

2010 Q: Quantitative electron microscopy for research and industry

This symposium aims at providing a forum for researchers interested in applying quantitative methods of electron microscopy and spectroscopy to materials research in the different technology fields, such as electronics, optics, magnetics, energy and environment, engineered materials, nanosystems, soft matter and bioscience. Current topics will be highlighted in keynote presentations given by leading invited experts.

Scope:

Materials research on thin films, bulk materials, surfaces, materials at the nanoscale and at the interface between physical and life sciences is of prevailing interest because of its fundamental importance in understanding the chemical and physical properties of materials and in evaluating their potential for technological applications. With the emergence of new electron optical components for energy filtering and aberration correction and the availability of improved software for data acquisition and analysis, new advanced quantitative high-resolution imaging, diffraction and spectroscopic techniques of electron microscopy have become available. These techniques play a crucial role in characterizing the microstructure and the structure-property relationships of materials, as well as in metrology.

The symposium will address but is not limited to the following topics of interest:

- advanced quantitative electron microscopy (EM) methods – strategies and applications: In-situ EM measurements, electron tomography, scanning-transmission EM, diffractive imaging and diffraction, aberration-corrected EM, electron holography, electron nanospectroscopic techniques for local bonding and elemental mapping, quantitative comparisons (experiment versus theory)
- the application of physical sciences techniques in electron microscopy to soft and biological materials
- metrology for thin layers, nanoscale materials, and interfaces
- electron microscopy for the characterization of the growth and structure of nanoscale materials, such as nanowires and nanotubes
- electron microscopy of the self-assembly of nanostructures on surfaces or in thin films
- the in-situ manipulation and characterization of nanomaterials and processes by electron and ion beam techniques
- electron microscopy of functional nanocomposite materials
- electron microscopy of organic-inorganic interfaces for molecular and electronic applications

Important topics will be highlighted in keynote presentations given by leading invited experts and will be targeted to a non-expert audience, designed to communicate the potential of quantitative EM to application-oriented researchers in all of the fields addressed above. Contributions are solicited that feature applications of quantitative electron microscopy to all different classes of materials.

Hot topics to be covered by the symposium:

The scientific sessions (incl. poster sessions) are grouped into the following clusters covering state-of-the-art characterization / using quantitative electron microscopy (EM) for topic areas of materials science:

- In-situ electron microscopy measurements
- Electron tomography, focused ion beam / scanning electron microscopy techniques (FIB-SEM) and scanning-transmission electron microscopy (STEM)
- Diffractive imaging and diffraction
- Aberration-corrected electron microscopy
- Electron holography
- Electron nanoscopic techniques for local bonding and elemental mapping
- Pump-probe electron microscopy
- Quantitative comparison: experiment vs theory

List of invited speakers:

- Florian Banhart Université de Strasbourg FR
- Juri Barthel ER-C Jülich / RWTH Aachen DE
- Pascale Bayle-Guillemaud CEA-Grenoble, Grenoble FR
- Hugo Bender IMEC Leuven BE
- Jose Calvino Universidad de Cádiz, Cadiz ES
- Aicha Hessler CIME-EPFL, Lausanne CH
- Wayne Kaplan Technion, Haifa IL

- Gerald Kothleitner FELMI Graz AT
- Dierk Raabe Max-Planck-Institut für Eisenforschung Düsseldorf DE
- John Walmsley Norwegian University of Science and Technology, Trondheim, Norway

Scientific Committee:

The symposium organizers agreed to not nominating a scientific committee.

Proceedings:

Accepted contributed papers will be published in

Journal of Materials Science (Springer)

The "instructions to authors" is available at the author's gateway:

<http://www.editorialmanager.com/jmsc/>

Sponsors:



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Symposium : Q

Quantitative electron microscopy for research and industry

[07 June 2010](#) [08 June 2010](#) [09 June 2010](#)

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start at	Subject	Num.
08:50	Welcome to Symposium Q: Rafael Dunin-Borkowski, Wolfgang Jaeger, Paul A. Midgley, Etienne Snoeck	
	Quantitative Methods I : W. Jaeger	
09:00	3D imaging for nano-electronics Authors : H. Bender imec, Kapeldreef 75, 3001 Leuven, Belgium hugo.bender@imec.be Resume : The evolution in electronics processing shows a decrease of the dimensions of the components to the nanometer range and a huge increase of the number of materials. On the other hand the introduction of stacked devices brings analysis needs also back to the 25-100 micrometer range. Both the small and large dimensions pose strong challenges to the structural characterization. For the cross-sectional metrology a high imaging resolution as well as a high accuracy of the positioning of the cross-sections are needed. The latter requirement can be somewhat released by applying a slice and view approach in combined FIB/SEM systems based on which a 3D view of the device configuration can be deduced. The analysis at large depths demands for optimized procedures and new tools to keep the analysis time acceptable. As the nanometer devices are smaller than the typical thickness of the TEM specimens, strong projection overlap effects occur that complicate the interpretation or even fully mask the information of interest. Electron tomography with thicker than usual TEM specimens, allows to release the requirements on site-specific preparation and to obtain 3D information at high resolution even from complicated device structures with many components. The challenges for the structural analysis of nano- and micro-scaled structures with FIB/SEM and TEM imaging and tomography will be discussed and illustrated by studies of nano-wire tunnelfet and finfet devices and stacked dies.	1 1
	add to my program	(close full abstract)
09:30	3D EBSD: tomographic orientation microscopy in a FIB SEM as a new dimension of microstructure characterisation Authors : D. Raabe, S. Zaeferrer, P. Konijnenberg Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany Planck-Str. 1, 40237 Düsseldorf, Tel. +49 211 6792 340, Fax. +49 211 6792 333, e-mail: d.raabe@mpie.de, s.zaeferrer@mpie.de, p.konijnenberg@mpie.de Resume : We give an overview of our recent progress in the development and optimisation of a technique for the 3D high resolution characterisation of crystalline microstructures using EBSD tomography. Our approach is based on automated serial sectioning using a focused ion beam (FIB) and the characterisation of the sections by orientation microscopy based on electron backscatter diffraction (EBSD) in a combined FIB-scanning electron microscope (SEM). We scan volumes of the order of 50 x 50 x 50 µm ³ at EBSD step sizes of 50 x 50 x 50 nm ³ . The technique has all the powerful features of 2D EBSD-based orientation microscopy and extends them into the third spatial dimension providing additional insights particularly into the character of the interfaces, microstructure percolation, and texture-related micromechanics relevant to representative volume element approximations. In order to highlight the capabilities of the method we discuss examples from such different microstructures as pearlite colonies in steel, orientation gradients at interfaces in dual phase high strength steels, twins in pseudo-nanocrystalline NiCo thin films and TWIP steels, Cu processed by ECAP, and micromechanically stimulated deformation and orientation patterning formed under nanoindentations	1 2

- copper single crystals.
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- 10:00 Coffee Break
- Quantitative Methods II : R. Dunin-Borkowski
- 10:30 In-situ electron microscopy in an aberration-corrected STEM
 Authors : Florian Banhart Institut de Physique et Chimie des Matériaux, UMR 7504, Université de Strasbourg, 23 rue du Loess, 67034 Strasbourg
Resume : Scanning transmission electron microscopes (STEM) with aberration correctors enable us to focus an intense electron beam onto a 1 Angstrom spot. Besides imaging and analysis with Angstrom resolution, the highly focused electron beam gives us the possibility of carrying out in-situ electron irradiation at the real atomic scale. Structural transformations can be induced in the specimen and \"nano-engineering\" becomes feasible. The presentation gives an overview of the first in-situ experiments with an aberration-corrected STEM. Materials on the basis of graphitic carbon such as carbon nanotubes or graphene are in the focus of interest. Single or multiple vacancies can be created in pre-selected positions with the focused beam [1]. This gives us the possibility of observing in real time the response of a material to the creation of point defects. The combination of high temperature and localized electron irradiation is a unique possibility of studying the formation and behaviour of point defects and of structuring materials at the atomic scale. In-situ experiments will be shown where the interaction between graphitic structures and metal atoms is studied [2]. Examples are the trapping of metal atoms in vacancies, the formation of covalent junctions between nanotubes and metals [3], and the growth of carbon nanotubes from catalytically active metal particles that can be induced by electron irradiation and observed in-situ in the electron microscope [4]. [1] J. A. Rodriguez-Manzo, F. Banhart, Nano Lett. 9, 2285 (2009) [2] F. Banhart, Nanoscale 1, 201 (2009) [3] J. A. Rodriguez-Manzo et al., Proc. Nat. Acad. Sci. 106, 4591 (2009) [4] J. A. Rodriguez-Manzo et al., Small 5, 2710 (2009) 2
1
- add to my program** **(close full abstract)**
- 11:00 Prospects for site-specific SE dopant mapping quantification in the SEM
 Authors : K.W.A. Chee, E.G.T. Bosch (1), R. Beanland (2), P. R. Wilshaw (3) and C. J. Humphreys University of Cambridge, Department of Materials Science and Metallurgy, Pembroke Street, Cambridge CB2 3QZ, United Kingdom (1) FEI company, FEI Electron Optics, Building AAE, Achtseweg Noord 5, Eindhoven, The Netherlands (2) University of Warwick, Department of Physics, Gibbet Hill Road, Coventry CV4 7AL, United Kingdom (3) University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH, United Kingdom
Resume : Dopant mapping using secondary electrons (SEs) in a field-emission gun scanning electron microscope (FEG-SEM) is a potentially useful technique for studying dopant distributions in semiconductor devices at high spatial resolution. We propose the combined use of in situ focussed ion beam (FIB) specimen preparation and SE microscopy as a potential solution for dopant mapping of the future, in two- and three-dimensions. However, its general implementation in industry is inhibited by the lack of an accurate and robust quantification procedure. Although it is known that SE dopant contrast observed across a p-n junction is a function of the surface built-in potential across the junction, surface band-bending and external local fields (patch fields) above the specimen, the lack of understanding of the relative contributions from all these factors has hampered accurate quantification. To analyse the problem, we have performed detailed computational modelling to investigate the effects of surface charges and dopant concentrations on surface band-bending, as well as on the surface junction potentials and external patch fields. We have also compared our experimental data from SE energy spectroscopy with the calculations and analysed the effects of surface damage that FIB specimen preparation produces. The analyses described in this work will advance the understanding of quantitative dopant profiling in the SEM where site-specific FIB specimen preparation is performed. 2
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- 11:15 Semi-empirical equation for electron scattering at low energies in thin films consisting of light elements
 Authors : Marina Pfaff, Erich Müller, Dagmar Gerthsen Laboratorium für Elektronenmikroskopie and Center for Functional Nanostructures (CFN),

Karlsruhe Institute of Technology, 76128 Karlsruhe, Germany

Resume : Scanning transmission electron microscopy (STEM) at low energies (<30keV) is used simultaneously for imaging as well as thickness and composition determination of different polymer and carbon films. High-angle annular dark-field (HAADF)-images of thin samples taken with a standard STEM detector show dominant material (Z-) contrast with a high lateral resolution. Parameters and alignment of the state-of-art instrument can be changed easily and no additional setup is needed. To quantify the measurements, image intensities are compared with results of Monte Carlo simulations and semi-empirical equations for multiple electron scattering. For the purpose of validation samples with well-known composition and geometry are examined. A new semi-empirical equation for electron scattering at low energies in thin self-supporting films consisting of light elements is elaborated. The formula enables fast preliminary and comparative calculations. Furthermore optimal experimental parameters like primary electron energy and detection-angle range of scattered electrons can be determined. The equation also permits a precise thickness determination by varying the imaging parameters and quantification of the different contrast levels.

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11:30

Composition quantification of InGaAs quantum wells by transmission measurements in a scanning electron microscope

Authors : T. Volkenandt 1), E. Müller 1), D. Gerthsen 1), D.Z.Hu 2), D.M. Schaadt 2) 1) Laboratorium für Elektronenmikroskopie und Center for Functional Nanostructures (CFN), Karlsruhe Institute of Technology (KIT), 76131 Karlsruhe, Germany 2) Institut für Angewandte Physik und Center for Functional Nanostructures (CFN), Karlsruhe Institute of Technology (KIT), 76131 Karlsruhe, Germany

Resume : Material-sensitive (Z-)contrast in scanning transmission electron microscopy (STEM) is more pronounced at low electron energies (<30keV) and can be used for quantitative composition analysis. Additionally, at low beam energies knock-on damage is reduced, enabling examination of radiation sensitive samples like semiconductor or biological materials. The presented method is based on high-angle annular dark-field (HAADF) images and the comparison of the measured intensities with Monte Carlo simulations. Electron transmission is measured with a common annular semiconductor detector in a state of art instrument. Parameters and alignment can be changed easily and no additional setup is needed. To verify the method, samples consisting of four In(x)Ga(1-x)As layers with well-known In-concentrations of x=10%, 20%, 30% and 40% are used. These layers, grown by molecular beam epitaxy (MBE) on a GaAs substrate are separated by thin layers of GaAs. The focused ion beam technique (FIB) is used to prepare wedge-shaped samples with defined geometries. Quantification of the electron transmission gives the possibility to determine the local thickness and wedge angle of the prepared samples. On the other hand the In-concentration can be determined by calculating intensity ratios among the layers containing In and the separating GaAs layers. Quantifications are performed with high lateral resolution and adequate signal-to-noise ratio.

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11:45

3D EDX microanalysis by FIB-SEM : Algorithm for elemental quantification enhancement

Authors : Pierre Burdet, CIME, EPFL Marco Cantoni, CIME, EPFL

Resume : The large scattering range of electrons in a bulk sample has always been a limiting factor for the spatial resolution quantitative X-ray analysis (e.g. EDX) in SEM. However, in some special cases, a good understanding of the variation of the X-ray ionization yield with depth had lead to procedures to get partially over this limitation. For example, in the case of multilayer (stratified) samples, it is possible to calculate both layer thicknesses and composition. In the general case, however, the structure below the surface is unknown which leads to an uncertainty on the quantified composition. Focus ion beam (FIB) nanotomography permits to access the volume in z direction through sequential milling through the volume of interest. Stacks of 2D elemental maps can be used to extend the EDX analysis to a full 3D technique. The extensive knowledge of the sample's 3D structure can be used to improve quantification. We discuss the use of a recursive approach to enhance accuracy of elemental quantification and based on existing algorithms. This approach is

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tested in a simplified case with a stratified material of known layer thicknesses and compositions. We compare experimental data and Monte-Carlo simulation.

