



D8 ADVANCE Plus

● Maximum flexibility meets unparalleled ease-of-use

The D8 ADVANCE Plus is the latest extension to the well-established D8 ADVANCE Multipurpose Solutions family.

The system is capable of automated switching between 6 beam path geometries from focusing Bragg-Brentano for powders to high-resolution parallel-beam $K\alpha_1$ geometry for epitaxial thin films and everything in-between.

It is therefore perfectly suited for all sample types including powder, bulk, fiber, sheet and thin-film (amorphous, polycrystalline and epitaxial) with investigations under ambient or non-ambient conditions.

Optimum resolution and system scattering geometry for any application is available with the push of a button, reliably and without the need for user participation in alignment or handling of expensive components.

The consistent implementation of DAVINCI design guarantees unlimited extension of capabilities to match all analytical needs – now and in the future.

Features

- Push-button configuration change using SmartCalib intelligence
- Eulerian cradle for powder, bulk, and thin-film applications
- Fast 0D/1D/2D-mode detector with sample fluorescence and $K\beta$ radiation rejection
- Real-time detection and configuration of all stages and components

Key Components

TRIO



**Motorized Slits
Focused Beam**



**Göbel Mirror
Parallel Beam**



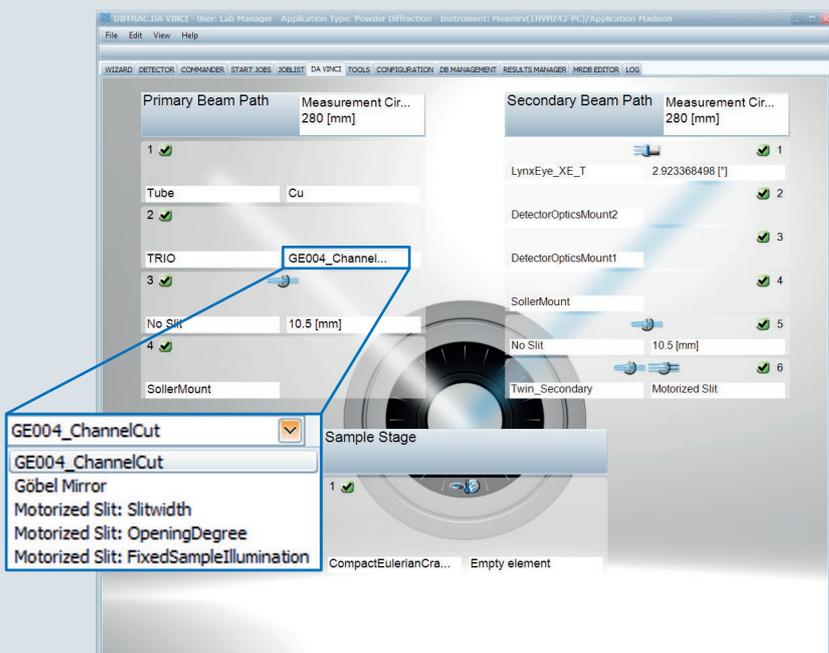
**Göbel Mirror +
monochromator
Highly Parallel K α 1 Beam**

The newly released TRIO optic uniquely enables push-button, automated switching between the three most commonly used primary beam geometries:

1. **Motorized slits** for a focused beam geometry used for conventional Bragg-Brentano powder diffraction
2. **Göbel Mirror** for high intensity Cu-K α parallel beam for capillary experiments, height insensitive measurements, surface sensitive grazing incidence geometry, coating thickness determination and micro-diffraction
3. **Göbel Mirror + 2-Bounce Ge Channel-cut monochromator** for highly parallel Cu-K α ₁ beam for high-resolution diffraction (HRXRD) of epitaxial thin films and pure K α ₁ diffraction patterns of powder samples

SmartCalib then takes care of any required system changes between these geometries without the need for any manual user intervention or special alignment.

The perfect match for the TRIO is the secondary side TWIN optic which has similar motorized switching between variable slits and a parallel beam Soller collimator. The TRIO / TWIN combination gives access to 6 configurations to match beam conditioning, scattering geometry, and resolution to sample requirements. Further, due to the compact design of the TRIO and TWIN modules, the system measurement diameter can be minimized, which leads to intensity gains for all scattering geometries; particularly Bragg-Brentano. Thanks to this innovative configuration, even a novice user has quick and easy access to the full range of X-ray diffraction and scattering techniques.



