MM 11: Topical Session: TEM-Symposium - STEM

Time: Monday 15:45–18:15 Location: H4

Topical Talk MM 11.1 Mon 15:45 H4

Scanning transmission electron microscopy at atomic resolution — •Ferdinand Hofer, Gerald Kothleitner, and Werner Grogger — Institut für Elektronenmikroskopie und Feinstrukturforschung, Technische Universität Graz, Steyrergasse 17, A-8010 Graz, Österreich

Advanced electron microscopy is making a vital contribution to the discoveries taking place in many areas of materials science. Especially, the advantages in aberration corrected TEM and STEM instrumentation now provide necessary background for solving materials science problems at the nanometer or even atomic scale. This is especially true of aberration corrected STEM which brings with it analytical techniques such as electron energy-loss spectroscopy (EELS) and the new silicon drift detector systems for x-ray spectrometry. In this paper we present how modern aberration corrected STEM systems can be used to examine the local chemistry and also the physical properties of energy-related materials, e.g. nanocomposite solar cells, solid oxide fuel cell cathodes and the optical properties of nanoparticles.

MM 11.2 Mon 16:15 H4

High Precision STEM Imaging by Non-Rigid Alignment and Averaging of a Series of Short Exposures — •Paul Voyles[1,2,3], Andrew Yankovich[1], Benjamin Berkels[4], Peter Binev[4], and Wolfgang Dahmen[3] — [1]University of Wisconsin, Madison, United States — [2]Forschungszentrum Jülich, Jülich — [3]RWTH Aachen, Aachen — [4]University of South Carolina, Columbia, South Carolina, United States

We have developed a method for non-rigid registration of a series of Z-contrast scanning transmission electron microscopy (STEM) images. The registered series can be averaged to improve signal to noise without loss of resolution. At very high dose to the sample, 10^5 C/cm^2, lattice images with reproducible precision in the atomic column positions better than 1 pm can be obtained. At an order of magnitude lower dose, images with 2-3 pm precision can be obtained. Lower dose images have been used to measure the displacements of atoms at the edges and corners of Pt on γ-Al_2O_3 nanocatalysts. Edge atoms experience displacements of 10-20 pm, consistent with previous reports. Corner atom displacements are 30-50 pm.

MM 11.3 Mon 16:30 H4

Characterisation of ultrathin ferroelectric film using scannning transmission electron microscopy — •Daesung Park[1], Anja Herpers[2], Tobias Menke[2], Regina Dittmann[2], and Joachim Mayer[1] — [1]Central Facility for electron microscopy (GFE), RWTH Aachen, Germany — [2]Institute of Solid State Research and JARA-FIT, Jülich Aachen Research Alliance, Fundamentals of Future Information Technology, Research Center Jülich, Germany

Ferroelectric thin films are attractive candidates for capacitors in random access memory (FeRAM), in which a reversible spontaneous polarisation is utilised to store information. However, below a critical thickness, the ferroelectric property usually disappears. Using smaller inplane lattice parameter of the substrate induces epitaxial strain which tends to enhance ferroelectric distortion. In this study, niobium doped SrTiO3 is used as a substrate due to its 2.2 % smaller lattice parameter in comparison to BaTiO3. To change and balance the possible mixed termination of BaTiO3, 1.5 additional unit cells of BaRuO3 are embedded between a BaTiO3 thin film (7 unit cells) and SrRuO3 top electrode. The critical thickness depends on the termination of the ferroelectric thin film between electrodes. To elucidate the termination at the interface, high angle annular dark filed (HAADF) imaging and StripeSTEM techniques are carried out in STEM mode due to the high sensitivity to the atomic number Z. Fine structure analysis of Ti-L23 edge is performed to account for Crystal field splitting effect which is a result of the distortion of Perovskite structure.

