MM 59: Topical session: In-situ Microscopy with Electrons, X-Rays and Scanning Probes in Materials Science VII - Nanomaterials

Time: Thursday 15:45-18:30

MM 59.1 Thu 15:45 H38

In situ off-axis electron holography of magnetic nanoparticles — •RAFAL E. DUNIN-BORKOWSKI, ANDRÁS KOVÁCS, and JAN CARON — Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons and Peter Grünberg Institute, Forschungszentrum Jülich, Jülich, Germany

Off-axis electron holography is a powerful technique for recording the phase shift of a high-energy electron wave that has passed through an electron-transparent specimen in the transmission electron microscope. The phase shift is, in turn, sensitive to the electrostatic potential and magnetic induction in the specimen, projected in the electron beam direction. We are currently using the technique to characterize the magnetic properties of individual and closely-spaced deep-submicronsized nanoparticles, nanostructures and thin films that are subjected to externally applied magnetic fields in situ in the transmission electron microscope, as well as to elevated and reduced temperature. We are also working on a model-based approach that can be used to reconstruct the three-dimensional magnetization distribution in a specimen from a series of phase images recorded as a function of specimen tilt angle using off-axis electron holography. We make use of a forward simulation approach within an iterative model-based algorithm to solve the inverse problem of reconstructing the three-dimensional magnetization distribution in the specimen from tilt series of two-dimensional phase images recorded about two independent tilt axes. In such applications of off-axis electron holography, the effects of electron beam induced charging and dynamical diffraction should be minimized.

MM 59.2 Thu 16:15 H38

Encapsulated silver nanowire electrodes: applications, computer based analysis and stability tests — •MANUELA GÖBELT¹, RALF KEDING¹, MATTHIAS BÜCHELE¹, BJÖRN HOFFMANN¹, and SILKE CHRISTIANSEN^{1,2} — ¹Max-Planck-Institute for the Science of Light, Erlangen, Germany — ²Helmholtz Zentrum Berlin für Materialien und Energie, Berlin, Germany

Transparent electrodes are a critical component in optoelectronic devices like solar cells. We developed a novel nano-composite transparent electrode material composed of a wet-chemically synthesized silver nanowire (AgNW) network encapsulated in a transparent conductive oxide which was deposited with nano-scale precision by atomic layer deposition. To demonstrate the performance of the AgNW/AZO electrode, it was used as a top electrode on different optoelectronic devices. With a combination of scanning electron microscopy images and computer based image analysis we can characterize the AgNW networks and determine the percolation of them on large areas. For the object recognition "ImageJ" serves as a basis and a combination of existing plugins and self-written software is used. The new software is capable of handling high aspect ratio objects in an interconnected network, where every single object has several intersections with others. It is possible to distinguish the single wires and to reconstruct their original shape although they are partly covered by others objects. We also take a detailed look at the stability of these nano-composite electrodes under ambient conditions and we will show how the AZO encapsulation shelters the AgNW networks from oxidation and sulfurization.

MM 59.3 Thu 16:30 H38

Structural and optical investigation of colloidal gold platelets for high-quality plasmonic applications — \bullet BJÖRN HOFFMANN¹, MUHAMMAD BASHOUTI¹, THORSTEN FEICHTNER^{2,1}, MIRZA MAČKOVIĆ³, CHRISTEL DIEKER³, AHMED SALAHELDIN⁴, PETER RICHTER⁵, OVIDIU GORDAN⁵, DIETRICH ZAHN⁵, ERDMANN SPIECKER³, and SILKE CHRISTIANSEN^{2,1} — ¹Max Planck Institute for the Science of Light, 91058 Erlangen — ²Helmholtz Zentrum Berlin für Materialien und Energie GmbH, D-14109 Berlin — ³Friedrich-Alexander-Universität Erlangen-Nürnberg (FAU), Lehrstuhl für Mikro- und Nanostrukturforschung (WW9) & Center for Nanoanalysis and Electron Microscopy, Department Werkstoffwissenschaften, 91058 Erlangen — ⁴FAU Erlangen-Nürnberg, Institute of Particle Technology, 91058 Erlangen — ⁵Semiconductor Physics, Technische Universität Chemnitz, 09107 Chemnitz

Wet-chemically synthesized ultraflat gold platelets are an ideal building block for high-quality plasmonic applications. They show superior Location: H38

properties compared to vapor-deposited gold layers and enable yet unknown fabrication precision. Even though gold platelets are already frequently used in the field of plasmonics, many details about their intrinsic structure as well as their optical properties are still unknown.

