MA 7: Analytical Electron Microscopy: SEM and TEM-based Material Analysis

Time: Monday 10:00–13:00 Location: MER 02

Invited Talk MA 7.1 Mon 10:00 MER 02

Point-group sensitive interpretation of EBSD patterns, and the impact of channeling-in and channeling-out of electrons — •Gert Nolze[1] and Aimo Winkelmann[2] — [1]Bundesanstalt für Materialforschung und -prüfung (BAM), Unter den Eichen 87, 12205 Berlin, Germany — [2]Bruker Nano GmbH, Am Studio 2D, 12489 Berlin, Germany

Recent studies contradict the common belief that electron backscatter diffraction follows Friedel's rule. The presentation will demonstrate that entire orientation maps collected under standard acquisition conditions can be processed by pattern matching of experimental with simulated patterns which enables to distinguish between (hkl) and $(\bar{h}\bar{k}\bar{l})$. However, the polarity determination for phases such as GaAs is very difficult since Ga an As have a similar contribution to the backscattered intensity of hkl and $\bar{h}\bar{k}\bar{l}$. We will show that in such case the energy-dispersive X-ray signal can be used, but presently for single orientations only.

It is also frequent practice that an EBSD pattern is mainly reduced to its backscattered diffraction part. This also called *channeling-out* signal is used for the orientation interpretation, phase interpretation etc. The presentation will prove that the *channeling-in* of the electron beam reacts clearly more sensitive regarding orientation variations and is responsible for the orientation contrast in images e.g. collected by a backscattered electron detector. Despite the fascinating misorientation sensitivity a quantitative evaluation seems to be very unlikely.

MA 7.2 Mon 10:30 MER 02

The Complicated Information Depth of EBSD — •Wolfgang Wisniewski — Otto-Schott-Institut, Fraunhoferstr. 6, 07743 Jena, Germany

The information volume of a method enables to estimate which part of a sample actually contributes to the given measurement and establishes boundaries concerning possible measurements. In the case of EBSD, the widespread opinion is that the information depth is limited to 10-40 nm or less. However, recent results show that this information depth depends not only on the material and the available technology, but also on the quality of the pattern being analyzed. In high quality patterns, the evaluated information indeed originates from a very thin layer of material, but the information depth may increase significantly for low quality EBSD-patterns. This aspect e.g. expands the possibilities of EBSD-measurements to materials covered by passivation layers.

MA 7.3 Mon 10:45 MER 02

Quantitative materials characterization at the nanoscale with TKD in SEM — •Laurie Palasse and Daniel Goran — Bruker Nano GmbH, Am Studio 2D, 12489 Berlin, Germany

Characterization of nanostructured materials requires high spatial resolution orientation mapping at large-scale for quantitative results. Because EBSD does not achieve such resolution on bulk samples, these kind of studies are often done using a TEM. However, TEM-based orientation mapping techniques suffer from small field of view. As a result, Transmission Kikuchi Diffraction (TKD) in SEM was developed as a technique capable of delivering the same type of results as EBSD but with a spatial resolution improved by up to one order of magnitude. TKD analysis is conducted on an electron transparent sample using the same hardware and software as for EBSD system. But when using conventional EBSD geometry, the transmitted patterns (TKP) are captured by a vertical phosphor screen with a considerable loss of signal and strong distortions induced by gnomonic projection. The limitations of such sample-detector geometry are overcome by an on-axis detection system. With a horizontal phosphor screen placed underneath the sample, the transmitted signal is captured where it is the strongest and TKPs will have minimal distortions. Using low probe currents, the spatial resolution is increased and the beam-induced specimen drift reduced. The improved stability and high spatial resolution allow the user to conduct large-area TKD orientation mapping, especially when combined with a fast and sensitive EBSD detector.

