Lab Report XRD 83

2D-Mode for LYNXEYE XE

Introduction

A position sensitive X-Ray diffraction (XRD) detector can be used to collect powder data very quickly by distributing its detection elements/strips along the goniometer scattering plane and scanning it through the measurement range. The LYNXEYE XE Energy-Dispersive 0D/1D/2D-mode detector is the latest generation compound silicon strip detector that can be used for this purpose. In addition to high speed data acquisition, it also features unmatched energy discrimination that results in high peak to background without the need for filters or monochromators.

In addition to scanning 1D mode, the detection elements or strips of this detector can be set to represent the same 2theta position; thereby allowing the detector to be used in "0D" mode for high-resolution parallel-beam geometries. Further, Bruker's patented 0 / 90 degree mount allows the detector to be quickly re-oriented with the strips perpendicular to the scattering plane (Figure 1). In this orientation, the scattering angle is determined by a physical slit in front of the detector and the intensity can be distributed along all of the detector strips allowing for a very high dynamic range 0D mode.

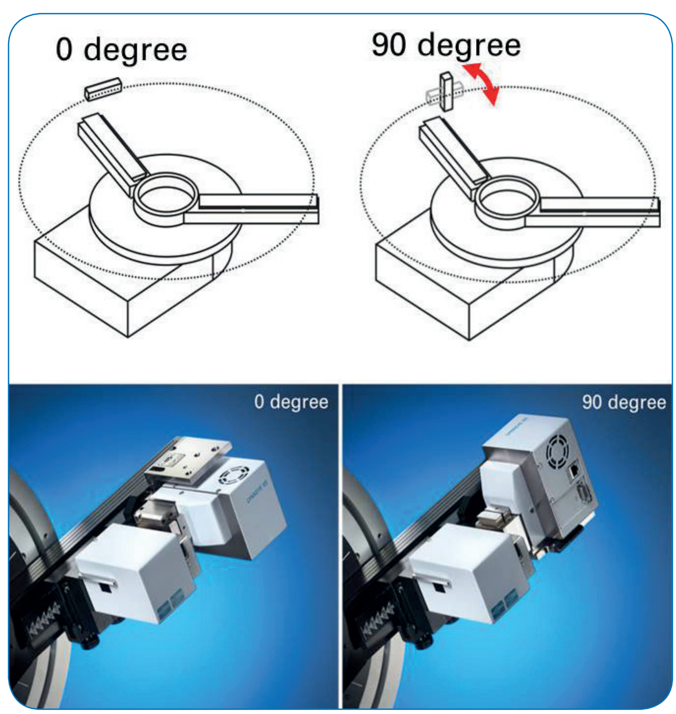

Figure 1: LYNXEYE XE in 0 degree and 90 degree orientations. The relationship of active area to scanning direction and scattering plane is shown.

However, if the LYNXEYE XE strips are set to retain their position sensitivity in this 90 degree orientation, a 2D scattering pattern can be constructed by scanning the detector along the 2θ direction. This new, 2D-mode of operation, supported by DIFFRAC.MEASUREMENT and DIFFRAC.EVA, allows access to scattering information not available to other 0D/1D detectors.

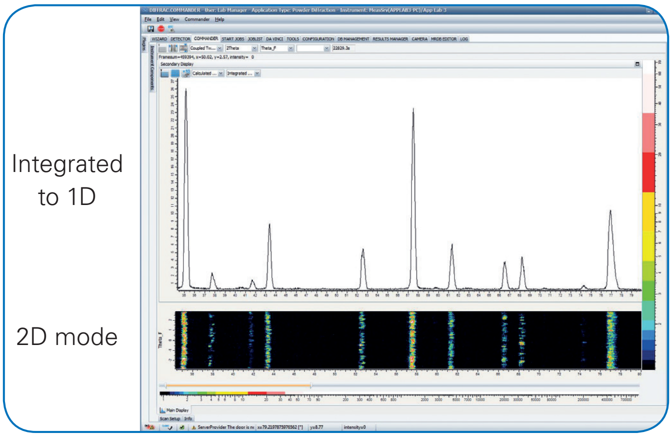

Figure 2: LYNXEYE XE 2D-mode data collection in DIFFRAC.MEASUREMENT. 2D data is integrated to 1D in real-time.

