



## Application Note XRD 602

# D8 DISCOVER with $I\mu S^{\text{High Brilliance}}$ Microfocus Source Application: Powder Diffraction

### Introduction

The D8 DISCOVER combined with the INCOATEC  $I\mu S^{\text{High Brilliance}}$  Microfocus source is an innovative x-ray diffraction (XRD) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a high resolution powder diffraction configuration to analyze powders or polycrystalline materials in reflection geometry for applications such as phase identification (Phase ID) and quantitative Rietveld<sup>1</sup> analysis.

In this report, the resolution and signal to background obtained on the SRM 1976b standard corundum sample will be shown along with the results from several real world experiments including the quantification of retained austenite, measurement of common pharmaceutical materials and the Rietveld refinement of a geological sample.

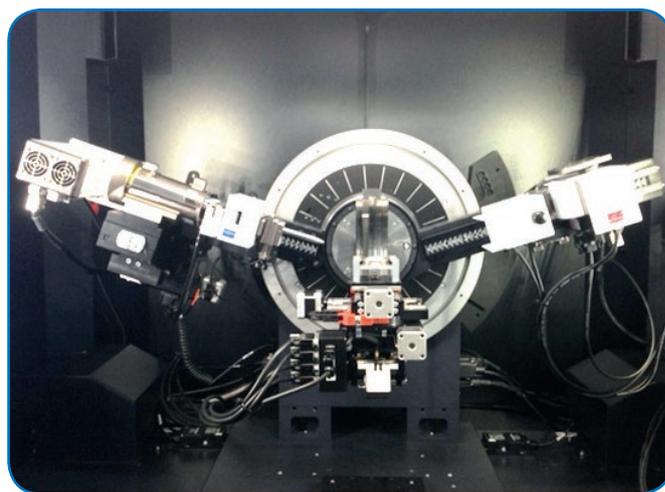


Figure 1: D8 DISCOVER with  $I\mu S^{\text{High Brilliance}}$  and LYNXEYE XE

## Powder Diffraction

The configuration used for powder diffraction is shown in Figure 1 and detailed in Table 1. The  $I\mu S^{\text{High Brilliance}}$  and integrated MONTEL-P optic produces a very brilliant primary beam with 1 mm diameter spot size. By utilizing a linear slit in the primary beam path, the beam can be optimized for maximum resolution (0.2 mm slit) or maximum flux on the sample (1 mm slit). For the smallest possible probe sizes, the collimator can also be exchanged, resulting in a minimum probe size of 50 microns. To improve particle statistics, larger samples can be “rastered” during measurement using the motorized XY translation of the Centric Eulerian Cradle (CEC) stage so that diffraction patterns are more representative of the phases that are present. Psi (tilt) and Phi (rotation) of the CEC stage can be combined with XY raster motion to present all crystal orientations to the beam and mitigate the effects of preferred orientation to produce a randomized powder pattern from samples of various sizes and morphologies. This method, similar to the classic Gandalfi camera<sup>2</sup> configuration, is not possible with line focused beams due to defocusing effects when the sample is tilted in Ps.

Source	$I\mu S^{\text{High Brilliance}}$ Microfocus (Cu)
Optics	Montel-P
Divergence slit	0.1 – 1.0 mm
Stage	Centric Eulerian Cradle (CEC)
Detector	LYNXEYE XE

Table 1: Typical powder instrument setup for the D8 DISCOVER with  $I\mu S^{\text{High Brilliance}}$

## Measurement of NIST SRM 1976b

The NIST standard reference material 1976b (NIST SRM 1976b) consists of a sintered plate of corundum. Figure 2 shows a coupled scan of SRM 1976b, measured from  $20^\circ$  to  $90^\circ$   $2\theta$ , with step size of  $0.01^\circ$  and time of 1 second per step resulting in a total scan time of 2 hours. The instrument resolution (defined by the minimum Full Width at Half Maximum, FWHM) is less than  $0.04^\circ$   $2\theta$ , as calculated from the (104) reflection. It is important to note that the peaks at low and high angle show no signs of asymmetric broadening due to axial divergence even though axial soller slits are not present on the primary or secondary side. This is due to the strong axial collimation produced by the integrated MONTEL-P optic. Peak to Background is also an important parameter as this determines sensitivity to weak peaks and trace phases. For the (104) reflection, the Peak to Background is found to be  $\sim 300$ . With this level of Peak to Background, reflections with small relative intensities, such as (211) with a rating of  $<1\%$  intensity in the powder diffraction database, are clearly differentiated from the background.

