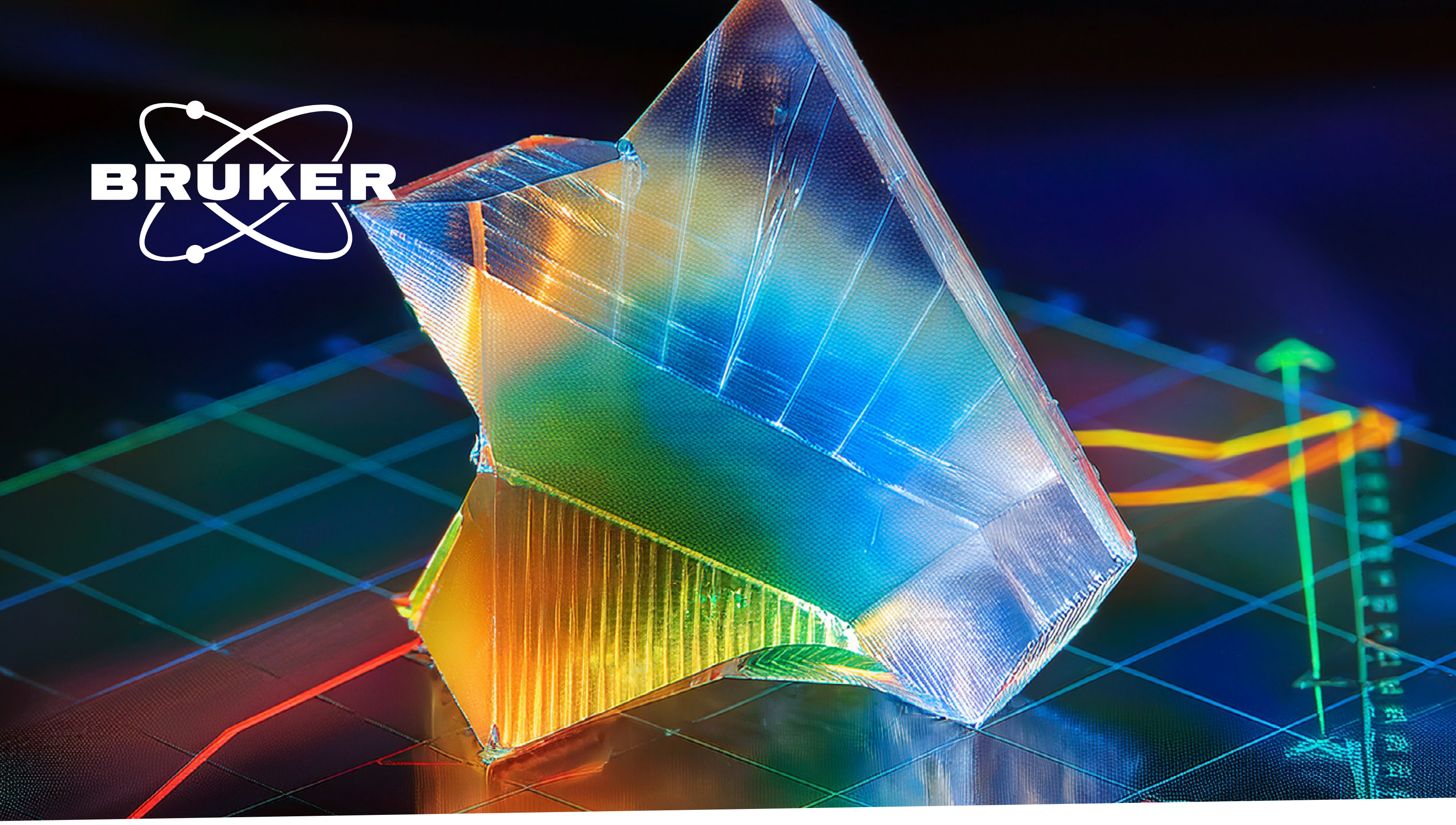


SINGLE-CRYSTAL X-RAY DIFFRACTION

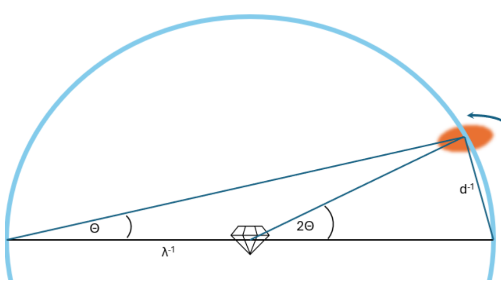
Direct Assessment of Crystal Mosaicity

Rocking Curve Measurements within the APEX Suite

Introduction

Rocking curve measurements are an excellent tool for gaining deeper insight into the quality and mosaicity of a given sample. Additionally, an instrument-specific "instrument broadening" contribution can be established. Measurements are straightforward and quick: By gradually changing the angle of the incident X-ray beam with respect to the sample, the profile of a given Bragg reflection is mapped, producing an intensity profile versus diffraction angle (Figure 1).

The first step in a rocking curve measurement is to carefully center the crystal in the X-ray beam. Once this is accomplished, a short orientation scan is used to select the desired Bragg reflection. Then, the sample is rotated in small angular increments around the diffraction angle, and a diffraction image is recorded at each step. Finally, for each frame, the diffracted intensity of the chosen Bragg reflection is plotted against the diffraction angle (2θ), resulting in the desired rocking curve. The full width at half maximum (FWHM) of this curve can be used to derive information about sample quality, crystal mosaicity, and inherent instrumental broadening. This experiment can be conducted on any Bruker X-ray diffractometer and is described here for a dedicated single-crystal XRD (SC-XRD) setup.



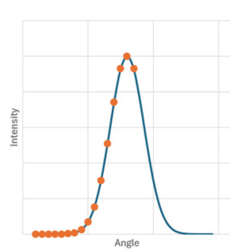


Figure 1
(top) Ewald representation of a rocking curve measurement on a SC-XRD diffractometer, (left): Intensity versus diffraction angle profile obtained in a rocking curve experiment.

Measurement and Evaluation

Collecting a rocking curve on an SC XRD setup is a simple process. The first step is to mount and center the crystal, then determine the unit cell. Identify a suitable Bragg reflection and obtain its corresponding goniometer angles from the image header. Enter these goniometer values with the PHI angle reduced by 