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12:00

Lunch Time

Quantitative Methods III : E. Snoeck

14:00

Materials Science Applications with a New Electron Energy-loss Spectrometer
 Authors : G. Kothleitner¹, M. Albu¹, J. Angseryd^{1,2} ¹ FELMI, Graz, University of Technology, Steyrergasse 17, 8010 Graz, Austria ² Department of Applied Physics, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden
Resume : Today analytical transmission electron microscopy (AEM, TEM) is one of the most powerful and widely used technique for the detailed structural and chemical investigation of heterogeneous materials. Applications range from precipitation analysis in steels or alloys and the defect analysis in semiconductor samples and ceramics to the localization of certain cell features in biological structures. Particularly electron energy-loss spectroscopy (EELS) and its imaging sibling EFTEM (energy-filtering TEM) with their ability to correlate element-specific, electronic and structural information at high spatial resolution has given interesting new insight to material properties. Performing truly quantitative measurements such as the measurement of precise core-edge energies (chemical shifts), or the absolute elemental quantification of single-scattering spectra with EELS (and EFTEM) on this kind of materials is still complicated, as the recording of the low-loss region compared to the core-loss requires completely different experimental conditions, due to the vast differences in intensity. Instrumental modifications to spectrometer system however, such as the addition of an electrostatic shutter plus an extra electrostatic vertical deflector and a new, fast CCD camera have been implemented for the first time world-wide at the FELMI in collaboration with Gatan. This setup now allows for the acquisition of the elastic and inelastic regime at nearly coincident times, with the benefit to use the ZLP (and low-loss) as an energy reference and deconvolution kernel for an unambiguous quantitative analysis of materials. The intention of this paper is to show recent results achieved in different fields of material research along with new data obtained from the modified energy-loss spectrometer.

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14:30

EFTEM study of surface plasmon resonances in silver nanostructures
 Authors : Peter A. van Aken, W. Sigle, C. T. Koch, B. Ögüt Stuttgart Center for Electron Microscopy, Max Planck Institute for Metals Research, Heisenbergstr. 3, 70569, Stuttgart, Germany, email: vanaken@mf.mpg.de
Resume : Understanding how light interacts with matter at the nanometer scale is a fundamental issue in nanophotonics and nanoplasmonics. The optical properties of metallic nanoparticles (NPs) are entirely dependent on collective excitations of their valence electrons, known as surface plasmon resonances (SPR), under electromagnetic illumination. Measuring these properties locally at the level of the individual NP in combination with spectral information over the entire visible range is important for linking the global response of a given assembly of NPs with the underlying structure and morphology. With the advent of monochromators and highly dispersive energy filters, energy-filtered TEM (EFTEM) has now become an excellent tool for probing the optical properties of metallic NPs in the ultraviolet–near-infrared (UV–NIR) domain with nanometer resolution. Here EFTEM studies of the dielectric response of Ag NPs, and of holes in a 100 nm thick Ag film, drilled by using a focused ion beam are presented. In the Ag NPs individual SPRs were measured with high spatial sampling. For the perforated Ag thin film, a number of localized SPRs are found which are due to the strong coupling effects between adjacent holes sensitively depending on the hole arrangement, apart from multipolar ring-shaped resonances visible at isolated holes. The experiments were carried out in the Sub-Electronvolt-Sub-Angstrom-Microscope (SESAM) equipped with an electrostatic monochromator and the in-column MANDOLINE filter. Financial support from the European Union under the Framework 6 program under the contract for an Integrated Infrastructure Initiative is acknowledged. Reference 026019 ESTEEM.

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14:45

Stress-strain effects of strained silicon nitride layers in Si structures
 Authors : S. Reboh¹, M.J. Hýtch¹, F. Houdellier¹, L. Clément², F. Morin², A.

Halimaoui2, A. Clavierie1 1 Groupe nMat, CEMES, 29 rue J. Marvig, 31055 Toulouse, France 2 STMicroelectronics, 850 rue Jean Monnet, 38920 Crolles, France

Resume : Stress-strain effects in the active semiconducting region of microelectronic devices have allowed improvements in carrier mobility and energy consumption. Traditionally, this goal has been achieved in p-MOS devices using SiGe graded alloys to tensile strain the Si channel. Alternatively, strain can be engineered using inherent steps of devices processing, such as capping contact etch stopping layers (CESL). Here, test structures are used to investigate the effects of strained silicon nitride CESL in Si by a combination of HoloDark, a transmission electron microscopy (TEM) based technique to locally map strain, and finite elements method (FEM) simulation. The result provides insights for future device developments.

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15:00

Strain Mapping by Dark-Field Inline Electron Holography

Authors : Vasfi Burak Özdöl, Christoph Koch, Peter A. van Aken

Resume : In order to characterize novel semiconductor devices, strain mapping with nanometer spatial resolution has become a key requirement. Although high-resolution transmission electron microscopy (HRTEM) coupled with geometric phase analysis (GPA) is a well established method to measure strain at the nanometer scale, the method requires very thin, high quality samples, is severely limited in its field of view, and prone to artefacts related to electron beam induced damage of the sample. To overcome these problems we have recently developed the dark-field inline holography (DIH) technique. This method relies on the reconstruction of the geometric phase from a focal series of dark-field images of 2 non-collinear reflections using a combination of the transport of intensity equation (TIE) and a non-linear exit face wave function reconstruction algorithm. In this contribution, we will demonstrate HRTEM and DIH approaches by applying them to strain-engineered MOSFET transistors with 45 nm node technology and commercially available high efficiency green LEDs with InGaN multi quantum wells . We acknowledge financial support from the European Union under the Framework 6 program under a contract for an Integrated Infrastructure Initiative Reference 026019 (ESTEEM).

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15:15

Wave-function reconstruction by phase-plate transmission electron microscopy

Authors : B. Gamm (1), J. Zach (2), K. Schultheiss (1), M. Dries (1), P. Donnadiou (4) R. R. Schroeder (3), D. Gerthsen (1) (1) Laboratorium fuer Elektronenmikroskopie, KIT, 76131 Karlsruhe (2) Ceos GmbH, 69126 Heidelberg (3) Bioquant Cell Networks, Universität Heidelberg, D-69120 Heidelberg, Germany (4) SIMAP, INPGrenoble-CNRS-UJF, BP 75, 38402 Saint Martin d'Hères, France

Resume : Phase-plate transmission electron microscopy is a promising technique for improving the contrast of biological samples. The basis for contrast formation is the shift of the relative phase between scattered and unscattered electrons by a physical phase plate and coherent interference in the image. Variable phase shifts can be generated by electrostatic phase plates without changing the other imaging parameters. Such a device can be used for a new method of inline holography which allows the analytical reconstruction of the wave function. The proposed method is not restricted to certain specimens, especially not to weak-phase objects or non-crystalline objects. To obtain phase-contrast images which are suited for reconstruction, a novel concept of such an electrostatic phase plate is presented. The phase plate can be considered as a micro-coaxial cable which overcomes the drawback of the spatial frequency-blocking ring electrode of micro-lens phase plates. The function of the phase plate is tested successfully for an amorphous test specimen. First experimental phase-contrast images are presented for PbSe and Pt nanoparticles with clearly varying contrast which depends on the applied voltage and corresponding phase shift of the unscattered electrons. With the new phase-plate design we show for the first time the reconstruction of an object wave function based on a series of only three experimental phase-contrast TEM images.

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15:30

Coffee Break

Quantitative Methods IV : F. Banhart

- 16:00 Magnetic imaging in a TEM on materials for future high density media
 Authors : C. Arm¹, A. Masseboeuf^{1*}, P. Bayle - Guillemaud¹, E. Gautier¹, B. Rodmacq¹, B. Dieny¹, A. Marty¹, C. Gatel^{*}, E. Snoeck^{*} 1CEA-Grenoble, INAC, 17, Rue des Martyrs, 38054 Grenoble cedex 9, France * CNRS-CEMES, 29, Rue Jeanne Marvig , 31055 Toulouse cedex, France
Resume : Magnetic imaging could be performed in a Transmission Electron Microscope to map the magnetic configuration of a sample or/and the stray field in vacuum. It is then of great interest to analyse new materials that could find applications for high density media. The so-called Lorentz microscopy and electron holography are used to image and quantify the magnetic induction. Experiments were done on a FEI-titan 80-300 kV equipped with a biprism and a Lorentz lens. We present here two examples on materials with perpendicular magnetic anisotropy exhibiting up and down domain configuration. The first one is a continuous thin film of chemically ordered FePd with the L10 structure. Magnetic configuration has been imaged and showed the domain shape and domain wall configuration. Magnetic defect as vertical Bloch lines where the sense of the magnetisation abruptly reverses, have been detected within the wall. The second sample is an array of bit patterned media. Perpendicular anisotropy multilayers of (Pt/Co)_{X10} were grown on a Si substrate patterned by nanoimprint lithography and pre-etched in the form of an array of Si pillars. Special sample preparation has been developed using a Focused ion Beam to prepare TEM sample with the cross section configuration and preventing any irradiation by the Ga ions. Electron holography has been used to identify the various configurations of the stray field.

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- 16:30 Influence of chemical bonding on HRTEM images of light elements
 Authors : Simon Kurasch¹, Jannik Meyer¹, H. J. Park², V. Skakalova², S. Roth^{2,3} and Ute Kaiser¹ 1. Central Facility of Electron Microscopy, Group of Materials Science Electron Microscopy, University of Ulm, 89069 Ulm, Germany 2. Max Planck Institute for solid state research, 70569 Stuttgart, Germany 3. School of Electrical Engineering, WCU Flexible Nanosystems, Korea University, Seoul, Korea
Resume : We used improved HRTEM image simulation where the electrostatic specimen potential is calculated by density functional theory (DFT) [1]. This approach takes into account the electron charge redistribution within the specimen due to chemical bonding. As only valence electrons are strongly influenced our study is focused on exclusively light element materials like graphene or h-BN. Furthermore these 2d crystals offer an outstanding specimen quality (known thickness, no amorphous cover layer) that can not be achieved for bulk materials. For both of them the DFT based simulation results in significant changes in the relative contrast: The slightly ionic character of the B-N bond makes it impossible to distinguish B from N atoms in h-BN (dZ=2) while the single atom nitrogen substitution in graphene is detectable (dZ=1) due to a strong charging effect on the neighbouring carbon atoms. As we show here, the bonding effect is clearly detected in experimental images of N-doped graphene and single layer h-BN. These experiments reveal that bonding has to be included in the TEM image simulation, and that a TEM can be utilized to obtain information about the electronic configuration of the specimen. This opens a way to discern electronic arrangements in point defects or other non-periodic objects that can not be analyzed in an diffraction experiment. [1] B. Deng and L.D. Marks, Acta Cryst. (2006). A62, 208-216

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- 16:45 Dispensing and Surface-Induced Crystallization of Zeptoliter Liquid Metal Alloy Drops
 Authors : Peter Sutter and Eli Sutter Center for Functional Nanomaterials, Brookhaven National Laboratory Upton, NY 11973, USA
Resume : Phase transformations of nanoscale objects play an important role in atmospheric physics, metallurgy, nanoscience and technology. In contrast to nanoparticle melting whose fundamental pathway was studied extensively, experiments on crystallization are hindered by the strong influence of support interfaces, which tend to induce the premature nucleation of the solid phase. Successful study of the phase transformations of free-standing nanoscale drops, not affected by support interactions, depends critically on developing the ability to dispense and manipulate such drops. Here we demonstrate the operation of a device (a pipette) specialized for the controlled delivery of

individual nanometer sized drops of liquid metals or metal alloys. The pipette, assembled and operated in-situ in a transmission electron microscope (TEM) [1-3], consists of a semiconductor nanowire (NW) that constitutes the pipette body, and whose tip provides a reservoir of a low-melting eutectic metal semiconductor (Au-Ge) alloy. The entire NW and the Au-Ge reservoir are encapsulated in-situ in a self-assembled graphene shell that drives the actuation of the pipette. The device delivers a metal alloy melt with zeptoliter (1 zL = 10⁻²¹ L) resolution, at least three orders of magnitude better than any existing pipette. Through quantitative analysis of data obtained by in-situ TEM, we explore the fundamental factors enabling the controlled zL fluid dispensing. We then use this exquisite control to produce individual, nearly free-standing nanoscale drops, and to image their phase transformations with high resolution. Our observations of the liquid-solid transition challenge classical nucleation theory by providing experimental evidence for an intrinsic crystallization pathway of nanometer-sized fluid drops that avoids nucleation in the interior, but instead proceeds via liquid-state surface faceting as a precursor to surface-induced crystallization. References 1. E. Sutter and P. Sutter, *Adv. Mater.* 18, 2583 (2006). 2. P. Sutter and E. Sutter, *Nature Mater.* 6, 363 (2007). 3. E. Sutter and P. Sutter, submitted.

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17:00

Quantitative STEM in ESEM at high vacuum and wet modes for three-dimensional particle characterization

Authors : Z. Barkay Wolfson Applied Materials Research Center, Tel-Aviv University, Tel-Aviv 69978, Israel (barkay@post.tau.ac.il)

Resume : Scanning electron microscope (SEM) with scanning transmission electron microscope (STEM) detector utilizes the transmitted electrons for two-dimensional imaging. It was shown that high STEM contrast in SEM is obtained for low atomic number materials including unstained biological samples[1] and polymers. This work focuses on quantifying the mass-thickness information of bright-field (BF) STEM images in the environmental SEM (ESEM) for three-dimensional (3D) characterization. For quantification, a Monte Carlo (MC) simulation program for transmitted electrons has been developed[2] and implemented for ellipsoidal particles. The 3D particle shape is obtained by measuring the spatial dimensions and fitting the measured BF-STEM intensity profile with the corresponding MC simulation profile using a spherical calibration particle. The quantification provides a distinction between various ellipsoidal shapes of a similar circular two-dimensional projected BF-STEM image. The 3D quantitative characterization is demonstrated on single particles and for initial steps of particle agglomeration using HV-STEM. Initial steps of water droplet condensation are in-situ studied using quantitative wet-STEM and possible biological applications are discussed. 1. "Three-dimensional characterization of drug-encapsulating particles using STEM detector in FEG-SEM", Barkay, Z., Rivkin, I., Margalit, R., *Micron* 40, 480-485 (2009). 2. "Quantitative STEM in SEM for three-dimensional nano-particle characterization", Z. Barkay, IMEC-14, Tel-Aviv, 13-14 Dec. (2009).

