectrometer. Fitting of the Mo 3d-, W 4f-, Nb 3d- and Ta 4f-core level spectra was essential for the estimation of oxidation states. This was done after the subtraction of Shirley background using Gaussian-Lorentzian sum function. It was found from the XPS data that the irradiation of MoO_3, WO_3, Nb_2O_5 and Ta_2O_5 by low-energy Ar^+ ions induces the surface reduction, metallization and formation of the layer consisting of MoO_x, MoO_2, Mo; WO_x, WO_2, W; NbO_2, NbO and TaO_2, TaO, Ta. The nature, mechanisms and features of oxide surface modification induced by the ion-beam irradiation are discussed.

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18:30 **Rapid thermal annealing effect on the optical properties of Au-doped ZnO thin films grown by rf-sputtering**

Authors : A.G. Rolo (1), C. Kubel (2), M. Buljan (3), S. Bernstorff (4), N. P. Barradas (5), N. Franco (6), and E. Alves (6)
Affiliations : (1) Centro de Física, Universidade do Minho, Campus de Gualtar 4710-057 Braga, Portugal (2) Karlsruher Institut fur Technologie, Institute of Nanotechnology, Eggenstein-Leopoldshafen, Germany; (3) Rudjer Boskovic Institute, Bijenicka cesta 54, 10000 Zagreb, Croatia (4) Elettra-Sincrotrone Trieste, SS 14 km163.5, Basovizza 34149, Italy (5) Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa, EN10 (km 139,7), 2695-066 Bobadela LSR, Portugal (6) Associação Euratom/IST, Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001, Lisboa, Portugal

Resume : In the present work we study nanocomposite thin films consisting of Au nanoparticles(NPs) dispersed in ZnO matrix. The influences of the magnetron sputtering deposition conditions on the structural and optical properties of the films are investigated. Different series of films were grown on glass substrate by co-depositing Zn and Au in an oxygen atmosphere with the magnetron RF-sputtering technique. The evolution of the average size of the Au NPs as a function of the deposition parameters is studied. The influences of the rapid thermal annealing (RTA) temperature and time on the properties are also summarized. We demonstrate the formation of size-uniform and homogeneously distributed Au nanoparticles in a ZnO superficial layer, which are crystalline after a short duration RTA treatment at 500ºC in an inert atmosphere. The optical properties of the films show the size-dependant surface plasmon resonance (SPR) of gold nanoparticles. The structural properties of the films are discussed in the light of the results given by a wide range of experimental characterization techniques such as Raman, SEM, XRD, GISAXS and TEM techniques. Work supported by KNMF and SPIRIT.

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18:30 **ARXPS study of vanadium surface oxidation under oxygen ion bombardment**

Authors : Nikolai Alov
Affiliations : Moscow State University, Faculty of Chemistry

Resume : The growth of oxide layer on vanadium surface induced by oxygen ion bombardment was studied by angle-resolved X-ray Photoelectron Spectroscopy (ARXPS). On the basis of fine structure analysis of V 2p spectra it was concluded that under the oxygen ion bombardment at room temperature the ion-beam induced surface oxidation of vanadium occurs and the oxides V_2O_3, VO_2 and V_2O_5 are formed in the surface layers. After long time of ion irradiation VO_2 is predominating in the oxide film. Different population of individual vanadium oxidation states is obtained with thin oxide films produced by thermal oxidation. The differences observed are caused by different radiation stability of vanadium oxides formed under the oxygen ion-beam irradiation. The distribution of individual vanadium oxidation states within the oxide film was determined. The spectra measured at different detection angles show that the distribution of individual oxide states within the oxide layer formed by oxygen ion bombardment is not homogeneous. The outer part of the oxide film being enriched by V_2O_5 and the lower oxides present predominantly in the inner region of the oxide film.

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18:30 **CASTOR: a new grazing incidence x-ray fluorescence (GIXRF) setup at LNHB**

Authors : Yves Ménesguen, Bruno Boyer & Marie-Christine Lépy
Affiliations : CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), F-91191 Gif-sur-Yvette, France

Resume : Studying new materials requires new techniques. A lot of new materials developed either in public research institutes or private companies are created for their new functionalities and are very often deposited as layers in stacks. Some of their properties can be affected by bad or uncontrolled interfaces as well as roughness, inhomogeneity. Grazing incidence x-ray techniques are of major interest as they can give information about in-depth or interfaces properties of materials. In collaboration with PTB, the LNHB decided to build a GIXRF setup designed to do metrology studies about these new materials. PTB, pioneer in this field, defined the specifications of the manipulator in order to meet up-to-date performance in positioning samples, detectors to deliver the best possible results. We report here on the assessment of the setup, mechanical, electrical, vacuum and software engineering. Mechanical performances were tested at PTB and met their specifications. The final assembly was done by LNHB and PTB. The setup will be equipped with several diodes, some of them calibrated by LNHB using the BOLUX setup. 1) P. Troussel,

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N. Coron, BOLUX : A cryogenic electrical-substitution radiometer as high accuracy primary detector in the 150-11000 eV range, Nuclear Instruments and Methods in Physics Research A, 614 (2010) 260-270

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18:30

Mapping nanoscale thermal transfer in a liquid environment -immersion scanning thermal microscopy (iSThM)

Authors : Peter D. Tovee and Oleg V. Kolosov
Affiliations : Physics Department, Lancaster University, Lancaster, LA1 4YB, UK

Resume : Nanoscale thermal properties are becoming extremely importantfor modern chips that dissipate increasing current densities. While Scanning Thermal Microscopy (SThM) that uses locally heated nanoscale probes is known to probe thermal properties of materials with nanoscale resolution, until today it was perceived impossible to use active SThM in a liquid environment due to heat dissipation into the surrounding liquid that would deteriorate SThM and spatial resolution. Nonetheless, our recent theoretical analysis of immersion SThM (iSThM) showed that for a resistive heater located near the tip apex, the probe's thermal signal is only moderately affected, on immersion in a dodecane environment, while spatial resolution is similar to in air SThM and the tip-sample thermal contact is beneficially improved. Our trials of iSThM were successful and here we report measurements of thermal conductivity of Ultra Large Scale Integration (ULSI) polymer-ceramic-metal interconnects and nanoscale graphene flakes immersed in liquid environment. The immersion directly thermally links the tip to the interconnect Al lead, allowing to avoid spurious presence of surface voids and roughness, while preserving thermal resolution down to 50 nm. iSThMopensa potentially broad range of applications from non-contact measurements of thermal transport in semiconductor devices to exploring catalytic reactions in liquid phase and heat generation in biological systems. Nanotechnology Vol. 24 (2013) 465706

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18:30

Enabling investigations of biomolecular films at liquid-solid interfaces

Authors : D. Grötzsch1, W. Malzer1,B. Kanngießer1, C.Streeck2, A. Nutsch2, B.Beckhoff2, C. Nietzold3, P. Dietrich3, W. Unger3
Affiliations : 1 Technische Universität Berlin, Hardenbergstr. 36, 10623 Berlin 2 Physikalisch Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin 3 Bundesanstalt für Materialforschung und –prüfung, Unter den Eichen 87, 12205 Berlin

Resume : Capturing biochemical markers by biomolecular films is one of the most promising approaches for the development of highly sensitive and highly selective diagnosis. In particular future innovative tools for in vitro or point of care diagnostics are expected to rely on this principle. Analytical techniques which can provide information on coverage, orientation and chemical state of biochemical films are capable of contributing to a purposeful development of such diagnostics. The liquid cell we present was designed to facilitate the application of soft X-ray spectrometry for the in-situ analysis of biomolecular films at solid-liquid interfaces. It allows for - the analysis through a silicon nitride window with a thickness of 150 nm - in situ preparation of successive layers by rinsing the window Currently, after the first successful soft X-ray experiments we are improving the versatility of the liquid cell. Spectrometry in transmission and in various emission geometries will be feasible. Further controls for the experimental conditions will be added.

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PROGRAM VIEW : 2014 Spring
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ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

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	JOINT SESSION SYMPOSIUM H AND DD: Correlating nanostructure and function in advanced organic electronic devices : Fernando Araujo de Castro	
08:30	**Quantitative Nano-scale Imaging at the Swiss Light Source** **Authors :** Benjamin Watts **Affiliations :** Paul Scherrer Institute **Resume :** Tbc	H13 1
09:00	**Modelling Photoconductive Atomic-Force Microscopy (Pc-AFM)** **Authors :** James C Blakesley , Fernando Castro, Alina Zoladek-Lemanczyk, Stephen Giblin, Alan Turnbull **Affiliations :** National Physical Laboratory, Teddington, TW11 0LW, United Kingdom **Resume :** Pc-AFM is a method with potential for imaging nanoscale features in photo-active thin films. It has the ability to image features that affect optoelectronic properties, even where these features are buried below the film surface. There is interest in applying the technique to the study of bulk heterojunction photovoltaics. Optimal photo-active layers comprise donor and acceptor phases intimately mixed on a < 10 nm scale. This mixing is too fine to be resolved by Pc AFM. However, defects in the morphology frequently occur on length scales of 10 – 1000 nm. These, being on a similar scale to the film thickness, can have a significant impact on the macroscopic device properties, while still being too small to identify with conventional optoelectronic methods. Pc-AFM has been used to image donor nanowires buried within an acceptor film [1]. It has also been used to identify sub-surface acceptor aggregate defects [2]. However, the interpretation of the high-resolution photocurrent images requires deep physical insight. We have developed a tool for simulating the processes of photocurrent generation and conduction under the AFM tip. We report on simulations of model photovoltaic systems and compare them to experiments. The tool is used to help interpret experimental results, and to determine the limitations of Pc-AFM as a instrument for imaging mesoscopic defects. [1] Tsoi et al., Energy & Environ. Sci. 4, 3646 (2011) [2] Coffey and Ginger, Nat. Mater. 5, 735 (2006)	H13 2
09:15	**Thermal degradation study of P3HT:PCBM solar cells using SPM techniques** **Authors :** Ravi C Chintala, Jeffry G Tait, Pierre Eyben, Eszter voroshazi and Wilfried Vandervorst **Affiliations :** IMEC, Kapeldreef 75, B-3001, Leuven , Belgium ; KU Leuven, Department of Physics and Astronomy (IKS), Celestijnenlaan 200D, 3001 Leuven, Belgium ; KU Leuven, ESAT, Kasteelpark Arenberg 10, B-3001, Leuven, Belgium **Resume :** Organic photovoltaics, especially solution processed solar cells, is a low-cost and complementary technology to currently deployed inorganic modules. One of the shortcomings of these devices is the thermal degradation of the active layer, morphological and compositional changes upon long-term exposure to elevated temperature. To improve the understanding of these changes, we studied the phase separation in accelerated conditions by annealing the samples at temperatures higher than the operating conditions (@130 C for 1h in a N2 atmosphere). In this work, various scanning probe microscopy (SPM) techniques (to probe electrical and mechanical properties) were used to investigate thermal degradation of a polythiophene: fullerene blend (P3HT:PCBM). The active material has been deposited by spray coating on a ITO	H13 3

(Between entries: add to my program — (close full abstract))

covered substrate. Using conductive AFM in peak-force mode (PF-CAFM), we observed, in agreement with earlier work, that a thin P3HT layer forms on top of the P3HT:PCBM blend during deposition (no contrast in current or in adhesion map). In order to investigate the vertical phase separation in the blend, craters have been dug using argon cluster beam. PF-CAFM and Kelvin probe force microscopy (KPFM) in the craters, respectively reveal contrast in the conduction (as well as in adhesion) and in the surface potential between the blend and the hillocks (formed due to annealing) corroborating the phase segregation and allowing us to demonstrate that these hillocks are PCBM rich. The segregation mechanism that we observe at the nm-size level using SPM explains the performance degradation observed at the macroscopic level.