0.5° and select a PHI scan from the operations table. Set the scan width to 0.03–0.05° and the scan sweep to 1°, ensuring the scan direction is positive. Choose an exposure time per frame that matches the crystal's diffraction strength. Click Execute to start the experiment, and the system will perform a final automated verification that the required scan ranges are physically feasible.

For proper evaluation, locate the frame with the highest visual intensity of the investigated reflection. Once this is done, mark the reflection with the rectangle tool, then select "Rocking Curve" from the mouse context menu. The rocking curve will be generated automatically and displayed in a separate window. The algorithm will take a default number of frames before and after the selected frame. You can modify the rocking curve frame interval as required via the slider at the bottom of the rocking curve window.

Instrumental broadening

In X-ray diffraction, instrumental broadening refers to the widening of diffraction peaks due to limitations or imperfections in the diffraction system. These imperfections can result from physical limitations, such as detector resolution or monochromator quality, or by design, as in the case of beam divergence in a dedicated single crystal diffraction system. "Instrumental broadening" excludes all impacts caused by the sample and is thus a fundamental parameter in XRD. It defines the best achievable angular resolution of a given system. Determining the instrumental broadening of a rocking curve experiment typically requires a sample with negligible intrinsic broadening. Repeating the experiment with different collimators, for example, is a powerful method for determining the beam divergence of a given setup.

Determination of the instrumental broadening of a D8 VENTURE

Instrumental broadening (beam divergence) was determined using the Si (111) reflection on a D8 VENTURE (Fig. 2) with a Mo IµS DIAMOND II and PHOTON IV. Three collimators were tested with a step size of 0.03° and an exposure time of 0.4 s per frame (Fig.3).

