Poster

Determination of the average crystallite size and the crystallite size distribution: The envelope function approach EnvACS

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A procedure is presented to exactly obtain the apparent average crystallite size (ACS) of powder samples using standard in-house powder diffraction experiments without any restriction originating from the Scherrer equation [1]. Additionally, the crystallite size distribution within the sample can be evaluated. To achieve this, powder diffractograms are background corrected and long-range pair-distribution functions $G(r)$ up to 300 nm are calculated from the diffraction data. The envelope function, f^{env}, of the $G(r)$, assuming spherical crystallites [2] is approximated by a procedure determining the absolute maxima of the $G(r)$ in a certain interval (r-range). Fitting of an ACS distribution envelope function to this approximation gives the ACS as well as its distribution. The method is tested on diffractograms of LaB_6 standard reference materials measured with different wavelengths, geometries, and instruments to demonstrate the validity of the approach and to clarify the influence of the used wavelength. The latter one results in the general description of the *maximum observable average crystallite size* (MOACS), calculated according to equation 1 derived from [3]:

$$MOACS = \frac{\lambda^2}{2\,\Delta\lambda} \qquad (1)$$

This depends on the used instrument and wavelength (see figure 1). Additionally, the crystallite site distribution is compared to particle size distributions based on TEM investigations, as shown in figure 2, enabling an approximation of the average number of crystallites perparticle.

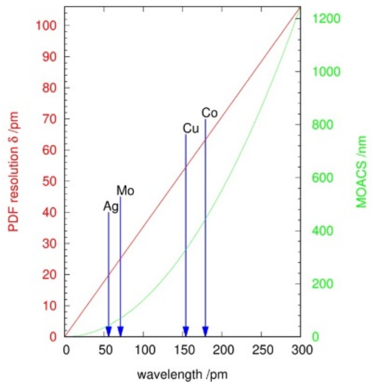

Figure 1. MOACS vs. wavelengths according to equation 1 using the natural spectral width of Cu radiation $\Delta\lambda = 3.615 * 10^{-5}\,nm$ [3] and PDF resolution δ assuming a maximum scattering angle 2θ of 135°

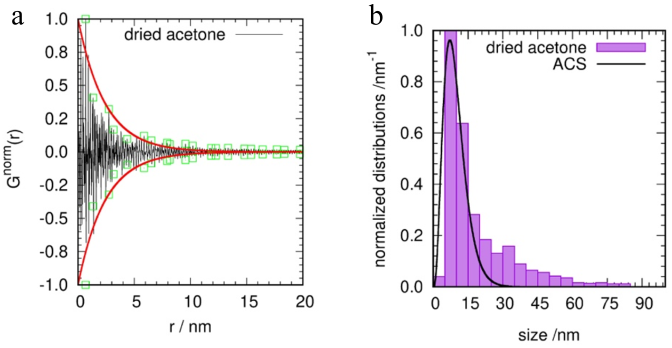

Figure 2. Normalized $G^{norm}(r)$ (black), determined maxima (green) and fitted envelope (red) function (a) and normalized particle size distributions (purple bars – taken from [4]) and the determined ACS distributions (black lines – calculated by the new procedure using data provided by B. Gökce, normalized) [4] (b).

[1] P. Scherrer, Bestimmung der Größe und der inneren Struktur von Kolloidteilchen mittels Röntgenstrahlen, Nachrichten von der Gesellschaft der Wissenschaften zu Göttingen, Mathematisch-Physikalische Klasse / Zeitschriftenband (1918) 98-100.

[2] R.C. Howell, T. Proffen, S.D. Conradson, Pair distribution function and structure factor of spherical particles, Physical Review B 73(9) (2006). Doi:10.1103/PhysRevB.73.094107.

[3] D. Rafaja, V. Klemm, G. Schreiber, M. Knapp, R. Kužel, Interference phenomena observed by X-ray diffraction in nanocrystalline thin films, Journal of Applied Crystallography 37(4) (2004) 613-620. Doi:10.1107/s0021889804012701.

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