Transmission electron microscopy and X-ray powder diffraction for solution of modulated structures: a synergetic approach.

Artem M. Abakumov[a], Joke Hadermann[a], Evgeny V. Antipov[b], Gustaaf Van Tendeloo[a]

[a] *EMAT, University of Antwerp, Groenenborgerlaan 171, B-2020 Antwerp, Belgium*
[b] *Department of Chemistry, Moscow State University, 119992 Moscow, Russia*

A priori extraction of information on the components of the modulation vector and superspace symmetry from X-ray powder diffraction patterns of incommensurately modulated materials is a challenging problem. The reflections on powder XRD patterns of incommensurate modulated phases form a dense set being projected on the 2θ axis, whereas for non-modulated or commensurate crystals this set is discrete. Frequent overlapping of weak satellites with intense main reflections, and a generally low peak-to-background noise ratio for satellite reflections, strongly reduce the accuracy to which the positions of the satellites that can be determined from the experimental XRD profile. Knowledge of the three-dimensional reciprocal lattice is still needed for an unambiguous determination of the length and orientation of the modulation vector in reciprocal space. Selected area electron diffraction and high resolution electron microscopy appear to be particularly useful for this purpose since they allow observing reciprocal space sections produced by diffraction from small areas of a few nanometers, i.e. from single crystallites that make up the powder sample. The usefulness of a combination of transmission electron microscopy and X-ray powder diffraction data for the solution of incommensurately modulated and composite structures will be illustrated using the examples of $Sr_{1+x}BO_3$ (B = Mn, Cu, Co) hexagonal perovskites, A_xMnO_2 and $[AF]_{2.5}[Mn_6O_{12}]$ (A = Ca, Sr) tunnel manganites, and anion-deficient perovskites modulated by periodically-spaced translational interfaces (crystallographic shear planes).