MI 3: TEM- and SEM-based Material Analysis

Time: Monday 14:30–16:45 Location: BEY 81

Invited Talk MI 3.1 Mon 14:30 BEY 81

Transmission electron microscopy of interface and defect phenomena of functional materials — •Wolfgang Jäger — Mikrostrukturanalytik, Christian-Albrechts-Universität zu Kiel

Advanced high-resolution imaging and spectroscopic techniques of electron microscopy play a crucial role in characterizing the microstructure and the structure-property relationships of inorganic materials and interfaces. The presentation will describe applications of quantitative transmission electron microscopy methods to investigations of surface and interface phenomena of nanostructured functional materials and technologically relevant layer systems. Examples are aberration-corrected high-resolution TEM of incommensurate interfaces in misfit layer compounds1, metal intercalation and formation of surface nanostructures on chalcogenide layered crystals2, electron tomography3 and interfaces4 of diffusion-induced nanoinclusions in III-V semiconductors, nanostructured oxide semiconductors5, defect engineering for high-efficiency semiconductor solar cells6, and multilayer systems for x-ray optics7.

1. M. Garbrecht, E. Spiecker et al., Ultramicroscopy (2011). 2. E. Spiecker, A. Schmid, A. Minor et al., Phys. Rev. Lett. 96, 086401 (2006). 3. Ch. Kübel, Ch. Dieker et al., Proc. 17th Int. Microscopy Congress, Rio de Janeiro, I7.30 (2010). 4. Ch. Jäger, E. Spiecker et al., Ultramicroscopy 92, 273 (2002). 5. Y. Ortega Villafuerte, Ch. Dieker et al., Nanotechnology 21, 225604 (2010). 6. J. Schöne, E. Spiecker et al., Appl. Phys. Lett. 92, 081905 (2008). 7. D. Häussler, Ch. Morawe, U. Roß et al., Surface & Coatings Technology 204, 1929 (2010).

Invited Talk MI 3.2 Mon 15:15 BEY 81

The contrast mechanisms of LL-BSE electrons in FE-SEM - Characterization of polymer, single proteins, and oxidization states of elements — •Heiner Jaksch — Carl Zeiss NTS GmbH, 73447 Oberkochen, Germany

Below landing energies of 4 kV, the backscatter coefficient becomes non linear and drops with increasing atomic number stronger than that from elements with low atomic numbers. At a certain landing energy we see equilibrium of backscatter yield and no contrast. Chromium is at 1 kV landing energy brighter than Gold. Carbon will be brighter than Gold around 400 eV landing energy! Due to this fact and the problem, that the mean free path length of BSE electrons from low-density materials, such as proteins or polymers, becomes extremely small, we have introduced new technologies to visualize these low intensity signals coming from electrons with very small energy loss. The low loss BSE electrons are now introduced in SEM. To understand the new contrast mechanisms experiments with hybrids, polymers and all kinds of different oxidization states of elements were made and will be shown. Essential for the contrast at low landing energies is not any more the atomic number or density as contrast mechanism, but only the bonding structure of outer shell electrons or plasmon losses. To get the information from the electrons a double stage filtering is necessary. In the examples these results are explained with the hybridization of carbon as sp^2 and sp^3 hybrids and shown with imaging examples. These hybrids are responsible for the contrast in all polymer and protein. These hybrids are responsible for the contrast in all polymer and protein. In general we have to consider the bonding-/ ionization energy or plasmon losses and not the nucleus charge as source of the contrast. In the shown examples it will be proven that density rules or z-number contrast fail in explaining the observed contrast. Monte Carlo simulations also are unable to model the fine contrast mechanisms. The sensitivity of the technology is explained with the detection of a single protein (8nm) in virus marked with GFP. The detection concept is verified with quantum dots (GaAlAs) of known band-gap with 2.5eV and 4.8eV. What we see there is the resonance of a more or less free electron, replaced by the primary electrons. Such "free" electrons typically show extreme high contrast due to the very small energy loss when replaced by primary electrons. One can use this contrast mechanism to detect functional groups in polymers or as described above, in fluorophors in live science. As an outlook the technology will be a big step forward for the characterization of anything in live science and material science.

