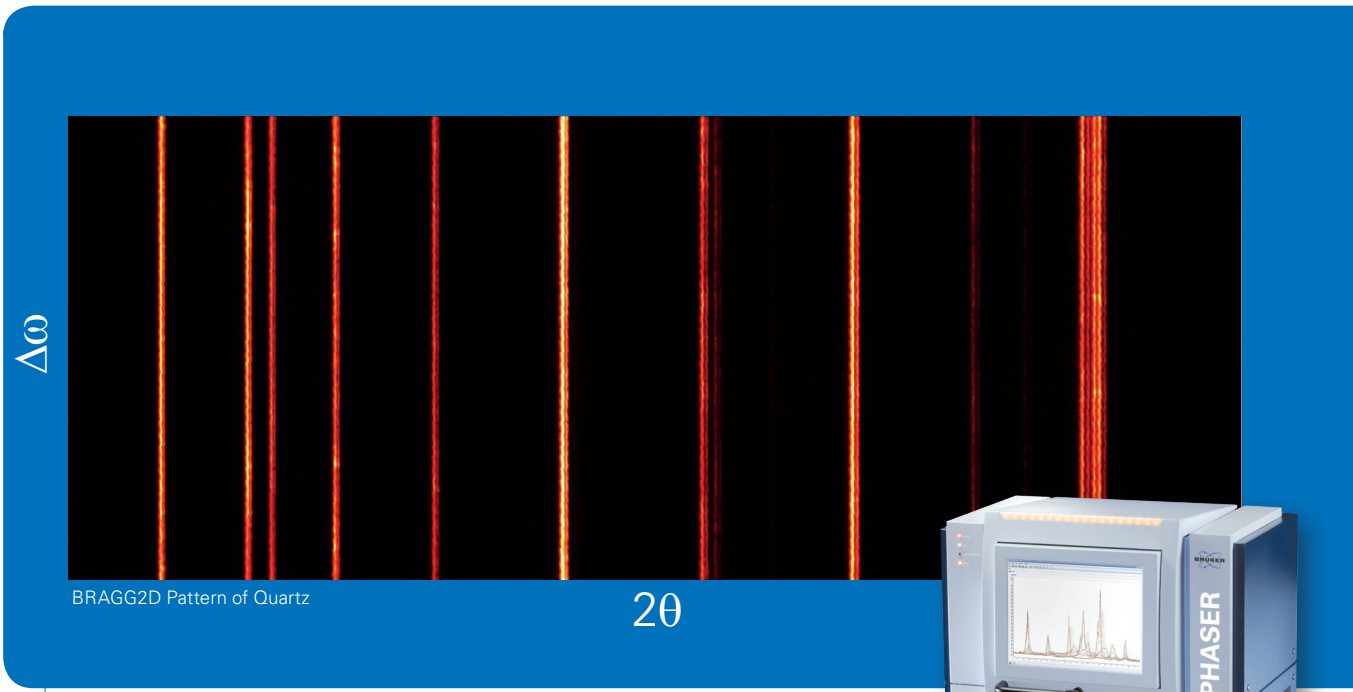


BRAGG2D Pattern of Quartz

Application Note XRD 615

BRAGG2D

- Two Dimensional Diffraction with the D2 PHASER

The D2 PHASER is a best-selling benchtop diffractometer featuring the most powerful detector line-up. In this application report we present the use of these advanced detectors in collecting two dimensional data sets for evaluating sample preparation effects including crystallite size and preferred orientation.

Detector	0D	1D	2D
Scintillation	✓		
SSD160	✓	✓	✓
LYNXEYE	✓	✓	✓
LYNXEYE XE-T	✓	✓	✓

Table 1. D2 PHASER Detection Modes

A common challenge encountered during powder X-ray diffraction experiments is ensuring the specimen consists of randomly oriented crystallites with appropriate dimensions. Two dimensional X-ray diffraction is a convenient way to visualize deviations from this ideal powder condition. In 2D data collection, diffracted intensity is collected as a function of 2θ and tilt with line uniformity at constant 2θ closely associated with the sample microstructure. BRAGG2D is a patent pending technique utilizing the parafocusing beam geometry with a new two dimensional data processing algorithm (available in EVA V5) allowing illumination of a large specimen area with the full X-ray beam resulting in rapid assessment of sample preparation.