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09:30 **Controlling bulk heterojunction photovoltaic morphology: The importance of solution phase aggregation**

Authors : Christopher J. Tassone, Kristin Schmidt, Jonathan Bartelt, Alan Yiu, Jeremy Niskala, Pierre Beaujuge, Jean M. Frechet, Michael McGehee, Michael F. Toney
Affiliations : Stanford Synchrotron Radiation Lightsource; Stanford Synchrotron Radiation Lightsource; Stanford University; UC Berkeley; UC Berkeley; King Abdullah University of Science and Technology; King Abdullah University of Science and Technology; Stanford University; Stanford Synchrotron Radiation Lightsource

Resume : Polymer bulk heterojunction (BHJ) solar cells have attracted significant attention in industry and academia because of their potential for achieving large-area, light-weight, and flexible photovoltaic devices. These devices consist of a blend of an donor (polymer) and an acceptor (fullerene), the molecular packing and phase segregation of which influence power conversion efficiency. We have used solution phase small angle x-ray scattering (SAXS) in order to investigate the self-assembly behavior of several different donor systems. We examine how the use of solvent additives, mixed solvent systems, and molecular weight tuning can induce ordering of the donor moiety within the casting solution. Using transmission SAXS on the as-prepared active layers of these BHJ systems, we observe that conditions which induce aggregation and ordering of the polymer chains within the casting solution, promote an ideal degree of phase segregation in the active layer of the device, which leads to enhanced power conversion efficiency (PCE). However, in cases in which no ordering of the donor polymer is observed in the solution phase, over phase segregated active layers are observed with corresponding low PCEs. This leads us to conclude that aggregation and ordering of the polymer chains in the casting solution is directly relrelated to the formation of an ideal three dimensional interpenetrating network.

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09:45 **Investigation of polymer organization for organic electronic devices using Langmuir-Blodgett methods**

Authors : Mariane Ouattara, Josée Brisson, Mario Leclerc
Affiliations : Université Laval, 1045 avenue de la medecine G1V0A6, Quebec, Canada

Resume : Investigation of Polymer Organization for Organic Electronic Devices Using Langmuir-Blodgett Methods Mariane Ouattara, Josée Brisson and Mario Leclerc Département de chimie, Université Laval, 1045 avenue de la Médecine, Québec, Québec, Canada G1V 0A6 http://www.chm.ulaval.ca/poly_conducteurs/ Research in the fields of plastic electronics generated an increasing interest in many scientific domains during the last decade. Contrary to inorganic semiconductors, organic semiconductors can be deposited as thin films on flexible supports by low cost processing techniques. The present work is aimed at investigating the possible performance improvement of polymer-based transistors through the control of morphology and orientation. It has been shown1 that carrier mobility can be increased by a factor of 1000 when conjugated units are organized in an edge-on conformation on the transistor surface. In order to achieve ordered polymers films fabrication, amphiphilic polymers based on thieno[3,4-c]pyrrole-4,6-dione (TPD) units2 were synthesized. Homogeneous and stable monolayers of TPD-based copolymers were prepared. Brewster angle microscopy (BAM) was utilized to characterize the morphology and topography of these Langmuir films. UV−vis absorption spectroscopy as well as atomic force microscopy has revealed a regular transfer of some copolymers on glass substrates. It was possible to obtain homogeneous. Langmuir−Blodgett films of up to 30 layers. Infrared dichroic measurements revealed an edge-on orientation. These Langmuir−Blodgett films made of conjugated polymers are therefore good candidates for organic field-effect transistors (OFETs). (1) Sirringhaus, H., Brown, P. J., Friend, R. H., Nielsen, M. M., Bechgaard, K., Langeveld-Voss, B. M. W.,Spiering, A. J. H.,

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Janssen, R. A. J., Meijer, E. W., Herwig, P. et de Leeuw, D. M. Nature 1999, 401,685. (2) Ouattara, M. P.; Lenfant, S.; Vuillaume, V.; Pézolet, M.; Rioux-Dubé, J.-F.; Brisson, J.; Leclerc, M. Macromolecules, 2013, 46, 6408.

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10:00 **Coffee break**

Advances in scanning probe microscopy : Ludger Koenders and Miroslav Valtr

10:30 **Nanoscale interfacial interactions of graphene with polar and non-polar liquids**

H14 1

Authors : Benjamin J. Robinson, Nicholas D. Kay, Oleg V. Kolosov
Affiliations : Physics Department, Lancaster University, Lancaster, LA1 4YB, UK

Resume : Graphene's nanomechanical behaviour in liquids, vital for its operation in rechargeable batteries, super-capacitors, and sensors, is still largely unexplored. Here we will discuss the results of recent studies investigating the nanomechanics of normal (adhesive and elastic) and tangential (friction) forces between a stationary, moving and ultrasonically excited nanoscale atomic force microscope (AFM) tip and exfoliated few layer graphene (FLG) on SiO_2 substrate as a function of surrounding media – air, polar (water) and non-polar (dodecane) liquids. We find that while the friction coefficient is significantly reduced in liquids, and is always lower for FLG than SiO_2, it is higher for graphene in non-polar dodecane than highly polar water. We also confirm that in ambient environment the water meniscus dominates high adhesion for both hydrophobic FLG and the more hydrophilic SiO_2 surface. By using nanomechanical probing via ultrasonic force microscopy (UFM) we observed profound reduction of graphene rippling and increase of graphene-substrate contact area in liquid environment. Friction force dependence on ultrasonic modulation amplitude suggests that dodecane at the graphene interface produces a solid-like "cushion" of approximately 2 nm thickness, whereas in water immersion, the same dependence shows remarkable similarity with ambient environment, confirming the presence of water meniscus in air, and suggesting negligible thickness of a similar water "cushion" on graphene.

add to my program **(close full abstract)**

10:45 **Fragmentation and exfoliation of low-dimensional materials; a statistical approach.**

H14 2

Authors : Konstantinos Kouroupis-Agalou1, Andrea Liscio1, Emanuele Treossi,1,2 Luca Ortolani3, Vittorio Morandi3, Nicola Maria Pugno4, Vincenzo Palermo1,2*
Affiliations : 1 Istituto per la Sintesi Organica e la Fotoreattività-Consiglio Nazionale delle Ricerche (ISOF-CNR), via Gobetti 101, 40129 Bologna, Italy.; 2Laboratorio MIST.E-R Bologna, via Gobetti 101, 40129 Bologna (Italy); 3 Istituto per la Microelettronica e Microsistemi-Consiglio Nazionale delle Ricerche (IMM-CNR), via Gobetti 101, 40129 Bologna, Italy.; 4 Dipartimento di Ingegneria Civile, Ambientale e Meccanica, Università di Trento, via Mesiano, 77 I-38123 Trento (Italia);

Resume : A main advantage for applications of Graphene and related 2-dimensional materials is that they can be produced on large scales by liquid phase exfoliation. The exfoliation process shall be considered as a particular fragmentation process, where the 2-dimensional (2D) character of the exfoliated objects will influence significantly fragmentation dynamics as compared to standard materials. Here, we used automatized image processing of Atomic Force Microscopy (AFM) data to measure, one by one, the exact shape and size of thousands of nanosheets obtained by exfoliation of an important 2D-material, Boron Nitride, and used different statistical functions to model the asymmetric distribution of nanosheets sizes typically obtained. Being the resolution of AFM much larger than the average sheet size, analysis could be performed directly at the nanoscale, and at single sheet level. We find that the size distribution of the sheets at a given time follows a log-normal distribution, indicating that the exfoliation process has a "typical" scale length that changes with time and that exfoliation proceeds through the formation of a distribution of random cracks that follow Poisson statistics.

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11:00 **Conductive and Photoconductive Atomic Force Microscopy parametric investigation of organic electronic ultra-thin films**

H14 3

Authors : Achilleas Sesis1, David Cheyns2, David. C. Cox1,3, Emmanuele Enrico4, Federico F. Lupi5, Luca Boarino4, Fernando A. de Castro*1
Affiliations : 1. National Physical Laboratory, Hampton Rd,Teddington, Middlesex, TW11 0LW,U.K. 2. IMEC, Kapeldreef 75, B-3001, Leuven, Belgium 3 Advanced Technology

Institute, University of Surrey, Guildford, Surrey, GU2 7XH, U.K. 4 Laboratorio MDM, IMM -CNR, Via C. Olivetti 2, 20846, Agrate Brianza (MB), Italy 5. NanoFacility Piemonte, Electromagnetism Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce 91 - 10135, Torino, Italy

Resume : Organic electronics applications range from display and lighting to solar cells and sensors. The active layer is often composed of small molecules or polymer blends exhibiting various optoelectronic properties. In order to optimise their performance in a device, manufactures must carefully consider their nanoscale architecture. Amongst the numerous characterisation techniques, atomic force microscopy (AFM) is perhaps the most popular, with its conductive (c-AFM), and more recently photoconductive (pc-AFM), variants being extensively used to comprehend and optimise the performance of active layers. However, many of the operational parameters dictating the quality of the images have not thoroughly been investigated. As a result, the images can be falsely interpreted, attributing measurement outcomes to the physical properties of the organic systems, where in fact the results may be ascribe to changes in the AFM operational conditions. In this study we present the analysis of c/pc-AFM images recorded at the surface of ultra-thin films deposited on glass/indium tin oxide substrates. The samples comprised individual fullerene and zinc phthalocyanine layers in addition to their combinatorial heterojunctions. We show the development of model samples with defined nanostructures to systematically investigate the effect of measurement operation conditions on the data. The study also includes the use of different conductive AFM tips tested in an atmospherically controlled environment for the tribological behaviour, an element believed to substantially influence the image quality.

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11:15 **FDTD modeling of photoconductive AFM experiments** H14 4

Authors : Petr Klapetek, Miroslav Valtr
Affiliations : Czech Metrology Institute, Okuzni 31, 638 00 Brno, Czech Republic

Resume : Photoconductive atomic force microscopy is a technique allowing us to map local charge generation on samples exposed to an illumination. In most cases this is done by conventional atomic force microscope equiped with additional source of illumination, conductive probe and very sensitive transimpedance amplifier. This techniques is particularly useful while studying local structure and functionality of solar cell materials. To estimate from which area the signal was collected is however not straightforward as the probe sample geometry is quite complex and light scatters in many ways while illuminating the sample. In this contribution we present computational approach to address the issues of light localisation, measurement resolution and measurement uncertainty in conductive AFM, using Finite Difference in Time Domain method for optical calculations and Finite Element method for electrical calculations. We will discuss effects of overall sample morphology, presence of thin films and nanoscale roughness, using realistic data including all these imperfections as model input and comparing outputs to experimental results.