1) Reimer L., Scanning electron microscopy 2nd Edition, Springer-Verlag Berlin Heidelberg New York 1998. 2) Jaksch H., Low Loss BSE imaging in a FE-SEM, Proceedings IMC 2010, Rio de Janeiro.

MI 3.3 Mon 16:00 BEY 81

TEM study on light induced crystallization of amorphous silicon — •Martin Schade[1], Teimuraz Mchedlidze[2], Martin Kittler[2], and Hartmut S. Leipner[1] — [1]Interdisziplinäres Zentrum für Materialwissenschaften, Martin-Luther-Universität Halle-Wittenberg, D-06099 Halle — [2]IHP/BTU Joint Lab, Konrad-Wachsmann-Allee 1, D-03046 Cottbus und IHP Microelectronics, Im Technologiepark 25, D-15236 Frankfurt (Oder)

HR-TEM and STEM-analytics have been carried out in order to characterize the influence of laser power during light induced crystallization (LIC) of amorphous silicon. Therefore, a 60 nm thick amorphous silicon layer and a 120 nm thick silicon dioxide cover layer have been deposited on quartz substrate by means of RPECVD. LIC was performed by treating the samples with a laser operating at a wavelength of 532 nm with a spot size of around 1 μm^2. The laser power was varied in order to achieve different crystalline fractions which could be controlled by Raman-microscopy.

TEM cross-sections exhibit that at an optimal laser power large undulating, mono-crystalline grains are formed. Lower laser powers lead to mixtures of defect rich crystalline grains and nano-crystallites. A higher laser power results in an ablation process with a massive formation of silicon dioxide.

MI 3.4 Mon 16:15 BEY 81

Enhancing electron diffraction through precession — •Giuseppe Pavia[1], Loic Patout[2], Gerd Benner[1], and Harald Niebel[1] — [1]Carl Zeiss NTS, Oberkochen, Germany — [2]ONERA, Paris, France

Nanostructures are often investigated in Transmission Electron Microscopy (TEM), and electron diffraction (ED) can be used to solve nanocrystals. Electrons interact very strongly with matter, and the diffracted intensities are highly dynamical. Precession Electron Diffraction (PED) is a recent technique delivering more kinematical diffraction patterns.

We have used an in column energy filtered TEM equipped with precession electron diffraction hardware, which allows working up to 3 deg precession angle, and energy filtering of the precession patterns. High Order Laue Zones, useful for space group symmetry determination and to enhance fine structure details, appear more clearly.

We have compared a microdiffraction pattern and a precession microdiffraction pattern performed along the orientation [010] of a sample TiSi2 with a space group Fddd. For cubic systems, this orientation allows to distinguish the Bravais lattice and the presence of glide mirrors. We show that with precession, we conserve the distinction of the gap and the difference of periodicity between the ZOLZ and the FOLZ is improved.

References

1. Vincent R. and Midgley P., (1994) Ultramicroscopy 53 271

MI 3.5 Mon 16:30 BEY 81

Indentation-induced dislocations and cracks in GaN bulk crystals — •Ingmar Ratschinski[1], Hartmut S. Leipner[1], Frank Heyroth[1], Wolfgang Fränzel[2], and Frank Habel[3] — [1]Interdisziplinäres Zentrum für Materialwissenschaften, Martin-Luther-Universität Halle-Wittenberg, Heinrich-Damerow-Straße 4, D-06120 Halle, Germany — [2]Institut für Physik, Martin-Luther-Universität Halle-Wittenberg, Von-Danckelmann-Platz 3, D-06120 Halle, Germany — [3]Freiberger Compound Materials GmbH, Am Junger-Löwe-Schacht 5, D-09599 Freiberg, Germany

GaN bulk crystals with a density of in-grown dislocations in the magnitude of 10^6 cm^{-2} have been deformed at room temperature using a Vickers indenter. The (0001) surface has been indented with loads in the range from 0.02 N to 4.90 N with two different orientations of the indenter. Dislocations and cracks at the indentations were observed by means of optical microscopy, scanning electron microscopy and cathodoluminescence. Dislocations occur at all indentations for the loads used in the investigations. The dislocation arrangement corresponds to the symmetry of the indented surface. Higher loads lead to radial cracks at the corners of the indentations as well as lateral cracks beneath the surface. The crack system is determined predominantly by the symmetry and orientation of the indenter. Geometrical relations have been found between dislocations and cracks.