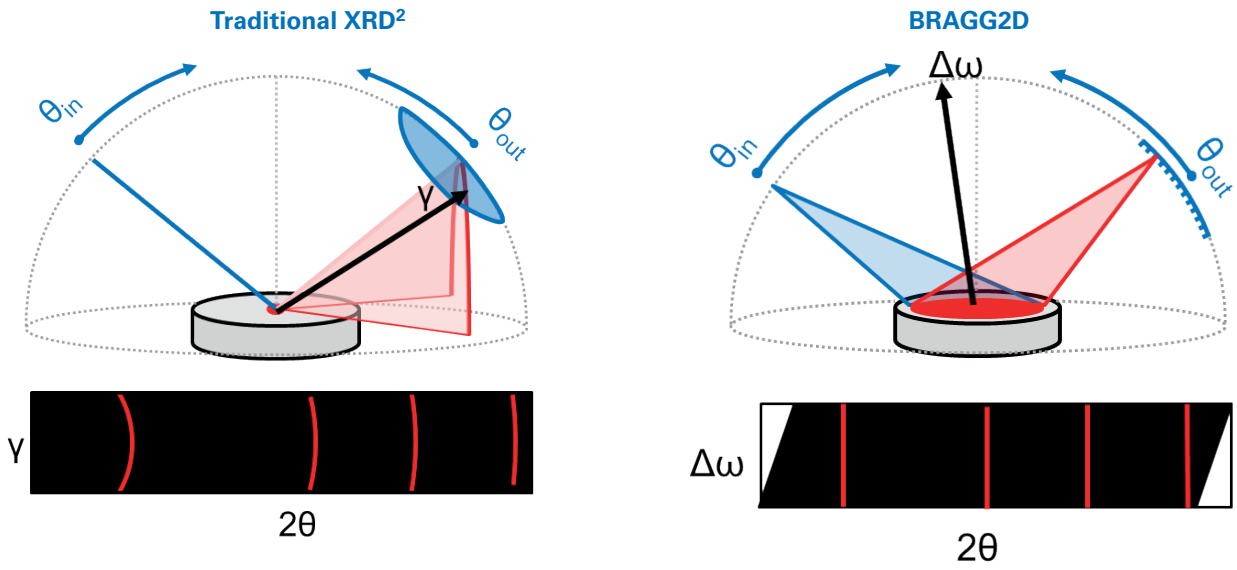


Figure 1. Geometry for Traditional XRD² (Left) and BRAGG2D (Right)

Perfect Powder

Large Crystallites

Preferred Orientation

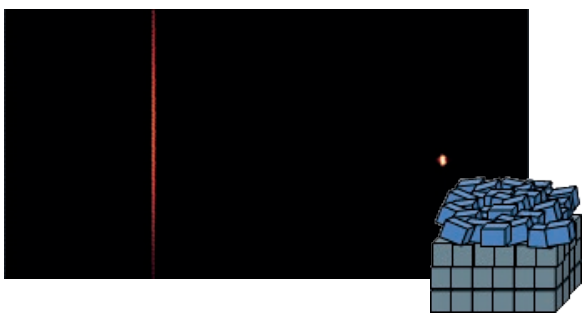


Figure 2. BRAGG2D measurements of various sample morphologies corundum (Top), table sugar (Middle) and oriented Cu film on Si wafer (Bottom)

What is BRAGG2D?

2D diffraction can be collected in two ways (Figure 1); Traditional XRD² and BRAGG2D.

Traditional XRD² utilizes a spot beam with tilt being recorded in a direction perpendicular to the goniometer plane. XRD² is often employed in floor-standing instruments with proper beam collimation, large area detectors and stages capable of complex motion for applications like texture, residual stress and high throughput screening. Due to the lack of beam conditioning leading to ring smearing and inability to control the angular and spatial position of the small beam, XRD2 is not recommended for compact diffractometer geometries where the primary application is phase identification and quantification.

BRAGG2D overcomes these issues by utilizing the large beam footprint of Bragg-Brentano combined with an asymmetric 1D detection geometry. Since the measured tilt is in the parafocusing plane, excellent resolution is achieved across the image. BRAGG2D utilizes the same line focus, divergence slits and axial sollers as a traditional 1D scan.

In both cases, Bragg2D and XRD², information about the morphological sample condition is collected.

For benchtop tools, BRAGG2D is preferred as it achieves maximum signal and resolution while utilizing the same beam condition used for subsequent phase identification and quantification.

The images in Figure 2 illustrate the challenges faced during sample preparation. A fine, randomly oriented crystallite structure results in smooth lines (top), a sample with large, randomly oriented crystallites will show random spots along the 2 Theta line (middle) and a fine grain oriented film on a single crystal substrate will show a broad streak (bottom).

