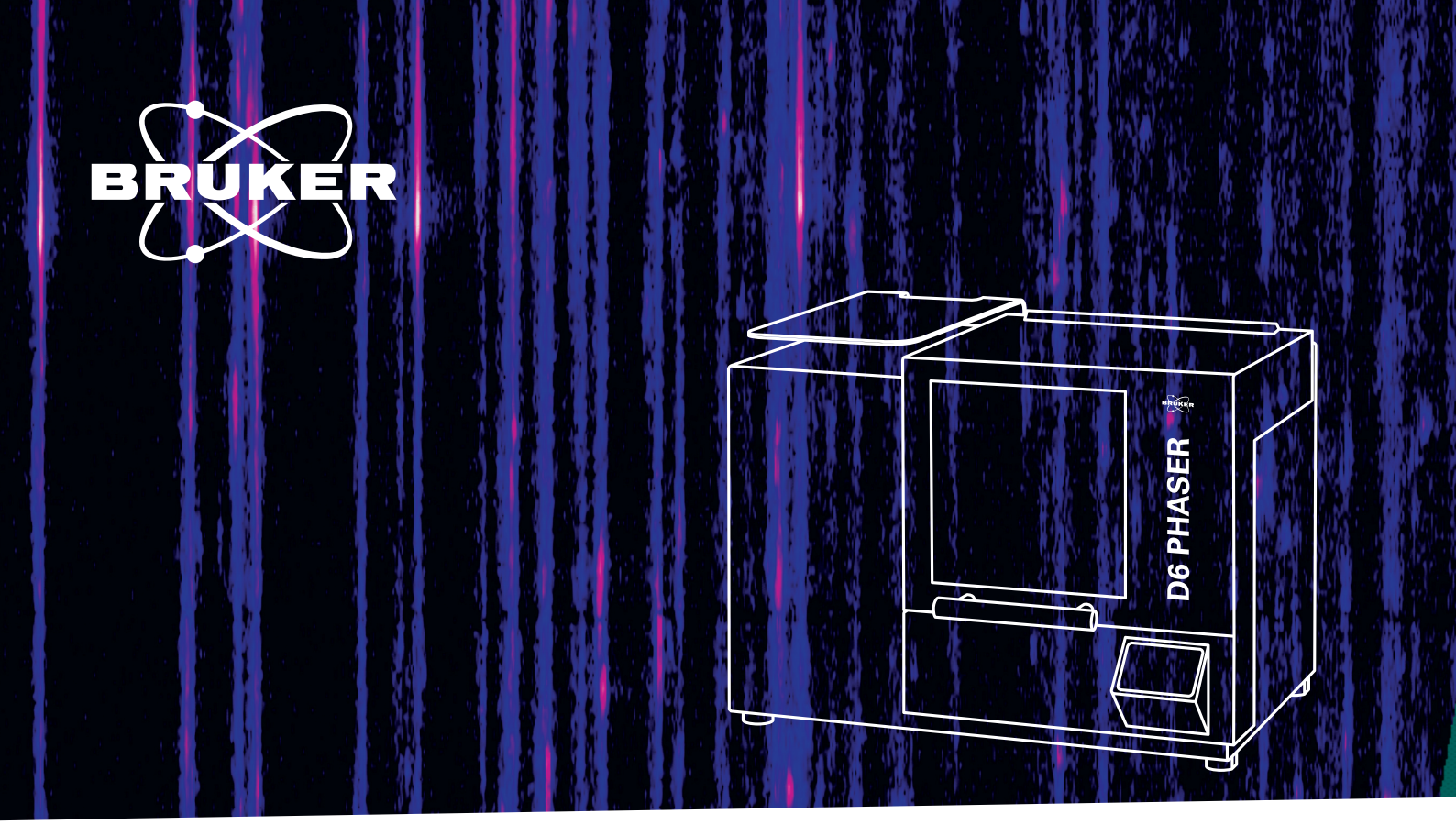

Bragg-2D representation in DIFFRAC.EVA of coarse-grained sugar powder, measured with LYNXEYE XE-T.