Scattering maxima from polycrystalline samples occurs at specific diffraction angles as defined by Bragg's Law. In 2D scattering space, the scattering maxima appear as Debye cone's which intersect a traditional 2D detector to form rings (Figure 3). The scattering intensity along the rings, referred to as the gamma (γ) direction, contains additional information about the sample including stress, texture, particle size, and phase type (single crystal, few crystal or polycrystalline).

Application Example: Phase ID using 2D-mode

A spot of interest on a rock obtained from a mine in Wausau, WI was analyzed using 2D mode. The results are shown in Figure 5. By inspecting the gamma profile of the 2D data, it is clear that the sample is made up of at least 2 crystalline phases; one that is fine grained and textured (blue arrows) as indicated by the continuous, but non-uniform, intensity distribution along gamma and one that is large grained (red arrows) as shown by the spotty and non-uniform intensity along gamma. This information allows peaks to be tentatively assigned to different phases before 1D search/match even begins. This simplifies and accelerates the search/match process as well as providing a clear explanation for the relative intensity deviations between raw data and reference patterns.

Figure 4: As measured rock from a mine in Wausau, WI

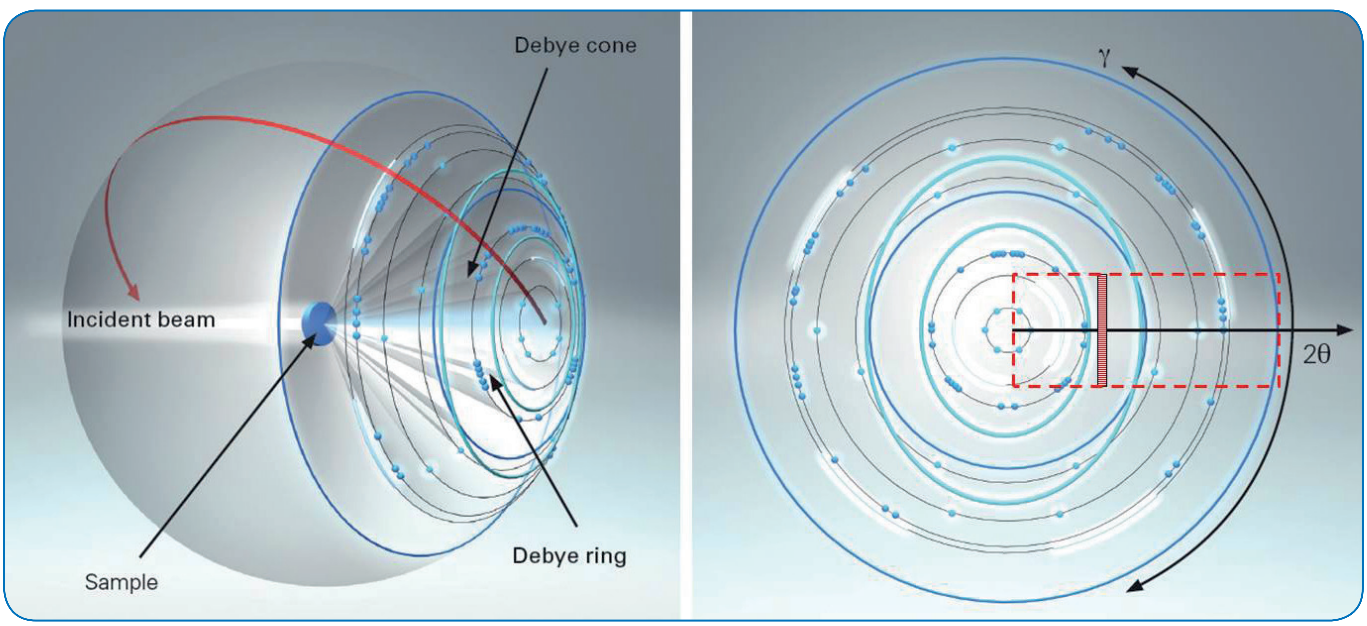

Figure 3: (Left) Illustration of 2D diffraction geometry, here shown in transmission geometry.
(Right) Examples of Debye rings exhibiting intensity distributions due to stress, texture, particle size, and number of particles. The red outline represents the region that can be scanned and displayed in 2D-mode.

