it is possible to sample and reconstruct the 3D diffraction space, to automatically determinate the unit cell parameters and to integrate electron diffraction intensity data sets able to deliver the crystal structure by direct methods. [1], [2]. 3D data are acquired through a sequential tilt of the selected nano-crystal around an arbitrary axis. The tilt step is usually 1° and tilt range of ± 60° can be reached. An exampled of 3D reconstructed diffraction space is shown in Figure 1. Such a data set contains nearly all the reflections present in the covered wedge of the reciprocal space. Combining electron beam precession (NanoMEGAS) with tomographic diffraction data acquisition principle allows proper integration of reflection intensities and drastically reduces dynamical effects. In the last three years more than 25 crystal structures (organic and inorganic) have been solved ab-initio with ADT. ADT is especially effective for data collection from beam sensitive materials because it uses low illumination conditions in STEM mode and includes devoted routines for electron dose distribution. Here we report for the first time ADT study on reference 6H-SiC semiconductor and NiTe binary compound samples. Those materials have physical properties which are suitable for applications into electronic systems as the wide energy gap of the 6H-SiC is suitable for UV-detectors and blue light lasers. Using ADT/PED we were able to reconstruct accurately their diffraction space, find their cell parameters and solve ab-initio their structure with a kinematical approximation (I proportional to F_{hkl}^2). All the atoms (C, Si and Ni, Te) were localized and the solution show a residual R of 13%, remarkably low for electron diffraction data.

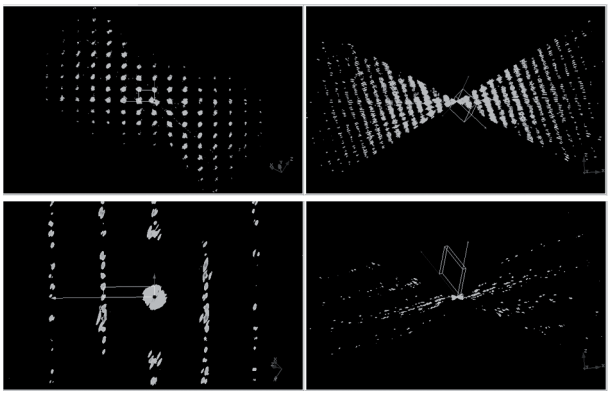


Figure 1: ADT reconstructed 3D diffraction space and unit cell for NiTe and SiC. Upper: NiTe along (001) (left) and tilt axis (right). Bottom: SiC along (001) (left) and tilt axis (right).

[1] U. Kolb, T. Gorelik, M. T. Otten, *Ultramicroscopy* **2008**, 108, 763-772. [2] E. Mugnaioli, T. Gorelik, U. Kolb, *Ultramicroscopy* **2009**, 109, 758-765

Keywords: electron diffraction, precession, structure solution

MS79.P05

Acta Cryst. (2011) A**67,** C694

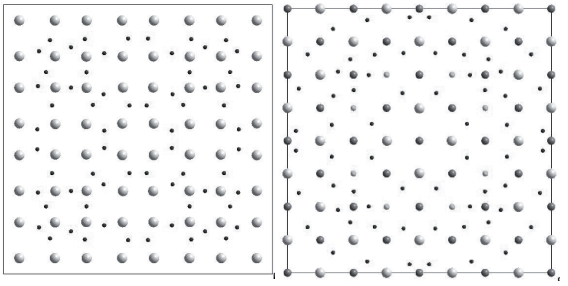
Huge unit cell $Sr_{64.1}Bi_{27.7}Ni_{8.2}O_x$ solved by precession electron diffraction

Holger Klein,[a] M. Bacia,[a] A. Rageau,[a] P. Strobel,[a] M. Gemmi,[b] [a]*Institut Néel, Grenoble, France* [b]*Dipartimento di Scienze della Terra 'Ardito Desio', Sezione di Mineralogia, Milano, (Italy).* E-mail: holger.klein@grenoble.cnrs.fr

Perovskite-like oxides of Sr–Bi–TM–O systems (with TM = transition metal) have been extensively studied but surprisingly no structures have been published in the Sr–Bi–Ni–O system. Samples with nominal composition $Sr_3Bi_{2-x}Ni_xO_{6-\delta}$ were prepared from nitride precursors and calcined in air or oxygen current at 900 °C during 10 – 15 hours, followed by 20–30 hours at 1000 – 1200 °C. In the present transmission electron microscopy (TEM) study precession electron diffraction was carried out on a Philips CM300ST equipped with the spinning star precession unit. Chemical analysis was achieved by EDS in a Tecnai F20ST. TEM showed the existence of at least 3 different phases: a tetragonal phase (a = 5.36 Å, c = 17.5 Å), a closely related orthorhombic phase ($a_o \approx a_t / \sqrt{2}$, $b_o \approx a_t * \sqrt{2}$, $c_o \approx c_t$) and a minority cubic phase (a = 33.7 Å, 8.2% Ni, 64.1% Sr, 27.7% Bi) which represents not more than a few percent of the sample. In the case of a minority phase with a large unit cell X-ray powder diffraction is useless for structure determination. We therefore conducted an electron crystallography study on the cubic phase.

