# Characterization of Primary Particle Size and Crystallinity by Using Small and Wide-angle X-ray Scattering (SAXS/WAXS)

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Primary particle size, agglomerate structure and crystallinity are crucial properties of nanoparticles especially in the particle synthesis processes, such as synthesized metallic nanoparticles. In-situ and on-line monitoring of these parameters allows a rapid feedback between the desired product quality and process parameters and thus minimizes reject in the continuous particle synthesis process improving the product quality and efficiency.

In this work, a modified laboratory X-ray (Cu-Kα, λ=0.154nm, line focus 0.4x12mm) camera was used for determination of the particle size and crystallinity. A focusing Goebel X-ray mirror and a 2-dimensional image plate X-ray detector [1] were utilized to increase the intensity of the primary X-ray beam significantly reducing the measuring time. This non-invasive and short-time measurement technique is suitable for in-situ and on-line characterization of nanoparticles. At small angles (SAXS) the primary particle size and fractal dimensions can be determined by using a unified fit model [2], while at wide angles (WAXS) the information on the crystallinity can be obtained simultaneously by only one measurement by means of a flexible camera detector. Dispersions and powders of different nanoparticles, e.g. TiO2 and Ag, were measured. The measured X-ray scattered intensity profile at low scattering vector q showed a Guinier's exponential decay, from which the primary particle radius of gyration was obtained showing good agreement with conventional methods like TEM. Then a Porod's power-law decay was observed at high q and the surface fractal dimension of the primary particles was obtained. At even larger q the measured scattering pattern exhibited some diffraction peaks, resulting from the crystallinity and crystal structures. These results obtained from WAXS also agree well with the standard database of X-ray diffraction Spectra. Therefore, it can be shown that this technique is very interesting for in-situ and on-line investigation of nanoparticles and their crystallite properties.

**Reference:**

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[2] G. Beaucage, J. Appl. Cryst. **28**, 717-728 (1995).