Here, we present a detailed TEM study that proves the existence of twin boundaries inside the particles. Furthermore, we have determined the complex dielectric function of single gold platelets by using microellipsometry. To demonstrate the outstanding quality of this material, we prepared ultrathin, single-crystalline layers as thin as 15 nm.

MM 59.4 Thu 16:45 H38 The role of CTAB micelles in gold nanorod synthesis - a combined SAXS/SANS study — •TILO SCHMUTZLER, TORBEN SCHINDLER, and TOBIAS UNRUH — Friedrich-Alexander-University Erlangen-Nuernberg, Chair for Crystallography and Structural Physics, Staudtstrasse 3, 91058 Erlangen, Germany

Au nanoparticles have been the subject of widespread research in the last two decades. Applications are expected in biological imaging, drug delivery and phototherapeutics.

The common wet chemical synthesis of gold nanorods (AuNRs) is the seed-mediated growth synthesis route.[1] Therefore small seed particles are used to grow nanorods in a solution of CTAB (Cetyltrimethylammonium bromide) as structure directing agent.

The behaviour of CTAB in solution and so the influence on the final AuNR formation can be influenced by additives like inorganic salts. Increasing concentrations of KBr for example lead to a slower formation kinetic of AuNRs which was investigated by time-resolved small angle X-ray scattering (SAXS) simultaneously to UV-Vis spectroscopy. Additionally the rod formation beside other morphologies is less pronounced compared to the synthesis in solutions without further KBr. The influence of CTAB as catalytically active component is obvious due to the change of the scattering related to the micelles itself. Therefore small angle neutron scattering (SANS) is perfectly suitable to analyze the structure of CTAB modified by different additives to understand the behavior during AuNR formation.

[1] C.J. Murphy et. al, J. Phys. Chem B. 2005, 109, 13857-13870.

15 min. coffee break

MM 59.5 Thu 17:15 H38 In Situ Methods for Studies of Mechanically, Thermally, Electron Beam and Liquid Induced Effects in Nanostructured Materials — •EVA OLSSON — Chalmers University of Technology, Göteborg, Sweden

In situ electron microscopy allows the imaging of transport of matter and charges in complex structures as well as heat spread. We can study mechanically and thermally induced changes of charge transport properties using holders designed to enable different stimuli allowing the direct observation and correlation between material structure and properties. This talk addresses examples of in situ mechanical, thermal and liquid studies. We have studied the effect of mechanical strain on the electrical resistance of nanowires. Electron energy loss spectroscopy was used to study the effect of strain on the electronic structure with emphasis on the low energy loss interval of 0 to 50 eV. Electron beam induced current measurements were also performed to study the effect of strain on the diffusion length of the charge carriers. Heating of a transmission electron microscopy specimen can be performed in several parallel modes and this talk will address three types of heating modes and show experimental results from nanostructured materials. The talk will also show a technique for in situ wetting of materials in the environmental scanning electron microscope and applied it to suspended coatings. The method uses a manipulator to bring the specimen in contact with a water reservoir in the microscope chamber and provides direct visual information about the water interaction with the material at a high spatial resolution.