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17:15

A quantitative study of optical losses in gallium oxide nano-waveguides

Authors : E. Nogales, B. Méndez and J. Piqueras Dpto. Física de Materiales, Facultad Cc. Físicas, Universidad Complutense, 28040 Madrid, Spain

Resume : Transparent conductive oxide (TCO) nanostructures are good candidates to develop efficient photonic and optoelectronic nano-devices, as they can guide, detect, generate or amplify a light signal. The wide band gap of some TCOs enables to tailor their optical properties in the ultraviolet, visible and infrared ranges. Monoclinic gallium oxide (β -Ga₂O₃) presents one of the widest band gaps, what makes it very suitable for ultraviolet-visible photonics, e.g. nano-waveguide applications (1). In this work, we study the light generation and transmission along β -Ga₂O₃ nanowires by cathodoluminescence (CL) in a scanning electron microscope. A special geometry of the CL acquisition system has been used to quantify separately, with a high spatial resolution, the luminescence emission efficiency and the optical losses of the light guided along the growth direction of the nanowires. These losses will be discussed as a function of the lateral dimensions of the nanowires and of their coupling with the substrate. It is well known that, in light guides, the confinement of light fails when the dimensions are smaller than the wavelength and, therefore, a complete study of this property is needed to assess the

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feasibility for their use as building blocks in photonic applications. Good UV light transmission is observed in nanowires with sub-wavelength cross-sectional dimensions, placed on a silicon wafer. [1] E. Nogales, B. Méndez, J. Piqueras, J.A. García, Nanotechnology 20, 115201 (2009)

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17:30

Precession electron diffraction for microstructure characterization: application to quartz polymorphs and Dauphiné twin

Authors : Jacob Damien, Cordier Patrick UMET, UMR CNRS 8207, University of Lille I

Resume : Precession Electron Diffraction is used to distinguish between the high-temperature hexagonal β and the low-temperature trigonal α phases of SiO₂ quartz. The two structures just differ by a kink of the SiO₄ tetrahedra arranged along spiraling chains, which induces a loss of the twofold axis and subsequent twinning in the low-temperature phase. Conventional selected-area electron diffraction (SAED) does not enable the phases distinction since their lattices are strictly superimposed and only their intensity of reflections are different. The distinction becomes possible with precession that drastically reduces the dynamical interactions between reflections and makes their intensity very sensitive to small variations of the electron structure factors. Distinction between the twinned individuals in the low-temperature phase is also easily made and the twin-law characterized using stereographic projections. The actual symmetry of precessed zone axis patterns is then examined in detail. Comparing our experimental data with dynamical intensity simulations, we show that under certain thickness conditions, the diffraction class symmetry can be revealed as in convergent beam electron diffraction patterns. This is particularly interesting for beam sensitive materials such as quartz for which selected area diffraction patterns are to be used to avoid the rapid amorphization of the sample under the electron beam.

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17:45

New unconventional reference materials for TEM-EDS quantitative analysis
Authors : M Nacucchi 1, 4 , M Alvisi 1, D Altamura 2, V Pfister 1, M Re 1, D. Valerini 1 and M Vittori Antisari 3 1 ENEA, Materials and Technologies Department, Research Centre of Brindisi, S.S. 7 Appia – km 706,00, I-72100 Brindisi, Italy 2 CNR- Institute of Crystallography, Via G. Amendola 122/O, I-70126 Bari, Italy 3 ENEA, Materials and Technologies Department, Research Centre of Casaccia, Via Anguillarese, 301, I-00123 S. Maria di Galeria (Roma), Italy 4 To whom any correspondence should be addressed

Resume : The Energy Dispersive X-ray Spectrometry (EDS) is employed in Transmission Electron Microscopy (TEM) to chemically characterize the sample on the nanometer scale, but there are limitations in the use of this technique for quantitative analysis due to the absence of reliable reference materials. The thin film approximation relates relative concentration of the elements to the ratio of their x-ray intensities through the so called Cliff-Lorimer factor, or k-factor. This work investigates the use of an unconventional reference material to determine experimentally the Cliff-Lorimer factor for TEM-EDS quantitative analysis with an uncertainty less than 5%. The results demonstrate the equivalence of a bi-layer of pure gold on pure silver, deposited by means of RF-sputtering, with respect to a binary alloy foil of the same elements. The availability of a calibrated set of similar bi-layer reference materials would allow direct measurement of the k-factors for a large number of elements. Furthermore, in order to perform an independent check of the new method, a quantitative comparison with results obtained by the extrapolation method based on thin films of pure gold and pure silver is also made. The use of this kind of standard would avoid the errors due to the use of theoretical k-factors extracted from the compilations available in the literature. A new expression of the correction factor for x-ray self-absorption of the sample has also been derived.

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Symposium : Q

Quantitative electron microscopy for research and industry

[07 June 2010](#) [08 June 2010](#) [09 June 2010](#)[hide all](#)

start at	Subject	Num.
08:30	<p>Electron Microscopy of Nanomaterials and Composites I : P. Bayle-Guillemaud</p> <p>Catalysts: Challenging Materials and Challenging Problems for Quantitative Electron Microscopy</p> <p>Authors : M. López-Haro, S. Trasobares, A.B. Hungría, J.J. Delgado, M.A. Cauqui, S. Bernal, J.A. Pérez-Omil and J.J. Calvino Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica y Química Inorgánica. Facultad de Ciencias. Universidad de Cádiz. 11510-Cádiz. Spain</p> <p>Resume : Real catalysts commonly contain so many morphological, structural and compositional heterogeneities that make them complex objects for most Electron Microscopy Techniques. Particle aggregation, steep local thickness or compositional variations, difficulties to reach a fully aligned situation, their multi-component formulation or the nano-sized nature of most of the objects of interest in these materials can be cited among the factors which call for both specific methodologies when Electron Microscopy is used for their characterization and for the development of new characterization tools of increasing resolution in spatial and energy domains. Thus, catalysts, whose characterization most of the times demands an insight at the atomic level, are continuously posing new challenges and calling for further improvements in Electron Microscopy techniques. In this context, this contribution will try to illustrate how recent developments in Electron Microscopy have been crucial to gain a proper understanding of the synthesis, activation, function and deactivation of some multicomponent catalysts, most of them of interest in the field of Environmental Catalysis.</p>	5 1
	<p>add to my program (close full abstract)</p>	
09:00	<p>Synthesis, Processing and Characterization of Cu-CNT Nano-composites</p> <p>Authors : Mendoza, M. E. (1)*, Solórzano, I. G. (1), Brocchi, E. A. (1), Costa Neto C.A. (2) (1)DCMM, Pontifícia Universidade Católica do Rio de Janeiro, e-mail:mmendozao@aluno.puc-rio.br (2)COPPE/ UFRJ. Universidade Federal do Rio de Janeiro</p> <p>Resume : This work reports some structural characteristics of a Copper-2% CNT nanocomposites synthesized by chemical method. Purified single wall Carbon nanotubes(SWCNTs) with diameters between 5 - 10 nm and few microns of length were used. The nano-composites powders were produced by dissociation of a homogeneous suspension containing Cu (NO₃)₂ · 3H₂O - SWCNT with an anionic tensoactive; followed by hydrogen reduction of the obtained CuO-SWCNT product. X ray diffraction and Transmission Electron Microscopy has been used as characterization tools. The former confirmed the presence of pure metallic copper with carbon (Fig. 1b). The later allowed the observation of a good dispersion as well as adherence between Cu particles onto CNT. The Cu powder particles were observed to be in the 50-300nm range. Bulk nano-composite pellets were obtained by a pre-compaction under uniaxial pressure of 60 MPa followed by isostatic pressure of 150MPa. Sintering of the compacted material was carried out at 650°C under Argon atmosphere. Studies show a heterogeneous growth of copper grains with sizes between 150nm and 3µm. Low temperature electric resistivity measurements show that the nanocomposite material has lower value (2x10⁻⁶ Ω.cm) at 83°K than the copper without carbon nanotubes (6x10⁻⁶ Ω.cm). Hardness and elastic modulus were determined by nano indentation. The composite displayed higher hardness (1,7GPa) compared with copper (1,2GPa). Nevertheless elastic modulus of the copper shows a higher value than copper,</p>	5 2

because the composite presented porosity at the end of the process. Functionalized multi wall carbon nanotubes (MWCNTs) were used to produce the composite Cu-MWCNT through the same chemical method. Transmission Electron Microscopy have shown a good adherence between MWCNT and heterogeneous sized copper particles. Hot sintering, volume fraction distribution and properties measurements are currently in progress.

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- 09:15 Genesis of a Cu/ZnO methanol catalyst followed by combined x-ray and electron spectromicroscopy
 Authors : Matti M. van Schooneveld (1), Emiel de Smit (1), Luis A. Corrales (1), Tadahiro Yokosawa (2), Henny W. Zandbergen (2), Jörg Raabe (3), Bert M. Weckhuysen (1), and Frank M.F. de Groot (1) 1. Inorganic Chemistry & Catalysis, Debye Institute, Utrecht University, Sorbonnelaan 16, 3584 CA, The Netherlands 2. Kavli Institute of Nanoscience, High Resolution Electron Microscopy, Lorentzweg 1, 2628 CJ Delft, The Netherlands 3. Paul Scherrer Institut, CH-5232 Villigen, Switzerland
- Resume** : The conversion of synthesis gas (a CO/H₂ mixture), to methanol is industrially catalyzed by Cu/ZnO at ~250 °C and 20-100 bar. Here the genesis of a Cu/ZnO catalyst by in-situ synchrotron-based scanning transmission x-ray microscopy (STXM) [1] and ex-situ transmission electron microscopy (TEM) is studied using microcells fabricated by Micro-Electro-Mechanical Systems (MEMS) technology [2]. Cu_xZn_(1-x)(NO₃)_u(CO₃)_v(OH)_w systems were prepared, calcined to CuO/ZnO, reduced to active Cu/ZnO, and used to convert 3/1 CO/H₂ into methanol. Chemical maps of the Cu and Zn L_{2,3}-edge, and the O K-edge, were obtained at 1 bar during these steps by in-situ STXM. The x-ray microscope allowed the visualization of single Cu/ZnO particles with 30 nm spatial resolution. The ability of the microscope to generate x-ray absorption spectra (XAS) at every 30x30 nm² allowed the monitoring of the chemical state of the different catalyst constituents. During the reduction the transition from Cu²⁺ and Cu⁺ to Cu⁰ in a spatially inhomogeneous way was followed. Combining this with high resolution TEM on the same particle revealed the Cu⁰ was segregated as ~5 nm crystallites. We show that industrially important reactions can, next to low pressure in-situ electron microscopy investigations, be studied efficiently with in-situ x-ray microscopy at atmospheric pressure with an additional gain in chemical information. 1. de Smit, E. et al., Nature 456 (2008) 222 2. Creemer, J.F., et al., Ultramicrosc. 108 (2008), 993

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- 09:30 Telling the core from the shell in MnO_x/MnO_y nanoparticles through Mn oxidation state quantification using EELS.
 Authors : S. Estradé^{1,2}, A. López-Ortega³, M. Estrader³, G. Salazar-Alvarez^{1,4}, M.D. Baró⁵, J. Nogués^{3,6}, F. Peiró¹ 1. LENS, MIND-IN2UB, Departament d'Electrònica, Universitat de Barcelona, Martí i Franquès 1, 08028 Barcelona, Spain. 2. TEM-MAT, SCT, Universitat de Barcelona, Solé i Sabarís 1, 08028 Barcelona, Spain. 3. Centre d'Investigació en Nanociència i Nanotecnologia (ICN-CSIC), Campus Universitat Autònoma de Barcelona, E-08193 Bellaterra, Spain. 4. Materials Chemistry Group, Dept. of Physical, Inorganic and Structural Chemistry, Arrhenius Laboratory, Stockholm Univ., Stockholm, Sweden. 5. Departament de Física, Universitat Autònoma de Barcelona, Bellaterra, Spain. 6. Institució Catalana de Recerca i Estudis Avançats (ICREA)
- Resume** : Bi-magnetic core-shell systems in which the core is ferromagnetic (FM) and the shell antiferromagnetic (AFM) have been widely investigated both theoretically and experimentally [1]. However, studies of "inverse" core-shell nanoparticles with AFM cores are rather scarce [2]. In this work we present the structural characterization of inverted MnO_x(AFM)/MnO_y(FiM) core/shell nanoparticles with different sizes, by means of (S)TEM-EELS. HRTEM does not allow distinguishing between the core and the shell in these nanoparticles, as the shell grows epitaxially on the core, and HAADF imaging does not yield any different contrast for the two manganese oxides. In this sense, quantitative EELS was applied to assess the local composition of the MnO_x/MnO_y nanoparticles, through a direct evaluation of Mn oxidation state, allowing to tell the core from the shell in the nanoparticles and to determine the nature of the involved manganese oxides. Mn oxidation state was estimated based on the Mn L₃/L₂ peak intensity ratio and from the Mn L₃

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peak onset using the home-made program MANGANITAS [3-5]. EELS analysis reveal that while the core corresponds to MnO phase the shell can be either Mn₂O₃ or Mn₃O₄ depending on the size of the nanoparticles, where the smallest particles have a pure Mn₂O₃ shell whereas larger ones have increasing amounts of Mn₃O₄ [6]. [1] J. Nogués, et al. Phys. Rep. 422 (2005) 65. [2] G. Salazar-Álvarez, et al. J. Am. Chem. Soc. 129 (2007) 9102. [3] S. Estradé et al., Appl. Phys. Lett. 91, 252503 (2007). [4] S. Estradé et al., Appl. Phys. Lett. 93, 112505 (2008). [5] S. Estradé et al., Appl. Phys. Lett. 95, 072507 (2009). [6] A. López-Ortega et al. submitted (2010).