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11:30 **Mechanical properties of polystyrene nanoparticles** H14 5

Authors : Joergen Garnaes
Affiliations : DFM - Danish Fundamental Metrology, Matematiktorvet 307, DK-2800 Lyngby, Denmark

Resume : Accurate characterization of the elasticity and plasticity of nanoparticles can be used to improve their function in engineered products. It will also help to better understand their basic properties such as adhesion and agglomeration which is important in health and environment issues. In general the material properties of nanoparticles can be different from the bulk properties of the same material as the nanoparticles can be synthesized in a different way than the bulk material and dominated by a surface region. In this paper Young?s modulus and the yield strength is estimated of polystyrene spheres with a nominal diameter in the range of 50 nm to 300 nm using atomic force microscopy. The measurements are based on force-distance curves recorded at each point on the surface of the spheres when they form a close-packed monolayer on a flat substrate. Accurate extraction of material properties from force-distance curves is limited by the knowledge of photodiode sensitivity and complicated by the presence of Van der Waals forces, capillary force and the curved surfaces of the spheres. In this paper the use of reference materials for the estimation elasticity is also explored and an uncertainty budget is given. The measured material properties are used to determine the deformation of the polystyrene spheres when they are in contact with each other or with the

substrate and the predicted deformation is discussed based on accurate measurements of the diameter.

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11:45 **Comparison of spherical nanoparticles size measurements by AFM and SEM**

Authors : Alexandra DELVALLEE, Nicolas FELTIN, S?bastien DUCOURTIEUX
Affiliations : Laboratoire National de m?trologie et d?Essais ? LNE, 29 avenue Roger Hennequin, 78197 Trappes Cedex

Resume : The LNE?s nanometrology team develops a platform dedicated to the metrological characterization of nanomaterials (CARMEN) and more specifically to the metrology of the mean diameter and size distribution of a population of spherical nanoparticles (NPs) by Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). The objective is to ensure the traceability of the measurements to the International System of Units and to be able to compare it. To determine the diameter of NPs by AFM, and due to the tip/surface dilation that leads to a broadening of the lateral measurement, we developed a method based on the measurement of NPs height with a expanded uncertainty of 0.8 nm for particles of 50 nm. On the contrary of AFM, the SEM is unable to give quantified information along Z axis. That is why the lateral diameter measurement is adopted for the size measurements of NPs. A partial uncertainty budget is given. A two step method has been developed for AFM and SEM image processing. The first step is realized with a commercial software and consists in levelling and binarizing images. The second is realized with a home-made software and consists in detecting, measuring and drawing NPs distribution. To compare both methods, a 80 nm SiO2 NP population was deposited on a silicate substrate. Measurement comparison was done from 1 SEM and 4 AFM images. NPs distribution shows a leading mode at 85.5 nm for SEM image and 79.1 nm for AFM. One explanation for this observation is the fact that not the same NPs are observed by AFM and SEM. So, the LNE is investigating colocalization techniques to image exactly the same NPs by both instruments.

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12:00 **Quantitative morphology characterization of PC-siloxane copolymer blends at nanometer scale**

Authors : Dr. Lanti Yang, Dr. Robert van de Grampel, Dr. Olivier Guise
Affiliations : SABIC Technology & Innovation (T&I)

Resume : SABIC recently developed polycarbonate-polysiloxane (PC-siloxane) copolymer blends which offer improved chemical resistance and low temperature impact compared to polycarbonate (PC) homo-polymers. Another advantage of the PC-siloxane copolymer blends compared to PC-siloxane copolymer is the improvement in aesthetic properties, an important requirement for mobile phone housing applications. In PC-siloxane copolymer blends, siloxane forms nano-domains (10–50 nm) and is dispersed in the PC matrix. As the siloxane nano-domain size and the dispersion have a significant influence on the material properties, it is critical to develop quantitative morphology characterization of PC-siloxane copolymer blends to offer accurate measurements of siloxane nano-domain size. We present here the new methods developed for the quantitative morphology characterization of PC-siloxane copolymer blends using a combination of AFM and image analysis. High resolution AFM phase images clearly show that siloxane nano-domains are dispersed in the PC phase. With these AFM images siloxane nano-domains were quantitatively measured using image analysis software fully automated. The accuracy of the measurements was improved by optimizing the analysis process using statistical method. The method developments and results for accurate quantitative morphology characterization at nanometer scale will be presented. The results help to better understand the correlation between morphology and material properties.

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12:15 **The cone embedded multi-ring grooves structure for Tip Enhanced Raman Spectroscopy**

Authors : Ruei Han Jiang, Ryan Chu,Ta-Jen Yen
Affiliations : National Tsing Hua University, Guangfu Rd.Sec.2 No. 101, Hsinchu, Taiwan

Resume : In this work, we have employed a tip centered in a metallic plate surrounded by also a metallic grating structure and then excite the structure by a radial polarized light for the proposed structure. Herein, we combine the two effects to enhance the local field intensity near the tip: (1) The excitation and the propagation of the SPP mode through the grating structure, and (2) the localized surface plasmon at the nano-sized apex of the tip. Owing to the coupling between the SPR and LSPR, the intensity of E field will be greatly increased. This proposed tip would be applied in the tip enhanced Raman scattering (TERS) for about the enhancement factor of 1010 since signals from

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Raman scattering is proportional to the local field in the order of 4. Moreover, the proposed tip could also be applied in the field intensity of a refraction index sensor with the sensitivity up to 3.23×105. In addition, the concentric ring grating structure can be utilized to be a filter to remove the background noise to evaluate the S/N ratio of TERS measurement. In conclusion, we presented an alternative method to obtain high local field based on the apex of an aperture-less metallic tip without background signal. Finally, the high sensitivity for reflective index changes can be applied to application of the sensor.

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12:30 **Lunch break**

Traceability in Nano Metrology: Reference Samples and Calibration : Marie-Christine Lépy and Sabine Zakel

14:00 **Dimensional metrology at nanoscale for surface topography and analysis**

Authors : L. Koenders, U. Brand, I. Busch, T. Dziomba
Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany

Resume : The core of nanotechnology is the linkage of material properties with dimensions in the nanometer range. To achieve a combined knowledge about the properties that are decisive for the envisaged purpose of the nanotech product, it is mandatory to investigate a variety properties, typically geometrical as well as physical and analytical properties. This includes also the investigation of small forces to measure binding forces of molecules, adhesion or mechanical properties. Scanning probe microscopes (SPM) are the tools to study such properties at high lateral resolution. However, to achieve reliable and comparable measurement results, such SPMs need to be calibrated both in their dimensional (e.g. instrument axes) as well as force properties (e.g. cantilevers) by standards. Over the last years, calibration procedures have been investigated that now form the basis for guidelines and documentary standards together with measurement standards for dimensional calibration of instruments. Furthermore methods to calibrate the stiffness of SPM cantilevers have been developed in order to ensure traceable nanonewton force measurements. The need for suitable standards, their calibration and application for instrument calibration will be shown. To complete the information, also analytical techniques, like ESCA, AES, XPS, etc. are needed. Beside the sensitivity of such instruments, the lateral resolution of their analytical measurements is a key figure for the accuracy of analysis and imaging. Consequently, the reliable determination of lateral resolution is of great importance for the application of surface analytical methods. Examples of newly developed resolution standards for surface analytical techniques will be shown.

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14:30 **Towards a standardised metrology for multi-scale characterisation of nanostructured durable hydrophobic coatings.**

Authors : Damaso M De Bono, Geraldine Durand, Alan Taylor
Affiliations : TWI Ltd., Cambridge, Great Abington, CB21 6AL, UK.

Resume : Nanostructured coatings are gaining an increasing importance in critical applications such as anti-fouling, low friction and boundary layer control applications, due to their ability to tailor roughness levels and to modify the energy of the underlying surface. The sustainability of a coating is linked to its lifetime and limited durability is one of the main drawbacks for nanostructured coatings. To improve performance and durability of nanostructured coatings a greater understanding of the structure at the nano-scale and the influence of this on the macro-scale behaviour is needed. As confirmation of this need, international bodies dealing with metrology standardisation have recognised that one of the main barrier limiting a larger scale exploitation of nanostructured coatings is the lack of understanding and testing methodologies able to link the nanostructure features of the coating with the final macro-scale performance of the coating itself. This paper will illustrate the work that is being carried out by TWI during an ongoing FP7 collaborative project called NATURAL. In this project, a real-world practical characterisation method for nanostructured coatings will be developed, through the design and engineering of a portable inspection tool based on a laser system and able to evaluate the performance of a low surface energy coating, linking the topology of its nanostructure to the macro-scale tribological performance. The project will also be supported by a comprehensive assessment of the coating systems in terms of their functional, chemical,

H15 2

mechanical, tribological and morphological properties both at nano-scale and macro-scale, by using advances in testing methodologies and standardised existing techniques.

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14:45

Thin film reference samples for (sub-)ng quantification in XRF and TXRF analysis

Authors : Markus Krämer1, Burkhard Beckhoff2, Reiner Dietsch1, Gerald Falkenberg3, Ursula Fittschen4, Thomas Holz1, Philipp Hönicke2, Matthias Müller2, Daniela Rogler1, Rolf Simon5, Danny Weißbach1

Affiliations : 1 AXO DRESDEN GmbH, Winterbergstr. 28, 01277 Dresden, Germany (markus.kraemer@axo-dresden.de); 2 Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; 3 HASYLAB at DESY, Notkestr. 85, 22603 Hamburg, Germany; 4 Institute for Applied Chemistry, Univ. of Hamburg, Martin-Luther-King-Platz 6, 20146 Hamburg, Germany; 5 Institute for Synchrotron Radiation, KIT, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein, Germany

Resume : The high sensitivity of modern XRF/TXRF instruments calls for reference samples of very low mass fractions (ng/mm² or less) with homogeneous area distribution of the element(s). Here standard droplet samples reach their limits due to agglomeration and crystallization in the drying process. Therefore we developed multi-element reference samples as thin films produced by physical vapour deposition techniques common in multilayer production. These deposition techniques assure very homogeneous layers and flexibility in the choice of elements and mass densities. The samples were characterized by AAS, ICP-OES and µ-XRF in the lab and by µ-XRF at several synchrotron sources. Evaluation of micro-XRF mapping data showed that lateral mass deposition heterogeneities are below 1-2% (rms). Possible applications of these thin film samples are calibration of the energy scales of EDXRF instruments, assessment of lower detection limits, as well as characterization of depth resolution and sensitivity of confocal µ-beam set-ups. Further, TXRF reference samples with layer type depositions in the atomic monolayer and sub-monolayer range (<<1e15 atoms/cm²) were developed and tested to meet the demands of the lower limits of detection of TXRF in terms of technical feasibility. It could be shown by synchrotron-based grazing incidence XRF analysis with a fundamental parameters approach that homogeneous layer-like structures with a mass deposition far below the monolayer range can be fabricated.

