KR 8: Poster Crystallography

Time: Wednesday 17:00–19:30 Location: P4

KR 8.1 Wed 17:00 P4

Analyzing high pressure diffraction data of perovskites with parametric Rietveld refinement and rotational symmetry modes of a rigid body — •Martin Etter[1], Melanie Müller[1], Michael Hanfland[2], and Robert E. Dinnebier[1] — [1]Max Planck Institut für Festkörperforschung, Stuttgart, Germany — [2]European Synchrotron Radiation Facility (ESRF), Grenoble, France

The high pressure behavior of crystals can best be observed with X-ray or neutron diffraction methods, as these methods allow the application of least square iteration processes (e.g. Rietveld method) to refine parameters, which are directly connected with structural and/or magnetic changes within the crystal. A challenge of the investigation of diffraction patterns under high pressure is that the data quality often decreases after only a few GPa, which makes it difficult for a least square iteration process to find the correct minimum. For this reason, adequate structural models are needed in order to stabilize the refinement and to decrease the number of free parameters. Such models can be provided by the application of rigid bodies, symmetry modes or the recently developed method of rotational symmetry modes of a rigid body which combines the advantages of both. Additionally, these models can be parameterized, treating different data sets simultaneously with the application of physical equations as constraints, which leads to a further reduction of refined parameters. In order to illustrate the power of this new approach, sequential and parametric Rietveld refinements of a LaFeO3 perovskite investigated with synchrotron powder X-ray diffraction under high pressure were carried out.

KR 8.2 Wed 17:00 P4

Using an innovative Vinet EoS approximation for parametric refinement of high pressure powder diffraction data — •Martin Etter and Robert E. Dinnebier — Max-Planck-Institut für Festkörperforschung, Stuttgart, Germany

The relationship between the thermodynamic state variables pressure, volume and temperature can normally be described with so-called equation of states (EoS), which are semi-empirical determined and which are often used with an isothermal description. Among this semi-empirical EoS, the Vinet EoS is believed to be the most accurate, allowing to describe the behavior of most material systems under high pressure up to a compression of 40%. Similar to other EoS like the Birch-Murnaghan EoS or the natural strain EoS, the Vinet EoS has an pressure on volume dependency, which makes it unsuitable for parametric refinement of high pressure powder diffraction data, where the pressure is the independent variable and the volume is the dependent which is a refineable quantity. Using an innovative Taylor series expansion of the Vinet EoS, it is possible to obtain an equation which is analytical invertible and therefore useful for a direct parametric refinement in analysis programs like TOPAS. By means of high pressure powder diffraction data of different material systems the validity of the newly developed EoS is evaluated and demonstrated.

KR 8.3 Wed 17:00 P4

Crystallografic orientation of hydrogen defects in lithium niobate and lithium tantalate — •Thomas Köhler, Erik Mehner, Juliane Hanzig, Günter Gärtner, Hartmut Stöcker, and Dirk C. Meyer — Institut für Experimentelle Physik, Technische Universität Bergakademie Freiberg, D-09596 Freiberg, Germany

Pyroelectric crystals are used in many optical devices, therefore, understanding the structural defects is helpful to control optical and electrical properties. It is easy to incorporate hydrogen in air-grown $LiNbO_3$ and $LiTaO_3$ crystals, however, the exact processes are only partially understood. The incorporation of hydrogen in the two materials is investigated crystal axis resolved and polarization-dependent by FT-IR spectroscopy. In the congruent material systems the hydrogen defect causes an OH band with several sub-bands, at different spectral positions for both materials.

The examined congruent crystals are cube shaped, cut and polished along the [001], [100] and [110] directions. The OH band show a significantly weaker signal for the [100] and [110] directions than for the [001] direction of the crystal cubes. Further measurements were made in the far infrared range. The aim of the crystal axis resolved and polarization-dependent study is the development of a structural model for the orientation of the OH defects of the two pyroelectric materials.

KR 8.4 Wed 17:00 P4

Transmission Kikuchi Diffraction and Selected Area Electron Diffraction studies of WS_2 nanotubes — Andrey Chkanov[1], Azat Khadiev[1], •Nathanael Jöhrmann[2], Dmitry Pashin[1], and Michael Hietschold[2] — [1]Kazan National Research Technical University named after A.N. Tupolev, Nanotechnology in Electronics Department, Kazan, Russia — [2]Institut für Physik, Technische Universität Chemnitz, D-09107 Chemnitz, Germany

Investigations of nanotubes have shown that tubes may differ not only in chemical composition but also in structure. Selected area electron diffraction (SAED) in TEM is a well-established method that allows distinguishing structural parameters of single nanotubes. However in the case of single tubes deposited on a bulk substrate TEM methods do not work. An alternative might be electron backscatter diffraction (EBSD). Clear understanding of peculiarities of Kikuchi diffraction found on nanotubes requires comparative studies. Using SAED and transmission Kikuchi diffraction (TKD) allows a comparison between a SAED pattern and an EBSD like Kikuchi diffraction pattern obtained from one and the same tube lying on a TEM grid.

We combine SAED and TKD measurements with pattern simulations for structural analysis of WS_2 nanotubes. SAED studies allowed determining parameters of the unit cell and chiral angle of the tube. In the TKD studies a peculiar intense band was observed for most nanotubes, which might also contain information about the chiral angle. In some cases two TKD patterns seemed to overlap. Structure models were proposed to explain this effect.

KR 8.5 Wed 17:00 P4

Combining SAXS and TEM to reveal the mechanism of nucleation and growth of anisotropic noble metal nanoparticles — •Tilo Schmutzler, Torben Schindler, Martin Schmiele, Thaer Kassar, Christian Bär, and Tobias Unruh — Chair for Chrystallography and Structural Physics, Friedrich-Alexander University Erlangen-Nürnberg, Staudtstr. 3, 91058 Erlangen, Germany

Anisotropic noble metal nanoparticles like nanorods or nanowires of gold and silver have been the subject of widespread research in the last two decades because of their possible applications i.e. in biological imaging and drug delivery. One common synthesis is the seed-mediated growth method, where a noble metal precursor is reduced in aqueous solution to form isomorphic seed particles that lead to anisotropic nanorods by further reduction of the precursor in the presence of cetyltrimethylammonium bromide (CTAB). Neither the exact formation mechanism of the seed particles nor that of the anisotropic growth in the presence of CTAB is completely understood up to now.

In situ time-resolved small angle x-ray scattering (SAXS) is a powerful tool to study the size and shape of nanoparticles in dependence of temperature and time during their formation and growth. In addition, transmission electron microscopy (TEM) reveals the current shape, polydispersity and size distribution of the particles. In the poster we will present studies to reveal the mechanism of nucleation and growth of anisotropic noble metal nanoparticles under the load of CTAB combining the results of the complimentary techniques of in situ SAXS and ex situ TEM.