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09:45

EELS-Spectrum Imaging – extended analysis for composite materials
 Authors : Mihaela Albu*, Jenny Angseryd**, Gerald Kothleitner* *Institute for Electron Microscopy Graz (FELMI) Center for Electron Microscopy Graz (ZfE) Graz University of Technology Steyrergasse 17, A-8010 Graz, Austria
 **Sandvik Tooling & Chalmers University of Technology, Göteborg, Sweden
Resume : This paper demonstrates absolute elemental quantifications of a composite material (50% polycrystalline cubic boron nitride in a TiCN matrix [1]) by the nearly simultaneous acquisition of a low-loss and high-loss spectrum image, a technique which is called DualEELS. This method combined with a multiple linear least square fitting procedure enables the calculation of elemental maps containing areal and/or volumetric densities [2] of each element in each pixel of the EELS-SI. Moreover, EELS fine structure [3] and absolute edge energy information can be added to map boron, nitrogen and oxygen bounding unambiguously in different phases. The measurements were performed on a monochromated Tecnai F20 microscope equipped with a Quantum Gatan energy filter with a new DualEELS acquisition system [4, 5]. Apart from c-BN, phases like TiC_{0.7}N_{0.3}, TiC_{0.5}N_{0.5} and Al₂O₃ (big areas or network-like), also small TiB₂ grains and different Al_xO_yN_z phases have been quantified. Volumetric densities (at/nm³) of B in BN and TiB₂, of N in BN, TiCN and AlN, of Ti in TiCN and TiB₂ and of O in Al_xO_yN_z and as surface oxidation have been successfully extracted. [1] J. Angseryd, Licentiate Thesis, Göteborg, Sweden, 2008 [2] J. Scott et al, Ultramicroscopy 108 (2008) 1586 [3] R. Arenal et al., Ultramicroscopy 109 (2008) 32 [4] C. J. Trevor, Ultramicroscopy EDGE 2009 proceedings to be published [5] G. Kothleitner, Ultramicroscopy EDGE 2009 proceedings to be published

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10:00

Coffee Break

10:30

Electron Microscopy of Nanomaterials and Composites II : A. Hessler-Wyser

High-resolution TEM and STEM investigations of gas-separation membrane materials

Authors : J. Barthel *(a), S. Roitsch (a), J. Mayer (a) (a) Ernst Ruska-Centre for microscopy and spectroscopy with electrons, Forschungszentrum Jülich, 52425 Jülich, Germany

Resume : The application of gas-separation membranes in fossil-fuel power plants enables several efficient routes to capture and store unwanted carbon dioxide from the combustion process. In future such novel techniques may contribute to the reduction of the global human carbon dioxide output while keeping up with the increasing energy consumption. The gas-separation modules can be applied at different stages of the combustion process using molecular sieving or ionic transport, where both separation mechanisms depend strongly on the atomic structure of the membrane material.

Aberration-corrected high-resolution transmission electron microscopy is a powerful technique to study the local microstructure of solid matter on the atomic scale. By using aberration-corrected imaging and scanning TEM we investigate in particular the structural and chemical stability of membrane materials prior and after exposure to realistic application conditions. Exemplary investigation results are presented for mixed oxygen-ion and electron conductors, e.g. La₂NiO_(4+d) and Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_(3-d), as well as for mixed proton and electron conductors from the system of the Lanthanide-Tungsten-Oxides.

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11:00

Morphological diversity of CeO₂ nanoparticles studied by Electron Tomography
 Authors : Ileana Florea(a), Cedric Feral-Martin(b), Jerome Majimel(b), Charles Hirlimann(a), Ovidiu Ersen(a); (a)-Institut de Physique et Chimie des

Matériaux de Strasbourg (b)-Institut de Chimie de la Matière Condensée de Bordeaux

Resume : Cerium oxide (CeO₂) particles with nanometric size have many potential applications in the field of heterogeneous catalysis, especially in the oxygen storage process. Using the micro-wave chemical synthesis process and adjusting adequately the synthesis parameters (pressure, time, heating temperature etc.), different morphologies can be obtained: cubes, bi-pyramids or rods. Based on electron tomography in STEM-HAADF mode, a consistent three dimensional characterization of these systems was achieved, allowing us to determine their exact morphology (the crystallographic type of the exposed faces and their proportions) and the internal structure. In the case of cubic CeO₂ nanoparticles, the analysis of the reconstruction shows the presence of truncated edges. For CeO₂ nanoparticles with a bi-pyramidal morphology, we demonstrate that ambiguous interpretation of the objects giving triangular views in classical TEM can be prevented and determine that the shape and the surface crystallography can be determined: the (111) planes were thus assigned to the exposed faces and the (100) to the basal plane. Finally, the shape of CeO₂ rods is much more complex than previously thought, exhibiting a roughly pentagonal structure but with morphology at the top depending on the rod size. Furthermore, the tomographic study reveals also the presence of a relatively ordered porosity inside the rods obtained by this preparation method.

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11:15

TEM study of In₂O₃ and Sn doped In₂O₃ nanostructures grown by thermal treatment

Authors : D. Maestre (1,2), D. Häussler (1), A. Cremades (2), J. Piqueras (2) and W. Jäger (1) (1) Institute of Materials Science, Christian-Albrechts Universität zu Kiel, 24143, Kiel, Germany (2) Departamento de Física de Materiales, Facultad de Física, Universidad Complutense de Madrid, 28040, Spain.

Resume : The nanoscale dimensions associated to nanostructures allow the exploitation of new properties and effects, hence considerable efforts are being invested in their synthesis and characterization. The growth and doping control of nanostructures reached during the last years enable to spread potential applications in many fields of research, such as opto- and nanoelectronics. This work reports on the synthesis by thermal treatment and the characterization of In₂O₃, as well as Sn doped In₂O₃ nanostructures, such as wires, rods and arrows. For the growth of In₂O₃ nanostructures, InN has been employed as a precursor, while a starting mixture of SnO₂ and InN has been used to grow Sn doped In₂O₃ nanostructures. The characterization has been carried out by means of transmission electron microscopy (TEM) and high-resolution TEM, selected area electron diffraction (SAED), high angle annular dark field - scanning TEM (HAADF-STEM) and x-ray energy dispersive spectroscopy (EDS). By combining these techniques the presence of nanopipes in the core of the undoped In₂O₃ elongated structures has been revealed. The presence of Sn as a dopant generates longer and thinner nanostructures, where loops and nano-precipitates associated with Sn (as confirmed by HAADF-STEM and EDS) are observed in the core region. A deeper understanding of the defects involved during the nanostructures growth and doping process can lead to improve future optoelectronic and field emitter applications.

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11:30

Electron Microscopy Characterisation of Horizontal Nanochannels Aligned by Physical Epitaxy and Guided Anodisation

Authors : Ciara T. Bolger,^{1,2} Richard A. Farrell,^{1,2} Gareth M. Hughes,⁴ Michael A. Morris,^{1,2} Nikolay Petkov,³ and Justin D. Holmes^{1,2} ¹Materials and Supercritical Fluid Group, Department of Chemistry and the Tyndall National Institute, University College Cork, Cork, Ireland, ²Centre for Research on Adaptive Nanostructures and Nanodevices (CRANN), Trinity College Dublin, Dublin 2, Ireland, ³Electron Microscopy and Analysis Facility (EMAF), Tyndall National Institute, Lee Maltings, Prospect Row, Ireland, and ⁴Department of Materials, University of Oxford, Parks Road, Oxford, OX1 3PH, United Kingdom

Resume : Synthesis of nanostructures within aligned mesoporous templates allows the highly ordered structures to be electrically and thermally isolated from each other, and hence addressed on an individual basis. Mesoporous

silica thin films prepared by Evaporation Induced Self Assembly (EISA)¹ are one potential sub-10 nm host due to the structural regularity and porosity of the material on the nanoscale². We have prepared aligned mesoporous silica by directed deposition of thin films within channelled substrates³. A systematic study of the deposition parameters, sol-gel concentration, and trench dimensions (cross sectional aspect ratio) required to produce well-aligned mesoporous silica channels on patterned substrates was carried out. In depth characterisation of the resultant films has established pore correlation lengths (length of a linear porous channel) of at least two micron. This characterisation was achieved by novel focused ion beam (FIB) sectioning and in situ SEM imaging, to our knowledge, a technique applied to such a system for the first time. Our findings establish that, under confinement, directed deposition of the sol within channelled substrates, where the cross-sectional aspect ratio of the channels approaches unity, induces alignment of the mesopores along the length of the channels. Such information on pore correlation lengths and defect densities is critical for subsequent nanowire growth within the mesoporous channels, contact layout, and possible device architectures. Anodised Aluminium Oxide (AAO) is another such host material, appealing due to the low cost and ease of processing⁴, along with its thermal and chemical robustness. The synthesis of extremely high aspect ratio, almost defect free AAO has already been achieved in a vertical orientation⁵, but there have been few reports on the synthesis of AAO in a horizontal orientation with respect to the supporting substrate.^{6, 7} An extensive study has been carried out on the anodisation of a series of Al fingers of different dimensions in a planar orientation. Characterisation has been carried out by SEM and TEM of sections taken through the pores, and most importantly along the pore lengths, revealing a branched porous nature not previously reported in the literature. 1. Brinker, C.J. Evaporation-induced self-assembly: Functional nanostructures made easy. *Mrs Bulletin*. 2004. 29: 631. 2. Petkov, N.; Platschek, B.; Morris, M.A.; Holmes, J.D.; Bein, T. Oriented Growth of Metal and Semiconductor Nanostructures within Aligned Mesoporous Channels. *Chem. Mater.* . 2007. 19: 1376. 3. Bolger, C.T.; Farrell, R.A.; Hughes, G.M.; Morris, M.A.; Petkov, N.; Holmes, J.D. Pore Directionality and Correlation Lengths of Mesoporous Silica Channels Aligned by Physical Epitaxy. *Acs Nano*. 2009. 3: 2311-2319. 4. Nielsch, K.; Choi, J.; Schwirn, K.; Wehrspohn, R.B.; Gosele, U. Self-ordering regimes of porous alumina: The 10% porosity rule. *Nano Letters*. 2002. 2: 677-680. 5. Jessensky, O.; Muller, F.; Gosele, U. Self-organized formation of hexagonal pore arrays in anodic alumina. *Appl. Phys. Lett.* 1998. 72: 1173-1175. 6. Cojocar, C.S.; Padovani, J.M.; Wade, T.; Mandoli, C.; Jaskierowicz, G.; Wegrowe, J.E.; Morral, A.F.; Pribat, D. Conformal Anodic Oxidation of Aluminum Thin Films. *Nano Lett.* 2005. 5: 675-680. 7. Gowtham, M.; Eude, L.; Cojocar, C.S.; Marquardt, B.; Jeong, H.J.; Legagneux, P.; Song, K.K.; Pribat, D. Controlled fabrication of patterned lateral porous alumina membranes. *Nanotechnology*. 2008. 19: 035303.

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11:45

Solving the chemistry of a 3D structure at the nanometre scale by analytical electron tomography
 Authors : L. Roiban(a),(b), L. Sorbier(b), C. Pichon(b), P. Bayle-Guillemaud (c), J. Werckmann(a), M. Drillon(a), O. Ersen(a); (a)-IPCMS, 23 rue du Loess, BP 43, 67034 STRASBOURG Cedex 2, France. (b)-IFP-Lyon, BP 3, 69360 Solaize, France. (c)-CEA/INAC, 17, rue des Martyrs, 38054 GRENOBLE Cedex 9, France
Resume : Combining the electron tomography and energy filtered imaging allows now to perform chemical selective 3D analysis with a resolution reaching the nanometre, pushing this new technique of analytical electron tomography at the cutting edge. The implementation of this powerful technique which provides a full characterisation of complex nanomaterials is challenging, requiring a careful and consistent optimisation of the acquisition, computing steps and analysis process. Furthermore, the chemical signal must satisfy the linear projection requirement for the tomography, which involves a correct estimation of the background for each pixel of the image. The main goal of this work was to map elemental concentrations on a geometrical complex surface. Obtaining this information at the cutting edge in the fields where the surface chemistry is crucial, as for example in catalysis. In particular, to obtain widespread catalytic materials, one of the ways is to

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combine materials with different properties, as the alumina and the silica. Depending on the preparation, their proportions at the surface of the porous material are different. The challenge was to implement the analytical electron tomography to determine their relative proportion in the whole grain and at the surface. A second breakthrough of the work was to illustrate the possibility to spatially discriminate in 3D two close chemical elements, here the silicon and the aluminium.

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- 12:00 Lunch Time
Electron Microscopy of Metals and Semiconductors : D. Raabe
- 14:00 Transmission Electron Microscopy studies of aluminium alloys
Authors : John C Walmsley (1,2) Calin D Marioara (1), Magnus H Larsen (2), Rangvald H Mathiesen (2), Kemal Nisancioglu (2) and Randi Holmestad (2) (1) SINTEF Materials and Chemistry, N-7465 Trondheim, Norway (2) Norwegian University of Science and Technology, N-7491 Trondheim, Norway
Resume : Aluminium is an important material with a wide range of applications including packaging, building and aerospace. Improvements in properties and alloy design require detailed microstructural and microchemical information for which Transmission Electron Microscopy (TEM) is an essential tool. For example, Mg and Si containing 6000 series alloys achieve desirable mechanical properties by the presence of metastable hardening phases. The influence of other elements, such as Cu, on the nanometre-sized precipitate structure and crystallography needs to be understood (1). At the same time, corrosion properties are influence by the segregation of elements to interfaces such as grain boundaries (2). It is increasingly important to combine quantitative TEM analysis with other techniques such as Atom Probe Tomography and ab-initio modelling (3). The approaches employed will be illustrated with examples from current research projects. (1) C.D Marioara, S.J. Andersen, T.N. Stene, H. Hasting, J. Walmsley, A.T. van Helvoort and R Holmestad, 2007, Philosophical Magazine, 87, (23), 3385-3413. (2) M.H. Larsen, J.C. Walmsley, O. Lunder and K. Nisancioglu, 2010, Journal of the Electrochemical Society, 157, 2, C61-C68. (3) H.S. Hasting, A.G. Froseth, S.J. Andersen, R. Vissers, J.C. Walmsley, C.D Marioara, F. Danoix, W. Lefebvre and R. Holmestad, 2009, Journal of Applied Physics, 106, 12, 123527.