Changing the scattering geometry is as easy as selecting the desired beampath from the TRIO drop-down menu. SmartCalib does the rest.

LYNXEYE XE-T 0D/1D/2D Detector

The LYNXEYE XE-T is a next generation compound silicon strip detector with superior energy resolution for ultrafast 0D, 1D and 2D X-ray diffraction. A key feature is its unprecedented energy resolution (< 380 eV for Cu radiation) which is 4x better than any other 1D or 2D detectors on the market. The benefit is that it allows the LYNXEYE XE-T to electronically discriminate unwanted radiation without $K\beta$ filters or secondary monochromators. This results in faster data collection, higher sensitivity, and no need for changing hardware in the case of fluorescing samples. The LYNXEYE XE-T supports multiple modes of operation that allow it to be used for many beam configurations and applications.

Modes of operation:

- Scanning 1-D mode for fast data collection
- Fixed 1-D mode measurements for ultra-fast measurements
- 0-D ("point detector") mode for high-resolution parallel-beam geometry
- 0-D mode and turned by 90° to cover an extremely large dynamic range
- 2D mode – turned 90° and scanned in 2θ to create 2D scattering images

These features make the LYNXEYE XE-T the perfect detector for the D8 ADVANCE Plus, delivering best data quality, versatile modes of operation, and minimal reconfiguration effort to support a wide range of measurements.

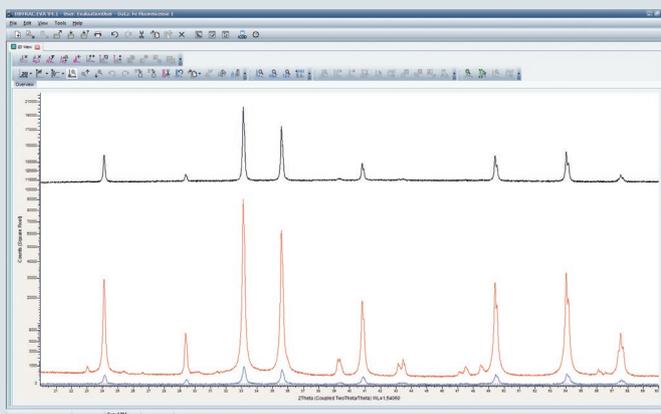


Figure 1: Unfiltered (black line) and filtered (red line) data demonstrating the superb filtering of fluorescence radiation by the LYNXEYE XE-T. The intensity gain over the secondary monochromator data (blue line) amounts to a factor of about 450.

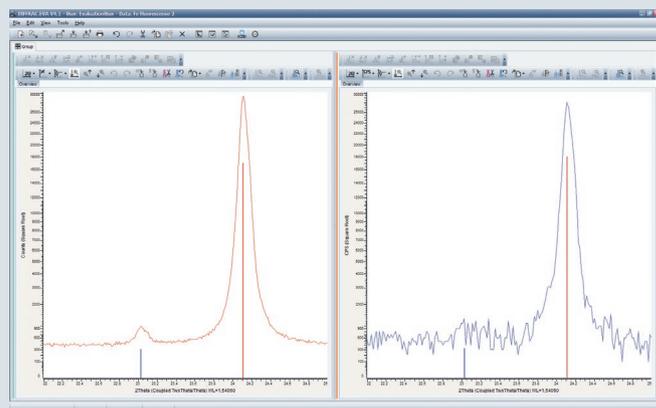
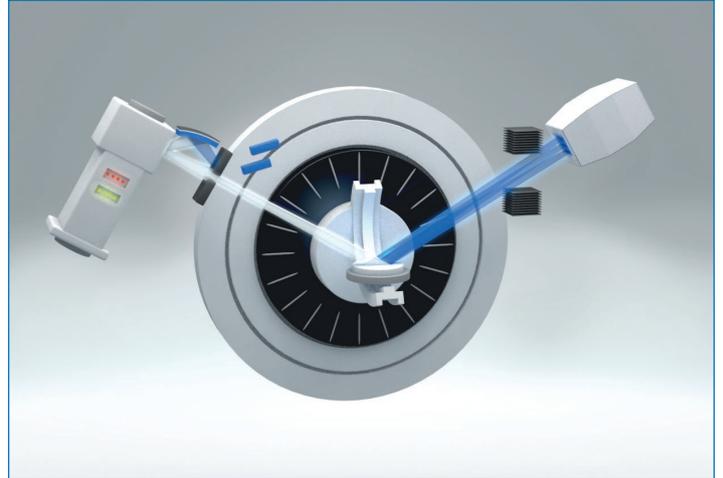