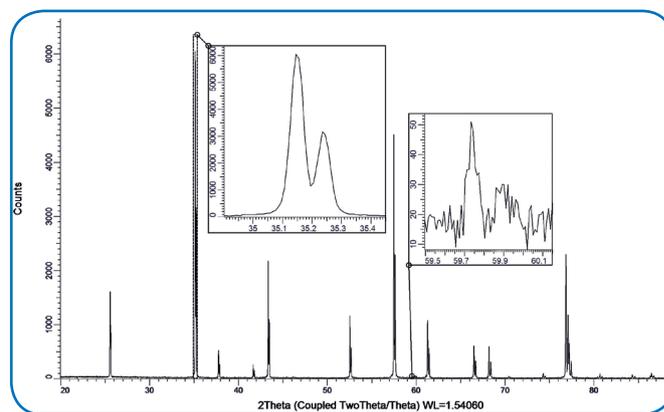


Figure 2: Measurement of NIST 1976b with the D8 DISCOVER with  $I\mu S^{\text{High Brilliance}}$  and LYNXEYE XE. The (104) reflection at  $35^\circ$   $2\theta$  has FWHM less than  $0.04^\circ$   $2\theta$ , while the  $<1\%$  relative intensity (211) reflection at  $60^\circ$   $2\theta$  demonstrates the exceptional Peak to Background.

## Measurement of Retained Austenite

In the metals industry, a common powder diffraction method is the quantification of retained austenite in steel. Austenite is a high temperature phase, which due to the kinetic limitations of processing can become trapped in the material. If the amount of retained austenite is too high, dimensional instability results due to the conversion of austenite to ferrite over time. Figure 3a shows a scan of a steel specimen containing ~5% retained austenite. Iron fluorescence from the Cu radiation is completely eliminated by the enhanced energy discrimination of the LYNXEYE XE detector resulting in very low background. The probe size used for this measurement was 0.5 mm by 1 mm enabling mapping of retained austenite over the surface. Alternatively, the sample can be moved in a raster motion with the CEC, resulting in a measurement of the global retained austenite content. Figure 3b shows quantitative Rietveld refinement performed in DIFFRACTOPAS of the austenite content.

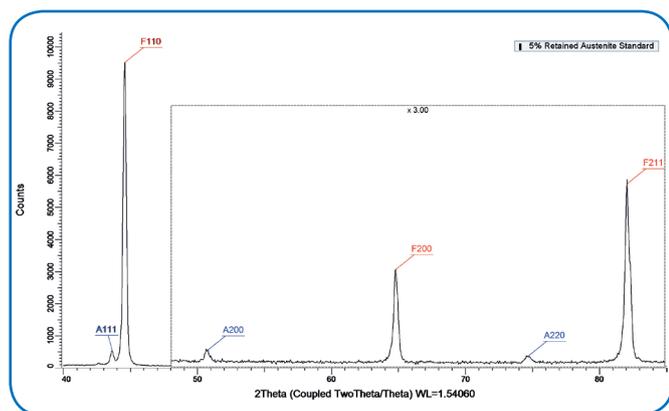


Figure 3a: Measurement of a steel specimen for retained austenite quantification with the D8 DISCOVER with  $I\mu S^{\text{High Brilliance}}$  and LYNXEYE XE. Background subtraction has not been performed.

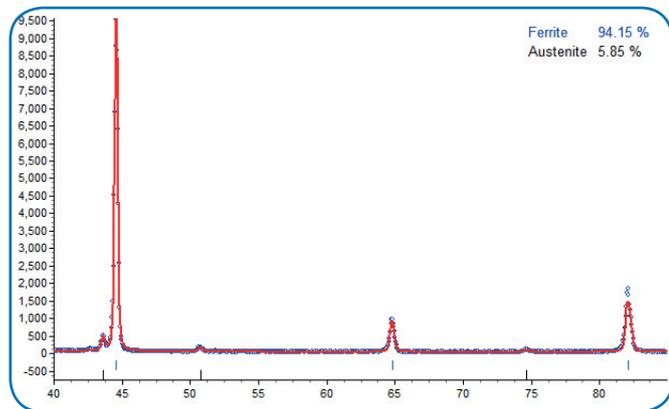


Figure 3b: Rietveld refinement of the retained austenite content using DIFFRACTOPAS

## Measurement of Pharmaceutical Materials

In the pharmaceutical industry, powder diffraction is commonly used for polymorph analysis to ensure phase purity in active ingredients. Since the unit cell of an organic pharmaceutical is very large, the majority of peaks are found at low angle with typical scan ranges spanning  $3^\circ$  to  $40^\circ$   $2\theta$ . Figure 4 shows scans of 1 milligram samples of 3 common pharmaceutical materials; Ibuprofen, Sucrose and Acetaminophen. Due to the beams small size and low divergence, the background stays very low as the scan approaches  $0^\circ$   $2\theta$ , giving the ability to identify phases at very low angles.