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- 14:30 Microstructure evolution of gold thin films under spherical indentation for micro switches contact applications
Authors : Brice Arrazat (1), Vincent Mandrillon (1,2), Karim Inal (1), Maxime Vincent (2,3), Christophe Poulain (2) (1) Packaging et Support Souple, Ecole Nationale Supérieure des Mines de Saint-Etienne, Centre de Microélectronique de Provence – George Charpak, Gardanne, France (2) Microsystems Characterization and Reliability Lab., CEA- Léti, Minatec, Grenoble, France (3) Innovation Division, Schneider Electric Industries, Grenoble, France
Resume : RF MEMS switches have demonstrated interesting performances in terms of power consumption and insertion loss. However, recent works on gold-on-gold contact switches reliability revealed two primary failure mechanisms during cycling that are contact resistance increase and stiction. Due to the complexity of involved phenomena, a first step towards failure understanding is the investigation of the contact metal microstructure evolution submitted to loads representative of micro-switches actuation. First, sphere / plane contact using a nanoindenter with a spherical diamond tip is studied on a 1 µm thick sputtered gold thin film with a <111> fiber texture. In addition, an apparatus dedicated to mechanical contact cycling is used to perform endurance testing up to half million cycles with a spherical tungsten tip. Contact area (including pile-up) analysis with high resolution Electron BackScatter Diffraction (EBSD) allows local crystallographic texture and microstructure determination. Results are analyzed in terms of equivalent pressure calculated as a function of load and contact curvature radius. Above an equivalent critical pressure of 1 GPa, spherical indentation induces grain rotation due to the plastic deformation at film surface corresponding to the degradation of the <111> texture. Such behavior is observed after endurance contact testing up to half million closures under a 500 MPa pressure due to cyclic work hardening demonstrating the interest of EBSD characterization.

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(close full abstract)

14:45 Quantitative study of GaN/AlxGa1-xN quantum dots microstructure using aberration-corrected transmission electron microscopy
 Authors : M. Korytov (1), M. Benaissa (2), T. Huault (1,3), J. Brault (1), T. Neisius (4), V. Grillo (5), J-L Rouvière (6), and P. Vennéguès (1) 1 CRHEA-CNRS, rue Bernard Grégory, Sophia Antipolis, 06560 Valbonne, France 2 CNRST, CNRST, angle Allal-Fassi/FAR, Madinat al-irfane, 10000 Rabat, Morocco 3 RIBER S.A., 31 rue Casimir Périer, BP 70083, 95873 Bezons Cedex, France 4 CP2M, Faculté Saint Jérôme, 13397 Marseille, Cedex 20, France 5 Center s3 INFN – CNR, via Campi, 213/A, 41100 Modena, Italy 6 CEA/Grenoble, 17 rue des Martyrs, 38054 Grenoble, Cedex 9, France
Resume : Comparing with AlN, which is the common template for the growth of GaN QDs, AlGaIn is better tailored to be a basis for optoelectronic devices, due to its availability to be heavily doped. The capping of GaN QDs by an AlxGa1-xN barrier results in changes of both QDs morphology and spacers microstructure. We present a quantitative study of composition distribution in these samples using various transmission electron microscopy (TEM) techniques. Studied GaN/AlGaIn QDs samples were grown on fully-relaxed Al0.5Ga0.5N (0001) templates by ammonia-assisted molecular beam epitaxy. Primary high-resolution TEM (HRTEM) observations revealed a nanoscale phase separation in the AlGaIn barriers. The value of the composition fluctuations was evaluated by strain analysis, obtained from HRTEM images acquired with a Cs-corrected microscope. The reliability of HRTEM image conditions was confirmed using image simulations. Study of the samples with probe-corrected scanning TEM in high-angle annular dark-field imaging (HAADF) mode evidenced phase separation phenomenon. Multislice HAADF simulations were performed for quantitative interpretation of the images contrast. Further electron energy-loss spectroscopy (EELS) was carried out with a monochromatized TEM. The Al composition in Al-rich zones was determined by analysing the plasmon peak shift. Based on these TEM analyses, different mechanisms for the observed phenomena have been evaluated and will be discussed in this presentation.

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(close full abstract)

15:00 A TEM investigation of the damage formation in thin GaN layers during rare earth ion implantation at medium range energy
 Authors : B. Lacroix (1), F. Gloux (1), M.P Chauvat (1), P. Ruterana (1), K. Lorenz (2) and E. Alves (2) (1) CIMAP, UMR 6252, CNRS-ENSICAEN-CEA-UCBN, 6, Boulevard Maréchal Juin, 14050 Caen, France (2) Instituto Tecnológico e Nuclear, Estrada Nacional 10,2 686-953 Sacavém, Portugal
Resume : Rare earth (RE) doped GaN has been the subject of research over the past few years due to promising light emission applications. In optoelectronics, particularly, interesting results have been recently obtained, for instance laser action at room temperature (RT) have been reported in Eu doped GaN by MBE. Ion implantation as a doping technique has interesting advantages, i.e. control of the doped area and easy lateral integration. However it causes a large amount of structural defects in the implanted layers. In this work, the nature of the damage in c- and a-GaN layers implanted by rare earth ions at 300keV and room temperature has been investigated by transmission electron microscopy versus the fluence, from 7×10^{13} to 2×10^{16} at/cm², using Eu ion implantation. The density of point defect clusters has been found to increase with the fluence. In GaN, from about 3×10^{15} at/cm², a highly disordered 'nanocrystalline layer' (NL) starts to form at the GaN surface. Its structure exhibits a mixture of voids and misoriented nanocrystallites. Basal stacking faults (BSFs) of I1, E and I2 types have been observed from the lowest fluence, they are I1 in majority. Their density increases and saturates when the NL appears. Many prismatic stacking faults (PSFs) with Drum atomic configuration have been observed. The I1 BSFs are shown to propagate easily through GaN by folding from basal to prismatic planes thanks to the PSFs. A mechanism based on the behaviour of the stacking fault is proposed to explain the non-amorphisation of the GaN.

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15:15 Direct mapping of strain depth- distributions in ion implanted Si and Ge
 Authors : N. Cherkashin*, S. Reboh*, F. Houdellier*, A. Claverie*, M. J. Hÿtch* * CEMES/CNRS, 29 rue Jeanne Marvig, 31055, Toulouse, France
Resume : Ion implantation into Si or Ge followed by annealing is a common process in modern semiconductor technology. Depending on the dose and

implant species, ion implantation can result either in the point defects being distributed all along the ion track, or being confined below the generated amorphous layer. During annealing, precipitation of all these species may occur, mostly driven by the reduction of the elastic energy stored by the matrix. Thus, mapping the strain (and stress) as a function of depth is essential to understand and model the evolution of such systems during annealing. In this work, we present direct quantitative mapping of the strain depth distribution obtained in Si and Ge implanted with H⁺ (non-amorphizing) and Ge⁺ (amorphizing) ions, respectively, using a new technique, HoloDark, based on electron holography in a transmission electron microscope (TEM). The effect of the thin-film strain relaxation in a TEM lamella was corrected for using finite element simulations. The results are discussed and compared to the depth distribution of vacancy/interstitials defects predicted by Monte Carlo simulations.

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15:30 Coffee Break

16:00

Quantitative transmission electron microscopy of single platinum atoms
 Authors : B. Gamm (1), H. Blank (1), R. Popescu (1), R. Schneider (1), A. Beyer (2), A. Götzhäuser (2), D. Gerthsen (1) (1) Laboratorium für Elektronenmikroskopie, KIT, 76131 Karlsruhe (2) Physik Supramolekularer Systeme, Universität Bielefeld, 33501 Bielefeld

Resume : Imaging single atoms or even monitoring the motion of single atoms on a substrate has always been an eminent goal in electron microscopy which has come into close reach by the developments in electron microscopy during the past decade. Imaging of single atoms and small clusters on a substrate is particularly interesting for the understanding of cluster nucleation and growth which are important aspects in catalysis, crystal growth and adsorption. An aberration-corrected FEI Titan operated at 300 keV was used to acquire images of single platinum atoms and planar Pt clusters on C nanosheets with a resolution of better than 1 Å. The platinum was deposited by electron-beam evaporation on the extremely thin substrate. Time series at constant defocus reveal the motion of single Pt atoms, which are highly mobile on the nanosheet. Through-focal series of single Pt atoms were taken which can be considered as ideal test objects for the determination of the point spread function (PSF). Simulations of the contrast of single Pt atoms were performed by the multislice implementation of the STEMsim program taking into account the measured aberrations and the modulation transfer function of the CCD camera. Quantitative agreement (limited by noise) is obtained between simulated and experimental contrast of single Pt atoms regarding intensity, shape and FWHM as a function of defocus.

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16:00

Compositional characterization of nickel silicides by HAADF-STEM imaging
 Authors : E. Verleysen (1,2), H. Bender (1), O. Richard (1), D. Schryvers (3), W. Vandervorst (1,2) (1) IMEC, Kapeldreef 75, 3001 Leuven, Belgium (2) Instituut voor Kern- en Stralingsfysica, K. U. Leuven, Celestijnenlaan 200D, 3001 Leuven, Belgium (3) EMAT, Universiteit Antwerpen, Groenenborgerlaan 171, 2020 Antwerpen, Belgium

Resume : A methodology for the characterization of Ni-silicides by HAADF-STEM imaging is developed. Initially, non-aligned HAADF-STEM images are taken at identical conditions from a set of Ni-silicide reference samples: Ni₃Si, Ni₃₁Si₁₂, Ni₂Si, NiSi and NiSi₂. The composition of these reference samples was determined in advance by EDS and EELS analyses. In order to extract quantitative information from the HAADF-STEM images, the intensities of the Ni-silicides are measured relatively to the intensity of the silicon substrate. This relative intensity can then be related to the average atomic number of the Ni-silicide phases. This allows us to quantify the relationship between the experimental Z-contrast intensities and composition of the analysed Ni-silicide samples. Furthermore, the effect of thickness on the HAADF-STEM intensities is examined, as well as the influence of Bragg contrast and channeling contrast. The experimentally determined relative intensities are compared to calculated values. This methodology is then applied to Ni-silicides with an unknown composition, present in CMOS devices. Since HAADF-STEM provides a very high spatial resolution by means of the nanosized electron probe, it is a powerful technique for the characterization of Ni-silicides and other transistor structures.

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- add to my program** **(close full abstract)**
- 16:00 Poster Session : R. Dunin-Borkowski, W. Jäger, P. A. Midgley, E. Snoeck
 MELTING TEMPERATURE OF GOLD NANOCCLUSERS AS MEASURED BY ELASTIC PEAK ELECTRON SCATTERING
 Authors : P.V. Borisyuk, V.D. Borman, M.A. Pushkin, V.N. Tronin, V.I. Troyan, National Research Nuclear University "MEPhI", 31 Kashirskoe chausse, 115409 Moscow, Russia
Resume : The results of the investigation of the elastic scattering of 500 eV electrons from Au nanoclusters pulsed laser deposited on highly oriented pyrolytic graphite HOPG, SiO₂ and Ni surface at room temperature are presented. The intensity of the EPES spectrum of Au nanoclusters was found to depend non-monotonically on clusters size with a minimum value lower than that of the clean substrate. The observed behavior can be caused by the change in the Debye-Waller factor of nanoclusters with decrease in their size. The analysis of the EPES spectra allowed to obtain the dependences of the Debye temperature and melting temperature T_m of Au nanoclusters on their size. The decrease in clusters size from 10 nm to 1 nm results in the decrease of T_m from the bulk value of 1337 K to 500 K. The melting temperature of Au clusters on HOPG is lower than that of Au on SiO₂ and Ni substrates, that can be caused by the roughness of non-regular boundary of Au clusters on HOPG demonstrated by STM images. The obtained results allow to propose a novel experimental technique for the measurement of the thermodynamic characteristics of supported metal nanoclusters. The advantage of this technique is the absence of direct temperature measurements and the possibility to investigate clusters on various substrates. The work has been partially supported by the Russian Foundation for Basic Research and the Russian Federal Agency of Education.

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- add to my program** **(close full abstract)**
- 16:00 Electron Tomography Studies for Assemblies of Shape-Controlled Platinum Nanocrystals
 Authors : Ileana Florea(a), Arnaud Demortiere(b), Christophe Petit(b), Charles Hirlimann(a), Ovidiu Ersen(a) (a)-Institut de Physique et Chimie des Matériaux de Strasbourg (b)-Laboratoire des Matériaux Mésoscopique et Nanometrique, Université Pierre et Marie Curie Paris 6
Resume : In the past years synthesis of individual Pt nanocrystals and their assembly in long-range ordered suprastructures has received an increased attention due to their new physical and chemical properties. Controlling their individual shape is crucial, since is the major parameter governing their use in applications such as catalysis or optics. Assembling the nanoparticles in suprastructures gives rise to other potential uses based on specific collective properties. A high long range order requires a narrow size distribution and a uniform shape. To precisely determine both individual parameters of the particles and of their periodic arrays, the use of electron tomography is unavoidable, since it is the unique technique allowing to retrieve the third dimension of the object with a nanometre resolution. For nanocrystals analysis, the STEM-HAADF mode is here the most appropriate thanks to its incoherent character and better resolution. STEM-HAADF tomography was applied to investigate the shape of Pt nanoparticles (with sizes ranging from 5 to 10 nm) and we could then allocate a cuboctahedron morphology with eight (111) and six (100) crystallographic planes. Furthermore, electron tomography performed in bright field mode on an assembly of Pt (NPs) allowed the determination of their individual 3D positions in the self-organized structure and to identify thus the type of structure (fcc) as well as the different packing defects.