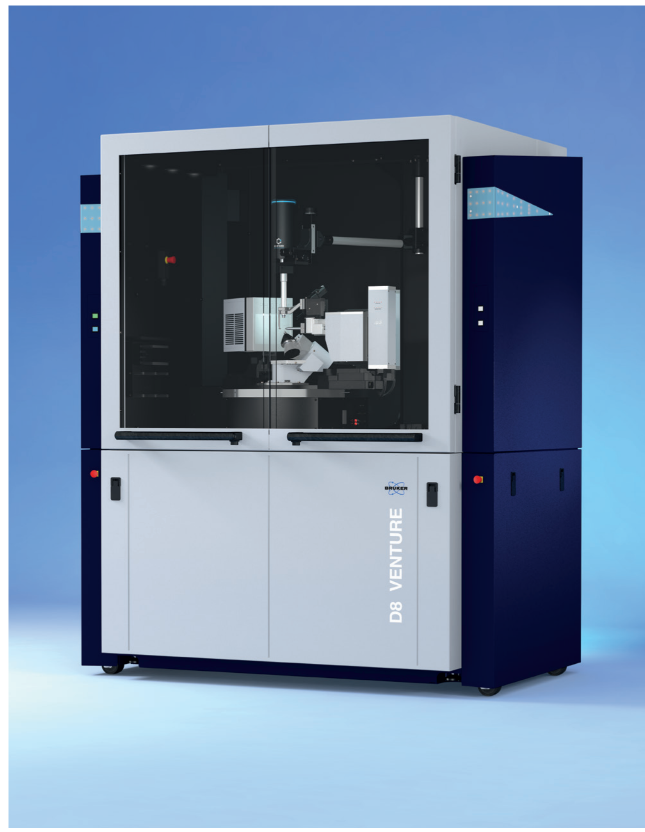


Figure 2
D8 VENTURE used for rocking curve measurement.

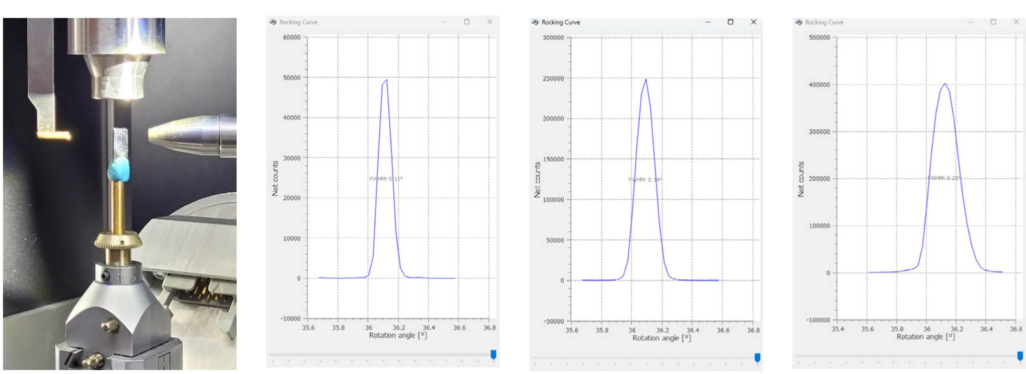


Figure 3
Silcon (Si) sample mounted on the D8 VENTURE (left); rocking curves of the Si (111) reflection using the 0.1mm, 0.2mm and 0.8mm collimator (next three to the right).