Due to the very large unit cell of the cubic phase the precession angle is limited by Laue zone overlap and was chosen as 0.8° or 1.3° depending on the zone axis. Systematic extinction and the symmetry of the higher order Laue zones indicated *Im3m* as the most probable space group. 6 zone axes yielded 1692 independent reflections. Using different input parameters for SIR2008 (starting composition, maximal resolution, applying or not a Lorentz correction) yielded the same cation positions with slightly different chemical order. The cations are ordered in layers alternating pure Sr layers and layers formed by Bi, Sr and Ni ions. In the mixed layers the cations are equally spaced forming a square lattice. In the Sr layers the distances between Sr rows alternate between 3.95 Å and 4.5 Å creating rectangles, small and large squares. The figure shows a Sr and a mixed layer.

Not all of the oxygen atoms were directly found but the ones present in the structure solutions clearly showed that the oxygen are in the center of the cation squares except for the 'large' squares in the Sr layers where the oxygen are on the square edges. The resulting composition is $Sr_{672}Bi_{272}Ni_{80}O_{1200}$ in agreement with the EDS measurements. To the best of our knowledge, this is the largest structure ever solved by precession electron diffraction.



Keywords: precession electron diffraction, structure solution, electron crystallography

MS79.P06

Acta Cryst. (2011) A**67,** C694-C695

Fourier images in coherent convergent beam electron diffraction and atomic resolution scanning transmission electron microscopy

Changlin Zheng,[a] Joanne Etheridge,[a,b] [a]*Monash Centre for Electron Microscopy, Monash University, Victoria 3800 (Australia).* [b]*Department of Materials Engineering, Monash University, Victoria 3800 (Australia).* E-mail: changlin.zheng@monash.edu

Fourier images (the Talbot effect) are a special self-imaging phenomenon arising from the coherent illumination of a periodic object. The formation mechanism has been carefully analyzed by J. M. Cowley and A. F. Moodie with diffraction theory in the 1950s [1].

Here we report the experimental observation of Fourier images in coherent convergent beam electron diffraction (CBED) patterns taken using high energy incident electrons. We use a transmission electron

microscope (TEM) fitted with a spherical aberration corrector on the probe forming lens to minimize the effect of lens aberrations on the Fourier image. As expected, these images bring different spatial frequencies into focus for different probe cross-over positions above the specimen. Under certain conditions, this enables specific families of atomic planes to be imaged individually. We discuss how this phenomenon can be used to image crystal defects in coherent CBED patterns. Furthermore, we show how, by using an appropriately configured detector in scanning transmission electron microscopy, this approach can be used to generate atomic resolution images with preferential contrast from selected atomic planes and defects. The applications of this scanning image mode will be further discussed at this congress.

[1] J.M. Cowley, A.F. Moodie, *Proceedings of the Physical Society of London Section B*, **1957**, *70*, 486-496, 497-504, 505-513, **1960**, *76*, 378-384.

Keywords: coherent CBED, fourier images, STEM

MS79.P07

Acta Cryst. (2011) A**67,** C695

"Transrotation" revealed by electron diffraction: perfect crystal in curved space

V. Yu. Kolosov, *Physics Dept., Ural State University, Ekaterinburg (Russia).* Vladimir.Kolosov@usu.ru

Earlier the bend-contour technique for direct lattice orientation analysis [1] by means of diffraction transmission electron microscopy had been developed. Using it we had discovered unusual micro crystals growing in thin (20-100 nm) amorphous films with strong (dislocation independent) internal bending of the crystal lattice planes [2]. Initially we observed the new phenomenon for Se (primarily *in situ*) and Te. Later on it has been revealed for many other substances and materials [3], [4], [5] of different chemical bonding and various preparation conditions (Se-C, Se-Te, Sb_2Se_3, Sb_2S_3, Ge-Sb_2Se_3, Ge-Te, Tl-Se, Cu-Te, α-Fe_2O_3, Cr_2O_3, Co-Pd, Re, W, carbides, amorphous metals, ferroelectrics, phase change materials for memory devices).

The main feature of novel micro-, nanostructure is the permanent regular bending/curving of the lattice planes (about axes primarily lying in the film plane) for the micro-, nanocrystals growing in amorphous film. Different geometries are revealed, Fig.1. Thus one can detect in a perfect crystal ("single crystal") usual translation complicated by relatively small rotation of the unit cell. Anyway more or less significant rotations, up to 300 degrees per 1 micrometer of the crystal length can be attained. Therefore the new term "transrotation" [5] was introduced for such novel crystals/structures. The geometry and gradient of lattice orientations depend upon crystallography of the substance, crystal growth rate (e.g., upon heating), film thickness (the thinner is the film, the stronger is the transrotation) and composition (for binary films with composition gradients).