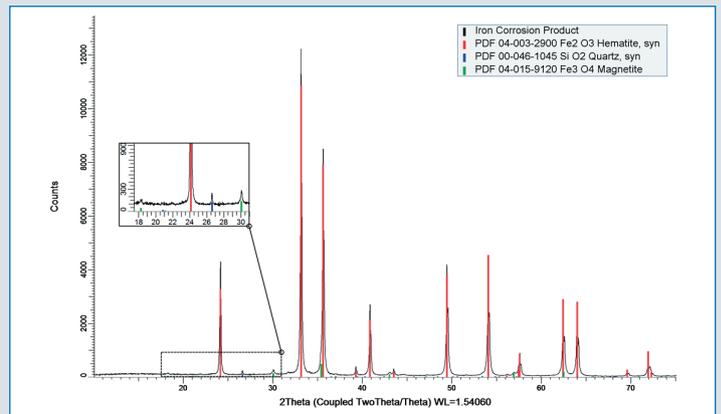
Figure 2: Zoomed region from Fig. 1 ($21^\circ - 27^\circ 2\theta$) illustrating the unparalleled lower limits of detection capabilities of the LYNXEYE XE-T. Secondary monochromator data (blue line) scaled to the same maximum peak intensity as the LYNXEYE XE-T data (red line). Calcite (blue stick) is clearly below the detection limit for the secondary monochromator data.

Bragg-Brentano

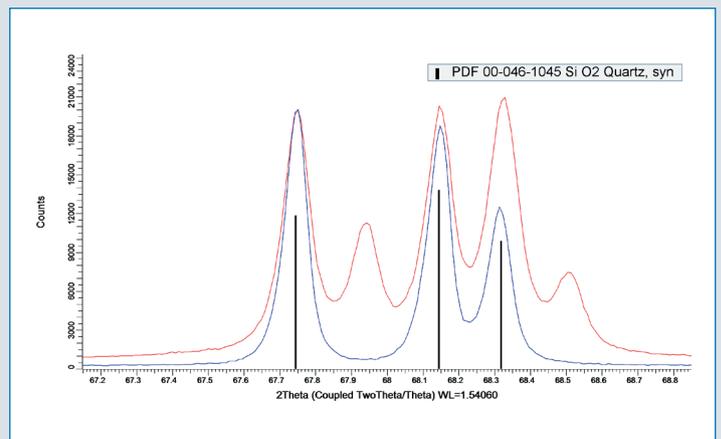
Conventional Bragg-Brentano powder diffraction geometry with TRIO (slit) and TWIN (slit) results in highest intensity and best peak resolution for flat powder samples due to parafocusing geometry. Sensitivity is maximized thanks to the LYNXEYE XE-T's ability to collect data in full 1D mode while electronically discriminating sample fluorescence and Cu K β .



This example shows phase analysis of a corrosion product powder sample. Despite the high iron content, which would typically result in a strong fluorescent background, the LYNXEYE XE-T is able to fully eliminate this unwanted radiation resulting in very low background and high signal to noise ratio. This enables the detection of all phases, including trace phases of magnetite and quartz.

