MS44-P8 Structural investigation of mullite-type $Al_4B_2O_9$ by electron diffraction

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Structural characterization of materials is a key step to understand their physical properties in order to optimize industrial applications. Electron diffraction, as opposed to X-ray powder diffraction, can provide atomic structure information from a single nanocrystal. It has advantages in the case of structures with large unit cells, pseudosymmetry, superstructures, low crystallinity and for impure or multi-phase samples. Automated electron diffraction tomography (ADT), combines nano diffraction, tilt series acquisition and electron beam precession to derive a three-dimensional atomic structure with data bearing minimal dynamic scattering artifacts.[1,2]

The published crystal structure of $Al_4B_2O_9$, an industrially important mullite-type ceramic material, was constructed from a model of the known boralsilite structure and refined from X-ray powder diffraction data.[3] Due to the pseudo-orthorhombic unit cell and the complexity of the structure, Rietveld refinement results were not ideal, leading to unclear occupancies of two oxygen atoms.

ADT data reconstruction delivered monoclinic unit cells in space group *C* 2/m (a = 14.8 Å, b = 5.5 Å, c = 15.1 Å, and β = 90.6°). *Ab-initio* structure solution of $Al_4B_2O_9$, using direct methods, delivered all atoms directly from solution. The well-resolved potential map clearly shows one fully occupied oxygen position but no significant signal for the second postulated oxygen in the other channel. For samples prepared with an initial excess of boron, some crystals revealed modulated streaking in b^* direction, which has been assigned to a superstructure with a threefold b-axis and an additional disorder. The modeling of different layer stacking along b direction and diffraction simulations were carried out with software package DISCUS[4] leading to electron diffraction patterns resembling the experimentally observed streaking.

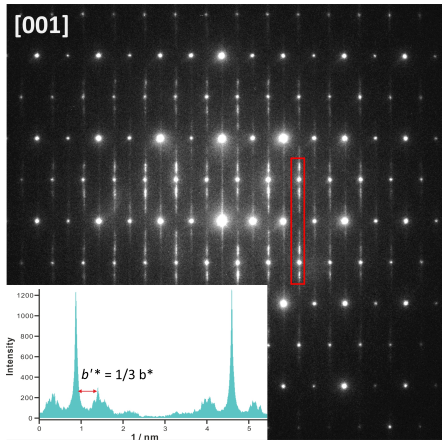

Figure 1. Modulated diffuse scattering in the electron diffraction pattern of $Al_4B_2O_9$.

Keywords: Electron crystallography, Automated electron diffraction tomography, Mullite-type structure

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