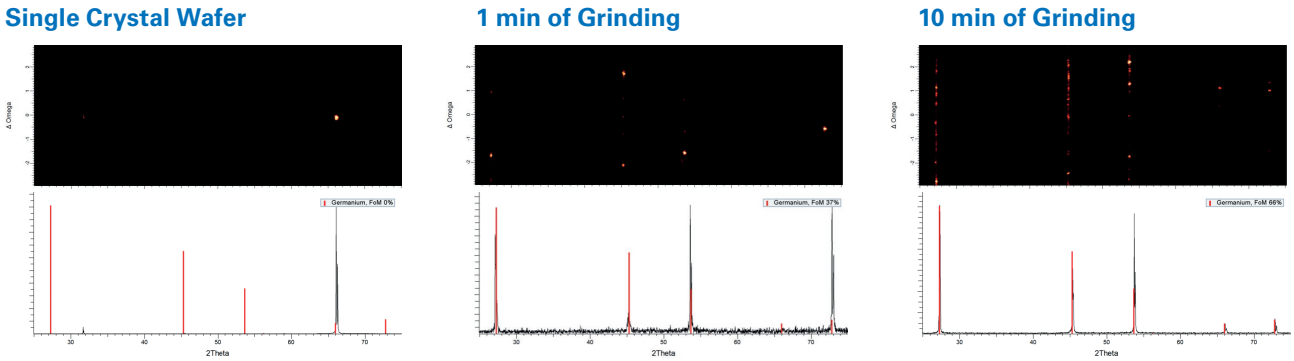


Figure 3. BRAGG2D and 1D scans of a single crystal wafer as it is ground to a fine powder. Search/Match of the 1D scan shows significant increase in FoM to the Ge powder pattern (FoM equal to 0%, 37% and 66% respectively) as line uniformity increases.

BRAGG2D Examples

Sample Preparation Assessment

BRAGG2D provides visualization of sample preparation quality. Adjustments such as additional grinding or back loading can be performed to increase success in phase identification and quantification. Figure 3 shows a single crystal wafer being ground into a fine powder. As grinding proceeds, the spot pattern converts to uniform lines. The Figure of Merit (FoM) for search/match to Ge strongly correlates with line uniformity.

Enhanced Phase Identification

The line morphology observed in BRAGG2D can be used to correlate lines originating from the same phase. Figure 4 shows phase identification of a geological specimen with the smooth lines associated with calcite and rough lines with quartz.

Increased Quantification Reliability

Classic quantification techniques such as Pattern Fit S-Q and Rietveld assume the sample consists of a randomly oriented finely sized crystallites. Deviations from this condition can be taken into account to reduce error between the measured data and fit, but this does not reduce the uncertainty to the actual phase quantity. Figure 5 shows BRAGG2D and semi-quant pattern fits of a nominal 50 wt% ibuprofen and sucrose mixture before and after grinding. The as received mixture shows strong spot reflections from large sucrose crystallites leading to a poor pattern fit (R_{WP} 85%) and overestimation of the wt% ibuprofen. After grinding, a clear line pattern is observed and an improved fit achieved (R_{WP} 24%) with semi-quant result of 46 wt% ibuprofen closer to the anticipated nominal value.

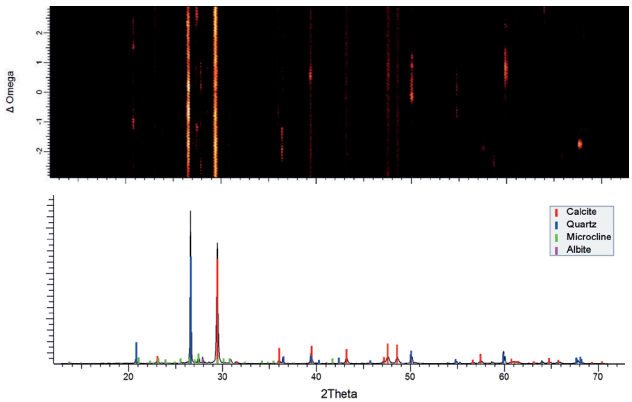


Figure 4. BRAGG2D and 1D scan of a geological specimen. Quartz matches the rough lines while calcite matches the smooth lines.