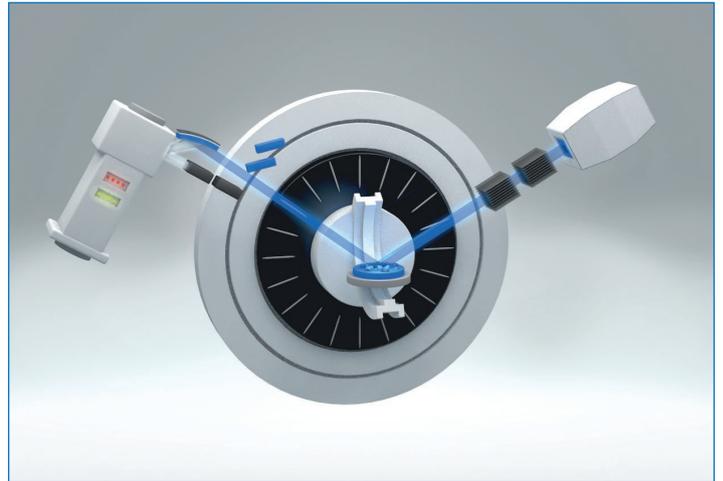


Standard Bragg-Brentano data contain the K α 1 and K α 2 wavelengths that may be resolved as separate peaks for each d-spacing, which can lead to difficulties in performing Search/Match. To help solve this problem, simply switch the TRIO to the high resolution monochromator to check the regions in question with pure K α 1 radiation. This example shows data from a quartz sample from both Bragg-Brentano (red) and monochromator mode (blue) clearly highlighting the advantages of K α 1 data.

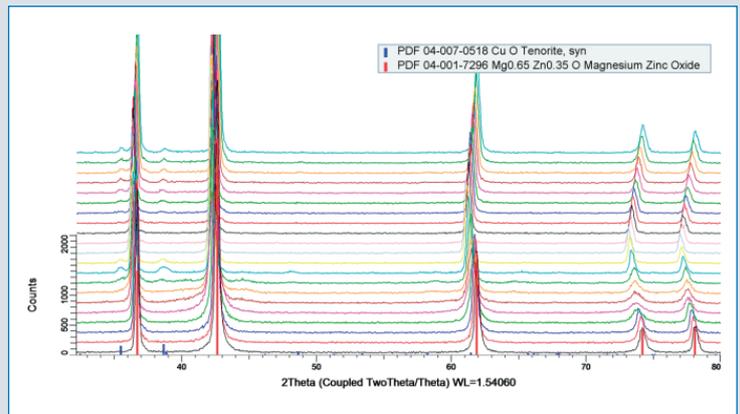


Parallel Beam

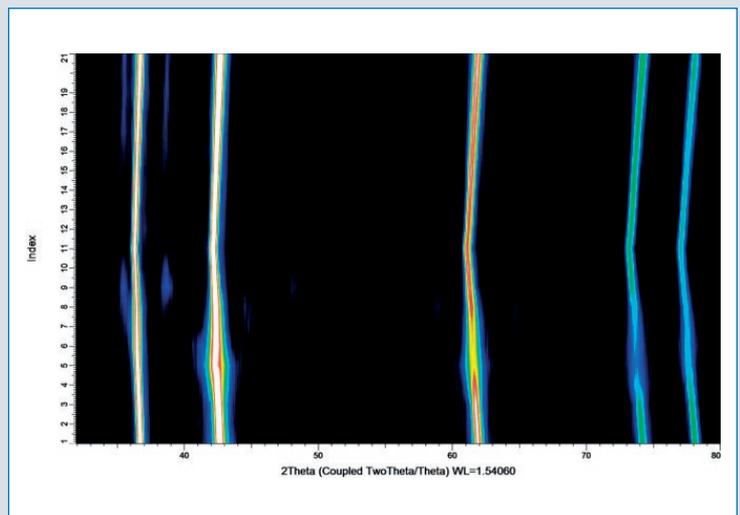
High intensity, parallel beam geometry in reflection mode is useful for measuring samples with uneven surfaces or sample height displacements (e.g. non-ambient induced) which can shift diffraction patterns too high or too low when using focusing geometry. Parallel-beam is insensitive to sample height displacement and therefore perfect to accurately determine lattice parameters from peak positions. This beam geometry is accomplished with TRIO (Göbel Mirror).