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15:00

Selection and characterization of candidate reference materials for nanoparticle shape analysis

Authors : Tsvetelina Gerganova a, Gert Roebben a, Vikram Kestens a, Yannic Ramaye a, Thomas Linsinger a, Andrea Held a, Eveline Veleyesen b, Jan Mast b, Hendrik Emons a

Affiliations : a) European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Retieseweg 111, 2440 Geel, Belgium b) Veterinary and Agrochemical Research Centre (CODA-CERVA), Service Electron Microscopy, Groeselenberg 99, 1180 Brussels, Belgium

Resume : Manufactured nanomaterials are an important product of nanotechnology, and enable innovations in many other technology sectors. In order to better understand and use these materials, an increasing number of measurement methods are being used to characterise nanoscale objects. The measurements in the nanometer range are challenging, and their limited accuracy may lead to disputes between suppliers and users of the material, or between producers and regulators. To be better comparable, the nanoscale measurement results should be traceable to internationally accepted units of measurement which requires common, validated measurement methods and calibrated scientific instrumentation. An important tool for instrument calibration, method validation and method verification are reference materials, i.e. materials with known (where possible certified) specified properties. Most of the available nanoparticle-based reference materials are developed for use in nanoparticle size analysis. But there is also a significant need for reference materials for nanoparticle shape analysis. Aspect ratio is well known as critical shape parameter which together with the minimum and maximum Feret diameter is influencing functional properties of nanomaterials. Progress in this field depends on a reliable assessment of nanoparticle shape, which can be improved by the development of appropriate reference materials. We illustrate here main criteria for the selection of suitable candidate reference materials for nanoparticle shape measurements and their further characterization.

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15:15 **Measurement of fundamental parameters for characterization of nanomaterials**

Authors : Yves Ménesguen, Bruno Boyer & Marie-Christine Lépy
Affiliations : CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), F-91191 Gif-sur-Yvette, France

Resume : Reference-free analysis based on X-ray fluorescence (XRF) can be used to characterize innovative materials in terms of impurity checking, depth profiling or interfaces structure. Nevertheless, this technique requires accurate knowledge of the atomic parameters characterizing photon-material interactions, such as attenuation coefficients, fluorescence yields and transition probabilities. Tabulated parameters are available; however, experimental data are scarce with high associated uncertainties, especially in the low-energy range (E < 30 keV). New measurements using monochromatic radiation from the Metrology beam line of the SOLEIL synchrotron facility have been carried out to determine mass attenuation coefficients with relative uncertainties around 1 % for several materials [1]. Fluorescence yields have also been measured using dedicated experimental setup and previously determined mass attenuation coefficients [2]. New experimental data will be presented and made available for users on the LNHB web site. This improved knowledge of the fundamental parameters will allow developing more accurate XRF characterization techniques. [1] Y. Ménesguen and M.-C. Lépy, X-Ray Spectrometry 40 (2011) 411-416. [2] J. M. Sampaio, T. I. Madeira, J. P. Marques, F. Parente, A. M. Costa, P. Indelicato, J. P. Santos, M.-C. Lépy and Y. Ménesguen, Physical Review A 89, 012512 (2014).

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15:30 **Spectroelectrochemistry of plasmonic nanoparticles**

Authors : Christy F. Landes
Affiliations : Rice University Department of Chemistry, Houston TX 77005 USA

Resume : Despite attempts to understand the effect of nanoparticle surface charge density on catalytic activity, controversy still remains, likely because of the sample heterogeneity that is intrinsic to ensemble measurements. To overcome this problem, a direct correlation between electrochemical activity and nanoparticle morphology and surface chemistry on the single particle level is required. In this work, the intrinsic surface plasmon resonance of gold nanoparticles is exploited as a high sensitivity local reporter on local charge density. To achieve single particle spectro-electrochemical microscopy, we performed single particle dark-field scattering spectroscopy of 20 - 50 nm gold nanoparticles in an electrochemical cell and monitored the charging and discharging of single nanoparticles through changes in the surface plasmon resonance that sensitively depends on the nanoparticle electron density.

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15:45 **Anti-site defects and self-doping in spinel oxide thin films: quantification by resonant X-ray diffraction and X-ray spectroscopy**

Authors : Yezhou Shi, Paul F. Ndione, Linda Y. Lim, Dimosthenis Sokaras, Tsu-Chien Weng, Arpun R. Nagaraja, Andreas G. Karydas, John D. Perkins, Thomas O. Mason, David S. Ginley, and Michael F. Toney
Affiliations : 1. SLAC National Accelerator Laboratory, Menlo Park, CA 94025 (USA) 2. National Renewable Energy Laboratory, Golden, CO 80401 (USA) 3. Northwestern University, Evanston, IL 60208 (USA) 4. NCSR Demokritos, Institute of Nuclear Physics,GR-15310, Athens (Greece) 5. Nuclear Spectrometry and Applications Laboratory, IAEA Laboratories, Austria A-2444 Seibersdorf (Austria)

Resume : The accurate quantification of defects in nanomaterials is essential to understand their functional properties. We describe the combined use of resonant X-ray diffraction (REXD) and X-ray spectroscopies to precisely determine anti-site defect concentrations in functional Ga_2ZnO_4 and Cr_2MnO_4 spinel oxide thin films. Cation lattice site swapping (e.g., tetrahedral (Td) cations occupying octahedral (Oh) sites) creates these defects and gives rise to self doping, which can result in charge carriers (holes) and modest conductivities. REXD probes structural and chemical information and quantifies lattice site-specific cation occupancies and oxidation states – quantities difficult to study with conventional XRD. The REXD is complemented with X-ray absorption and emission spectroscopies (overall oxidation states), which are in good agreement with the REXD. For stoichiometric Ga_2ZnO_4 films, we find the anti-site defects are equal in concentration and electrically compensate each other. But for non-stoichiometric Cr_2MnO_4, excess Mn on the Td sites becomes electrically inactive as the Mn species switch from (III) to (II), resulting in an insulating film. This quantitative approach enables us to gain a better understanding of the nature of the anti-site defects and how they affect the electrical behavior of functional materials.

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16:00 **PLENARY SESSION**

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PROGRAM VIEW : 2014 Spring
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Symposium : H

ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

26 May 2014	27 May 2014	28 May 2014	**29 May 2014**	30 May 2014

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start at	Subject	Num.
	Characterization of Nano Materials with Advanced X-Ray Technologies 3 : Wolfgang Malzer and Emmanuel Nolot	
09:00	**Influence of annealing conditions on the structural and photoluminescence properties of Ge quantum dot lattices in continuous Ge + Al2O3 films** **Authors :** M. Buljan1, N. Radić1, I. Bogdanović-Radović1, Z. Siketić1, K. Salamon2, M. Jerčinović1, M. Ivanda1, G. Dražić3, S. Bernstorff4 **Affiliations :** 1 Ruđer Bošković Institute, Bijenička c. 54, 10000 Zagreb, Croatia; 2 Institute of Physics, Bijenička cesta 46 ,10000 Zagreb, Croatia; 3 Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia; 4 Elettra-Sincrotrone Trieste, 34149 Basovizza, Italy **Resume :** The structural and photoluminescence (PL) properties of regularly ordered lattices of Ge quantum dots (QDs) formed by self-assembly in thick films of Ge + Al2O3 mixtures were investigated. For this, we applied Grazing Incidence Small Angle X-ray Scattering (GISAXS), to study the synthesis and arrangement of QDs through the proper analysis and modeling of the corresponding 2D scattering pattern. Further, the samples were characterized also by complementary methods including transmission electron microscopy, x-ray diffraction and time of flight elastic recoil detection analysis. The effects of annealing at different temperatures and different annealing environments (vacuum and air) are studied. We have shown that the regular ordering of QDs remains unchanged for annealing up to 800 oC in vacuum, while it is preserved up to 700 oC when annealing is performed in air. The inner / crystalline structure of Ge QDs and their shape were found to depend on the annealing conditions. Crystalline grains of γ-Al2O3 form in the initially amorphous Al2O3 matrix after annealing at 800 oC. The PL spectra show two main contributions, one from the matrix and the other from Ge QDs. Both contributions depend strongly on the annealing conditions.	H16 1
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09:15	**Advanced structural characterisation of micro- and nano-electronics using neutrons and synchrotron X-rays** **Authors :** E.Capria 1, J.Beaucour 2, R.Kluender 3, E.Mitchell 1, J.C.Royer 3, J.Segura-Ruiz 2 **Affiliations :** 1 European Synchrotron Radiation Facility - Grenoble (France), 2 Institut Laue Langevin - Grenoble (France), 3 LETI - Grenoble (France). **Resume :** There is an increasing realisation worldwide that large-scale research infrastructures are a key component, not only of academic and fundamental research, but also of the innovation cycle and industrial research and development of micro- and nano-electronic devices and systems. However, working with industry has its own special requirements and research infrastructures are often based on an academic modus operandi which can limit their industrial impact. The IRT Nanoelec, is public-private partnership which comprises 18 public- and private-sector research partners - including the CEA-LETI (Laboratoire d'?lectronique des technologies de l'information), the ESRF (European Synchrotron Radiation Facility) and ILL (Institut Laue-Langevin). This platform, being created as a pathfinder project, it is focused on a core technology programme encompassing 3D assembly integration, nanophotonics on silicon and via technologies. This talk will depict the first results from this programme of industrial characterisation. Apart from the presentation of the main scientific results, emphasis will be given to the opportunities, limitations	H16 2

and viability of these characterisations, putting them into their economic context, driven by the industrial needs, and the valorisation of the products throughout their value chain. Finally, the perspectives for future developments, coherent with the electronic technology roadmaps and the actual main trends will be given.

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09:30 **Characterization of luminescent multilayered nanoperiodical structures containing Si nanocrystals by means of synchrotron X-ray absorption technique**

Authors : D.A. Koyuda *, S.Yu. Turishchev *, V.A. Terekhov*, E.V. Parinova *, D.E. Spirin *, D.N. Nesterov*, A.V. Ershov **, D.A. Grachev **, A.I. Mashin **, E.P. Domashevskaya *

Affiliations : * - Voronezh State University, Universitetskaya pl.1, 394006, Voronezh, Russia. ** - Lobachevsky State University of Nizhni Novgorod, pr. Gagarina 23, 603950, Nizhni Novgorod, Russia.

Resume : Multilayered nanoperiodical structures (MNS) of X/SiOx/.../X/SiOx/c-Si were studied by means of synchrotron X-ray absorption near edge structure (XANES) spectroscopy technique. As the "X" interlayer we used different wide gap materials: SiO_2, Al_2O_3 and ZrO_2. 1100 C annealing for MNS may lead to SiOx interlayer decomposition followed by silicon nanocrystals (ncSi) formation. It makes this kind of structures attractive because of visible photoluminescence property realization in framework of silicon technology. Another task here is the determination of the optimal interlayer type for size-limited ncSi formation due to possible interatomic interaction at layers boundaries under annealing. The MNS formation study necessitates the use of nondestructive methods sensitive to the surface structure, phase composition and local atomic surrounding of given sorts of atoms (here Si, Al and Zr). XANES technique satisfies all of these criteria. But the apparent disadvantage of XANES is fixed probing depth for each kind of spectrum type determined by excited core level. This can be avoided by taking different core levels for the spectrum excitation that is demonstrated in the present work. XANES spectra were obtained at the SRC synchrotron (USA). Probing depth was ~5 nm for L2,3 and ~70 nm for K XANES of Si. ncSi formation was demonstrated for all "X" types and interlayer interaction was studied. Al_2O_3/SiOx MNS showed the most significant ncSi formation.