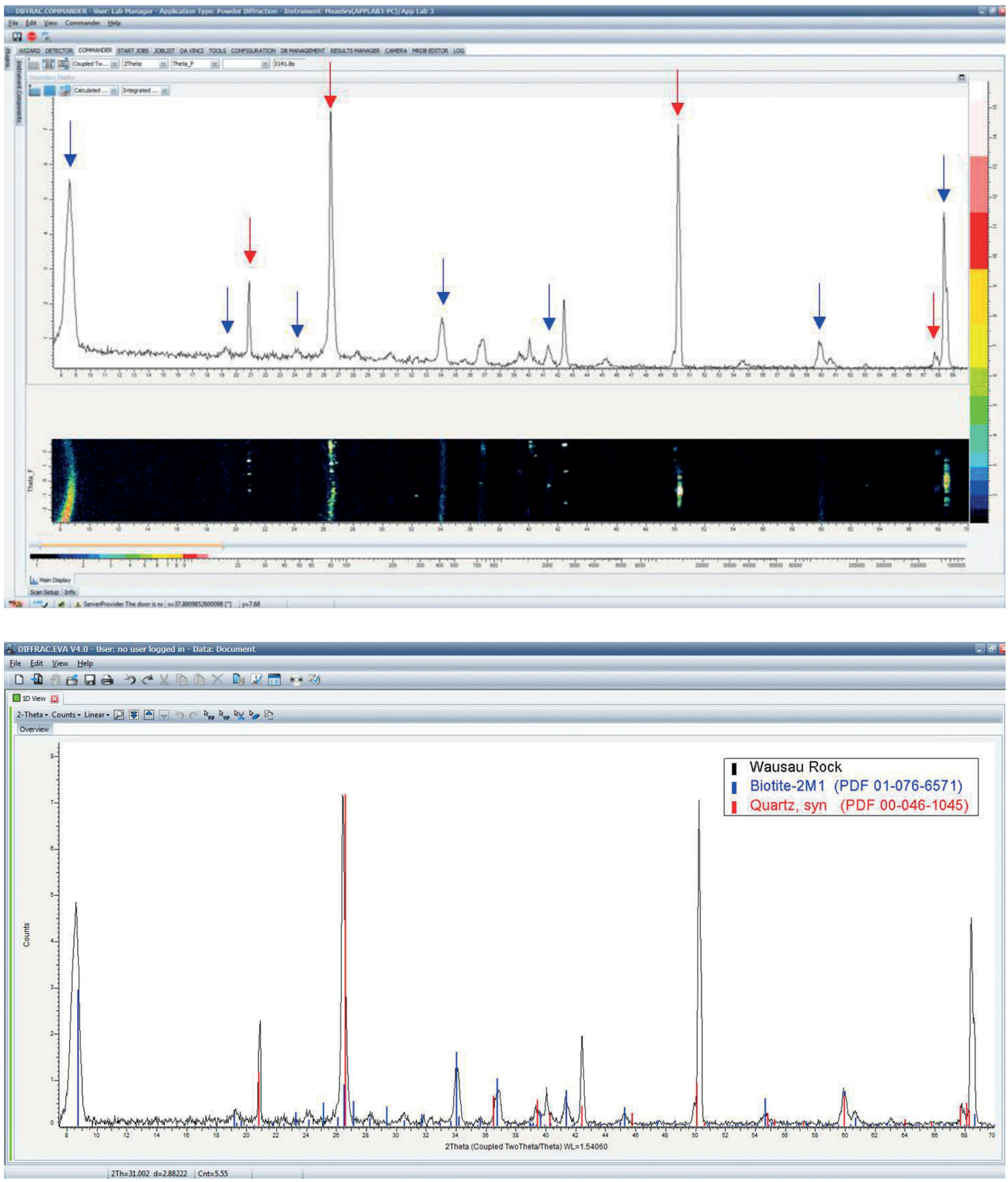

Figure 4: (Top) 2D-mode scan and real-time 1D integration of Wausau, WI mine rock. Based on gamma scattering profile, the peaks are grouped into phase 1 (red arrows) and phase 2 (blue arrows).
(Bottom) DIFFRAC.EVA Search/Match results in phase identification of Quartz for phase 1 and Biotite for phase 2. Relative intensity differences can be explained by referencing the 2D data

An important advantage of using the LYNXEYE XE for this application is the superb energy resolution well beyond the capabilities of traditional 2D detectors that allows collection of 2D images without sample fluorescence that can elevate background and restrict trace phase sensitivity (Figure 6).

