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.

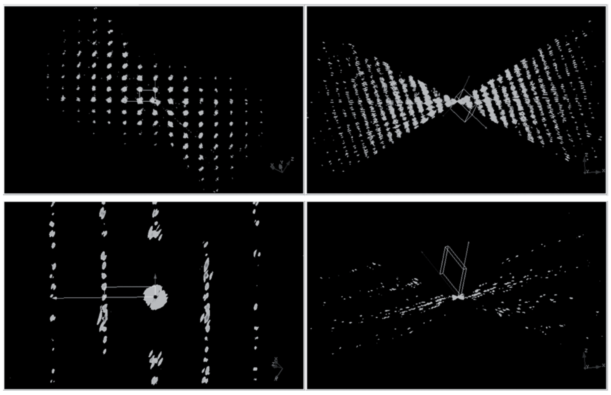

Figure 1: ADT reconstructed 3D diffraction space and unit cell for NiTe and SiC. Upper: NiTe along (001) (left) and tilt axis (right). Bottom: SiC along (001) (left) and tilt axis (right).

[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

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

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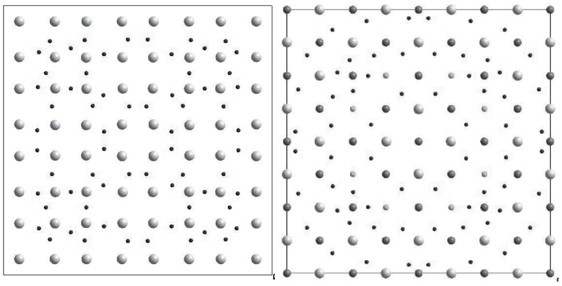
Perovskite-like oxides of Sr–Bi–TM–O systems (with TM = transition metal) have been extensively studied but surprisingly no structures have been published in the Sr–Bi–Ni–O system. Samples with nominal composition $Sr_3Bi_{2-x}Ni_xO_{6-\delta}$ were prepared from nitride precursors and calcined in air 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

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

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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