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09:45 Coffee break

Joint session with symposium A - Metrology in solar cells and Highlights of EMRP projects : Daniel Abou-Ras and Burkhard Beckhoff

10:15 **Diffusion of buffer-layer and substrate impurities in solar-grade CIGSe and epitaxial CISe layers**

Authors : N. Stolwijk1, J. Bastek1, R. Wuerz2, S. Sadewasser3

Affiliations : 1Universitaet Muenster, Institut fuer Materialphysik, 48149 Muenster, 2Zentrum fuer Sonnenenergie- und Wasserstoff-Forschung Baden-Wuerttemberg, 70565 Stuttgart, 3International Iberian Nanotechnology Laboratory, 4715-330 Braga, Portugal

Resume : We investigate the diffusion properties of several technologically relevant impurities in polycrystalline $Cu(In,Ga)Se_2$ (CIGSe) structures using depth profiling of radiotracers and stable isotopes upon isothermal heat treatments. For comparison, similar experiments are carried out in single-crystal $CuInSe_2$ (CISe) layers epitaxially-grown on GaAs wafers. Pertinent results are obtained for the buffer-layer elements Cd [1] and Zn [2] as well as for the substrate elements Fe [3] and Na. Diffusion coefficients and activation energies are deduced in the temperature range from 200 ?C to 400 ?C from front-side penetration profiles of the radioisotopes Cd-109, Fe-59, Zn-65. The results for Fe and Zn are confirmed by the diffusion of the natural elements using in-depth SIMS analysis. Information on concentration levels and near-surface solubilities is obtained as well. Several anomalous features may relate to the presence of grain boundaries or grown-in defects. Zn-65 profiles in CIGSe exhibit double-hump shapes with a second maximum near the interface to the Mo contact layer. Similar observations are made for Zn in epitaxial CISe close to GaAs substrate. Experiments with the radiotracer Na-22 turn out to be difficult, because of its extremely fast transfer across the CIGSe film and the high solubility of Na in the glass substrate. Our results will be compared with data in the literature and discussed in terms of diffusion mechanisms. [1] K. Hiepko, J. Bastek, R. Schlesiger, G. Schmitz, R. Wuerz, N.A. Stolwijk, Appl. Phys. Lett. 99

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(2011) 234101 [2] J. Bastek, N.A. Stolwijk, R. Wuerz, A. Eicke, J. Albert, S. Sadewasser, Appl. Phys. Lett. 101 (2012) 074105 [3] N.A. Stolwijk, Sh. Obeidi, J. Bastek, R. Wuerz, A. Eicke, Appl. Phys. Lett. 96 (2010) 244101

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10:30 **The role of Ga content in CIGSe efficiency : an atom probe study** H17 2

Authors : Mohit Raghuwanshi, Emmanuel Cadel, Sébastien Duguay, Philippe Pareige, Nicolas Barreau
Affiliations : Groupe de Physique des Matériaux (GPM), University of Rouen; University of Nantes

Resume : Very high absorption coefficient and cost effective production makes Copper Indium Gallium Selenide (CIGS) semiconductor one of the most promising thin films photovoltaic device. With efficiency > 20%, it is currently the most efficient thin film solar cell produced. Na segregation along the Grain Boundaries (GBs) in polycrystalline CIGS is well known to improve efficiency. Efficiency of CIGS is very sensitive to Ga content in CIGS and is most efficient for $x \approx 0.25$. In the present study, GB nano-chemistry for various Ga-concentrated CIGSe samples (from Ga/In+Ga=0 to Ga/In+Ga = 1) has been investigated by Atom Probe Tomography (APT). APT provides 3D atomic mapping of elements in a material at sub nanometer resolution and can therefore accurately characterize the composition profile across GB in CIGS cells. APT has already put into evidence the modification of the GB chemistry at different stages of the CIGS-growth process suggesting an important role of the In/Cu-CIGSe-content on its final efficiency. In the present study, a clear influence of the Ga content over the GB chemistry was observed by APT. The results will be discussed in terms of cell efficiency and quantum efficiency.

add to my program **(close full abstract)**

10:45 **Strain measurements in CuInSe2 absorber layers by several diffraction techniques** H17 3

Authors : Norbert Schäfer1, Daniel Abou-Ras1, Manuela Klaus1, Christoph Genzel1, Julien Marquart1,2, Susan Schorr1,2, Thorsten Rissom1, Angus Wilkinson3, Tobias Schulli4
Affiliations : 1. Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin, Germany; 2. Freie Universitaet Berlin, Institute of Geological Sciences, Malteserstr. 74-100, 12249 Berlin, Germany; 3. Department of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, U.K.; 4. European Synchrotron Radiation Facility, BP 220, Grenoble Cedex, France

Resume : Thin-film solar cells based on CuInSe2 absorber layers, produced by co-evaporation, are subject to strain due to the evolution of the microstructure. For the investigation of the macrostrain and microstrain within the completed CuInSe2 thin films, electron and X-ray diffraction (XRD) measurements were performed. Since the ternary CuInSe2 layers do not exhibit substantial chemical gradients and a crystal structure with low pseudocubic distortion, this compound is suitable to compare different diffraction techniques applied at various scales. Also, coevaporated CuInSe2 thin films feature average grain sizes of typically more than 1 µm, which is feasible for X-ray microdiffraction within individual grains. While macroscopic XRD experiments were performed on a five-circle ETA diffractometer from GE Inspection Technologies, microdiffraction within individual grains was conducted at the ID01 Microdiffraction Imaging Beamline at the ESRF, Grenoble. In addition, information on strain was extracted by the Williamson-Hall method from XRD data acquired under grazing incidence using a PANalytical X'pert Pro diffractometer. Electron backscatter diffraction (EBSD) maps were measured using a Zeiss Ultra Plus scanning electron microscope equipped with a NordlysNano EBSD detector as well as using the Oxford Instruments AZtec acquisition and evaluation software. The recorded EBSD patterns were evaluated by means of the software CrossCourt (BLG Productions). Shifts in EBSD patterns can directly be correlated with strain distributions within individual grains. Based on a specific reference pattern, strain distributions can be calculated within individual grains with a spatial resolution of about 50 nm. The results on the strain distributions obtained by the different diffraction techniques were compared, also with respect to the relationships between macrostrain and microstrain. It is the aim to correlate the local strain distributions in CuInSe2 thin films to the optoelectronic properties of the corresponding solar cells obtained by means of electron-beam-induced current and cathodoluminescence measurements

add to my program **(close full abstract)**

11:00 **EMRP - Thin Films - Project: Traceable Raman mappings on solar cell thin-film materials** H17 4

Authors : S. Zakel, S. Wundrack, B. Güttler, R. Stosch
Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany
Resume : Micro-Raman spectrometry has become a meaningful method in thin-film analysis, but variation in the measurement conditions as well as a lack of standards for effective integration into the process are still factors which have to be overcome to enable the routine use of this technique. A 2D-Scan representing the spatially resolved scattering intensity is easily created from an individual vibrational mode, separating the object from the non-object areas. Here, well known structured reference samples were assessed for the uncertainty analysis of such segmented areas to provide traceability of the results to the meter. Real CIGSe samples were then investigated with regard to inhomogeneities resulting from the production process. In this context, data pretreatment is a major issue as mapping spectra often shows a low S/N ratio due to the limited integration time. As the optical resolution is diffraction limited, blurry edges influence the area determination. It is crucial for the quality of the segmentation as well as for the size of the segmented areas to choose the right threshold value by the assessment of the histogram. Traceability of the chemical composition was achieved for the CIGSe thin films via high purity reference compounds, their spectra being traceable to existing standards with regard to the instrument response and Wavenumber accuracy. This work is funded through the European Metrology Research Programme (EMRP).

add to my program **(close full abstract)**

11:15 **EMRP - Thin Films - Project: Reference-free quantification of in-depth matrix gradients – the uncertainty dependencies of the effective solid angle of detection**

H17 5

Authors : C. Streeck 1, C. Herzog 2, B. Kanngießer 2, B. Beckhoff 1
Affiliations : 1 Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin 2 Technische Universität Berlin, Hardenbergstr. 36, 10623 Berlin
Resume : Synchrotron-radiation based Grazing Incidence X-ray Fluorescence analysis (GIXRF) with varying excitation angles provides non-destructive access to the compositional depth profile of thin film matrix elements in the nano- and micrometer range. Reference-free GIXRF in conjunction with fundamental parameter based quantification allows for an analysis without the need for any calibration standards. This XRF-methodology can be used e.g. for the non-preparative determination of elemental depth gradients with an In to Ga gradient in $Cu(In,Ga)Se_2$ thin film solar cell absorber layers. As a key metrological aspect, the uncertainty of the components of the effective solid angle of detection and its impact on the uncertainty of the detected count rate will be presented: with varying angle of incidence the irradiated area on the sample changes over two order of magnitude, the Gaussian shape of the beam leads to an intensity distribution and the field of view of the detector is dependent on the distance from the sample. The uncertainty of all components shows a different angle dependency. Therefore, a detailed uncertainty analysis and their implication is prerequisite for a reliable calibration procedure.

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11:30 **5 minutes for transfer from symposium A lecture hall to symposium H lecture hall**

Highlights of European Metrology Research Programme (EMRP) Projects 2 : Andreas Hertwig and Beatrix Pollakowski

11:35 **EMRP - Thin Films - Project: Development of a facility for thermal conductivity measurement by modulated photothermal radiometry**

H18 1

Authors : Bruno Hay, Nolwenn Fleurence, Guillaume Davée
Affiliations : Laboratoire national de métrologie et d'essais (LNE) Photonic-Energetics Division Centre for Scientific and Industrial Metrology 29, avenue Roger Hennequin 78197 Trappes cedex
Resume : The understanding of the thermal behaviour of thin films, used for example in micro-electronics, solar cells and information storage, is essential to support new technological developments in these fields. Depending on the way they are deposited, thin films (from few nanometers to few micrometers thick) contain various degrees of disorder, which may influence their thermal conductivity. The thermal transport properties of thin films differ from those of bulk materials of similar chemical composition, due to their specific microstructure. It is therefore important to determine their thermal conductivity,

as well as the interface thermal resistances with the substrate on which they are deposited, under temperature conditions encountered in real applications. In the framework of the European project "Metrology for the manufacturing of thin films", LNE has developed a metrological facility based on modulated photothermal radiometry technique (PTR) for the thermal conductivity measurement of thin films up to 1000 °C. The method consists in illuminating the specimen front face by an intensity modulated laser beam and to detect the oscillating component of the temperature rise of the same face by means of an infrared detector. This metrological tool, directly traceable to the international system of units (SI), aims to improve the reliability and accuracy of this type of measurement at sub-micrometer scale. This paper presents a detailed description of the facility developed at LNE, and the first results obtained. This work was funded through the European Metrology Research Programme (EMRP) Project IND07 Thin Films. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

add to my program **(close full abstract)**

11:50 **EMRP - Thin Films - Project: Optical characterization of non-ideal samples** H18 2

Authors : D.M. Rosu1, A. Hertwig1, P. Petrik2, U. Beck1
Affiliations : 1BAM -Federal Institute for Materials Research and Testing, Unter den Eichen 87, 12200 Berlin, Germany; 2Research Centre for Natural Sciences – Institute for Technical Physics and Materials Science, Konkoly Thege Rd. 29-33, 1121 Budapest, Hungary

Resume : Consistent product quality is one of the main concerns nowadays in semiconductor industry and microelectronics. Therefore the development of techniques able to detect and inspect variations, defects and contaminations of devices is essential. A set of "real-world" samples with various non-idealities (thickness inhomogeneity, structured surface, non-stoichiometry) will be presented in the current work. These samples cover a range of materials relevant for semiconductor industry such as photoresist, structured silicon oxide thin layers, unusually thick silicon oxide layers, and silicon nitride. Spectroscopic ellipsometry in the visible range, mapping ellipsometry and IR spectroscopy were the investigation techniques used to obtain qualitative and quantitative information about our set of samples. The use of these ellipsometric methods is evaluated. Combined methodologies with e.g. X-ray measurement methods as complementary techniques are introduced. The discussion of these methodologies is focused on the possibilities for future development of reference samples and standard methods for calibrating optical surface measurement techniques.

add to my program **(close full abstract)**

12:05 **EMRP - Thin Films - Project: Polarization-encoded ellipsometry for large-area samples characterization** H18 3

Authors : Richard Koops, Petro Sonin, Omar El Gawhary
Affiliations : VSL, National Metrology Institute in the Netherlands Thijsseweg 11, 2629 JA Delft, the Netherlands

Resume : The characterisation of properties of materials through optical investigation means offers several appealing advantages compared to other competing techniques. First of all, at the common level of optical power used, such techniques are mostly not invasive and not destructive. Also, they are non-contact techniques which allows for a fast analysis of the sample of interest. Additionally, since electronic transitions in matter mainly fall in the visible range, the index of refraction of materials show a notable spectral diversity at optical frequencies, which increase the applicability of optical inspection methods and their sensitivity as well. In the last decades, ellipsometry and scatterometry have grown and developed in a way to become indispensable tools for the characterisation of physical properties of materials and their geometry. In this paper we will describe the design and realization of a compact polarization-encoded ellipsometer module that can be attached to a translation mechanism to expand the lateral measurement range up to meter level. The module is developed as part of the EMRP project IND07 ``Metrology for the manufacturing of thin films'' in order to implement traceable metrology tools for film thickness and refractive index measurements on large surfaces. Next to the actual realization and implementation, we will describe the modelling and the method used to retrieve the targeted material properties.