Here a (MgZn)O pellet is heated from room temperature to 1100° C and back to study phase transitions and accurately determine lattice parameters as a function of temperature. Parallel beam geometry is used to collect this data. The lattice parameter of the main cubic phase changes by 1.2% and then returns to the initial value on cooling back to room temperature.

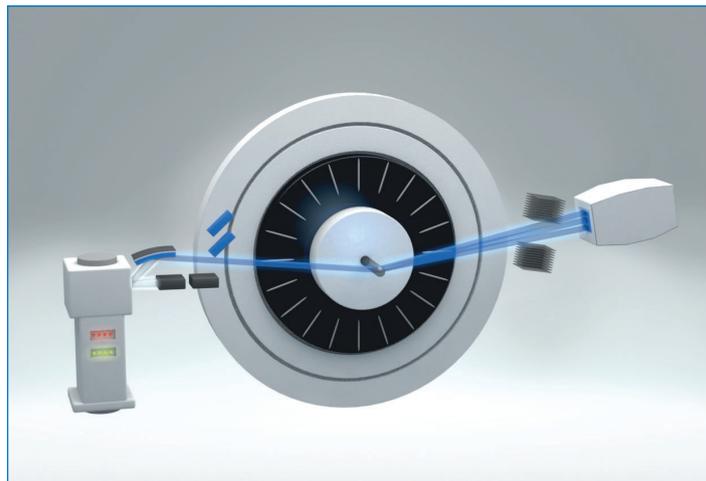


The 2D view of the peak intensity as a function of temperature clearly shows the shift of the main phase peaks and also the appearance of an additional phase, identified as CuO, which appears at 750° C and then again at 650° C on cool down.

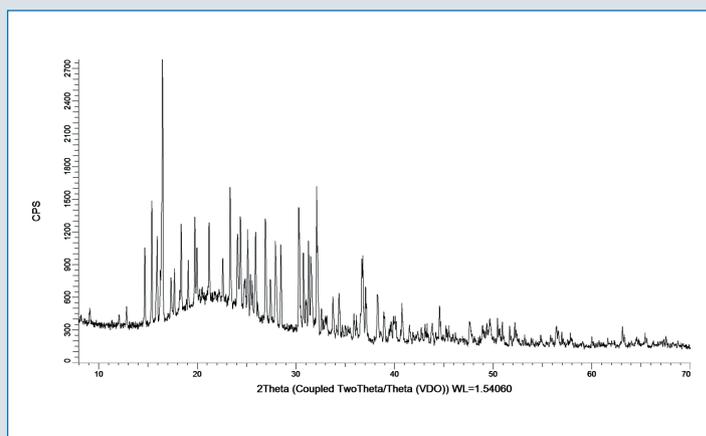


Powder Transmission

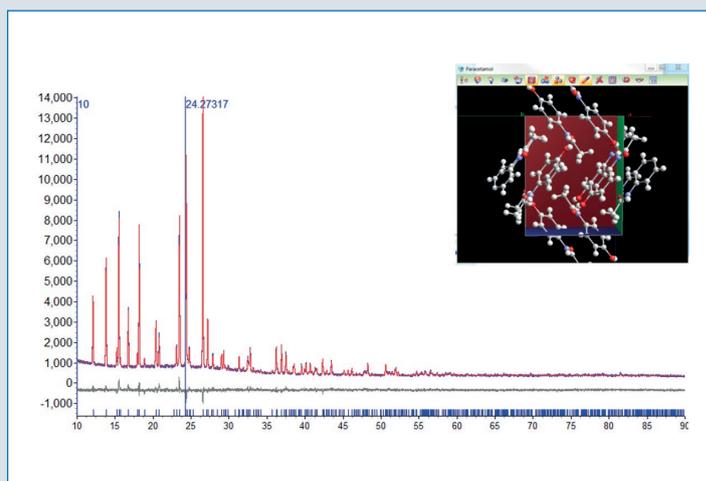
For small sample quantities, preferred orientation, materials with low absorption coefficients, and air-sensitive samples, etc. measurements in transmission geometry are the best choice. The TRIO set to Göbel mirror provides a high intensity, collimated beam. In combination with TWIN (slit) and LYNXEYE XE-T in 1D mode this is the ideal configuration to measure such powder samples in capillary or flat plate orientation.



