MM 22: Topical Session TEM VI

Time: Wednesday 11:00–13:00 Location: IFW B

Topical Talk MM 22.1 Wed 11:00 IFW B
In-situ TEM: Atomistic Insights into Crystallisation — •Christina Scheu — Department of Chemistry & Center for NanoScience (CeNS), Ludwig-Maximilians-University, Munich, Germany

Ultrathin non-conducting nanowires are potential candidates for application in electronic devices and for medical treatments. Recently we studied the self-catalytic vapor-liquid-solid (VLS) growth of alumina nanowires in-situ at 750 degrees Celsius in a high voltage transmission electron microscope [1]. The atomic scale observation revealed that the growth of the nanowire in [0001] direction takes place layer-by-layer involving a two-step mechanism. Oscillatory growth and dissolution reactions alternate at the top rim of the nanowire which is in contact to the liquid Al droplet and the vapor phase leading to periodical changes of the triple junction configuration. The dissolution reaction of the crystalline top rim supplies the oxygen which is required to grow a new (0006) alumina layer [1]. The growth of the (0006) layer is relatively fast since the Al atoms in the liquid adjacent to the crystalline alumina wire possess already similar position as in the solid [2]. This leads to an easy pathway for the interfacial diffusion of oxygen. The rate limiting step for growth of the alumina nanowire is the oxygen transport after completion of a new (0006) alumina layer when the crystalline rim is formed.

[1] Oh, Chisholm, Kauffmann, Kaplan, Luo, Rühle, Scheu, Science 330, 489 (2010). [2] Oh, Kauffmann, Scheu, Kaplan, Rühle, Science 310, 661 (2005).

Topical Talk MM 22.2 Wed 11:30 IFW B
Quantitative Nanoscale Analysis in 3D using Electron Tomography — •Christian Kübel — Karlsruhe Institute of Technology, INT, 76344 Eggenstein-Leopoldshafen, Germany

State-of-the-art electron tomography has been established as a powerful tool to image complex structures with nanometer resolution in 3D. Especially STEM tomography is used extensively in materials science in such diverse areas as catalysis, semiconductor materials, and polymer composites mainly providing qualitative information on morphology, shape and distribution of materials. However, for an increasing number of studies quantitative information, e.g. surface area, fractal dimensions, particle distribution or porosity are needed. A quantitative analysis is typically performed after segmenting the tomographic data, which is one of the main sources of error for the quantification. In addition to noise, systematic errors due to the missing wedge and due to artifacts from the reconstruction algorithm itself are responsible for these segmentation errors and improved algorithms are needed.

This presentation will provide an overview of the possibilities and limitations of quantitative nanoscale analysis by electron tomography. Using catalysts and nano composites as applications examples, intensities and intensity variations observed for the 3D volume reconstructed by WBP and SIRT will be quantitatively compared to alternative reconstruction algorithms; implications for quantification of electron (or x-ray) tomographic data will be discussed and illustrated for quantification of particle size distributions, particle correlations, surface area, and fractal dimensions in 3D.

MM 22.3 Wed 12:00 IFW B
In-situ Transmission Electron Microscopy of Material Transport and Crystallization during the Al induced layer exchange (ALILE) process — •Balaji Birajdar[1], Tobias Antesberger[2], Martin Stutzmann[2], and Erdmann Spiecker[1] — [1]Center for Nanoanalysis and Electron Microscopy (CENEM), Materials Science Department VII, University of Erlangen-Nürnberg, Germany — [2]Walter Schottky Institut and Physics Department, Technische Universität München, Germany

The ALILE process enables fabrication of thin polycrystalline Si films at relatively low temperatures, making it highly promising for applications in thin film photovoltaics. While the driving forces for the metal-induced crystallization are rather well understood, the details of the material transport during the layer exchange are largely unknown. In this work, the microstructure of a stack of a-Si(100nm)/Al(50nm)/quartz, annealed at 450°C, has been investigated at different length scales by combining optical microscopy, SEM, and TEM. The results indicate that the layer exchange and crystallization proceeds by forming 20-50 μm wide dendritic cells with Al deficient centers. Excessive upward transport of Al by epitaxial growth out of the existing Al grains into the a-Si was observed in a rim of about 10 μm width around these cells and, to a smaller extent even beyond. Using in-situ TEM, the lateral and vertical transport of Al at the expanding crystallization front could be directly visualized for the first time and is proposed to be caused by Coble-type diffusion of Al driven by the compressive stress in the Al layer.