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12:20 **EMRP - Thin Films - Project: Characterization of thin films for thickness non-uniformity** H18 4

Authors : F. Manoocheri, S. Pourjamal, H. Mäntynen, P. Jaanson, E. Ikonen

Affiliations : Metrology Research Institute, Aalto University, P.O. Box 13000, FI-00076 Aalto, Finland
Resume : In this contribution, we present the characterization methods and the instrumentation for obtaining experimental results for the optical parameters and thickness profile of SiO2 thin-film samples. These results are based on spectral reflectance data at multiple sample spots and angles of incidence including 10, 30, and 56.4 degrees. The optical parameters of the SiO2 coatings derived from the reflectance results at various spots are compared with those determined from the near-normal and oblique incidence of the central spot of the samples. Preliminary results indicate that the characterizations are consistent and agree within 0.9 nm and 17 nm for the nominal film thicknesses of 300 and 6000 nm, respectively. The consistency among the determined optical parameters of the thin-film layers using a purpose-built gonioreflectometer and a commercial spectrophotometer used for these characterizations also confirms the accuracy of the spectrophotometric measurements. The effect of the systematic factors in the measurements is also discussed. The analysis of the determined refractive indices for each set of the reflectance data did not reveal any reasonable absorption in the SiO2 layer. The physical thicknesses of the layer derived from the oblique-incidence spectrophotometric data for several spots are compared. As in the case of the refractive indices, both physical and optical thicknesses yielded by the reflectance data agree within 0.9 nm.

add to my program (close full abstract)

12:35 **Lunch Break**

Highlights of European Metrology Research (EMRP) Programme Projects 3 : Fernando Araujo de Castro and Wolfgang Unger

14:05 **EMRP - Thin Films - Project: Chemical analysis of nano-scaled materials by x-ray spectrometry under grazing incidence condition**

Authors : R. Unterumsberger1, B. Pollakowski1, Christiane Becker2, Marcel Pagels3, Carolin Zachäus2 , Birgit Kanngießer3, B. Beckhoff1, Bernd Rech2
Affiliations : 1 Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany 2 Helmholtz-Zentrum für Materialien und Energie, Institut Silizium Photovoltaik, Kekuléstr. 5, 12489 Berlin, Germany 3 Technische Universität Berlin, Institut für Optik und Atomare Physik, Analytische Röntgenphysik, Hardenbergstr. 36, 10623 Berlin, Germany
Resume : The development of improved characteristics of functional nanoscaled devices involves novel materials, more complex structures and advanced technological processes, requiring analytical methods to be well adapted to the nanoscale. Thus, non-destructive and non-preparative techniques for chemical nanometrology providing sufficient sensitivity, reliable quantification and high information depth dynamics to reveal interfacial properties are needed for an interfacial analysis. Appropriate measurement strategies adapted to a nanoscaled stratified sample enables the combined technique of Near-Edge X-ray Absorption Fine Structure (NEXAFS) and Grazing Incidence X-ray Fluorescence (GIXRF) to provide interfacial species information. GIXRF-NEXAFS is a non-destructive and non-preparative technique which has the advantage that the interfacial chemical bonds remain unchanged by the measurement [1]. By means of two examples the methodology will demonstrated. In particular high-temperature processed polycrystalline silicon thin-film solar cells were analyzed focusing on the interface between absorber and the transparent conductive oxide material [2]. A reliable depth-resolving analysis of the elemental composition and chemical species close to the poly-Si/(SiN)/ZnO:Al interface was carried out by using the combined method GIXRF-NEXAFS. References: [1] B. Pollakowski et al., Anal. Chem. 85, 193 (2013), [2] Ch. Becker et. al., J. Appl. Phys. 113, 044519 (2013)

H19
1

add to my program (close full abstract)

14:20 **EMRP - Thin Films - Project: Probing organic semiconductor microstructures by Raman Spectroscopy**

Authors : Dr Ji-Seon Kim
Affiliations : Department of Physics & Centre for Plastic Electronics, Imperial College London, UK

H19
2

Resume : Organic semiconductors such as small molecules and conjugated polymers have been demonstrated as the active material in light-emitting

diodes, transistors and photovoltaic cells. The main challenge still remains to find a way of controlling molecules from a solution while maintaining their optical and electrical functionalities, and moreover probing these molecules in thin films and devices. Here, I will discuss the key advances on our fundamental understanding of the structure-property relationships of organic semiconductors with a particular focus on the effects of molecular order and thin film morphology. As one of the most valuable structural probes for organic semiconductors, we have extensively used Raman spectroscopy [1-6]. Raman spectroscopy is a non-destructive technique which delivers valuable chemical/ structural information together with optical properties of materials with sub-micrometer spatial resolution from surface films as well as from buried layers in the devices. I will show some examples of our Raman studies performed on various organic microstructures in thin films and devices. The application of non-resonant, resonant and polarized Raman spectroscopy to the characterisation of reaction, composition, crystallinity and orientation of molecules will be discussed. References [1] James et al., ACS Nano (2013) http://dx.doi.org/10.1021/nn403073d [2] Wood et al., J. Chem. Phys. (2013) DOI: 10.1063/1.4816706 [3] Tsoi et al., ACS Nano, 6(11), (2012), 9646-9656. [4] Tsoi et al., J. Am. Chem. Soc., 133, (2011), 9834-9843 [5] James et al., ACS Nano 5 (12), (2011), 9824-9835 [6] Kim et al., J. Am. Chem. Soc., 130, (2008), 13120-13131

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14:35 **EMRP - Thin Films - Project: Development of optical metrology for thin films** H19 3

Authors : P. Petrik1, B. Pollakowski2, S. Zakel4, A. Nutsch3, G. Roeder4, T. Gumprecht4, B. Fodor1,5, E. Agocs, G. Juhasz1, O. Polgar1, C. Major1, Z. Labadi1, Z. Baji1, M. P. M. Jank4, M. Schellenberger4, B. Beckhoff2, M. Fried1

Affiliations : 1Institute for Technical Physics & Materials Science (MFA), Research Centre for Natural Sciences, Konkoly Thege u. 29-33, 1121 Budapest, Hungary 2Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany 3Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany 4Fraunhofer Institute for Integrated Systems and Device Technology IISB, Schottkystrasse 10, 91058 Erlangen, Germany 5Faculty of Science, University of Pécs, 7624 Pécs, Ifjúság útja 6, Hungary

Resume : With shrinking device dimensions and layer thicknesses, there is an increasing need for the accurate measurement of thin and ultrathin films in a broad range of key technologies including biology, semiconductor device technology, photonics, photovoltaics, and sensor technology. The major challenge of optical ultrathin film metrology is the appropriatemodeling of interfaces, as well as of potential lateral and vertical layer inhomogeneity of the optical properties. For the determination and interpretation of optical properties of layers with thicknesses of several nanometers, even surface structures or interface roughness at nanometer scale play a crucial role. The way the interfaces are modeled also influences the determined bulk optical properties of the thin films to a large extent. Organic surface contamination has been revealed by vacuum ultraviolet reflectometry. Lateral inhomogeneity is a major concern whenever a comparative study is preformed involving different methods. For a reliable study, the lateral homogeneity has to be carefully checked and taken into account. The vertical inhomogeneity of the layer has a similar importance in comparative measurements if the information depths of the applied methods are different. A further problem with ultrathin films is that their optical properties are different from bulk values, and their dispersion needs to be modeled. We show for the example of ZnO that modeling of the dielectric function is a complex task, especially for photon energies around the band gap and at critical points. Around the critical point energies, the penetration depth of light strongly depends on the wavelength, which allows a depth scan by changing the wavelength, but also sets limitations in terms of film thickness, and requires proper modeling of the vertical inhomogeneity.

add to my program **(close full abstract)**

14:50 **EMRP - Thin Films - Project: Traceable water vapour measurements to underpin the development of barrier layer technology** H19 4

Authors : Valerio Ferracci, Paul Brewer

Affiliations : National Physical Laboratory

Resume : Flexible organic electronics offer an attractive opportunity for the semiconductor industry; however, the performance and lifetime of these devices is critically affected by the ingress of water vapour. Barrier films have been developed to reduce the transport of water into the operational parts of the device; the efficacy of such barrier layers is measured in terms of their water vapour transmission rate (WVTR). A range of different techniques are currently

used to measure the WVTR of barrier layers, which include the calcium test, radioactive methods with tritiated water or most commonly, coulometric methods (ASTM F1249). However, as the performance of barrier layers improves, more sensitive techniques are required. NPL has developed a method for providing a traceable measurement of WVTR by means of cavity ring-down infrared spectroscopy with a detection limit of < 5 × 10−5 g m−2 day−1. It has been used to characterise a range of high-performance multi-layer and single layer barrier materials. This work has enabled several fabrication processes to be optimised. The capability has also been used to study the influence of defects in the barrier material on performance.

add to my program **(close full abstract)**

15:05 **EMRP - Q-AIMDS - Project: Development and characterization of model samples systems for improved surface chemical analysis of medical devices.** H19 5

Authors : B. J. Tyler, R. T. Steven, S. J. Spencer, A. Shard
Affiliations : National Physical Laboratory, UK

Resume : Roughly 1 in 5 European adults have a permanent implanted medical device such as an artificial joint, cardiovascular stent, or intraocular lens. Because surface contaminants and defects can lead to life threatening device failures, the industry is facing increasingly stringent regulatory requirements for surface characterization. Meeting these demands is a daunting challenge. The well-established surface chemical characterization techniques are poorly suited to high throughput analysis in the manufacturing environment. Raman and ambient mass spectrometry are promising but the underpinning metrology needed to provide the required reproducibility and traceability is lacking. The goal of this project is to develop the protocols, standards, and techniques needed to support the device industry in meeting these requirements. We are developing a series of well-defined model systems which include thin films of common contaminants on metal and polymeric substrates. Protocols have been developed for producing nano-scale films of ethylene-bis-stearamide and sodium dodecyl-sulphate on polyethylene, titanium, 316 stainless steel and silicon. The uniformity, reproducibility and stability of these model systems has been investigated with ellipsometry, AFM, ToF-SIMS and XPS. Details of the characterization of these samples as well as recommendations for using these systems to improve reproducibility and quantitation of ambient surface analysis methods will be presented.