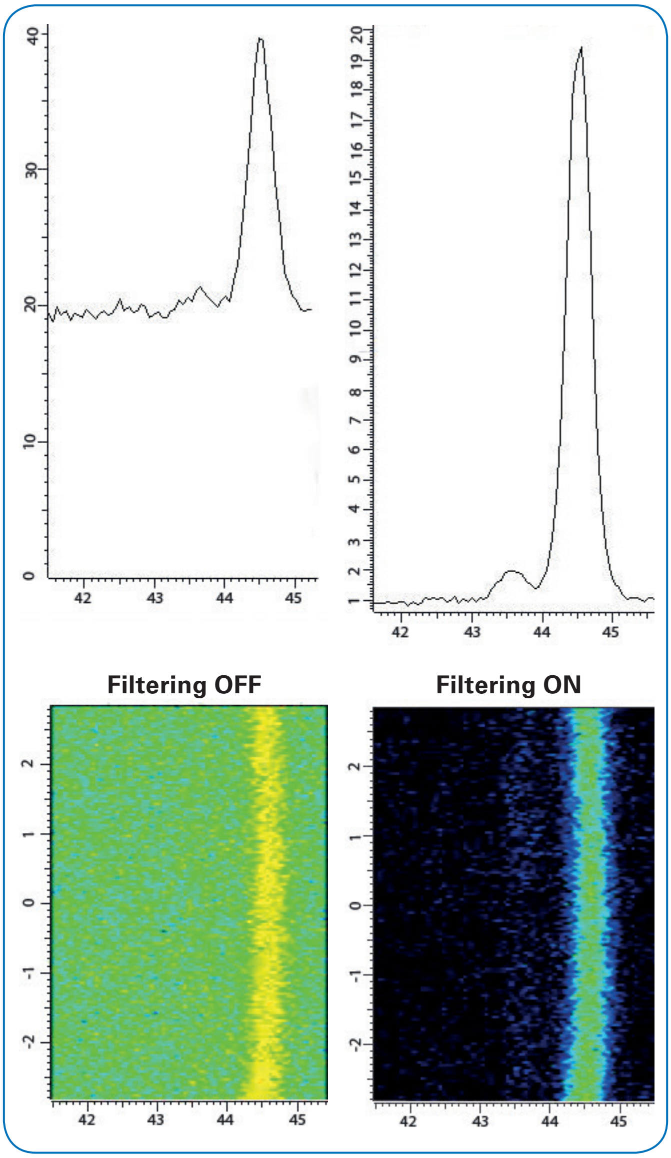

Figure 6: Fluorescence Filtering. 2D-mode scan (bottom) with resulting integrated 1D data (top) of Iron sample with Copper Kα primary radiation. Fluorescence filtering set of OFF to simulate a traditonal 2D detector and ON showing results from LYNXEYE XE stadard setting.

Conclusions

Thanks to a 0/90 degree detector mounting system combined with an innovative scan type, the LYNXEYE XE, energy discriminating detector, is capable of a new and powerful mode of operation: 2D-mode. In this mode, 2D scattering images can be produced which reveal information about the sample not available to standard 0D/1D detectors.

Advantages of using LYNXEYE XE in 2D-mode include:

- Eliminates defocusing by using symmetrical scanning mode
- Anti-scatter slits reduce background by blocking air-scatter
- Adjustable resolution via slit and scan settings
- Sample fluorescence removed via superior energy discrimination
- No compromise 0D/1D performance with same detector
- Variable positioning to optimize 2θ and gamma
- Operation with all common wavelengths, including hard radiation

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Patent

LYNXEYE turned 90°: EP 1 647 840 A2 patent;
EP 1 510 811 B1 patent

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