This $\text{Na}_2\text{S}_2\text{O}_3$ powder sample is run in capillary mode to minimize preferred orientation and produce best peak resolution as it is a low symmetry unit cell. The primary beam is matched to the capillary diameter and the LYNXEYE XE-T 1D detector accelerates data collection to achieve required signal/noise. The large hump at about 22° 2θ is the amorphous scattering signal from the glass capillary.

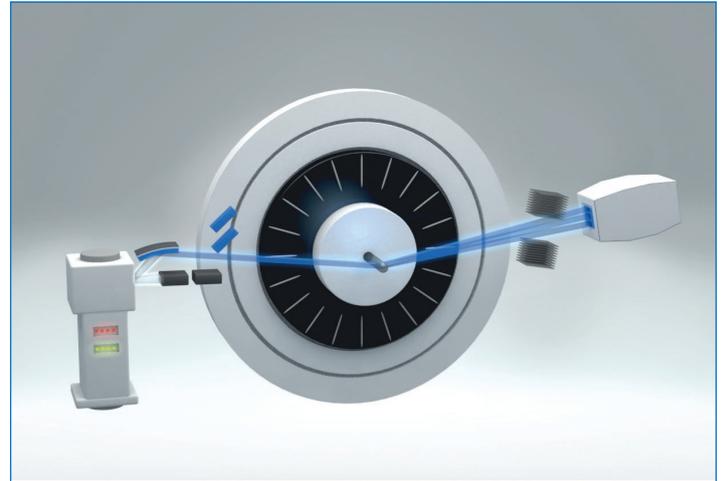


As with flat plate powder measurements, a $K\alpha_1$ high resolution mode is available (TRIO, Göbel Mirror + Monochromator) for transmission measurements which produce data that is ideal for structure solution and subsequent Rietveld refinement. In this example, paracetamol ($\text{C}_8\text{H}_9\text{O}_2\text{N}$) powder in a capillary was analyzed and structure solution using simulated annealing is carried out with DIFFRAC.TOPAS. Even tiny diffraction peaks close to major diffraction peaks can be perfectly resolved and good residuals are obtained for a fit covering the whole data range.

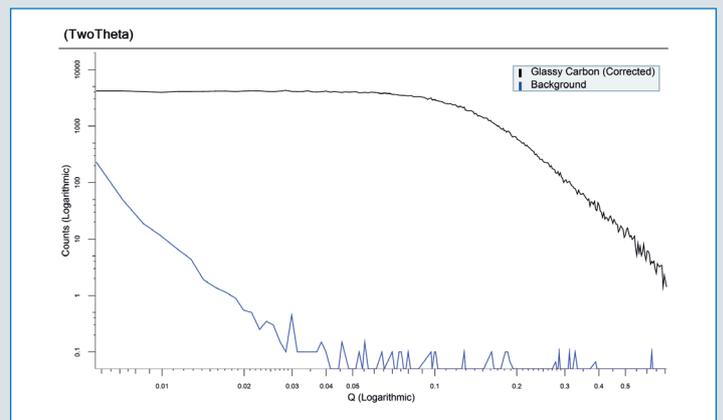


Small Angle X-Ray Scattering (SAXS)

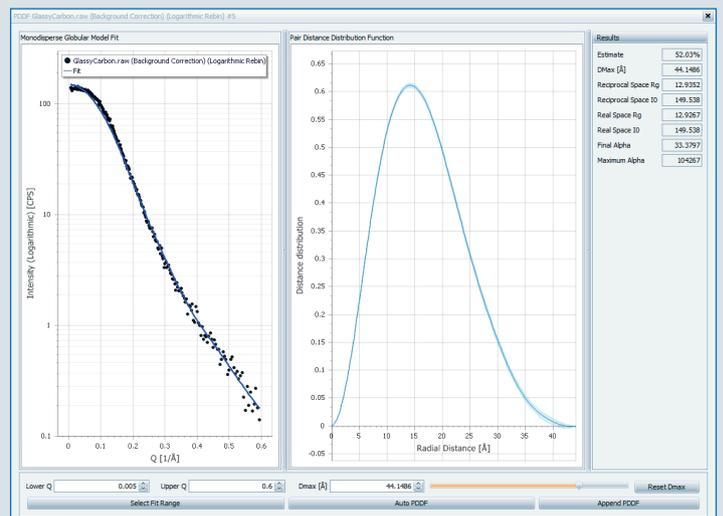
The Small Angle X-Ray Scattering (SAXS) geometry is similar to powder transmission, except that the measurement range is in the very small angle region (typically $0 - 10^\circ$ 2θ) which probes length scales from a few up to about 100 nm. Small slits in the primary and secondary side are used to give access to sample scattering very close to the direct beam. SAXS data is typically shown with the x-axis given in $q = (4\pi \sin\theta)/\lambda$. DIFFRAC.SAXS features straightforward data reduction and analysis.