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- add to my program** **(close full abstract)**
- 16:00 Morphology of lamellar delta-alumina support and spatial distribution of Pd catalyst nanoparticles studied by electron tomography
 Authors : L. Roiban(a),(b), L. Sorbier(b), C. Pichon(b), I. Florea(a), F. Tihay (b), J. Werckmann(a), M. Drillon(a), O. Ersen(a) (a)-IPCMS, 23 rue du Loess, BP 43, 67034 STRASBOURG Cedex 2, France. (b)-IFP-Lyon, BP 3, 69360 Solaize, France.
Resume : The electron tomography is now one of the most employed technique to study the morphology of heterogeneous catalysts as well as the spatial distribution of their different components at the subnanometer scale. Its applications to catalyst materials are very broad, as an accurate knowledge

of the 3D characteristics aids in understanding their catalytic properties. In this framework, we present a high-resolution electron tomography study applied to a system composed by Pd nanoparticles supported on a high surface area delta-alumina support. In a first step, the morphology and the porosity of the support was determined: it presents a lamellar morphology, with broad terraces on the top and various steps at the edges. A global analysis performed on an assembly of lamellas allowed us to statistically determine the step density and their position with respect to the alumina body. In a second stage, the mean size and the spatial localization of the Pd nanoparticles on the support was quantitatively determined. We found that, due to the interaction of Pd with the alumina steps, 70% of the particles are localized at the edges of the platelets.

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16:00

Analytical STEM investigations of Sn-Pd Nanoparticles with Core-Shell Structures

Authors : D. Häussler(1), B. Schaffer(2,3) , F. Liu(1,4), F. Hofer(3), X. B. Zhang(4), W. Jäger(1) 1-Microanalysis of Materials, Christian-Albrechts-University Kiel, 24143 Kiel, Germany, 2-SuperSTEM Facility, Daresbury Laboratory, Warrington, WA4 4AD, U. K., 3-Institute for Electron Microscopy, Graz University of Technology, 8010 Graz, Austria, 4-Department of Materials Science and Engineering, Zhejiang University, Hangzhou, 310037, China

Resume : Metallic core-shell nanoparticles for applications in catalysis and as data storage materials offer the possibility to tailor macroscopic properties generally not obtained by the single-component particles. Pd-Sn core-shell nanoparticles were fabricated by a solution impregnation method on multi-wall carbon nanotubes and characterized by a combination of various transmission electron microscope (TEM) methods. Our methodological approach demonstrates the feasibility to precisely analyse and map structure, morphology, and chemical composition of such particles. Composition analyses by electron-energy loss spectroscopy are aggravated by overlapping of ionization edges and have limited detection sensitivity whereas energy-dispersive X-ray microanalyses reveal small spectral signal-to-noise ratios at limited spatial resolution. The scanning TEM dark-field image contrast allows distinguishing Sn from Pd and other lighter elements due to the strong atomic number (Z) dependence of electron scattering to high angles (HAADF contrast) and allows atom column imaging. Electron nano-diffraction patterns formed by electrons scattered to smaller angles provide additional information about the particle structure. Our analyses of a number of Sn-Pd particles with diameters as small as 20 nm reveal formation of both polycrystalline particles with Pd-rich cores and oxidized shells that mainly contain Sn as well as polycrystalline alloy particles.

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16:00

STRUCTURE, MORPHOLOGY AND MAGNETIC PROPERTIES OF CoPt NANOPARTICLES AND THIN LAYERS

Authors : Marzena Materska(1,4), Ileana Florea(1), Damien Alloyeau(2), Nadi Braïdy(2,3), Dorin Geiger(5), Daniel Wolf(5), Ovidiu Ersen(1), Corinne Ulhaq-Bouillet(1), Cyril Langlois(2), Christian Ricolleau(2), Yann Le Bouar(3), Manuel Acosta(1), Annick Loiseau(3), Rafal Kozubski(4), Hannes Lichte(5), Véronique Pierron-Bohnes(1) (1) IPCMS, (CNRS-UdS) Strasbourg, 23 rue du Loess BP 43 67034 Strasbourg Cdx 2 France (2) MPQ (Un. Paris 7-CNRS) Bât. Condorcet, Case courrier 7021 75205 Paris Cdx 13 (3) LEM, CNRS/ONERA BP 72 92322 Châtillon Cdx France (4) M. Smoluchowski Inst. of Physics, Jagiellonian Un., Reymonta 4 30-059 Krakow, Poland (5) Triebenber Lab. Un. of Dresden 50 D-01328 Dresden- Zschendorf, Germany

Resume : CoPt is a promising material for application as high density storage media. The crucial condition, that must be fulfilled for such an application is that small nanoparticles must remain ferromagnetic. In this work the size effect is considered. It is due to the ratio between the amount of surface and volume atoms (additional surface energy) that chemically ordered phase for small nanoparticles is stable at a lower temperature than for bulk samples [1]. L10 phase has high magnetocrystalline anisotropy (ensuring higher superparamagnetic limit), Pt and Co-rich monoatomic layers are perpendicular to the easy magnetization axis. The samples were prepared by PLD and magnetron sputtering on different substrates: in epitaxy (MgO, NaCl) or not (Si, Al₂O₃, amorphous C) at several temperatures. Growth at high

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temperature or post-preparation annealing (with or without external magnetic field) yields chemical ordering of nanoparticles and thin layers. The long range order has been studied using X-ray diffraction and STEM analysis with a corrected probe, and the morphology using STEM-HAADF tomography. The magnetic properties were investigated using SQUID magnetometry (magnetization curves), torque (direct evaluation of the magnetocrystalline anisotropy) and, at the nano-scale, off-axis electron holography. [1] Alloyeau et al. Nature Materials (2009) 8, 940–946 (2009).

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[\(close full abstract\)](#)

- 16:00 TEM holder generating in-place magnetic field and its application on thin permalloy films
 Authors : Ryohei Tokuda, Kouichi Hamada, Masashi Arita, Yasuo Takahashi
 Graduate School of Information Science and Technology, Hokkaido University
Resume : In recent years, functional magnetic devices such as magnetic random access memories (MRAM) are studied in various fields. Their physical properties, such as electric resistance, are strongly influenced by the magnetic microstructure. However there are a few reports on in-situ experiments to correlate the magnetic microstructure and the electric property. For easy performance of such experiments, we have developed a special specimen holder for transmission electron microscopy (TEM), which has electromagnets generating magnetic field on ferromagnetic samples and four leads for electric measurements to investigate the magnetoresistance (MR) effect. In this research, we evaluated this TEM holder by experiments using Py pattern. It appears to be possible to apply the in-plane magnetic fields at any direction and intensity.

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- 16:00 I-V measurement of nano-region NiO in transmission electron microscope
 Authors : T. Fujii, H. Kondo, T. Kawanishi, H. Kaji, K. Hamada, M. Arita, Y. Takahashi
 Graduate school of Information Science and Technology, Hokkaido University
Resume : NiO thin films show resistance switching characteristics induced by voltage application. This phenomenon can be used to the nonvolatile memory named as the resistance random access memory (ReRAM). Although this phenomenon may relate to the nanostructure in the device, the mechanism of resistance switch is still obscure. To investigate the structural change during application of voltage, we observed NiO film by in situ transmission electron microscopy (TEM). To perform such experiments, a custom-made TEM holder was used, with which electric measurements can be done during TEM observation. Electrodes are movable and are controlled on the nanometer order. They were two sharp tips set in the TEM holder. One was a thin Pt-Ir tip fabricated by ion milling on which a NiO thin film was sputter deposited and annealed in air. The counter electrode was a sharp Pt-Ir tip made by an ion shadow method. After moving the counter electrode to contact to NiO, I-V measurements were performed during TEM observations. As recognized in TEM image, the Pt tip was covered by a about 25nm-thick NiO polycrystalline film. When we measured I-V characteristics during TEM observation, NiO film resistance changed from high state to low state at about 1 V. In the corresponding TEM image some contrast change was confirmed. It seems that some structure change occurred in NiO film. In addition, we confirmed the difference of local conducting characteristics in NiO film.

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- 16:00 Imaging of complex nanoparticles using low voltage SEM in scanning transmission electron image working mode
 Authors : A.L. Tóth (1), G. Filipcsei (2), G. Battistig (1), F. Darvas (2), L. Illes (1), G. Heltovics (2), I. Bársony(1) (1) MTA MFA, H-1121 Budapest, Konkoly-Thege u 29-33., Hungary (2) Nangenex Zrt, H-1031 Budapest, Záhony u. 7., Hungary
Resume : The scanning transmitted electron (TE) imaging (STEI) is a well established auxiliary working mode of scanning electron microscopes (SEM). Several technical solutions have been developed, like building a whole dedicated scintillator - multiplier combination for TE detection, substages with conversion of transmitted electrons into secondary ones (SE) and then using the Everhart-Thornley SE detector, or inserting a semiconductor charged particle detector under the thin sample. In this work a semiconductor detector was chosen for investigating complex active pharmaceutical nanoparticles

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using a LEO 1540XB Gemini LV-SEM. The STEI was found an optimal tool for characterization of these micro and nanosized powder samples. The shadow-like contrast of grains on uniform white background is well suited for applications like quantitative image analysis (where the surface morphology of the usual SE images is a drawback). Carefully setting the parameters the selective absorption of electrons within a complex grain provides information about the internal structure, unavailable from conventional secondary and backscattered electron images of the SEM.

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16:00 The structure of dislocations in metamorphic III-V heterostructures grown by molecular beam epitaxy
 Authors : Yi Wang¹, M.P. Chauvat¹, P.Ruterana¹, L. Desplanque², X. Wallart²
¹CIMAP UMR 6252 CNRS-ENSICAEN-CEA-UCBN, 6, Boulevard du Maréchal Juin, 14050 Caen Cedex, France ²Institut d'Electronique, de Microélectronique et de Nanotechnologie, UMR-CNRS 8520, BP 60069, 59652 Villeneuve d'Ascq Cedex, France

Resume : Lattice-mismatched epitaxy of Sb-based materials on GaAs is attracting much attention due to the numerous advances in optoelectronic devices that can be enabled. Between these compounds and the substrate, the mismatch is high (6-10%), and one expects a relaxed interface with misfit dislocations. In such zinc blend materials, the $\frac{1}{2}\langle 110 \rangle$ interface dislocations can be of Lomer type or 60° , probably depending on the growth conditions. In this work, we carry out an iterative investigation of the dislocations versus the growth conditions. At the interface, we determine their atomic structure. We also analyze the threading dislocation density all along the heterostructure using plan view as well as cross sectional TEM samples. Our aim is to contribute to finding the optimal conditions for the formation of a perfect network of Lomer interface dislocations, thus minimizing the density of the threading dislocations inside the active layers. The preliminary results indicate that the large majority of the interface dislocations are of Lomer type and the quantitative image analysis using the geometrical phase method shows that many are probably dissociated. Depending on the growth condition, the measured residual strain changes as well as the threading dislocation densities. Acknowledgment: This work is supported by project MOS35, project No.: ANR-08-NANO-022.

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16:00 Determination of the composition of a-Si/SiO₂ multilayer structures after light induced crystallization
 Authors : M. Schade¹, T. Mchedlidze², M. Kittler², B. Berghoff³, and H.S. Leipner¹
¹ Interdisziplinäres Zentrum für Materialwissenschaften, Martin-Luther-Universität Halle-Wittenberg, D-06099 Halle, Germany ² IHP/BTU Joint Lab, Brandenburgische Technische Universität Cottbus, D-03046 Cottbus, Germany ³ Institut für Halbleitertechnik, Rheinisch-Westfälische Technische Hochschule Aachen, D-52056 Aachen, Germany

Resume : High resolution transmission electron microscopy (HRTEM), electron energy loss spectroscopy (EELS) and Raman spectroscopy have been performed on individual layer structures consisting of silicon nano-crystallites embedded in a SiO₂ matrix. The nano-crystallites have emerged from amorphous silicon (a-Si) layers by performing light induced crystallization (LIC) [1] using a laser system with a power of around 310 mW. By deviating slightly certain parameters, i.e. the laser power, a modified degree of crystallization of the a-Si layers can be obtained. Raman-spectroscopic measurements (confocal spot size 1 μm^2) have been performed on these layer structures in order to determine their degree of crystallinity on a macroscopic level. In addition, EELS investigations were carried out in order to verify the Raman results on a nanoscopic level. Therefore, multiple least squares fitting was applied by comparing spectra of the silicon L_{2,3} edges measured from the nano-crystallites with separately measured reference spectra of bulk silicon, a-Si and SiO₂. The investigations were completed by HRTEM micrographs in order to determine the size distribution of the nano-crystallites. A correlation between the degree of crystallization and the size of the nano-crystallites is supposed. References: [1] T. Mchedlidze, T. Arguirov, S. Kouteva-Arguirova, M. Kittler, R. Rölver, B. Berghoff, D. Bätzner and B. Spangenberg, Phys. Rev. B 77 (2008) R161304

