

Nanopowder crystallite sizes and shapes as viewed from XRD and TEM: examples of EMT zeolite, Ti- and Mn-oxide nanoparticles

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Crystallites with sizes down to one or several unit-cells give rise to strongly broadened diffraction lines, often discouraging any quantitative treatment of the patterns. Full-profile analysis of these patterns obtained from nanopowders however still offers quantitative characterization, provided a robust implementation of crystallite sizes and shapes is operated. Rietveld analysis combined to shape modelling by spherical harmonics as proposed by the Popa approach is used in this work. It proved to allow discrimination between two closely related zeolite phases, namely FAU and EMT, obtained as nanopowders using a specific technique. Mean crystallites not larger than 2 unit-cells of EMT could be quantified using laboratory XRD. In cases for which not enough powders could be obtained, i.e. for which XRD signal becomes too small, we used an equivalent approach on TEM ring patterns. In this latter case only small powder volumes are required and the large TEM signal becomes advantageous. When calibrated using XRD and a given powder, the microscope can be used for further nanopowder characterization, allowing crystallite sizes, shapes and local crystallographic textures.