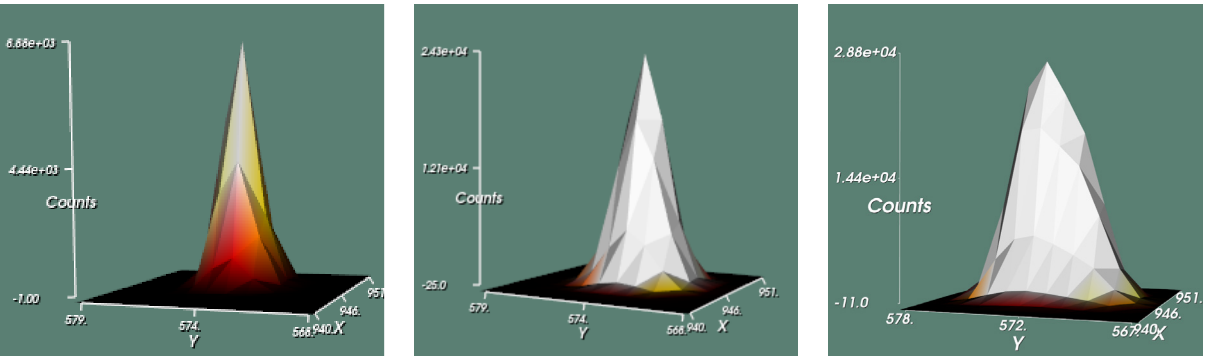


Figure 4
Intensity profile of the Si (111) reflection collected with the 0.1 mm, 0.2mm, and 0.8mm collimator

Collimator geometry	Measured [°]	Measured [mrad]	Nominal [mrad]
0.1mm 3mrad	0.11	1.8	1.5
0.2mm 6mrad	0.14	2.8	3.0
0.8mm	0.23	4.0	4.9

The results confirm the nominal divergence values within the usual experimental variations. For small deviations, the dominant effect is the actual alignment of the respective collimator.

Determination of the sample quality

In crystallography, "mosaicity" refers to small angular misorientations between different domains within a single crystal. Rather than being perfectly uniform, most real crystals consist of slightly misaligned blocks or mosaic domains. Low mosaicity indicates well-aligned crystal domains, which generally correlates with high crystal quality. Conversely, high mosaicity suggests significant internal disorder or strain, which can lead to broadened diffraction peaks, reduced resolution, and challenges in data interpretation.

APEX automatically calculates the average mosaicity during indexing and scaling. However, rocking curve measurements, as described above, provide a powerful tool for directly measuring the apparent mosaicity of a specific reflection in a single scan. For demonstration purposes, this was done on a $Cu(NC_5H_5)_2(C_7H_5O_3)_2$ crystal.

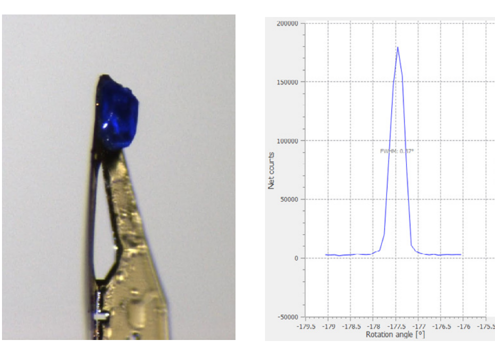


Figure 4
(left) $Cu(NC_5H_5)_2(C_7H_5O_3)_2$ crystal mounted on a loop, (right) automatically analysed rocking curve of the (5 0 -4) reflection.

Generally, a rocking curve analysis can be performed on any reflection. To obtain the true reflection characteristics and mitigate other effects, the reflection under investigation should be located in the equatorial plane and not on the rotation axis. In this case, the rocking curve of the (5 0 -4) reflection was analyzed, determining an FWHM of 0.37°. This value is in excellent agreement with an overall average mosaicity (RMS) of 0.83°, as calculated by APEX based on all harvested reflections. For Gaussian signals, the relationship between FWHM and RMS is defined as $FWHM = 2\sqrt{(2\ln 2)} \cdot RMS$, which is approximately $2.355 \cdot RMS = 0.87°$.

Conclusion

Rocking curve measurements can easily be performed on the D8 VENTURE. These measurements are a powerful tool for determining instrumental broadening, mainly the instrument's beam path characteristics, and sample properties. The APEX software suite provides all the necessary tools to perform these measurements conveniently, reliably, and quickly, as demonstrated by two experiments: the dependency of instrumental signal broadening on the collimator and the direct assessment of crystal mosaicity for a given sample.

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