X-RAY DIFFRACTION PRECISION, ACCURACY, AND CONFIDENCE INVESTIGATION FOR DETERMINING CRYSTALLITE SIZE IN NANOPOWDERS

Alexander P. Moore*, Martin B. Nemer*, Mark A. Rodriguez*, Christine C. Roberts*, Patrick F. Fleig*, and Hans W. Papenguth*

***Sandia National Laboratories, 1515 Eubank, Albuquerque, NM 87123**

X-ray diffraction (XRD) is often utilized as a method to determine bulk sample crystallite size in powder characterization. A sample's crystallite size affects the XRD spectrum by broadening the diffraction peaks. However, microstrain and instrumentation set-up also broaden diffraction peaks. Therefore, to determine crystallite size, the instrument, microstrain, and crystallite contributions to peak broadening need to be deconvoluted. This deconvulation complicates crystallite size estimation and adds uncertanty.

While it is generally accepted that XRD peak broadening allows for qualitative crystallite size comparisons, its use for quantitative information is still debated. In this study, the precision, uncertainty, and accuracy in estimating crystallite size via X-ray diffraction was investigated using whole pattern (WP) weighted least squares (wLSQ) and Williamson-Hall methods. The methods' precision is investigated by re-preparing, re-running, and re-analyzing identical samples. The crystallite-size-determinations were compared against independent crystallite-size analyses using visual particle identification of scanning-electron-microscope (SEM) micrographs and from indirect calculations using Brunauer-Emmett-Teller (BET) adsorption determined surface area. Finally, the methods' confidence is investigated using both a frequentist F-test approach and a Bayesian Inference statistical inversion method. An open-source code-package for use with GSASII has been written to aid in this analysis for the XRD community.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc., for the US Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.