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**Keywords: quantitative convergent beam electron diffraction, DFT, charge density studies**

## FA4-MS25-T03

**Structure solution in multiphase Sr-Bi-Ni-O powder by electron crystallography.** Holger Klein[a], Pierre Strobel[a], Jérémy David[a], Amélie Rageau[a], Mauro Gemmi[b], Vladimir V. Pankov[c], Masha Golikova-Novitskaya[c], Leonid V. Makhnach[c]. [a]*Institut Néel, Grenoble, France.* [b]*Dipartimento di Scienze della Terra 'Ardito Desio', Sezione di Mineralogia, Milano, Italy.* [c] *Inst. of General and Inorganic Chemistry, National Academy of Sciences of Belarus, Minsk, Belarus.*
E-mail: holger.klein@grenoble.cnrs.fr

In multicomponent oxides of the transitional and rare-earth elements with perovskite structure ($ABO_3$, A = rare-earth, B = transition metal) it is possible to improve the physicochemical and electrochemical properties with cation substitution in sites A and B. In this aim perovskite-like oxides of Sr–Bi–Me–O systems (with Me = Cu [1-3], Co [4], Fe, Cr [5], Mn [6]) have been extensively studied. In this work we investigate the Sr–Bi–Ni–O system where no structures have been published in spite of the fact that it should be possible to obtain multicomponent oxides which exhibit superconducting properties or are suitable for oxide fuel cell and membrane production. The 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. Transmission electron microscopy 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 ($a$ = 33.6 Å). EDX yielded the cation ratio to be 22.0% Ni, 64.2% Sr and 13.8% Bi for the tetragonal phase, the oxygen being too light to be analyzed. In the case of such a mixture of phases with related cell parameters X-ray powder diffraction is useless for structure determination. We therefore conducted an electron crystallography study on the tetragonal phase. Due to the close relationship with the orthorhombic phase only few zone axes permit to distinguish between these two phases. Therefore, we only considered data from a single particle clearly identified as the tetragonal phase in this study. A total of 13 different zone axes were each recorded in selected area electron diffraction mode and with different precession angles up to 4.1°. From the observed extinctions the space group was determined to be *I4/mmm* or *I4mm*. For the structure solution we extracted the intensities from the 8 main zone axes yielding a total of 109 independent reflections with a resolution of 0.8 Å. The data were corrected by a geometrical Lorentz type factor and the structure was solved ($R$ = 29%) using the SIR2008 program [7]. The solution contained all atoms except for one oxygen position. The structure can be described as formed by layers of edge sharing oxygen octahedra. The layers are connected via octahedron corners. In this contribution we compare the obtained structure to oxides containing other transition metal ions and discuss the subject of the missing oxygen position.

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**Keywords: electron crystallography, crystallographic structure, oxide**

## FA4-MS25-T04

**Structure Analysis of Titanate Nanorods by Electron Diffraction.** Iryna Andrusenko[a], Enrico Mugnaioli[a], Tatiana Gorelik[a], Ute Kolb[a], Dominik Koll[b], Martin Panthöfer[b], Wolfgang Tremel[b]. [a]*Institute of Physical Chemistry, Johannes Gutenberg-University, Mainz, Germany.* [b]*Institute of Inorganic and Analytical Chemistry, Johannes Gutenberg-University, Mainz, Germany.*
E-mail: andrusen@uni-mainz.de

Structural information is essential for understanding physical and chemical properties of materials. In order to gain structural information from nano crystalline materials diffraction data from single nano grains should be collected. In these cases nano electron diffraction is the technique of choice, as it can probe single crystals as small as 20 nm. Nevertheless structure solution by electron diffraction is hampered by strong dynamical effects and limited number of sampled reflections. In order to reduce these drawbacks, routines for a quasi-continuous sampling of reciprocal space were developed by Kolb at al. (Automated Diffraction Tomography – ADT) [1, 2]. Since the first report on the "Formation of Titanium Oxide based Nanotubes" [3] this type of nano materials has been discussed intensively for its use in photocatalysis or as semiconductor electrode in dye sensitized solar cells. Preliminary investigations showed that there are different crystallographic phases formed in nano sized titania systems depending on the synthetic route. Structures of all these phases are important for understanding the mechanism of nano architectural formation and physical properties of the product. Here we present the structure analysis of the primary product in the synthesis of titania nano rods from titania upon hydrothermal treatment in concentrated caustic soda solution. The structure analysis was performed by a combination of ADT and precession electron technique [4]. Quasi-kinematical 3D electron diffraction data sets from single nano crystals were collected with a FEI TECNAI F30 transmission electron microscope. All analyzed crystals exhibited diffuse scattering. Nevertheless, an almost ordered crystal was selected for diffraction data collection. Lattice parameters were determined automatically by devoted routines. The structure was solved using direct methods implemented in SIR2008 and refined by ShelxL. The disorder was visualized and described by the 3D reconstructed reciprocal space.

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