MM 11.4 Mon 16:45 H4

A transmission electron microscopy study on highly strained BiFeO3 thin films — •Young Heon Kim[1,2], Akash Bhatnagar[1], Ji Hye Lee[1], Marin Alexe[1], Eckhard Pippel[1], and Dietrich Hesse[1] — [1]Max Planck Institute of Microstructure Physics, Weinberg 2, D-06120 Halle (Saale), Germany — [2]Korea Research Institute of Standards and Science, Daejeon 305-340, Republic of Korea

BiFeO3 (BFO) has been widely studied for its astounding magnetoelectric properties, such as antiferromagnetism coupled with ferroelectricity. Although BFO is a rhombohedrally distorted multiferroic perovskite (R3c) in the bulk form, its structural stability is unclear in the form of thin films under strain. The misfit strain by lattice mismatch, one of constraints in thin film growth, causes the distortion of the bulk structure and/or the stabilization of novel phases. Several research groups have recently reported the formation of tetragonally distorted BFO phase (P4mm) on highly-lattice-mismatched substrates. In this talk, we present detailed investigation on the atomic structure and the phase behavior of highly strained BFO thin films, based on a transmission electron microscopy study. The transition information of the rhombohedral to tetragonal-like phase is considered to be a critical issue in achieving highly polarized BFO phase. The morphological and microstructural properties of BFO thin films were studied by various transmission and scanning transmission electron microscopy techniques (specially, with a probe Cs-corrector for atomic resolution in the latter case). We will also show geometrical phase analysis results adopted to determine strain distribution.

MM 11.5 Mon 17:00 H4

Microscopic origin of the giant ferroelectric polarization in strained BiFeO3 thin films — •Marta D. Rossell — Electron Microscopy Center, Empa, Swiss Federal Laboratories for Materials Science and Technology, CH-8600 Dübendorf, Switzerland

Because of their astounding electromechanical properties, BiFeO3 (BFO) thin films are promising candidates for the replacement of lead-based ceramics in microelectromechanical system devices. However, a full understanding of the piezoelectric properties reported for these ceramic materials is still missing. In particular, polymorphs of BFO stabilized under epitaxial strain are not yet fully understood. Two distinct structures are known to evolve above and below a 4.5% critical compressive strain. They are pseudotetragonal (T phase) and pseudorhombohedral (R phase), respectively. The T phase shows a unique structure characterized by a strongly elongated unit cell with a c/a axial ratio close to 1.3. The structural information of this metastable polymorph is particularly relevant because it is predicted to have a giant polarization roughly 1.5 times of the bulk material.

We determine the atomic structure of the T phase and the R phase in highly strained BFO thin films by using a combination of atomic-resolution scanning transmission electron microscopy and electron energy-loss spectroscopy (EELS). The coordination of the Fe atoms and their displacement relative to the O and Bi positions are assessed by direct imaging. These observations allow us to interpret the electronic structure data derived from EELS and provide evidence for the giant spontaneous polarization in strained BFO thin films.

MM 11.6 Mon 17:15 H4

STEM HAADF characterization of dilute Bi containing GaAs — •Nikolai Knaub, Andreas Beyer, Peter Ludewig, and Kerstin Volz — Structure and Technology Research Laboratory, Materials Science Center and Faculty of Physics Philipps-Universität Marburg, Germany

Incorporating small amounts of Bi in III/V semiconductors has a huge influence on the energetic position of the valence bands, mainly also of the spin-orbit split-off band. Therefore, dilute bismides such as Ga(AsBi) are promising materials for optical and electronical devices. For a sufficent incorporation of Bi in GaAs, the growth temperature of MOVPE (metal organic vapour phase epitaxy)-grown samples has to be low, typically between 375° C and 450° C. Because of such relatively low growth temperatures point defects, such as Bi or As antisites, can arise and influence the crystal structure. We present the results of STEM (scanning transmission electron microscopy) high angle annular dark field (HAADF) measurements on a MOVPE-grown Ga(AsBi) sample. We used a spherical aberration corrected JEOL JEM 2200 FS with an annular dark field detector for the high resolution. For quantitative comparison with the experimental images, an absorptive potential approximation based simulation software was used for simulations of antisite defects in GaAs and Ga(AsBi). The present contribution will show how to gain information of crystal stochiometry and composition out of experimental as well as simulated HAADF images by using the Voronoi map method. It will be shown that it is possible to describe the influence of point defects on an atomic scale quantitativley.