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD Two-Dimensional Diffractometry

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The basic requirement for detecting all reflections with representative intensity in powder diffraction is the presence of a sufficient number of crystallites of appropriate size and their statistical arrangement in the sample holder. Two-dimensional diffractometry is a method to easily visualize the presence of too large or too few crystallites, or their preferred arrangement.

This report shows that one-dimensional LYNXEYE detectors in combination with the line focus of the X-ray tube can be used efficiently to monitor the quality of sample preparation without the need for expensive two-dimensional detectors. The real strength of 2D detectors lies in the fact that in combination with the point focus of the X-ray tube as well as a variable sample to detector distance they allow quantitative powder diffraction, stress and texture analysis. However, this requires a certain size of 2D detectors, which is not feasible in benchtop XRD instruments anyway.

The D6 PHASER offers two elegant realizations of 2D diffraction for reflection geometry, the Bragg-2D method and Phi-1D scans. Bragg-2D does not require the sample to be moved. Instead, a large area of the sample is exposed to the X-ray beam by choosing a wide primary divergence and the diffraction signal from different crystallites is visualized in $\Delta\omega$ vs. 2Theta space. The Phi-1D method requires a rotating stage. The sample is illuminated with a narrower X-ray beam, the detector is positioned at a specific 2Theta peak position, and the crystallites are imaged by rotating the sample while continuously taking detector snapshots.

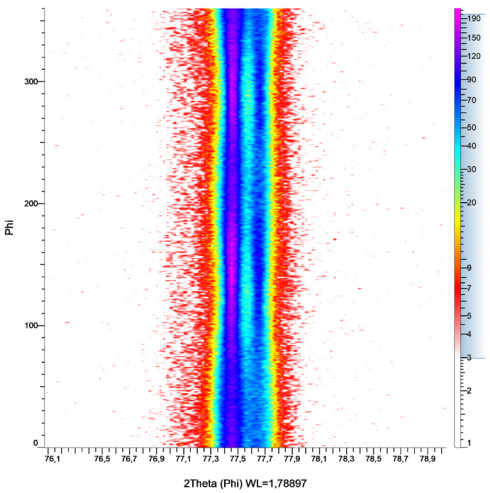
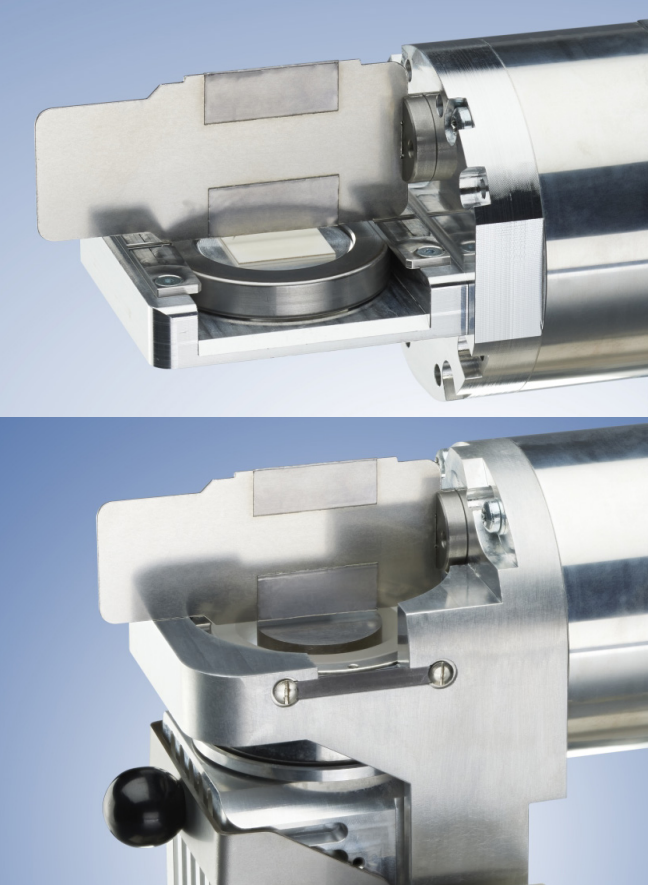


Figure 1
DIFFRAC.EVA 2D representation of a Phi-1D scan of a rolled aluminum sheet sample, measured with Co radiation.



