Morphological reconstruction from powder diffraction data

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Traditionally, the analysis of powder diffraction data through whole pattern fitting has either focused on structural analysis (through Rietveld refinement) or through peak shape analysis for size/strain studies (through the independent fitting of individual peaks). We have recently pioneered methods that fuse these two approaches [1]. This has advantages both for structural analysis / phase quantification (which can be done more effectively when the particle size and/or strain is complex, as seen in Figure 1) and for peak shape analysis (in which the constraint of intensities based on a structural model allows peak shapes to be more effectively extracted when peak overlap exists). Since this approach enables us to rapidly and robustly quantify the shapes of a hundred or more peaks automatically refined from a powder diffraction pattern (Figure 2), we can use it to effectively reconstruct the morphology (size and shape) of the particles comprising the powder. Some examples of these novel methods applied to the characterization of functional materials (batteries, catalysts, thermoelectrics) will be discussed.

Figure 1. Comparison of different approaches to whole-pattern fitting for synchrotron diffraction data from a sample containing a polymorphic mixture of nanoscale Co: Rietveld refinements with an isotropic size model (top), Pawley fits with an isotropic size model (middle), and our modified Rietveld approach using separately refined peak shapes for each diffraction peak (bottom).

Figure 2. Top: Independent peak shape refinement (IPSR) fit shown on a log scale along with a comparison of the difference plots from IPSR ($R_{wp} = 7.4\%$) and conventional isotropic fits ($R_{wp} = 14.7\%$). Bottom: Morphological information obtained from 100+ automatically extracted peak integral breadths fit while using the constraint of a structural model, shown in a Williamson-Hall type plot.