

A small- and wide-angle X-ray scattering (SWAXS) laboratory camera for simultaneously determining the size, morphology and crystallinity of nanoparticles

Xiaoai Guo, Alexander Gutsche, Hermann Nirschl

Institute for Mechanical Process Engineering and Mechanics, Karlsruhe Institute of Technology,
Strasse am Forum 8, D-76131 Karlsruhe, Germany
Email: xiaoai.guo@kit.edu

Abstract

This paper reports a modified small- and wide-angle X-ray scattering (SWAXS) laboratory camera, which allows simultaneous determination of the size of the primary particle or pore, and fractal dimension, and identification and quantitative determination of the crystallite properties of nanoparticles. The SWAXS laboratory camera used in the current work (KFLCu2K, $\lambda=0.154\text{nm}$) has a monochromatic high-intensity primary beam, which has been realized by using a focusing Göbel mirror and a slit collimation system, and a flexible 2-dimensional imaging plate X-ray detector with a wide dynamic range and a high spatial resolution of $25\mu\text{m}$. The increase in the primary beam intensity led to a significant reduction of the exposure time (120-180sec in this work). This compact laboratory camera system makes a simultaneous and continuous measurement possible, i.e., with one detector the complete information from a single sample at different detection angles (SAXS and WAXS, $2\Theta < 90^\circ$) can be simultaneously obtained by one measurement. Different nanostructured particles, such as pure metallic nanopowders, metallic and oxide nanoparticles, were investigated with this SWAXS laboratory camera. The results of primary particle or pore size obtained from the SAXS spectra were compared to those obtained with TEM. Meanwhile, different phases, namely fingerprints indicating the crystal morphology, were identified from the WAXS spectra. Furthermore, these Bragg peaks were used to quantitatively determine the crystallite size. These results were compared to the XRD reference patterns, showing good agreement. This unique technique is very suitable for fast in-situ characterization of nanostructured powders and dispersions as well as process monitoring.

Keywords: Small- and wide-angle X-ray scattering (SWAXS), nanoparticles, primary particle size, fractal dimension, crystallinity