The cover image and Figure 3 show the 2D diffraction pattern of a sample that was powdered too coarsely. This results in the spots visualized here. In a regular 1D powder measurement, the signal would be integrated along the diffraction lines without the user realizing that the grain size is not homogeneous. As a consequence of the fast 2D measurement, the sample should be pulverized more before a quantitative 1D XRD measurement is performed.

The second example (Figure 1) shows the preferential orientation of sufficiently small crystallites. The vertical line shows a broad intensity modulation, whereas for a perfectly randomly oriented material the intensity should be constant. In addition, the diffraction signal has different widths, indicating the presence of microstrains. For the Al sheet measured here, a better averaging can only be achieved by different orientations of the sample. When measuring a powder sample, try to reorient the crystallites more randomly during preparation, or compact the powder with less contact pressure.

Figure 2
D6 PHASER fix stage (top) capable of Bragg2D diffraction and a rotation stage (bottom) for Bragg2D and Phi-1D two dimensional diffraction.

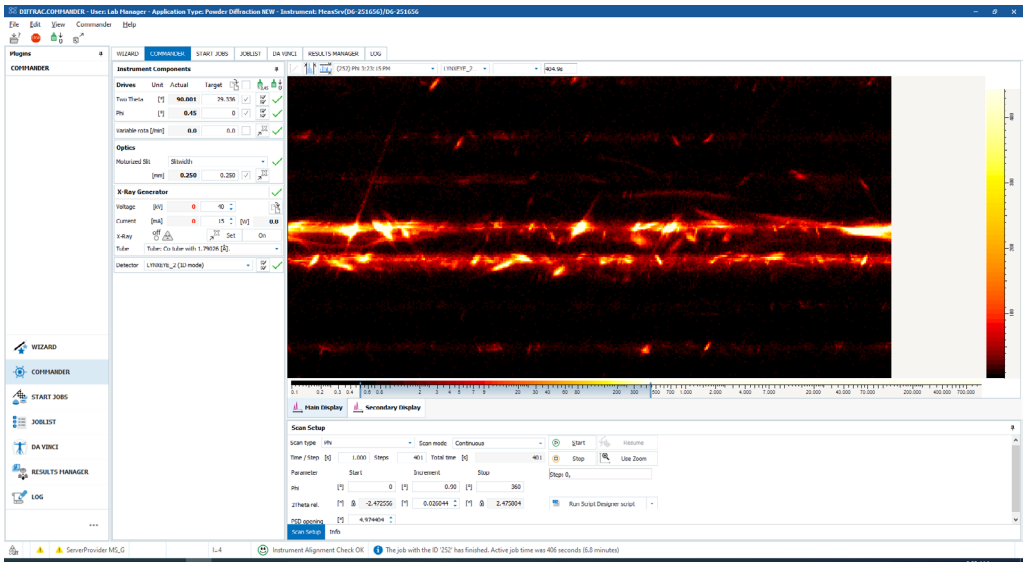


Figure 3
DIFFRAC.COMMANDER showing a Phi-1D scan of a coarse-grained sugar sample. The horizontal axes of the image corresponds to the Phi rotation while the vertical shows the detector snap-shots.

Data were collected with a D6 PHASER 600W, Co radiation, K-beta filter, 2.5° Soller collimators, primary variable divergence (constant opening mode, 0.25 mm), no air scatter screen. The measurement was performed as continuous phi-scan with 0.9 deg increment, 1 sec exposure, total scan time 401 sec with the LYNXEYE-2, fully opened covering 4.97 °2Theta.

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