MA 7.4 Mon 11:00 MER 02

Cryo-EBSD on $BaFe_2As_2$ single crystals — •Aurimas Pukenas[1], Paul Chekhonin[1], Ellen Hieckmann[2], Martin Meissner[2], Saicharan Aswartham[3], Jan Engelmann[3], Bernhard Holzapfel[4], Sabine Wurmehl[3], Bernd Büchner[3], and Werner Skrotzki[1] — [1]Institut für Strukturphysik, Technische Universität Dresden, 01069 Dresden, Germany — [2]Institut für Angewandte Physik, Technische Universität Dresden, 01069 Dresden, Germany — [3]Leibniz-Institut für Festkörper- und Werkstoffforschung (IFW) Dresden, 01069 Dresden, Germany — [4]Institut für Technische Physik, Karlsruher Institut für Technologie, 76344 Eggenstein-Leopoldshafen, Germany

$BaFe_2As_2$ belongs to the family of iron-based high temperature superconductors. In previous publications it was reported that superconductivity occurs under certain conditions, e.g. by chemical doping, pressure or epitaxial strain. Undoped $BaFe_2As_2$ orders antiferromagnetically at $T_C \approx 140$ K and simultaneously undergoes a tetragonal $I4/mmm$ to orthorhombic Fmmm structural phase transition. The orthorhombic structure leads to the formation of twin lamellae. Investigations reported so far using transmission electron microscopy and polarized light microscopy show inconclusive results with respect to the domain size. To achieve high spatial resolution ($\leq$ 100 nm) an experimental setup was used consisting of a scanning electron microscope with sample holder on a helium cryostat and electron backscatter diffraction (EBSD) technique. EBSD mappings of domains below T_C and after a cooling-warming cycle will be presented and discussed.

15 min. break.

MA 7.5 Mon 11:30 MER 02

Angle-resolved X-ray fluorescence spectroscopy for elemental depth profiling with nanometer resolution — •Ioanna Mnatouvalou, Jonas Baumann, Veronika Swedowski, Malte Spanier, Steffen Staeck, Daniel Grötzsch, Wolfgang Malzer, and Birgit Kanngiesser — Institut für Optik und atomare Physik, Technische Universität Berlin, Deutschland

X-ray fluorescence (XRF) spectroscopy is a well-established analytical tool for the non-destructive investigation of elemental distributions. Typically, the measured fluorescence intensities are converted to elemental concentrations using tabulated atomic cross sections, thus rendering reference-free quantification feasible. With adapted X-ray lenses lateral micro- or even nano-analysis is possible. Depth information, though, is not readily available. Recent development is directed toward angle-resolved (AR) XRF for the derivation of elemental depth profiles with nm resolution. Here, the angle of incidence or emission is varied, thereby changing the fluorescence information depth. The comparison of measured and simulated angular profiles yields information about the stratigraphy of technologically relevant specimen such as multilayer structures, solar cells or transistor gate stacks. We present our lab-based instrumentation in the soft and hard X-ray range. With a flexible spectroscopy chamber and various sources (X-ray tubes, laser-produced plasma source) and detectors (SDD, CCD) AR and especially grazing emission measurements show the feasibility of the analysis independent on large scale facilities such as synchrotron radiation sources.

MA 7.6 Mon 11:45 MER 02

Analysis on nanostructures and samples with high topography using low acceleration voltages — •Max Patzschke — Bruker Nano, Berlin, Germany

Continuing technological advances require the elemental analysis of increasingly smaller structures in many industrial fields, including biological applications, semiconductors, and nanotechnology in general. This confronts the otherwise well proven electron microscope based energy dispersive spectroscopy (EDS) with new challenges. Most of these challenges are due to physical conditions, such as limited resolution and radiation yield in the low energy range requiring the analysis on bulk samples with low accelerating voltages. The necessary of low probe current would give low X-ray count rates with traditional EDX detectors, and only low to intermediate energy X-ray lines with many peak overlaps can be evaluated.

The XFlash FlatQUAD Silicon Drift Detector (SDD) allowing us to overcome these limitations, and offering additional benefits. Using the FlatQuad detector with low accelerating voltages, the element distribution of nanometer-sized structures can be displayed in a short time.

Peaks with several overlapping elements (e.g. Co-L, Ni-L, Fe-L) can be deconvolved using the improved atomic database with 250 additional L,M and N lines below 4 keV.

Examples for nanotechnological applications will be presented: mapping of nanoparticles down to 4nm, biological application, samples with high topography and specimen where sample preparation like coating is excluded.