The background-corrected SAXS scattering from a solid piece of glassy carbon is shown here along with the background scattering for comparison. The data corresponds to a 2θ range of <0.1 to 8 degrees. The minimum value of Q is around 0.006 \AA^{-1} and corresponds to a length scale of about 100nm.

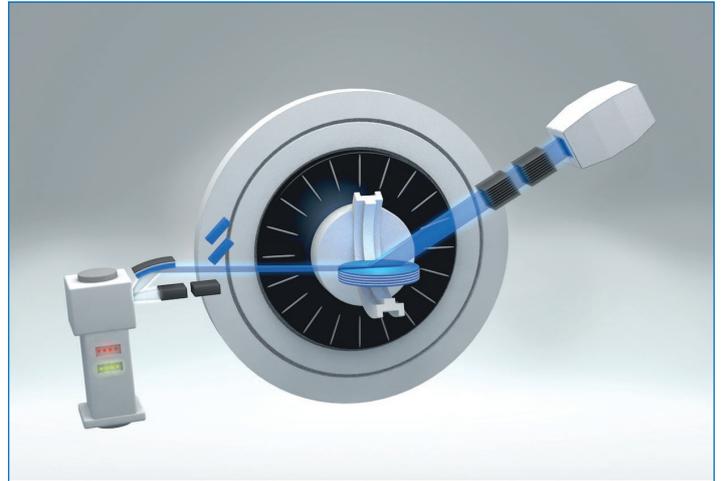


Analysis of the scattering data is done with DIFFRAC.SAXS. Here the pair distance distribution function (pdf) is shown along with the corresponding globular model fit assuming a monodisperse distribution of pores. Parameters such as the radius of gyration (R_g) and D_{max} , the maximum particle (or pore) dimension can be extracted from the pdf as indicators of pore size. Further, the distribution the pdf gives an indication of the pore shape, which was determined here to be ellipsoid.

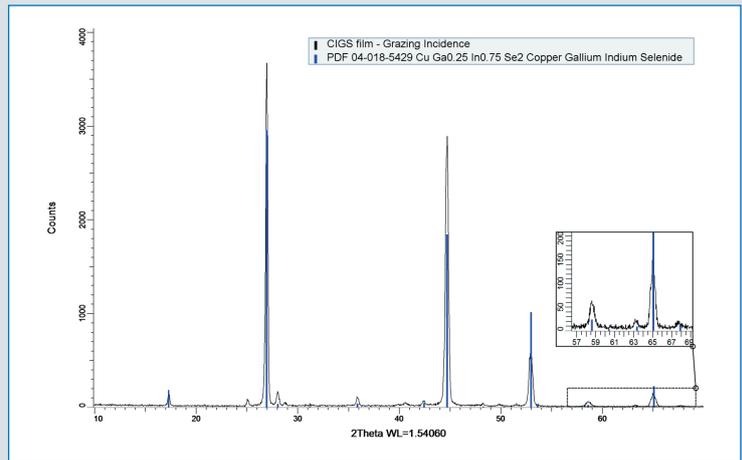


Grazing Incidence Diffraction (GID)

