MS40-P08 | Morphology characterization of anatase TiO_2 nanocrystals by advanced Rietveld refinement of powder X-ray diffraction data including anisotropic size broadening models

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Control of the crystalline nanocrystal morphology is important since it is known that certain facets substantially promote catalytic activity. It is, however, in itself challenging to determine nanocrystal morphology to provide a rational basis for the synthesis control. One widely used technique to characterize morphology of crystalline nanocrystal is Tranmission Electron Microscopy (TEM) and High Resolution Transmission Electron Microscopy (HRTEM). While the main challenge of TEM and HRTEM is the so-called 3D-to-2D dilemma: the TEM and HRTEM images are the two-dimensional (2D) projections of the real three-dimensional (3D) nanocrystal along the direction of the incident electron beam (EB), and accurate reconstruction of the 3D morphology requires additional crystallographic knowledge and insight to the material under study.

Here, an alternative method, namely incorporation of an anisotropic size broadening model based on a linear combination of symmetrized spherical harmonic functions into the Rietveld refinement of synchrotron PXRD data is utilized to establish the sample averaged 3D morphology of anatase TiO_2 nanocrystals obtained from supercritical flow synthesis. This procedure can not only overcome the intrinsic limitation of utilizing Scherrer equation on a non-refined diffraction pattern, but also intuitively display the average 3D morphology of nanocrystals by using the refined parameters. The robustness of this strategy is verified by comparing the anisotropic peak broadening models with results obtained from HRTEM and Raman spectra. In addition, it is shown that 3D morphologies determined from laboratory PXRD data give nearly identical results to reconstruction from synchrontron PXRD, demonstrating the broad applicability of the approach.