Earlier hypothetical mechanism of unusual phenomenon based on surface nucleation has been improved and supported by atomistic model of transrotational microcrystals. The last is based on mathematic instruments of conformal transformations. Generally transrotational crystals/structures revealed by TEM can be considered as a new state intermediate between glassy and crystalline ones (similarly to the structure of liquid crystals intermediate between crystalline and liquid). Alternatively transrotation can be regarded as an example of new kind of extended defect in condensed matter. In this sense transrotations (in thin crystals) supplement dislocations (in crystals) and disclinations (in liquid crystals). For the simplest case of cylindrical lattice bending small transrotational "single" crystal has organization of the atoms similar to the hypothetical 2.5D endless plane semicircular multi-walled nanotube.

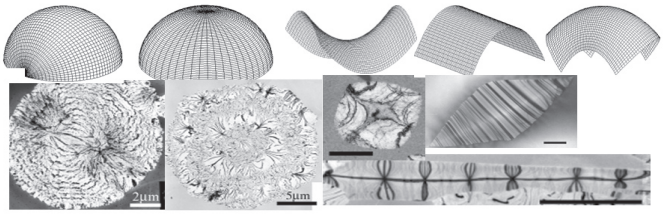


Fig.1 Schemes of lattice transrotation geometry with crystal TEM images below: Se, Fe_2O_3, Ta_2O_5, C+Se+C, Cu-Te. Bar = 1 μm (if not specified).

[1] I.E. Bolotov, V.Yu. Kolosov, *Phys. Stat. sol.* **1982**, *69a*, 85-96. [2] I.E. Bolotov, V.Yu. Kolosov, A.V. Kozhyn, *Phys. Stat. sol.* **1982**, *72a*, 645-654. [3] V.Yu. Kolosov, *Electron Microscopy* **1990** Proc. XII ICEM, *1*, 574-575. [4] V.Yu. Kolosov, *Acta Cryst*. **1990**, *46A*, Bordeaus, 398. [5] V.Yu.Kolosov, A.R. Thölén, *Acta Mater*. **2000,** *48*, 1829-1840.

Keywords: microdiffraction, crystallization, nanocrystal

MS79.P08

Acta Cryst. (2011) A**67,** C695

Transmission electron microscopy studies on $Bi_{1/2}Na_{1/2}TiO_3$-$Bi_{1/2}K_{1/2}TiO_3$-$K_{0.5}Na_{0.5}NbO_3$ ceramics.

Ljubomira Ana Schmitt,[a] Eva-Maria Anton,[a] Wook Jo,[a] Joe Trodahl,[b.], Jacob Jones,[c.] Jürgen Rödel,[a.] Hans-Joachim Kleebe,[a.] [a]*Faculty of Materials- and Geo-Sciences, Technische Universität Darmstadt, 64287 Darmstadt (Germany)*. [b]*MacDiarmid Institute of Advanced Materials and Nanotechnology, Victoria University, PO Box 600, Wellington (New Zealand)*. [c]*Department of Materials Science and Engineering, University of Florida, Gainesville, Florida 32611 (USA)*. E-mail: Ljuba@st.tu-darmstadt.de

For the last decades, lead-zirconate-titanate (PZT)-based materials have been the material of choice for high-performance actuator, sensor and transducer applications. Due to detrimental effect of lead on the environment, it has to be replaced by lead-free non-hazardous materials in the near future. Among the various lead-free systems, the $Bi_{1/2}Na_{1/2}TiO_3$-$Bi_{1/2}K_{1/2}TiO_3$-$K_{0.5}Na_{0.5}NbO_3$ system seems to be a promising candidate for actuator applications.

For this study, lead-free (1-y)($Bi_{1/2}Na_{1/2}TiO_3$-x$Bi_{1/2}K_{1/2}TiO_3$)-y$K_{0.5}Na_{0.5}NbO_3$ specimen with x=0.2 or x=0.4 and y=0, 0.02 or 0.05 were prepared by the conventional solid state reaction method [1]. For transmission electron microscopy (TEM) investigations, samples were polished, dimpled and ion thinned.

Selected area electron diffraction (SAED) revealed the presence of superlattice reflections of the type ½{ooe} and ½{ooo} for specific compositions, where o and e denotes odd and even Miller indices, respectively. For higher $Bi_{1/2}K_{1/2}TiO_3$ content, the appearance of domains was observed. The corresponding SAED patterns showed reflection splitting. This was further confirmed by X-ray diffraction (XRD) measurements that showed peak splitting of (200) pseudo cubic reflections, implying the presence of a tetragonal distorted phase. In contrast, $K_{0.5}Na_{0.5}NbO_3$ addition destabilised the ferroelectric order indicated by the absence of domains and a decrease of peak splitting in XRD. A clear correlation between the aforementioned microscopic features and macroscopic measurements such as polarisation and strain could be drawn.

[1] E-M. Anton, W. Jo, J. Trodahl, D. Damjanovic, J. Rödel, *Jpn. J. Appl. Phys.* accepted 2011

Keywords: Microscopy, Diffraction, Ferroelectricity