 $\begin{array}{c} MM \ 59.6 \quad Thu \ 17:45 \quad H38 \\ \mbox{Interaction of commensurate charge density waves with an$ $ionic point defects in electron beam irradiated 1T-TaSe2 \\ \mbox{and 1T-TaS2} & - \mbox{$^{-}$Michael Kinyanjui}^{1,2}, \ Pia \ Kynrim^2, \ Tibor \\ Lehnert^2, \ Janis \ Koster^2, \ and \ Ute \ Kaiser^2 & - \ ^1 Helmholtz \ Insitute \\ \end{array}$ Ulm, Helmholtzstraße 11, 89081 Ulm, Germany — ²Central Facility of Electron Microscopy, Ulm University, Albert Einstein Allee 11, 89068 Ulm, Germany

Low dimensional transition metal dichalcogenides (MX2, M = transition metal e.g Nb, Mo, Ti X= chalcogen e.g S, Te, Se) are of great interest due to some of their unique properties including metal-insulator transitions and charge density waves (CDW) observed as a function of temperature, pressure, and doping. Here we report on the interaction of commensurate charge density waves and anionic point defects in 1T-TaSe2 and 1T-TaS2. By using atomic resolved high resolution transmission microscopy (HRTEM) we show the loss of CDW long range order as a result of the interaction with S and Se vacancies generated by the electron beam irradiation. We discuss the loss of long range order as a result of the interaction between the CDW and the Friedel oscillations arising from the point defects.

MM 59.7 Thu 18:00 H38

X-ray Nanodiffraction for in situ Microscopy — •CHRISTINA KRYWKA^{1,2}, STEPHAN V. ROTH³, and MARTIN MÜLLER¹ — ¹Helmholtz-Zentrum Geesthacht, Max-Planck-Straße 1, D-21502 Geesthacht — ²Christian-Albrechts-Universität, Leibnizstraße 19, D-24118 Kiel — ³DESY, Notkestraße 85, D-22607 Hamburg

The origins of the macroscopic behavior of synthetic and natural high-performance materials can often be found on no less than atomistic length scales. Access to these dimensions is barred for light microscopes while electron microscopes suffer from the low penetration depth of electrons. Consequently, high resolution structural data recorded with external stimuli modified *in situ* is rare to find. X-ray nanodiffraction is able to overcome these hurdles. That's because a sub-micrometer sized hard X-ray beam can extract local structural information residing within bulk volumes and from samples inside extended sample environments - given a sufficiently long focal distance. The Nanofocus Endstation of P03 beamline (PETRA III) is a dedi-

cated X-ray nanodiffraction setup. Tensile and indentation stresses, magnetic and electric fields, hydrostatic pressure and fluid shear have all been applied *in situ* in past nanodiffraction experiments at P03, i.e. while high resolution structural information data were recorded. Not only do these results emphasize our focus on materials science but they also demonstrate why X-ray Nanodiffraction is a genuine *in situ* microscopy technique.

MM 59.8 Thu 18:15 H38 $\,$

A setup for AFM-based pick-and-place handling of nanoobjects inside an SEM — •UWE MICK^{1,2}, PETER BANZER^{1,2,3}, SILKE CHRISTIANSEN^{1,4}, and GERD LEUCHS^{1,2,3} — ¹Max Planck Institute for the Science of Light, Erlangen, Germany — ²Institute of Optics, Information and Photonics, University Erlangen-Nuremberg, Erlangen, Germany — ³Max Planck - University of Ottawa Centre for Extreme and Quantum Photonics, Ottawa ON, Canada — ⁴Institute of Nano-Architectures for Energy Conversion, Helmholtz-Zentrum Berlin, Berlin, Germany

Employing AFM technology for nanomanipulation is well-established. However, as a stand-alone technology, the AFM lacks immediate visual feedback on the manipulation in progress. Here we present a Dual-AFM system that is integrated into an SEM and dedicatedly designed to enable pick-and-place handling of nano-objects under the in-situ visual control of the SEM. Several usage scenarios for handling nanowires and nanoparticles down to below 100 nm in diameter are shown; including general schemes for adapting the shape of AFM probe tips by FIB milling to specific manipulation tasks. As the main application, the prototyping of plasmonic particle patterns is presented and specifically the capability to select building blocks from different sources to fabricate heterogeneous nanoparticle patterns. When preparing glass samples for photonic applications in an SEM, additional challenges arise. Therefore, a toolchain of methods and instruments for precharacterizing and selecting suitable nano-building-blocks and mitigating charging effects during sample preparation are outlined.