add to my program **(close full abstract)**

15:20 **EMRP - Q-AIMDS - Project: Reference samples for quantification of trace elements in biomaterials by solid state spectroscopy** H19 6

Authors : Jan Thieleke, Anja Dreyer, Carla Vogt
Affiliations : Leibniz University Hannover, Institute for Inorganic Chemistry, Callinstr. 9, 30167 Hannover, Germany, vogt@acc.uni-hannover.de

Resume : Elemental analysis with high lateral resolution in biomaterials is important to gain information about biochemical pathways, nutrition status or enrichment or depletion of certain elements in organs as result of diseases or implant degradation. For the calibration of trace metal concentrations and determination of their distribution in biomaterials matrix matched standards are preferred which are often not commercially available. Matrix matched biomaterial standards could be prepared from agarose polymers with adapted concentrations of water and carbon, or homogenized sample material with originally low background values for the elements under investigation which were afterwards doped with the elements of interest. In addition to these two methods in this study also the preparation of films from chitosan biopolymer and of acrylate lacquer polymers was included. In this work we optimized synthetical parameters to produce reference materials with wide variations in concentration, elemental distribution and homogeneity, species and oxidation states of the elements under investigation (Al, Mg, Fe, Cu, Zn). Applications will be shown for LA-ICP-MS, SIMS and 3D-XRF measurements. In addition conditions for a matrix -independent calibration by LA-ICP-MS have been investigated. For this samples with agarose, root material, chitosan and acrylate lacquer were prepared which contained different contents of carbon, water and the analytes. Results will be presented for different calibration strategies applied for the determination of trace elements in root and bone sections. The results show that quantification in biomaterials is possible with determination coefficients > 0.99 regardless of the sample preparation and sample matrix.

add to my program **(close full abstract)**

15:35 **EMRP - Q-AIMDS – Project: Characterization of advanced biomaterials for the medical device industry using Synchrotron Radiation based FTIR microspectroscopy** H19 7

Authors : Andrea Hornemann, Kelim Vano Herrera, Carla Vogt, Bonnie Tyler, Arne Hoehl, Peggy Emmer, Burkhard Beckhoff
Affiliations : Physikalisch Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; Leibnitz University Hannover, Callinstr. 9, 30167 Hannover, Germany; Leibnitz University Hannover, Callinstr. 9, 30167 Hannover, Germany; National Physics Laboratory, Hampton Road, Teddington TW11 0LW, United Kingdom; Physikalisch Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; Physikalisch Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany; Physikalisch Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany;
Resume : Since its early development implantable medical devices had tremendous impact on the quality of life. But still today many implants cause inflammations due to incompatibility with human tissues or due to device related infections. Hence, there is a need for exploring new advanced biomaterials that comprise thin film coatings, surface grafted biomolecules, nanoparticle coatings, drug eluting films, and especially bioresorbable components. Reliable metrological tools for the rapid characterization of medical devices are essential to probe their physicochemical properties. Both vacuum-based and ambient techniques can successfully characterize surface layers, contaminants on the surface of medical devices in the manufacturing environment, and enable the detection of defects and chemical constituents in the near-surface region. The identification of thin organic surface layers at solid state/organic interfaces, being relevant for the manufacturing device industry, is addressed by Synchrotron Radiation based FTIR microspectroscopy as a qualified ambient technique. The model systems studied are classified into three major categories: surface contaminants, protein films and polymer thin films. In addition to the geometric challenges, device development and failure analysis often requires the analysis of explanted devices and biological tissues. Therefore, our systematic analysis focuses on 'real production line' medical devices, i.e. stent systems in/outside biological interfaces.

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15:50 **Coffee Break**

Highlights of European Metrology Research Programme (EMRP) Projects 4 : Bonnie Tyler and Carla Vogt

16:20 **EMRP - Q-AIMDS - Project: Surface analysis of biomedical device materials by Raman Spectroscopy**

H20 1

Authors : A.M. Giovannozzi*, C. Portesi**, A.L. Hook***, M.R. Alexander***, R.T. Steven****, B. Tyler****, S. Pascale***** and A.M. Rossi*
Affiliations : A.M. Giovannozzi*; A.M. Rossi* Thermodynamic Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce, 91 10135, Torino, Italy C. Portesi** Electromagnetism Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce, 91 10135, Torino, Italy A.L. Hook***; M.R. Alexander*** Laboratory of Biophysics and Surface Analysis University of Nottingham Nottingham, UK R. Stevens****; B. Tyler**** Surface & Nanoanalysis National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK S.Pascale***** Cardiac Surgery B.U. - Heart Valve Sorin Group Italia S.r.l. Strada per Crescentino,13040 SALUGGIA (VC), Italy
Resume : The global medical device industry is estimated at over 200 billion € annually and European manufacturers currently hold 35 % of the market. Although implantable medical devices improve quality of life for millions of people, the rates of complications and failures due to incompatibility of the devices with human tissues and device related infections are unacceptably high. Medical device companies and biomaterials scientists have developed a range of novel materials that may reduce complications and failures of implanted devices by using surface treatments, thin film over-layers and drug eluting coatings1. Manufacture and certification of devices that employ these advanced biomaterials will require traceable, reliable metrology tools that are able to measure surface layers, surface contaminants, defects and the 3-D distribution of chemical constituents in the near-surface region. Although the established surface analysis methods, such as XPS and ToF-SIMS, have demonstrated their value for the development of these advanced biomaterials2, 3, they are very poorly suited for routine monitoring of medical devices because they must be operated in high ultra-high vacuum, often require special sample preparation, have a slow turn-around and are very expensive. Furthermore, these methods are very poorly suited for in-line analysis in the manufacturing environment. They are also generally incapable of handling the complex geometry of complete

medical devices. Emerging ambient techniques that employ either optical spectroscopy or mass spectrometry are far better suited to the manufacturing environment but at this time these techniques lack the reproducibility, traceability and standard materials needed for the characterisation of advanced biomaterials. Recent advances in micro-Raman spectroscopy offer great promise for meeting the medical device industry needs for in-line surface quality assessment4. In addition to providing detailed chemical information, Raman is a non-contact, non-destructive chemical analysis tool well suited to in-line analysis. The aim of this study is to develop the fundamental metrology needed to provide robust, reproducible, surface sensitive analysis of medical device materials using micro-Raman spectroscopy. This technique was used to perform chemical analysis of the molecular composition of surface layers of some model and real systems. Traceable model systems for relevant surface treatments and contaminants were used for the quantification and quality assurance of medical device surface chemistry, and they are representative of key issues expressed by industrial stakeholders. In particular, Raman analysis was focused on the chemical characterisation of antibacterial polymers films onto silicone catheters, whose in vivo efficacy has been previously demonstrated5, 6, and on N′,N′-ethylene bis(stearamide) films on Silicon and HDPE (high density polyethylene), which is a common contaminant in biomedical devices fabrication7. Surface analysis of films with different thicknesses (4, 0.2 and 0.1 μm for antibacterial polymers and 100, 50 and 10 nm for stearamide coating) was conducted in order to check the homogeneity of the films, the analysis reproducibility and the technique sensitivity. Raman mapping characterisation will also be performed in order to evaluate the presence of defects or other contaminants which might induce polymer delamination. A preliminary study on a real system biomedical device provided by Sorin group S.r.l., i.e. heart valve Perceval S, that is formed by functional component in bovine pericardium fixed on a metal stent made of shape memory alloy, will be also shown. Raman characterisation will be exploited for the analysis of the anticalcification molecule on the valve. Further studies using a surface enhanced Raman spectroscopy (SERS) approach will be conducted in order to detect alcohols or amino-acids in solution after the fixing of the anticalcification molecule on the pericardium in order to increase the technique sensitivity. References 1. Hetrick, E.M., Schoenfisch, M.H.. Chemical Society Reviews 2006, 35, 780. 2. Tilinin, I.S., Jablonski, A., Werner, W.S.M. Progress In Surface Science, 1996, 52, 193. 3. Belu, A.M., Graham, D.J., Castner, D.G. Biomaterials, 2003, 24, 3635. 4. Challa S. S. R. Kumar, Raman Spectroscopy for Nanomaterials Characterization, Springer-Verlag Berlin Heidelberg 2012. 5. Hook, A.L., Chang, C.Y., Yang J., Atkinson S., Langer R., Anderson D.G., Davies M.C., Williams P., Alexander M.R. Advanced Materials 2011, 25, 2542. 6. Hook A.L., Chang C.Y., Yang J., Luckett J., Cockayne A., Atkinson S., Mei Y., Bayston R., Irvine D.J., Langer R., Anderson D.G., Williams P., Davies M.C., Alexander M.R. Nature Biotechnology 2012, 10, 868. 7. Cai, Y.H. Asian Journal of Chemistry, 2013, 25, 6219.

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16:35

EMRP - TReND - Project: Self-assembled diblock copolymers as reference model systems for metrology and surface analysis.

Authors : L. Boarino 1, E. Enrico 1, N. De Leo 1, G. Aprile 1, M. Perego 2, F. Ferraresi Lupi 2, M. Laus 3, K. Sparnacci 3, I. Gilmore 4, R. Havelund 4, A. Sesis 4, and F. Castro 4
Affiliations : 1 NanoFacility Piemonte, Electromagnetism Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce 91 - 10135 Torino (I); 2 Laboratorio MDM, IMM-CNR, Via C. Olivetti 2, 20846 Agrate Brianza (MB), (I); 3 DiSIT, Università del Piemonte Orientale, viale T. Michel 11, 15121 Alessandria (I); 4 Materials Division National Physical Laboratory, Hampton Rd, Teddington, UK;

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Resume : In recent years, an increasing interest was addressed to self-assembly processes at the nanoscale. Diblock copolymers, under proper conditions, are able to self-organize into ordered nanostructures. Depending on their composition and molar mass characteristics lamellae, cylinders and gyroids morphologies featuring typical size in the 50 to 10 nanometer range are obtained. As indicated by International Technology Roadmap of Semiconductors, the directed self-assembly based on diblock copolymers (BCP) could reveal as an alternative to immersion extreme UV lithography for the 32 nanometer node. In this respect, detailed structural information are very important for these systems to exploit their full technological potential. The state-of-the-art in 3D structural analysis is through the combination of high-resolution microscopies, such as electron microscopies and AFM, with diffraction techniques such as GISAXS. Recent developments of photoconductive-AFM (PC-AFM) allow for current maps of the surface and near surface with resolutions of about 30 nm. SIMS, combined with sputtering with argon clusters, has become a powerful

technique for 3D molecular imaging. The goal of this work is to provide different sample models based on self-assembled polystyrene nanospheres and PS-r-PMMA diblock copolymers for PC-AFM and SIMS in order to develop novel quantitative methods for direct subsurface nanoelectrical measurement.