MM 22.4 Wed 12:15 IFW B
In situ tensile testing of Au nanowires — •Burkhard Roos[1], Bahne Kapelle[1], Gunther Richter[2], and Cynthia A. Volkert[1] — [1]Institut für Materialphysik, Universität Göttingen — [2]Max-Planck-Institut für Metallforschung, Stuttgart

Increasing strength with decreasing size is a common phenomenon in metals, and is often explained in terms of dislocation pile-ups and interactions. However, for free standing samples with dimensions below 150 nm, dislocation storage is hard to envision and a convincing explanation for the size-dependent strength is still missing. The goal of this study is to directly observe dislocations in small volumes, using in situ TEM during deformation. Single crystal Au nanowires with diameters between 40 and 250 nm have been used for this study. In wires with diameters above 180 nm, full dislocation based deformation and dislocation storage is observed. Wires with diameters below 180 nm show a different deformation behaviour. Stacking faults appear during deformation as a result of the nucleation and motion of partial dislocations. The stacking faults form homogenously along the wire length and appear and disappear in less than 50 ms. The stacking faults do not move as the wire is further deformed, but may eventually thicken into nanotwins through the sequential activation of partial dislocations on neighbouring (111) planes. Post-deformation TEM studies show that fracture often occurs at a nanotwin. A possible explanation for the dependence of the deformation mode on wire diameter and stress will be discussed in terms of the splitting distance of partial dislocations.

MM 22.5 Wed 12:30 IFW B
Optimization of STEM Tomography Acquisition - A Comparison of Convergent Beam and Parallel Beam STEM Tomography — •Johannes Biskupek, Jens Leschner, Paul Walther, and Ute Kaiser — Central Facility of Electron Microscopy, Ulm University, Ulm, Germany

In this work two imaging modes available in state-of-the-art scanning transmission electron microscopes (STEM) are compared: conventional STEM with a convergent beam (nanoprobe) and STEM with a parallel beam (microprobe). The effect and influence of both modes with respect to their depth of field are investigated using standard gold cross grating TEM samples. It will be shown that microprobe posses a large depth of field (up to +/-30 micron), thus, all features are kept in focus even at high tilt without the necessity to apply specimen topology-dependent dynamic focus. A decrease of measured FWHM of gold beads at high tilt by a factor of three shows a gain in resolution by microprobe STEM and the advantage over conventional nanoprobe STEM imaging. Test tomograms are acquired, aligned, reconstructed, and evaluated using both modes. It is shown that STEM using the microprobe mode produces tomograms with fewer distortions and artifacts and allows resolving finer features. Microprobe STEM tomography is advantageous when semi thin TEM samples (ca. 500 nm thick) are imaged at relatively low magnification with a large field of view (more than 3 by 3 micron).

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MM 22.6 Wed 12:45 IFW B
3D electron tomography of biological photonic crystals — •Benjamin Butz[1], Benjamin Winter[1], Benito Vieweg[1], Isabel Knoke[1], Stefanie Spallek[1], Gerd Schröder-Turk[2], Klaus Mecke[2], and Erdmann Spiecker[1] — [1]CENEM, Universität Erlangen-Nürnberg — [2]Theoretische Physik I, Universität Erlangen-Nürnberg

Photonic crystals, i.e. periodical nanostructures of materials with different dielectric constants, are highly interesting for applications in optics, optoelectronics, and sensing. By tailoring the geometrical parameters radically different and improved optical properties (e.g., optical band-gap structure, extreme refractive indices, or high anisotropy) can be achieved. Naturally occurring photonic crystals, like butterfly scales, exoskeletons of insects (chitin), or seashells (nacre), can serve as model systems for understanding the relationship between structure and optical properties. Butterfly scales are studied by TEM using a FEI Titan^3 80-300 instrument. An optimized FIB technique or ultramicrotome sectioning were used to prepare the sensitive specimens with desired thickness. Since the periodical structures have dimensions on the sub-μm scale, HAADF-STEM tomography was employed for obtaining extended tilt series under conditions of atomic-number sensitive imaging. Since the solid crystal consists of chemically homogeneous chitin while the pores are unfilled, the distinct contrast in the images can easily be interpreted in terms of the local projected mass density allowing to reconstruct the chitin distribution within the optical unit cell of the scales with high 3D resolution.