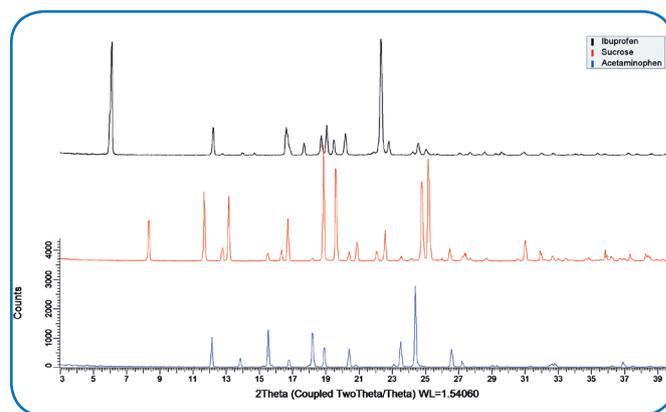


Figure 4: Measurement of various pharmaceutical materials with the D8 DISCOVER with  $I\mu S^{\text{High Brilliance}}$  and LYNXEYE XE.

## Measurement of Geological Materials

Powder diffraction can be used for quantitative mineralogy of geological samples. These samples often contain iron and multiple crystallographic phases so fluorescence elimination and good instrument resolution are necessary to achieve reliable results. Figure 5a shows the phase identification in DIFFRAC.EVA of 1 mg of a standard geological sample. Peaks from each phase are narrow and well separated indicating good instrument resolution and the background is very low even with the presence of a Pyrite phase. The small sample amount couples very well to the beam size of the  $\mu\text{S}^{\text{High Brilliance}}$  resulting in data that is comparable to results from a larger sample run in a conventional divergent beam geometry. Figure 5b shows the quantitative Rietveld fit of the data in DIFFRAC.TOPAS. Due to the extremely low axial divergence of the primary beam produced by the MONTEL-P optic, the instrument function is free from aberrations such as axial divergence that can result in asymmetric peaks. This reduces the number of parameters that must be refined to account for the peak shapes, dramatically simplifying the quantitative fitting process.

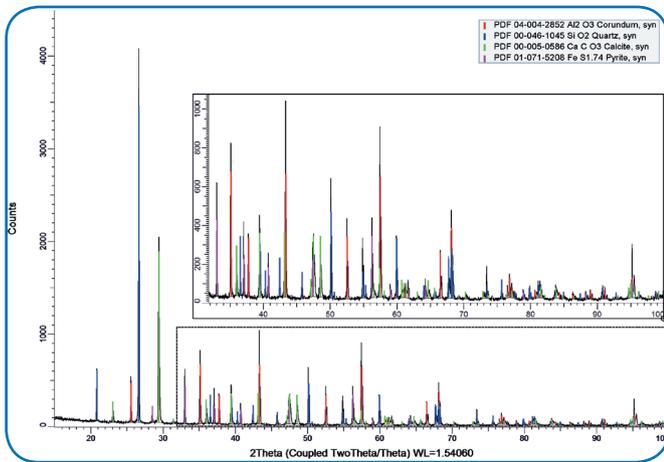


Figure 5a: Measurement of a geological sample with the D8 DISCOVER with  $\mu\text{S}^{\text{High Brilliance}}$  Microfocus source and LYNXEYE XE. Phase identification was performed in DIFFRAC.EVA

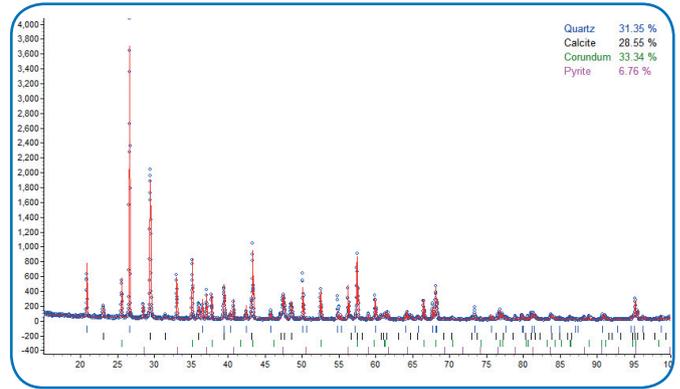


Figure 5b: Rietveld Refinement of the geological sample using DIFFRAC.TOPAS

## Conclusion

Powder samples from a variety of industrial applications were successfully measured with the D8 DISCOVER with  $\mu\text{S}^{\text{High Brilliance}}$  and LYNXEYE XE detector.

The  $\mu\text{S}^{\text{High Brilliance}}$  combined with MONTEL-P optic creates a beam with extremely high brilliance and low divergence, resulting in an ideal configuration for measuring samples as small as 1 milligram. The Centric Eulerian Cradle stage gives the ability to raster the small probe over large areas in both X and Y to increase particle statistics and to remove preferred orientation effects through sample rotation and tilt. Using the LYNXEYE XE position sensitive detector, with 192 channels and enhanced electronic energy discrimination, results in scans with exceptional peak to background and signal to noise. The D8 DISCOVER with  $\mu\text{S}^{\text{High Brilliance}}$  and LYNXEYE XE detector allows the use of conventional powder diffraction analysis without the limitations of large area conventional powder diffraction measurement geometries.

## References

1. Rietveld, H.M. (1967): Line profiles of neutron powder-diffraction peaks for structure refinement. - Acta Cryst., **22**, 151-152.
2. Gandolfi G. Miner. Petrogra. Acta (1967). 67-74

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