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16:50 **EMRP - TReND - Project: Comparison of lab-based and synchrotron-based quantification of Al in thin ALD Al2O3 films**

Authors : B. Detlefs ,E. Nolot , H. Fontaine, T. Lardin, A. B. Fadjie Djomkam, M. Mueller, P. Hoenicke, B. Beckhoff
Affiliations : CEA-LETI, 17 rue des Martyrs, 38054 Grenoble, France; Physikalisch-Technische Bundesanstalt, Abbestraße 2- 12, 10587 Berlin, Germany

Resume : Use of the atomic layer deposition (ALD) technique for fabrication of nanoelectronics devices requires an accurate characterization of the deposited thin layers. Here we present quantification of Al in thin ALD Al2O3 films and we compare analysis performed by synchrotron-based TXRF/GIXRF at PTB-BESSY II, measurement with a standard lab-based TXRF setup at CEA-LETI and LPD-ICPMS. The comparison's emphasis is on the dose calculation, limits of detection and various background contributions that influence the precision of the quantification. Both lab-based techniques use calibration standards contrary to the reference-free synchrotron-based XRF. Advantages and shortcomings of each of the method are discussed in the case of Al quantification.

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EMRP Tutorials: Characterization of Nano Materials 1 : Blanka Detlefs and Andreas Nutsch

17:05 **EMRP - Nanostrain - Tutorial: Focused Ion Beam machining for site-specific nanocharacterization - advances and drawbacks**

Authors : Werner Österle, Nicole Wollschläger
Affiliations : BAM Federal Institute for Materials Research and Testing

Resume : An increasing demand for site-specific nanocharacterization evolves from the following reasons: Firstly, since devices become smaller and smaller, material characterization on the nanometre scale becomes increasingly important. Secondly, not so obvious but important as well: Even for conventional technologies microstructural changes at critical sites are not well realized and understood yet. Site-specific nanocharacterization is based on the combination of three microscopic techniques: Scanning electron microscopy (SEM), focused ion beam machining (FIB) and analytical transmission electron microscopy (AnaTEM). The tutorial will be organized in three parts. The first part will descibe the method and its merits by presenting a number of case studies in which several technical and scientific problems could be solved. The scope involves shear banding in metals, functional coatings and metrology of multi-layer thin film structures. The second part highlights the problems which may occur due to the interaction of the ion beam with the materials under investigation. It will be pointed out how these features can be distinguished from "real" structures. In the third part a parameter study of ion implantation into a piezoelectric thin film (PZT) will be presented. The latter study is part of the recently launched EMRP-project "Nanostrain" which aims at supporting the development of nanoscale piezoelectrics by applying novel characterization techniques at the nanometre scale

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17:45 **EMRP - Nanostrain - Tutorial: Advanced XRD methodologies**

Authors : Thomas Hase
Affiliations : University of Warwick

Resume : X-ray diffraction is a widely used metrology tool applied to a wide range of crystalline and polycrystalline materials. The position of diffraction peaks is proportional to the long-range periodicity of the crystal. As such, the inter-atomic distances can be determined to high precision and the strain state of the material determined. In this tutorial presentation we will discuss the appropriate tools to determine the strain in a range of crystalline materials ranging from bulk crystals to thin films and multilayers. The different experimental geometries that can be exploited on both laboratory and synchrotron sources will be highlighted with topical examples. The tutorial will concentrate on the basics of the techniques however the advanced methodologies required for accurate and precise measurements of nanoscaled structures will be highlighted. As part of the tutorial, the realistic measurement limits and precision for the determination of strain, particle size and strain

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dispersion will be discussed. The tutorial will not assume any in-depth X-ray knowledge, but will highlight the current methodologies being implemented to study samples under the influence of external stimuli such as magnetic and electric fields.

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PROGRAM VIEW : 2014 Spring
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Symposium : H

ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials

26 May 2014 | 27 May 2014 | 28 May 2014 | 29 May 2014 | **30 May 2014**

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	EMRP Tutorials: Characterization of Nano Materials 2 : Birgit Kanngießer and Petr Klapetek	
08:30	**EMRP - Nanostrain - Tutorial: Characterization of Si-based nanostructures by near-field imaging and nano-FTIR spectroscopy** **Authors :** Peter Hermann, Arne Hoehl, Bernd Kaestner, Gerhard Ulm, Burkhard Beckhoff **Affiliations :** Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany **Resume :** Conventional optical methods such as infrared and Raman micro-spectroscopy are non-destructive and highly sensitive to chemical and structural properties of matter. The achievable lateral resolution of these techniques is however limited by optical diffraction, thus preventing the investigation of structures on a nanoscale. This limitation can be circumvented by applying near-field based approaches where a near-field probe e.g. a metal coated tip is brought into close proximity to the sample surface. The irradiation of such a scanning near-field probe by a focused photon beam leads under optimized conditions to a strong electric field enhancement around the tip apex which improves the lateral resolution and increases the sensitivity for spectroscopic investigations. Aim of this tutorial is to provide a brief introduction and to demonstrate the capability of near-field techniques for the characterization of different silicon-based samples by high-resolution imaging and spectroscopy. Additionally, the use of broadband synchrotron radiation for performing nano-FTIR measurements is discussed in detail.	H22 1
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09:00	**EMRP - Q-AIMDS - Tutorial: Ambient mass spectrometry for surface characterization of medical devices.** **Authors :** B. J. Tyler, T.L. Salter, I.S. Gilmore **Affiliations :** National Physical Laboratory, UK **Resume :** Ambient mass spectrometry (AMS) is a powerful and rapidly growing field that provides high sensitivity mass spectrometry directly from surfaces at ambient pressure with minimal sample preparation. The number of AMS techniques is growing rapidly and includes Desorption Electrospray Ionization (DESI) and Plasma Assisted Desorption Ionization (PADI) among many others. AMS has been successfully used to study explosives, pharmaceutical tablets, drugs of abuse, metabolites, surface contamination and fingerprint identification. These techniques offer high speed and minimal sample preparation and are readily adapted to analysis of complex geometries, which makes them highly promising for routine surface analysis in the manufacture of medical devices. Although AMS is very promising, the underpinning metrology of AMS must be better understood to improve the robustness and reliability of the. NPL is involved in inter-laboratory research efforts in the fundamentals of AMS in order improve the reproducibility and robustness of the techniques. This tutorial will provide an introduction to AMS, including an overview of existing methods and details of instrumentation and analysis procedures. The state of the art in reproducibility, sensitivity and data interpretation will be presented along with example applications that will illustrate the potential of ambient mass spectrometry for a range of applications including medical device, forensic, personal care and biological analysis.	H22 2
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09:30 **EMRP - Thin Films and Q-AIMDS - Tutorial: Quantitative X-ray fluorescence analysis under grazing incidence condition for stratified materials based on X-ray Standing Wave field calculations**

Authors : B. Pollakowski, J. Eilbracht, B. Beckhoff
Affiliations : Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany

Resume : Many functional nanoscaled devices have a complex structure consisting of several thin layers. For a quantitative analysis of the elemental composition of this kind of specimens X-ray spectrometry under grazing incidence conditions is a beneficial method. Under these conditions interference effects of the incoming and the reflected radiation occur at smooth interfaces of stratified materials. The related spatial intensity distribution has a nanoscaled periodicity and is named the X-ray Standing Wave (XSW) field. The mean penetration depth can be tuned to a buried nanostructure of interest. The information depth follows the penetration depth that can be adapted by changing either the angle of incidence or the photon energy. The information depth can vary from a few to several hundreds of nanometers. Quantitative GIXRF leads to most reliable results when the actual XSW field intensity is taking into account the excitation conditions determined by both the angle of incidence and the incident photon energy. By means of a nanolayered specimen consisting of light elements and transition metals the XSW based GIXRF quantification procedure is demonstrated and compared to other approaches.

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10:00 Coffee Break

EMRP Tutorials: Characterization of Nano Materials 3 : Marie-Christine Lépy and Burkhard Beckhoff

10:30 **EMRP - Thin Films - Tutorial: Metrology for quantitative Raman mapping evaluation**

Authors : R. Stosch, C. Frank, B. Güttler, S. Pape, S. Wundrack, S. Zakel
Affiliations : Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany

Resume : Two dimensional (2D) Raman mapping is a versatile spectroscopic tool that enables chemical (composition, stoichiometry) and mechanical (stress) properties of a surface to be measured with sub-µm spatial resolution. While the identification of compounds from characteristic spectral patterns relies on the determination of Raman band positions, the stoichiometry of a surface is derived from particular band intensities or intensitiy ratios. Therefore, accurate calibration of the Raman instrument with respect to wavenumber shift and instrument response is an indispensable prerequisite for a reliable data analysis. This tutorial provides an overviev of reference standards and calibration schemes used for both Raman shift and instrument response calibration. A step-by-step guide is presented on how a two-point Raman shift calibration is carried out using sulfur and polystyrene as Raman shift standards and how the response function of a dispersive Raman instrument is measured with the help of a traceable white light source approximated by a tungsten lamp. It is further demonstrated that traceability of Raman maps to SI units of length can be established by comparison with data obtained from a 2D Raman reference standard developed at PTB and how image processing tools and procedures are applied to Raman maps for the reliable determination of percent surface coverage including measurement uncertainties, e. g. for particular chemical compounds, crystalline phases or sum of impurities.

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11:00 **EMRP - TReND - Tutorial: 3D secondary ion mass spectrometry imaging of organic materials**

Authors : R. Havelund
Affiliations : National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK

Resume : SIMS has become a powerful technique for the label-free analysis of organics from cells to electronic devices. The development of cluster ion sources has revolutionised the field, increasing the sensitivity for organics by two or three orders of magnitude and for large clusters, such as C60 and argon clusters, allowing depth proiling of organics. The latter has provided the capability to generate stunning 3 dimensional images with depth resolutions of around 5 nm, simply unavailable by other techniques. In this tutorial, we show the fundamentals that allow 3D molecular imaging and the optimisation for state

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-of-the-art performance. We also highlight the capability with examples from molecular and polymer devices, including organic photovoltaic devices.

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11:45 **EMRP - TReND - Tutorial: Synchrotron Radiation based X-Ray Spectrometry for nanoscaled materials**

Authors : Matthias Müller, Philipp Hönicke
Affiliations : Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587, Berlin, Germany

Resume : The tutorial will focus on the quantitative analysis of layered materials for nanoelectronics and of surface contaminations on semiconductor substrates by X-ray spectrometry. In general, reference materials and calibration standards are used to achieve quantitative results. In Total Reflection X-Ray Fluorescence analysis (TXRF) dried droplets are commonly used for calibration. It will be demonstrated that such calibration standards are often not appropriate for a reliable characterization of ultra-trace contaminations [1]. Reference-free TXRF quantification [2] based on radiometrically calibrated instrumentation will be presented. The availability of appropriate reference materials and calibration standards for the reliable analysis of nanolayered systems is rather limited and cannot cover the increasing variety of layer and substrate materials. Reference-free grazing incidence XRF including X-ray standing wave field calculations or experimental XRR data can serve as a self-contained method to quantitatively analyze nanolayered systems. This will be demonstrated by several systems for novel gate stacks including high-k and interface passivation layers. Furthermore, the chemical speciation of a passivated interface buried below a several nanometer thick layer by X-ray absorption spectroscopy will be presented [3]. [1] M. Müller et al., Solid State Phenom. (2012) 187, 291 [2] B. Beckhoff et al., Anal. Chem. (2007) 79, 7873 [3] M. Müller et al., Solid State Phenom. (2013) 195

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12:30 Lunch Break

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