MA 7.7 Mon 12:00 MER 02

Identification of laminates in 10 M martensite Ni-Mn-Ga magnetic shape memory single crystals — •Jaromír Kopeček[1], Ladislav Klimša[1], Ladislav Straka[1], Jan Drahokoupil[1], Petr Veřtát[1], Vít Kopecký[1], Hanuš Seiner[2], Martin Zelený[1], and Oleg Heczko[1] — [1]Institute of Physics of the AS CR, Na Slovance 2, Prague, 182 00, Czech Republic — [2]Institute of Thermomechanics ASCR, Dolejškova 5, 182 00 Prague, Czech Republic

Ni-Mn-Ga is the most studied magnetic shape memory alloy and the most effective example of magnetic shape memory effects. Used single crystals with composition Ni50Mn28Ga22 have monoclinic structure at room temperature, nevertheless the monoclinicity is very weak with respect to both monoclinic angle and lattice parameters a and b. Such structure allows two types of mobile twinning boundaries in magnetic field. The Type I with mirror plane symmetry and more complicated, extremely mobile Type II twinning boundary. Generally, thanks to monoclinicity there exists a hierarchy of twinning on different scales: a-c laminate; monoclinic lamellae - compound twinning; a-b laminate. The complex structure of macrotwinning lamellae and monoclinic twinning was observed many times. However, the third level of lamination, i.e. a-b laminate is hard to observe and describe correctly. We observed whole hierarchy of all three types of laminates by particular SEM observation and manage to identify them using our previous knowledge from optical microscopy, X-ray diffraction and theory of martensite. The identification includes the branching of lamellae on twinning boundaries.

Invited Talk MA 7.8 Mon 12:15 MER 02

Microstructural characterization of non-metallic precipitates in silicon crystallization processes for photovoltaic applications — •Susanne Richter, Martina Werner, Sina Swatek, and Christian Hagendorf — Fraunhofer Center for Silicon Photovoltaics CSP, Otto-Eißfeldt-Str. 12, D-06120 Halle (Saale)

During the directional crystallization of silicon the formation of non-metallic precipitates may occur due to enrichment and segregation of carbon and nitrogen. Different types of precipitates lead to different defects in the later processed solar cells. Extensive material analyses were performed to obtain micro structural, chemical and electrical properties of all occurring precipitate types including analyses via IR microscopy, ToF-SIMS, FIB target preparation for TEM combined with nanospot-EDS and SAED. As a result in addition to the previous state of knowledge a precipitate classification is deduced. Selective material properties are correlated to individual precipitate types such as morphology, crystal structure (and polytype) or the presence of certain impurities. These properties can be used for precipitate identification and prediction of expected defect behavior. Especially the correlation between the found impurities, its concentrations within the precipitates analyzed by ToF-SIMS, EDS and ICP-MS, and the resulting crystallographic microstructure investigated by TEM and SAED are presented in detail.

MA 7.9 Mon 12:45 MER 02

Quantitative high-resolution off-axis electron holography of 2D materials — •Florian Winkler[1,2], Juri Barthel[1,3], Sven Borghardt[4], Amir H. Tavabi[1,2], Emrah Yucelen[5], Beata E. Kardynal[4], and Rafal E. Dunin-Borkowski[1,2] — [1]Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons (ER-C), Forschungszentrum Jülich, D-52425 Jülich, Germany — [2]Peter Grünberg Institute 5 (PGI-5), Forschungszentrum Jülich, D-52425 Jülich, Germany — [3]Gemeinschaftslabor für Elektronenmikroskopie (GFE), RWTH Aachen University, D-52074 Aachen, Germany — [4]Peter Grünberg Institute 9 (PGI-9), Forschungszentrum Jülich, D-52425 Jülich, Germany — [5]FEI Company, Achtseweg Noord 5, Eindhoven 5600 KA, The Netherlands

Usually, phase information in conventional transmission electron microscopy (TEM) is lost. A fully recorded electron wave function with its amplitude and phase would allow for post-acquisition removal of residual aberrations and thus an accurate quantitative description of a material's atomic structure.

Here, we present electron wave functions reconstructed from high-resolution electron holograms of two-dimensional WSe_2. We show that a very precise knowledge of microscope and sample-related parameters, such as image spread, Debye-Waller factor and specimen tilt, can be obtained by comparing experimental wave functions with simulations. Furthermore, we are able to remove residual aberrations from the experimental data, which enables a quantitative description of the atomic structure, including the detection of structural defects.