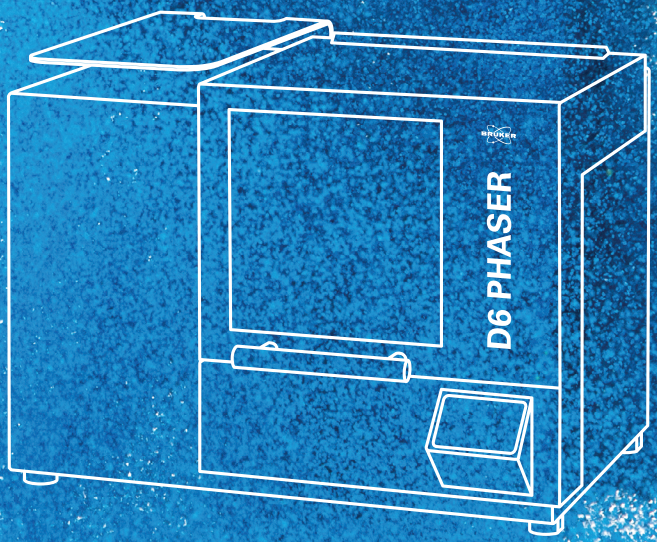

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD for the Analysis of Crystallite Sizes

Application Report 38

The D6 PHASER is ideally suited for rapid crystallite size analysis using powder X-ray diffraction. The analysis is based on the evaluation of the diffracted peak profiles. These show a characteristic broadening that increases as the crystallites become smaller. This broadening must be separated from the instrumental line broadening, which determines the upper limit of the crystallite size. The lower limit of the analysis is determined by the ability to separate broad, low intensity signals from the instrumental background.

Key characteristics of the D6 PHASER for this application include

- Angular resolution better than 0.03° 2Theta
- High X-ray flux, achieved by the 600 W or 1.2 kW generator, the compact goniometer radius, and the motorized divergence slit that illuminates a large and constant sample area at all measurement angles.
- Dynamic Beam Optimization (DBO) provides tight control of the instrument background to enable separation of broad peaks.

These features make the D6 PHASER an excellent tool for studying crystallite size in the chemical or pharmaceutical industries, where process parameters or material properties are closely related to crystallite size or surface area. Another typical application is LC analysis (ASTM D5187). It relates the Full Width Half Maximum to the quality of calcined anode coke used in the electrolytic smelting of metals.

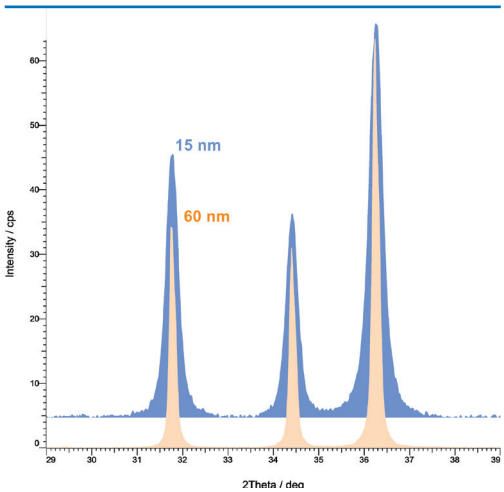

Figure 1
Two scans of NIST SRM1979 schematically showing the peak broadening for the smaller particles.

Figure 2
D6 PHASER equipped for Dynamic Beam Optimization (DBO) with Variable Divergence Slit (VDS), Motorized Air Scatter Screen (MASS) and LYNXEYE XE-T detector.

The instrument performance of the D6 PHASER for crystallite size analysis was verified by evaluating the NIST Powder Diffraction Line Profile Standard Reference Material SRM 1979 (Figure 1). Thin sample films were prepared on low background silicon sample holders. Data were collected in the configuration shown above and evaluated using DIFFRAC.TOPAS v7. Line shape analysis was performed similarly to the approach described in the NIST certificate, using Pawley fitting and the fundamental parameter peak model to deconvolute crystallite size from instrumental line broadening. The table below shows the excellent agreement of the crystallite size with the data from the certificate.

For the determination of isotropic crystallite sizes it is sufficient to evaluate a single peak in either DIFFRAC.EVA or DIFFRAC.TOPAS. The latter also allows to refine the directional dependence of the crystallites (or the shape of the crystallites) by evaluating the whole diffraction pattern (Figure 3).

	Sample A	Sample B
Certificate LVol / nm	31.65(46)	97.2(14)
Fixed Divergence Slit data	29.46(50)	107.2(16)
Variable Divergence Slit data	26.5(18)	104(1)

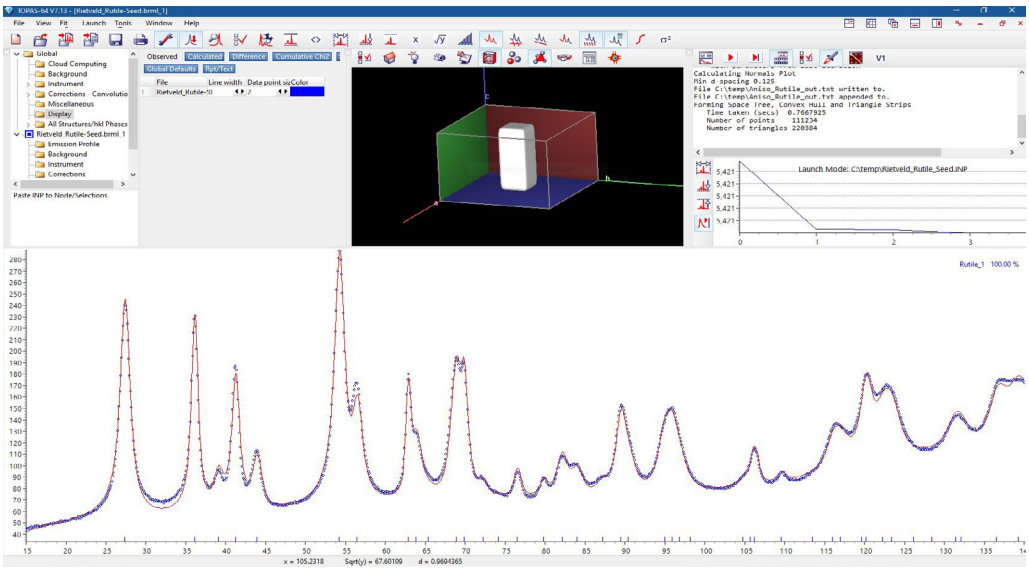

Figure 3
Crystallite size anisotropy of cuboidal rutile, TiO2, analyzed with DIFFRAC.TOPAS. The crystallization seeds show an x:z aspect ratio of about 1:2.5.

Data were collected with a D6 PHASER 1.200W, Cu radiation, no K-beta filter, 2.5° Soller collimators, primary variable divergence (constant illumination mode), motorized air scatter screen, scan range 15 to 140° 2Theta, total scan time 214 sec with the LYNXEYE XE-T detector in high resolution mode.

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