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Combining powder diffraction with TEM for solving modulated structures

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In many materials competing interactions of different nature may give rise to incommensurate modulations causing extreme structure complexity. Ab initio solution of the modulated structures even with using high quality synchrotron X-ray and/or neutron powder diffraction data appears to be a very challenging problem due to weakness of the satellite reflections, ambiguity in the determination of the modulation vector(s) and superspace symmetry and difficulties in building the initial model for further Rietveld refinement. These problems can be resolved or, at least, mitigated if the diffraction, imaging and spectroscopic advanced transmission electron microscopy techniques are combined with the analysis of powder diffraction data. Complete reconstruction of the reciprocal space, structure solution using quasi-kinematical electron diffraction data, mapping projected scattering density in the unit cell, visualization of the light atoms, displacive and occupational ordering, mapping chemical composition and coordination number can be utilized to reveal the nature of incommensurate modulations and construct the reliable model for the refinement from powder diffraction data. The benefit of the strategy of combining the powder diffraction data with the reciprocal and real space information obtained using aberration-corrected scanning transmission electron microscopy will be illustrated on the examples of the transition metal oxides: $\text{Li}_3\text{xNd}_{2/3-\text{x}}\text{TiO}_3$ perovskites with frustrated incommensurately modulated octahedral tilting pattern [1]; perovskites $(\text{Bi,Pb})_{1-\text{x}}\text{Fe}_{1+\text{x}}\text{O}_{3-\text{y}}$, modulated by crystallographic shear planes [2]; $\text{CaGd}_2(1-\text{x})\text{Eu}_2\text{x}(\text{MoO}_4)_4(1-\text{y})(\text{WO}_4)_4\text{y}$ scheelites with incommensurately modulated ordering of cation vacancies [3].

[1] A.M. Abakumov, R. Erni, A.A. Tsirlin et al, *Chem. Mater.*, 2013, 25, 2670, [2] A.M. Abakumov, D. Batuk, J. Hadermann et al, *Chem. Mater.*, 2011, 23, 255, [3] V.A. Morozov, A. Bertha, K.W. Meert et al, *Chem. Mater.*, 2013, 25, 4387

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