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- 16:00 Scanning electron microscopy and related Raman mapped images of YBCO films
 Authors : M. Branescu, National Institute for Materials Physics, Bucharest, Romania. I. ward, EAG, Sunnyvale, CA, USA. E. Leroy, Horiba Jobin Yvon Inc., Edison , NJ, USA.
Resume : Recently we presented and developed a new nanoscale approach regarding 3D structures configured on YBCO films by Raman profiling system. Now we present and review our experiments on Raman mapped images and correlated SEM images showing the submicron boundaries between grains with different crystalline orientations and the surface aspect of the film, respectively. We used Raman mapped images of a predominantly c-axis oriented thin YBCO film with few a-axis and mixed phases grains. We analyze and detail new progress in engineering 3D quantum electronic devices by using both in-depth Raman profiling spectra (for passive components patterned on a predominantly c-axis oriented film) and Raman mapped images (for active components, patterned at a clear delimited planar boundary between an a-axis and a c-axis oriented grains).
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- 16:00 Multiscale STEM characterization of nanostructured Al-Si-Zr alloys obtained by rapid solidification method
 Authors : Mariusz Andrzejczuk, Małgorzata Lewandowska, Jerzy Latuch, Krzysztof Jan Kurzydłowski; Warsaw University of Technology, Faculty of Material Science and Engineering, Woloska 141, 02 – 507 Warsaw
Resume : The properties of engineering metallic alloys (e.g. fracture toughness, corrosion resistance) are often limited by the presence of primary intermetallic particles which form as a result of segregation of impurities in conventional solidification. Rapid solidification brings about much more chemically homogenous amorphous and/or nanocrystalline structure. Rapidly solidified thin ribbons obtained by melt spinning are usually considered as intrinsically homogenous. However, due to different cooling conditions at the wheel surface and on the side exposed to the ambient environment, structure of such ribbons may vary significantly across its thickness. The materials studied in the present study were 30-40 μm thickness ribbons of nanocrystalline hyper- and hypoeutectic Al-Si-Zr alloys produced by melt spinning method. X-ray diffraction examinations revealed in the ribbons nanocrystalline α -Al phase, with the grain size in the range from 20 to 50 nm, depending on the composition of the solidified alloy. High resolution STEM Hitachi HD2700 was used to quantitatively characterize the structure homogeneity across the ribbons. Thin foils for STEM observations were prepared by Focused Ion Beam (FIB) system. Microstructural observations confirmed nanocrystalline character of Al-Si-Zr alloys. However, these observations revealed inhomogeneity of the structure across the ribbon width. The results were correlated with mechanical properties determined via microhardness measurements.
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- 16:00 Fabrication and Characterisation of different families of ordered Nanostructures by Self-Assembly and Metal Assisted Etching
 Authors : L. BOARINO1, E. ENRICO1, N. DE LEO1, F. CELEGATO1, P. TIBERTO1, N. PUGNO2, AND M. LAUS3 1 NanoFacility Piemonte, Electromagnetism Division, Istituto Nazionale di Ricerca Metrologica, Strada delle Cacce 91 - 10135 Turin, Italy; E-mail: l.boarino@inrim.it; Tel: +39 (11) 3919640. 2 Laboratory of bio-inspired Nanomechanics "Giuseppe Maria Pugno", Dept. of Structural and Geotechnical Engineering, Politecnico di Torino, C.so Duca degli Abruzzi 24 - 10129 Turin, Italy. 3 Dipartimento di Scienze dell'Ambiente e della Vita, Università del Piemonte Orientale Amedeo Avogadro, Viale Teresa Michel 11 – Alessandria, Italy.
Resume : The combination of two recent techniques developed in the last years demonstrates the possibility to obtain regular and semi-ordered nanostructures like nanowires, nanopillars, mesopores or nanostructured thin films on any type substrates and with holes diameter ranging from 800 nm to 100 nm. Self-assembly of polystyrene, PMMA and PTFE nanospheres in a hexagonal close packed structure is obtained by floating technique, then the 2D crystal is lifted on a silicon substrate. The nanospheres can be reduced in diameter by Reactive Ion Etching in O₂ atmosphere, then thin films deposition is performed. In this way a typical "antidot" structure is obtained. To obtain 8
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the complementary "dot" structure, a thin continuous film is deposited before nano-spheres self-assembly, then few minutes of sputter-etching treatment in Ar+ removes the thin film in the zones not masked by the nanospheres. By these two main procedures, different families of nanostructures can be obtained, from metallic or magnetic nano-arrays, to nanopillar and nanowires by means of a successive step of Metal Assisted Etching (MAE). Ordered and regular nano-pillars, nanowires and mesopores have been fabricated with this method on a large area. In this field, electron microscopy is playing a major role, thanks to the direct inspection and morphological analysis, but also to the direct modification of the soft masks by Electron or Ion Beam, and in-situ nanomanipulation and electrical characterisation.

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- 16:00 Analytical Electron Tomography Mapping of the SiC Pore Oxidation at the Nanoscale
 Authors : I. Florea(a), L. Roiban(a), A. Deneuve(b), K. Chirazi(b), I. Janowska (b), D. Ihiwakrim(a), C. Hirlimann(a), C. Pham-Huu(b), O. Ersen(a) (a)-Institut de Physique et Chimie des Matériaux de Strasbourg (b)-Laboratoire des Matériaux, Surfaces et Procédés pour la Catalyse, Strasbourg
Resume : Silicon Carbide is a ceramic material that has been widely studied for its potential applications, ranging from electronics to heterogeneous catalysis. Recently, β -SiC, has attracted overgrowing interest as a new class of catalyst support with high specific surface area and thermal conductivity. A primary electron tomography study has revealed a dual surface structure defined by networks of connected channels with sizes larger than 50 nm and ink-bottled pores with sizes going from 4-50 nm. Inserting Pd nanoparticles in this porous network by means of a wetness impregnation method, their selective localisation with respect to the two surfaces depends on the surface tension of the solvent, illustrating a selective wetting titration of the two types of surfaces by different liquids. The hypothesis that has been putted forward was that this selectivity against solvents could be related to the pore surface reactivity, in particular to the oxidation degree of the two types of pores. The new technique of analytical electron tomography, where the series of projections used to reconstruct the volume is recorded in energy filtered mode, has been implemented to map the pore oxidation state and to correlate it with the morphology and the accessibility of the porous network. In this framework, our study highlights the great interest of this method by accessing the pore oxidation state of a material; we found thus that the interconnected channel pores are more oxidized than the ink-bottled pore.

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Quantitative electron microscopy for research and industry

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start at	Subject	Num.
08:30	<p>Electron Microscopy of Interfaces : J. Calvino</p> <p>Using Electron Microscopy to Measure Interface Energy Authors : M. Baram, H. Meltzman, and W.D. Kaplan Department of Materials Engineering, Technion – Israel Institute of Technology, Haifa, 32000, Israel Resume : While sessile drop experiments provide important information on solid-liquid interfaces, the solid-solid interface energy and its influence on adhesion is important for fundamental science and technological applications. It is the purpose of this presentation to explore methods to measure solid-solid interface energies, specifically applied to metal-ceramic systems. Samples were produced by dewetting thin solid metal films (Au and Ni) on single crystal sapphire substrates, which leads to the formation of sub-micron solid particles equilibrated with the substrate. In addition to pure metal-ceramic systems, Au films were dewetted in the presence of anorthite glass. This allowed for the comparison of interfacial energies for clean metal-ceramic interfaces, and interfaces with an intergranular film. Cross-section transmission electron microscopy (TEM) samples of specific particles were prepared using a dual-beam focused ion beam system, complimented by low-voltage ion polishing. The convoluted Wulff shape of the particle-substrate and thus the interface energy, dihedral angles providing estimates of the torque terms, the orientation relationship between the particles and the substrate, and the interface structure and chemistry were characterized using a monochromated and aberration corrected TEM. The existence of nanometer-thick intergranular films at Au-sapphire interfaces has been experimentally verified. Aberration corrected TEM proved the existence of structural order at the interface, which serves as part of the entropy term defining the interface energy. Formation of an intergranular film significantly reduces the interfacial energy, increasing the thermodynamic work of adhesion, offering a unique method to improve adhesion at metal-ceramic interfaces.</p>	9 1
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09:00	<p>Cross-sectional TEM investigations of metallic nanoparticle arrays formed by nanosphere lithography Authors : J.K.N. Lindner 1,2,3, M. Weini 1,2, C. Seider 2, D. Gogel 2, D. Kraus 2, F. Reichardt 2, A. Sesselmann 2, B. Stritzker 2 1 University of Paderborn, Department of Physics, 33098 Paderborn, Germany 2 University of Augsburg, Institute of Physics, 86135 Augsburg, Germany 3 CeOPP Center for Optoelectronics and Photonics Paderborn, 33098 Paderborn, Germany Resume : Metallic nanoparticle arrays on semiconductor surfaces exhibit exciting optical properties due to plasmonic effects and are therefore intensively studied at present. A fast and cost-effective technique to fabricate such nanoparticle arrays is nanosphere lithography (NSL), in which a self-assembled monolayer of equally sized spheres from a colloidal suspension is used as a shadow mask for a metal deposition process, resulting in the formation of nanoparticles with a roughly triangular foot print and a seemingly simply predictable 3D-shape. In order to understand the optical properties of such particle arrays in detail, exact knowledge of the particle morphology, composition as well as inner and interfacial structure is essential. Since nanomasks are often made from polystyrene spheres just weakly adhering to the substrate, they are difficult to prepare for cross-sectional TEM (XTEM). This may be the reason why so far the NSL process has not been studied by TEM. Here it is shown by conventional, high-resolution and analytical XTEM</p>	9 2

that the particle morphology strongly depends on the particle material (Au, Ag, Co, Ti, ZnO) and deposition technique (PVD, sputtering). Some particles exhibit compositional changes due to high chemical reactivity. It is demonstrated that modifications of the mask shape using ion beam and plasma techniques can be monitored by XTEM and that ultimate control of nanoparticle morphologies requires control of the nanoparticle/substrate interfaces.

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09:15

Dopants segregation analysis on Sb:SnO₂ nanocrystals

Authors : Daniel G. Stroppa¹, Luciano A. Montoro¹, Armando Beltrán², Rafael O. da Silva³, Juan Andrés², Edson R. Leite³, Antonio J. Ramirez^{1*} ¹ Brazilian Synchrotron Light Laboratory, Campinas, SP, Brazil. ² Departament de Química Física i Analítica, Universitat Jaume I, Castellón de la Plana, Spain. ³ Department of Chemistry, Federal University of São Carlos, São Carlos, SP, Brazil

Resume : The development of novel and reliable nanostructured devices requires the ability to synthesize and characterize materials on the atomic scale. Among the most significant challenges in nanostructural characterization is the evaluation of crystal growth mechanisms and their dependence on the distribution of doping elements. This work describes a new methodology for the evaluation of Sb dopant segregation on SnO₂ nanoparticles (3-5 nm) and discusses its effects on nanocrystal morphology and growth mechanism. Pure and Sb-doped (7%atom, 18%atom) SnO₂ nanocrystals obtained by a nonaqueous synthesis route were characterized by the combined use of HRTEM characterization and surface energy ab initio calculations. HRTEM characterization was performed using a JEM-3010 URP at 300 kV with a LaB₆ electron gun. Wulff construction using the ab initio calculated surface energies was applied to model the nanocrystals. The results show a composition-dependent dopant segregation behavior towards different nanocrystal facets and its effect on the Sb:SnO₂ nanoparticle morphology. These findings were evaluated in terms of overall surface energy minimization. In addition, oriented attachment was identified as the predominant growth mechanism for the studied systems and the observed preferential growth directions could be successfully described on the light of the ab initio calculated surface energies. Keywords: HRTEM, Dopant Distribution, Oriented Attachment, Surface Energy ab initio Calculation. 1- Stroppa, D. G., Montoro, L. A., Beltran, A., Conti, T. G., da Silva, R. O., Andres, J., Longo, E., Leite, E. R., Ramirez, A. J.; Unveiling the Chemical and Morphological Features of Sb:SnO₂ Nanocrystals by the Combined Use of High-Resolution Transmission Electron Microscopy and ab Initio Surface Energy Calculations. Journal of the American Chemical Society, v.131, p.14544 - 14548, 2009. ramirez@lnls.br – Caixa Postal 6192, Campinas-SP 13083-970, Brazil

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09:30

Mechanical Deformation of Gold Atomic-size Nanorods Studied by Time-resolved HRTEM

Authors : M.J.Lagos ^{*}, F.Sato ^{**}, D.S.Galvao [^], D.Ugarte ^{^*} Brazilian Synchrotron Light Laboratory - Campinas, Brazil ^{**} Universidade Federal de Juiz de Fora - Minas Gerais, Brazil [^] Universidade Estadual de Campinas - Campinas, Brazil

Resume : The mechanical properties of a strained nanoscale volume of matter represent a fundamental issue for understanding phenomena such as friction, fracture, adhesion, etc. Miniaturization levels are raising the need of accurately characterizing nanodevices and nanomaterials, to develop models and predictions of their mechanical performance and reliability. But, we must also consider that the deformation of macroscopic matter continues to be a very interesting and dynamic research field displaying open questions and polemical issues. In this work, we have analyzed the atomistic aspects of the elongation of one-nm-wide metal rods as function of temperature using in-situ high resolution electron microscopy. For gold nanorods(NR) no extended defect could be observed at room temperature, while at 150 K stacking faults (SF) and twins (TW) are generated frequently. In fact, we have observed that as their size is reduced, the energy barriers became very small that thermal ambient energy is sufficient to overcome them. Then, NRs display an extended elastic regime until a mechanism with high enough blocking barrier

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can be nucleated. We have also determined experimentally the barrier energy associated to SF recombination process, which is about 40 meV. From our TEM observations we have determined the mechanism of deformation which is based on compact slip over (111)planes. We have also developed ab initio calculations to derive the total energy changes associated with SF formation in NRs. Ab-initio calculations revealed that contribution from surface steps overrule stacking fault energetics in NRs, in such a way that system size and shape determines preferred faults gliding directions. The relation between morphology and surface steps can produce anisotropic behavior and, even large differences in elastic or plastic response for elongation or compression.

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09:45

Understanding Growth and Doping in Group IV Semiconductor Nanowires
 Authors : Eli Sutter and Peter Sutter Center for Functional Nanomaterials, Brookhaven National Laboratory, Upton NY 11973, USA
Resume : Semiconductor nanowires (NWs), self-assembled nanostructures that can be prepared in high-yield processes [1] based on vapor-liquid-solid (VLS) growth, could become the basis for novel electronic device architectures that avoid complex top-down processing. The successful realization of practical devices requires a fundamental understanding of NW growth and doping. Central to the VLS growth process is a liquid metal-semiconductor alloy seed drop whose phase diagram at or close to equilibrium with the adjacent NW governs important aspects of the growth. For example, for a fixed amount of metal the equilibrium composition of the binary alloy defines the size of the seed drop, which in turn controls the diameter of the NW. Given the small size of the alloy drop, typically few tens of nanometers, the known binary bulk phase diagrams cannot provide a reliable basis for predicting such growth phenomena. Hence, we have developed in-situ microscopy techniques to establish the nanoscale phase diagrams of alloy seed drops used in VLS NW growth. Variable-temperature transmission electron microscopy on individual Au-Ge alloy seeds at the tips of Ge NWs was used to measure key features of the phase diagram of the nanoscale alloy [2, 3]. To illustrate the predictive power resulting from knowledge of the true phase diagram of the nanoscale VLS seed drop, we demonstrate control over the local, position-dependent diameter of the growing NW. The phase behavior of the VLS seed will also be key to developing strategies for doping of NWs. To ensure homogeneous doping of the growing NW, rather than conformal deposition of a doped shell around it, the preferred incorporation of dopant is via the VLS drop. We demonstrate successful in-situ Sb-doping of Ge NWs during their synthesis by thermal evaporation at moderate temperatures using Ge and Sb powders [4]. We use our in-situ microscopy method to identify the mechanisms of doping during NW growth. We study the phase behavior of the catalyst alloy drops, which now include a dopant species in addition to the metal and semiconductor, and show that dopants are incorporated in the growing NW via the ternary alloy VLS drops. [1] E. Sutter, B. Ozturk, and P. Sutter, Nanotechnol. 19, 435607 (2008). [2] E. Sutter and P. Sutter, Nano Lett. 8, 411 (2008). [3] E. Sutter and P. Sutter, submitted. [4] E. Sutter and P. Sutter, Appl. Phys. A (published on-line).

