

Applications of Ultra-small angle X-ray scattering in the characterization of nanomaterials

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Ultra-small angle X-ray scattering (USAXS) instruments using Bonse-Hart geometry – both synchrotron based and desktops - are unique among SAXS instruments in their ability to characterize large sizes – over 1 micrometer for the APS instrument. They are also unique in their ability to measure over 3-4 decades of scattering vector (q) range, compared to about 1.5 decades for a typical single configuration of a pinhole SAXS or SANS instrument. The use of USAXS instruments for nanomaterial analysis may seem unnecessary, given the nanostructures of interest. However, experience from over 10 years of operation of the APS USAXS instrument (now located at 15ID beamline) shows, that for many – if not most – nanomaterials there are important and interesting features at small scattering vector (“low- q ”) values inaccessible in regular pinhole SAXS instruments. These features may, for example, reflect agglomeration or long-distance arrangements present in the materials. Our experience shows, that understanding of these low- q data is often imperative for a fully quantitative analysis of nanomaterial samples.

This presentation will review interesting examples from the last about 10 years of APS USAXS operations and provide some insights into the challenges encountered in the characterization of different nanomaterial types. The examples will include previously published studies of powders, nanoporous ceramics, metals, aerogels, and gels.

Furthermore, recent upgrades of the USAXS instrument at APS will be highlighted. These provide improved capabilities to measure high- q data, of particular interest for the study of weakly scattering nanomaterials, such as polymer systems or solutions. Again, the new capabilities will be demonstrated with recent examples of user science.

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