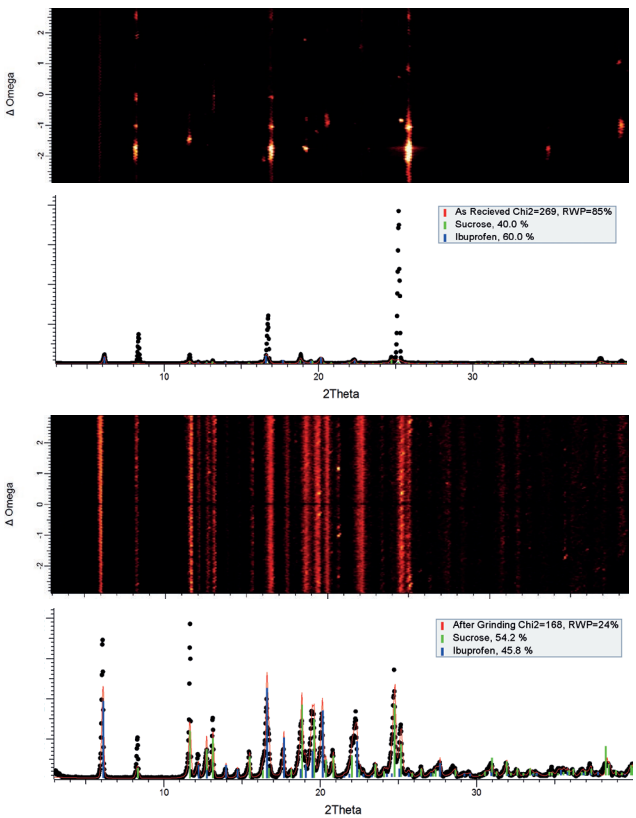
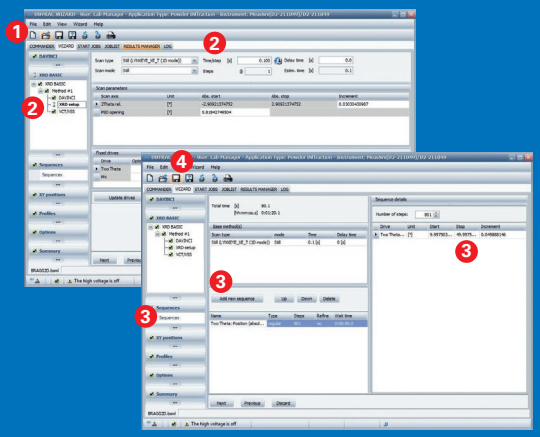


Figure 5. BRAGG2D and 1D data with S-Q fit of as received (top) and ground (bottom) mixture..

DIFFRAC.SUITE Workflow for BRAGG2D

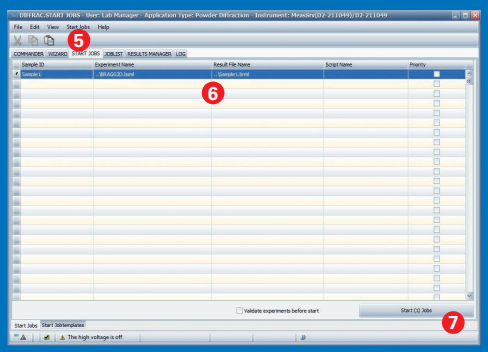
PLAN in DIFFRAC.WIZARD

1. Create a new XRD method
2. Under XRD setup choose the scan type Still (LYNXEYE 1D Mode)
3. Under Sequences insert a Two Theta sequence with start, stop and increment of interest
4. Save the method



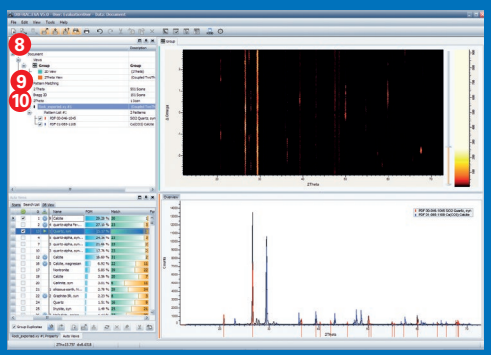
MEASURE in DIFFRAC.COMMANDER

5. Click on the START JOBS Tab
6. Choose the method created in step 4 and set the desired result file location
7. Click Start and monitor progress in COMMANDER



ANALYZE in DIFFRAC.EVA V5

8. Open DIFFRAC.EVA and import the result file
9. Right click on the 2Theta node and choose Tool → BRAGG2D View Realignment
10. Right click on the BRAGG2D node and choose Create → 2D View



Bruker AXS GmbH
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/xrddetectors

www.bruker.com