

# Oral Contributions

## [MS16-04] Complex $\text{Sr}_{64.1}\text{Bi}_{27.7}\text{Ni}_{8.2}\text{O}_x$ Revisited and Refined

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Perovskite-like oxides of Sr–Bi–TM–O systems (with TM = transition metal) have been extensively studied but only recently the structure of tetragonal  $\text{Sr}_5\text{BiNi}_2\text{O}_{9.6}$  has been published in the Sr–Bi–Ni–O system [1]. The structure of this phase was solved by precession electron diffraction (PED, [2]) in a transmission electron microscope (TEM) and subsequently refined from laboratory X-ray powder diffraction.

In the same powder a minority phase was identified yielding complex electron diffraction patterns. Its cubic unit cell has a cell parameter of 33.7 Å. Chemical analysis by energy dispersive spectroscopy (EDS) in a TEM gave a cation ratio of 8.2% Ni, 64.1% Sr and 27.7% Bi. From the rare particles of this phase in the powder only an incomplete data set could be obtained. Nevertheless, the space group was determined as  $Im\bar{3}m$  and a structure model was obtained by direct methods (SIR2008 [3]) from PED data. This structure model contained reasonable cation positions, but only part of the oxygen positions were found. This first result permitted us to obtain a powder mainly composed of this cubic phase and a more extensive PED study and an X-ray powder diffraction (XRPD) study could be conducted which will be presented in this contribution.

The intensities observed in PED patterns don't depend critically on the exact orientation of the crystal with respect to the incident beam as is the case in conventional electron diffraction. The intensities can therefore be used for the

determination of the symmetry of a crystal. A close inspection of the [111] zone axis revealed a very slight deviation from the 6mm symmetry expected for the  $Im\bar{3}m$  space group. The true symmetry of the [111] is only 6, indicating the space group  $Ia\bar{3}$ . This result was then confirmed from the extinction conditions observed in the [001] zone axis, which was previously unavailable.

PED data was then used to obtain a new model of this phase. Even though PED data is quite capable of solving even such complex structures, dynamical diffraction is still present and it is not possible to extract precise structure factors from the diffracted intensities. For structure refinement we have used XRPD data using the structure model obtained from the PED as a starting point. This presentation will highlight the complementarity of PED and XRPD and discuss why a reasonable model of the structure could be obtained in a wrong space group.

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[2] Vincent, R., and Midgley, P.A., (1994) *Ultramicroscopy* **53**, 271-282

[3] Burla, M.C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G.L., De Caro, L., Giacovazzo, C., Polidori, G., Siliqi, D., and Spagna, R., (2007) *Journal of applied Crystallography* **A40**, 609-613

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