MM 11.7 Mon 17:30 H4

Analytical transmission electron microscopy in the third dimension — •Bert Freitag, Arda Genc, Jonathan Winterstein, Huikai Cheng, Lee Pullan, and Joerg Jinschek — FEI Company, Eindhoven, The Netherlands

As the feature sizes in material science continue to decrease to nanometer regime, the techniques solely based on 2-dimensional (2D) imaging fail to provide a full characterization of the nanoscale materials. We employed a new tomography technique for STEM XEDS which utilizes the combination of a four silicon drift detector (SDD) system and a high brightness electron gun (XFEG) optimized for high X-ray collection efficiency [1]. Three dimensional tomograms are obtained when the sample is tilted and images and EDS maps are acquired from all angles. The EDS signal can be processed like normal z-contrast images since the EDS signal increases monotonously with the concentration of the element like z-contrast signal increases monotonously with the mass thickness. Examples of 3D chemical mapping using XEDS are given on (InGa)N Nanopyramid LEDs, NiAl3 super alloy material for aircraft turbine blades, high-k dielectric transistor and catalytic particles. In summary, this new technique enables a larger field of view and reduces the acquisition time of a complete XEDS mapping tilt series to hours instead of days, which were impractical before. Even the use of conventionally prepared FIB foils for 3D chemical mapping is possible, which overcomes the difficulties related to background changes with thickness increase known in EELS. [1] P. Schlossmacher et al., Microscopy Today 18(4) (2010) 14-20.

MM 11.8 Mon 17:45 H4

High-resolution HAADF-STEM analysis of hetero-interfaces — •Anna Moros, Harald Rösner, and Gerhard Wilde — WWU Münster

Nanoparticles embedded in an inert immiscible matrix offer ideal conditions to study the impact of hetero-interfaces on reversible phase transitions as for instance melting. Al-Pb composites consisting of nanometer-sized Pb inclusions embedded in a polycrystalline Al matrix serve as model systems for such structural studies. The large lattice constant mismatch of 22.2% between Al and Pb leads to strain at the hetero- matrix-particle interfaces, which is accommodated by misfit dislocations. In order to correlate the thermodynamic properties and the atomic structure of the hetero-interface between the Pb particles and the matrix, an addition of Ga (1 and 3 at. %) selectively into the Al matrix was implemented. Since the lattice constant of the Al(Ga) matrix is extended in comparison with the pure Al matrix, the lattice mismatch between the matrix and the embedded Pb nanoparticles should be reduced. To investigate the interface structure and the particle morphology, aberration-corrected high-resolution HAADF-STEM was performed using the ultra stable stage of the TEAM I microscope. These results will be discussed with the focus on the arrangement of misfit dislocations at the particle-matrix interfaces. The authors acknowledge support of the National Center for Electron Microscopy, Lawrence Berkeley Lab, which is supported by the U.S. Department of Energy under Contract # DE-AC02-05CH11231. Funding by DFG is gratefully acknowledged.

MM 11.9 Mon 18:00 H4

Electrostatic Phase Plates for Transmission Electron Microscopy — •Simon Hettler[1], Manuel Dries[1], Nicole Frindt[2], Rasmus R. Schröder[2], and Dagmar Gerthsen[1] — [1]Laboratorium für Elektronenmikroskopie, KIT, Karlsruhe, Germany — [2]BioQuant CellNetworks, Universität Heidelberg, Heidelberg, Germany

Physical phase plates (PP) for transmission electron microscopy (TEM) enhance phase contrast of (weak-)phase objects by inducing an additional relative phase shift between the scattered and unscattered electrons. The phase shift can be varied by the use of electrostatic PPs, which generate a variable electrostatic field at the zero-order beam position in the back focal plane. An important property of the PP is the cut-on frequency which limits the maximum size of the objects to be imaged with phase contrast. The cut-on frequency is determined by the spatial localization of the electrostatic field which can be optimized by adequate PP design. Moreover, obstruction of scattered electrons in the back focal plane by the PP structure as well as contamination and charging of the device additionally affect PP TEM.

We implemented a PP optimized in size and shape in a Zeiss 912 Omega transmission electron microscope. The obtained images of different samples show contrast enhancement and inversion. Charging and contamination is minimized using an integrated heating device in the vicinity of the PP. The inhomogeneous potential is analyzed and compared to simulations. The effect of inelastic scattering on phase contrast is studied.