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10:00

Coffee Break

Electron Microscopy of Thin Layers and Nanomaterials : H. Bender

10:30

QUANTITATIVE ELECTRON MICROSCOPY TO CHARACTERIZE SOLID OXIDE FUEL CELL DEGRADATION

Authors : A. Hessler-Wyser¹, A. Faes^{1,2}, J. A. Schuler^{1,2}, Z. Wuillemin², J. Van herle²
¹ Interdisciplinary Centre for Electron Microscopy (CIME), Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015 Lausanne, Switzerland
² Laboratory of Industrial Energy Systems (LENI), Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015 Lausanne, Switzerland

Resume : Clean energies are a clear objective for most countries and many efforts are made to reduce energy consumption and carbon dioxide emission. Amongst the numerous options, solid oxide fuel cells (SOFC) represent an attractive conversion technology, as they are able to cogenerate electricity and heat with especially very high electrical efficiency (60%) already at small scale (kW). They are considered as an answer for decentralised production at small to medium scale and represent a step towards sustainable development.

A solid oxide fuel cell is composed of (i) a porous ceramic cathode, (ii) a dense membrane made out of an ion conducting ceramic (the solid electrolyte), and (iii) a composite ceramic-metal anode. It allows to generate electricity by electrochemical reaction between a fuel and an oxidant gas, and their operating temperature may range between 600 and 1000 °C. In a stack, cells are separated by interconnects that avoid mixing of fuel and air and ensure electrical connection. The performance of a stack degrades during operation and occurs at different levels. Cell degradation is a crucial technological issue prior to SOFC commercialization, and considerable progress has been achieved. A number of degradation pathways has been identified, amongst them microstructural changes, poisoning effects and formation of less conductive phases. In the case of this work, the mechanical support of planar SOFCs is the anode, fabricated as a nickel and yttria stabilized zirconia composite (YSZ). It is co-sintered with a few micron thick membrane of dense YSZ electrolyte, on top of which a 20-40 µm layer of lanthanum strontium manganite (LSM) cathode is subsequently deposited. The interconnects are usually Cr-rich ferritic steels. Electron microscopy and its related techniques is a powerful tool to quantify microstructural changes and pollution effects occurring during SOFC operation, at different levels of the cell. In the anode, low accelerating scanning electron microscopy (SEM) imaging is used to separate the three anode phases (nickel, YSZ and porosity). Microstructural quantification is obtained using a software code that yields phase proportion, particle size, particle size distribution and a direct measurement of triple phase boundary (TPB) density. A model that describes the gradual degradation of the anode due to nickel particle sintering and the concomitant loss of TPB has been considered, and the combination of experimental results and modelling allows separating the degradation due to sintering of nickel from the total stack degradation of stacks tested for medium duration (2000 h) [1]. On the cathode side, pollution and secondary phase formation may alter the cell performances. In particular, pollutant species contained in the air flow can deposit at the cathode/electrolyte interface or at the current collection surface layer and reduce either the catalysis or mass transfer e.g. by pore blocking. Cr, even in small quantities, is known to be a severe pollutant, but its detection along with LSM appeared to be challenging by energy dispersive spectroscopy (EDS), because of strong overlaps of the X-ray emission lines. Systematic SEM, EDS and spectral simulations performed in house demonstrated it was possible to quantify the amount of Cr deposited in such polluted cathodes when operating the SEM at 30 kV, at the expense of the spatial resolution. As a complementary method, wavelength dispersion spectroscopy (WDS) gave the Cr localization at the cathode/electrode interface [2]. In order to investigate secondary phase formation at the cathode/electrolyte interface, TEM lamellae, extracted from a cell (operated for 2000 h) by focussed ion beam (FIB), have been analysed by scanning transmission electron microscopy (STEM) and EDS (see figure below). Zirconate formation has been evidenced in the polarised regions of the cell, together with delamination of the sintered grains. Moreover, very thin Cr oxide layers have been localized at the LSM/zirconate interface. Energy filtered TEM (EFTEM) has been undertaken to remove any ambiguity on the Cr detection. Furthermore, Cr-S compounds have been found, revealing the impact of sulfur poisoning, even from ambient air, on the cathode microstructure [3]. A main source of Cr is the metallic interconnect (MIC) and possible protection layers against Cr evaporation have been tested. Elemental depth profiling and microstructural analyses on MIC cross-sections have been carried out, revealing interfacial reactions (SrCrO₄ formation) and subsurface silica formation as potential problems. 1] Faes A, Hessler---Wyser . A, Presvytes D, Vayenas CG, Van Herle J. Nickel---Zirconia . Anode Degradation and Triple Phase Boundary Quantification from Microstructural Analysis. Fuel Cells 2009;9:841. [2] Schuler AJ, Wullemin Z, Hessler--Wyser A, Van Herle J. Sulfur as pollutant species on the cathode side of a SOFC system. vol. 25. Vienna, 2009. p.2845. [3] Wullemin Z, Nakajo A, Müller A, Schuler AJ, Diethelm S, Van Herle J,

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11:00

Structural characterization of thin Co₂FeSi films on GaAs(111)B
 Authors : Bernd Jenichen, Jens Herfort, Kazuhide Kumakura, Achim Trampert.
 Paul-Drude-Institut für Festkörperelektronik, Hausvogteiplatz 5-7,D-10117

Berlin, Germany.

Resume : Co₂FeSi is a ferromagnetic half-metal with a Curie temperature larger than 1100K. The lattice parameter matches that of GaAs. Therefore, Co₂FeSi is suitable for spin injection into GaAs-based structures such as spin light-emitting diodes. Co₂FeSi/GaAs(111)B hybrid structures are grown by molecular beam epitaxy and characterized by transmission electron microscopy. Our films on GaAs(111)B have a stable interface up to a growth temperature $T_{\{S\}}=275^{\circ}\text{C}$, which is 75 K higher than the temperature guaranteeing a stable interface during growth on GaAs(001). Despite the vanishing misfit between the Co₂FeSi film and the GaAs buffer layer, we found a typical grain structure of the film, which is strong evidence for three-dimensional island growth during the stage of heteroepitaxy before a continuous film is formed. The films contain the fully ordered L2_{1} and the partially ordered B2 phases. The long-range order of the Co₂FeSi lattice improves with increasing growth temperature $T_{\{S\}}$. However, near the FM/SC interface we often find some disordering (B2 phase) especially at higher $T_{\{S\}}$. The spatial distribution of long-range order in the films is imaged using a comparison of superlattice and the corresponding fundamental reflections. The order parameters, i.e. the fraction of Si atoms occupying the Fe and the Co sublattices are determined quantitatively. Spatial inhomogeneities of long-range order can be explained by local non-stoichiometry.

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- 11:15 TEM investigation of Sn-ZnO nanorod heterostructures
 Authors : Y. Ortega^{1, 2}, Ch. Dieker¹, W. Jäger¹, J. Piqueras² and P. Fernández²
¹ Institute of Materials Science, Christian-Albrechts-University of Kiel, 24143 Kiel, Germany ² Departamento de Física de Materiales, Facultad de Ciencias Físicas, Universidad Complutense de Madrid, 28040 Madrid, Spain
Resume : The synthesis and characterization of one dimensional monocrystalline metal/semiconductor heterostructures is a subject of increasing interest due to their potential applications as electrical nanocomponents and thermal nanodevices. In the present work, Sn doped ZnO nanostructures grown by thermal treating of compacted ZnS and SnO₂ powders, were investigated by TEM imaging, selected area electron diffraction (SAED), and spatially-resolved energy-dispersive x-ray spectroscopy (EDS). TEM investigations reveal that the grown structures are nanorods with a complex and inhomogeneous radial distribution of extended defects, which includes dislocations, precipitates and voids of different sizes and shapes. A detailed investigation of some of the as-grown structures reveals also the presence of ZnO nanotubes partially filled with single-crystalline Sn. In-situ TEM experiments demonstrate that under proper electron beam focussing, the Sn melts and expands when the Sn core is connected with a large hollow channel. The reversible thermal expansion behaviour of Sn, that was directly monitored and recorded, is a promising approach for applications of these heterostructures as nanothermometers. In addition, when the Sn core ends at the tip of the rods the controlled formation of a Sn nanodrop, which emerges at the front face of the rod, can lead to future technological applications as nano-soldering devices.

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- 11:30 Compositional Distribution on InAs-GaSb quantum dots by Low Loss Electron Energy Loss Spectroscopy
 Authors : Ana M. Beltrán^{1*}, Ana M. Sánchez², Teresa Ben¹, Mhairi H., Gass^{3†}, Alfonso G. Taboada⁴, José M. Ripalda⁴ and Sergio I. Molina¹
¹ Departamento de Ciencia de los Materiales e I.M. y Q.I., Facultad de Ciencias, Universidad de Cádiz, Campus Río San Pedro, s/n, 11510 Puerto Real, Cádiz, Spain ² Physics Department, University of Warwick, Coventry CV4 7AL, United Kingdom ³ UK SuperSTEM, Daresbury Laboratory, Daresbury WA4 4AD, United Kingdom. ⁴ Instituto de Microelectrónica de Madrid (CNM, CSIC), Isaac Newton 8, 28760 Tres Cantos, Madrid, Spain *Corresponding author: ana.beltran@uca.es †now at: Serco, Technical Services, Warrington, WA3 6GA, United Kingdom
Resume : Self-assembled InAs(Sb)GaAs quantum dots (QDs) have received much attention since they are able to emit at 1.3-1.55 μm even at room temperature. This emission range is very interesting for telecommunication lasers and opto-electronic applications. The analysis of these semiconductor

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quaternary alloys by core-loss Electron Energy Loss Spectroscopy (EELS) is difficult because the signals associated with the elements that constitute these materials are overlapped and/or they exhibit a similar shape. In this case, it is necessary to use an alternative technique, such as Low Loss Electron Energy Loss Spectroscopy (L-L EELS) to analyse the presence of these elements. This energy range (0-60 eV) is dominated by the zero loss peak, the collective excitations known as plasmons and of the ionisation electrons in the valence bands to empty states in the conduction bands. The application of a methodology for the analysis of L-L EELS has allowed the compositional distribution of III-V semiconductor ternary and quaternary alloys containing In, Ga, As and Sb to be determined. The heterostructure to be analysed here consists of InAs quantum dots (QDs) grown by molecular beam epitaxy (MBE) and covered by a GaSb layer, prior to the deposition of a final GaAs capping layer. The formation of the quaternary alloy $\text{In}_x\text{Ga}_{1-x}\text{Sb}_y\text{As}_{1-y}$ has been detected due to In and Sb segregation. Results have been confirmed by the combination of several techniques such as conventional transmission electron microscopy or high-angle annular dark field.

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11:45

Quantitative analysis of the interfacial intermixing in InAs/GaSb short-period-superlattices using transmission electron microscopy
 Authors : E. Luna, B. Satpati, J.B. Rodriguez*, A.N. Baranov*, E. Tournié* and A. Trampert Paul-Drude Institute for Solid State Electronics, Berlin, Germany
 * Université Montpellier 2, CNRS-IES, Montpellier, France
Resume : InAs/GaSb short-period-superlattices show unique potential device applications, which are strongly dependent on the quality of the interfaces. While most of the studies focus on the interfacial roughness (i.e., steps and islands), little is known about the interfacial intermixing (i.e., composition profile). We propose an innovative method for a reliable and systematic characterization of these noncommon-atom interfaces. It is based on transmission electron microscopy (TEM) and, although a dedicated data analysis is required, all the steps are straightforward to implement. In particular, the compositional sharpness is obtained from the comparison of the experimental contrast in chemical sensitive g002 two-beam dark-field (DF) TEM micrographs with simulated intensity profiles. The later are calculated assuming that the element distribution profiles are described by sigmoidal functions, with the interface width as the main fitting parameter. The procedure (i.e. search of the composition profile that after being input into the calculation best fits the experiment) would resemble the analysis of x-ray diffraction data. The method is of general applicability and not only restricted to the InAs/GaSb case. The interfacial intermixing is determined for different case studies samples, even in the extreme case where nominally less than 3 monolayers (ML) thick layers are concerned. The limitations of the method are discussed, but it should be mentioned that it allows for the detection of variations in the interface width that are smaller than the resolution of the 002 DF TEM imaging. Moreover, it is possible to identify the presence of nominally 1 ML-thick interfacial layers and to evaluate the existence of In and/or Sb segregation.

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12:00

Quantitative Transmission Electron Microscopy of W/C Multilayer Coatings for X-ray Optics
 Authors : D. Häussler(1), U. Ross(1), U. Heidorn(2), F. Hertlein(2), J. Wiesmann(2) and W. Jäger(1) 1 - Microanalysis of Materials, University of Kiel, 24143 Kiel, Germany, 2 - Incoatec GmbH, 21502 Geesthacht, Germany
Resume : Multilayer coatings consisting of ultrathin bilayers on the nanometer scale are essential components of X-ray optics for advanced X-ray analytical equipment and for synchrotron beam lines. Periodic multilayer systems constitute the basis for monochromators of small spectral bandwidth whereas aperiodic multilayer systems are used as advanced X-ray optical components for large spectral bandwidth synchrotron applications. The development of novel multilayer systems requires precise monitoring of the multilayer parameters such as layer thickness, layer periodicity and uniformity, and interface quality. High-angle annular dark-field scanning TEM (HAADF-STEM) was applied to cross-section imaging of periodic and aperiodic W/C multilayer systems and their interfaces and was combined with analyses of image intensity profiles. It is shown that the layer thickness on a local scale

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can be determined with sub-nanometer precision. Generally good agreement with nominal data is obtained except for systems containing near-substrate multilayer regions with growth-induced thickness fluctuations. Analyses of the position and the width of the interface contrast were employed for the quantitative determination of the interface roughness. This procedure yields separate values for the lateral interface roughness and for interface broadening. For periodic multilayer systems the result shows excellent agreement with data obtained from X-ray reflectivity measurements.

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12:15 Lunch Time

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