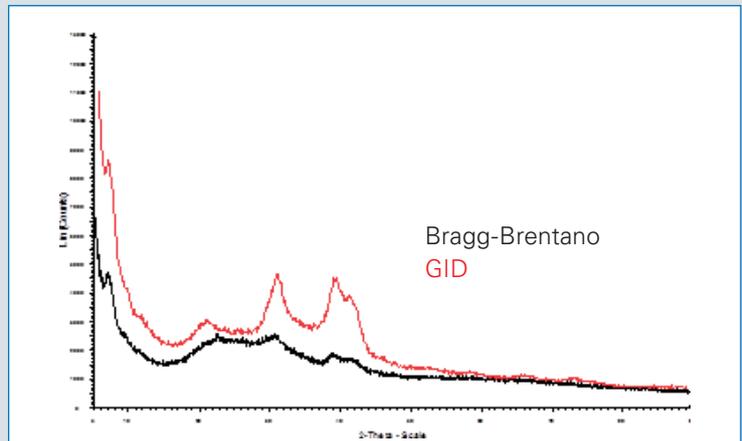
Grazing incidence diffraction utilizes a fixed, low (or grazing) incident angle to maximize signal from thin polycrystalline films while minimizing or eliminating the interfering signal from the substrate. The TRIO (Göbel Mirror) produces a high intensity, collimated primary beam to control penetration depth and the TWIN (Soller collimator) produces good peak resolution in this asymmetric diffraction geometry.



The application example is a Copper Indium Gallium Selenide (CIGS) film used for solar cell technology, which is effectively characterized using grazing incidence diffraction (GID). Here a shallow incident angle is used to virtually eliminate the diffraction pattern from the substrate and give a strong, representative pattern from the polycrystalline thin film which allows the most complete match to the solid solution reference pattern; even at high 2Theta angles as shown in the inset.

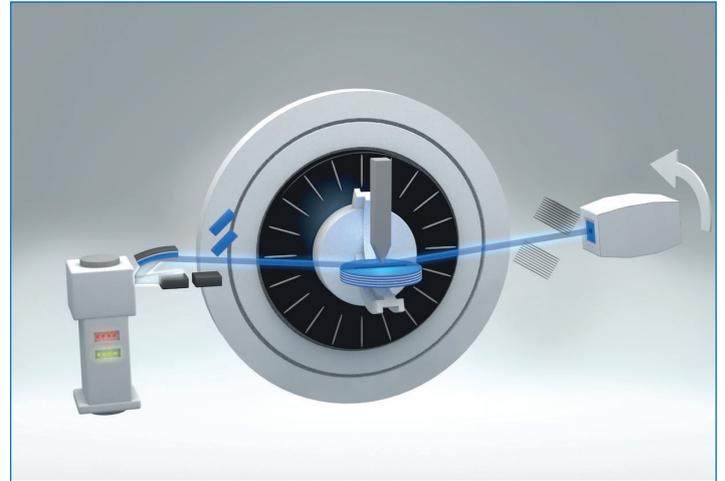


As a comparison, an Ag_2Te thin film on glass was measured with both Bragg-Brentano (black) and GID (red) geometry. Note how the GID data more clearly shows the diffraction pattern from the Ag_2Te nanocrystallites and the glass substrate signal is reduced.

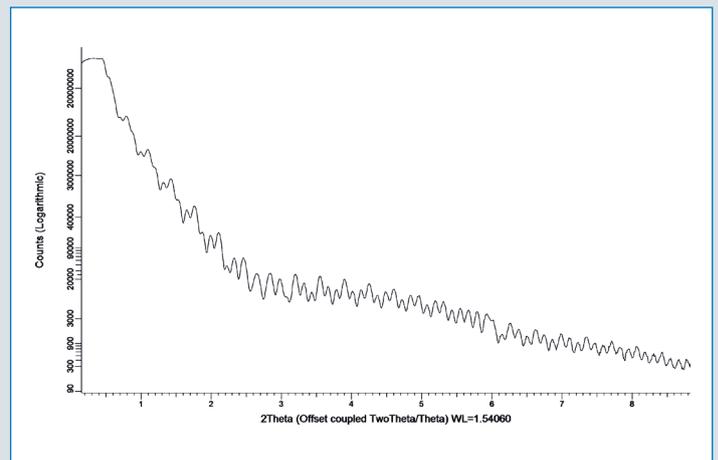


X-Ray Reflectivity (XRR)

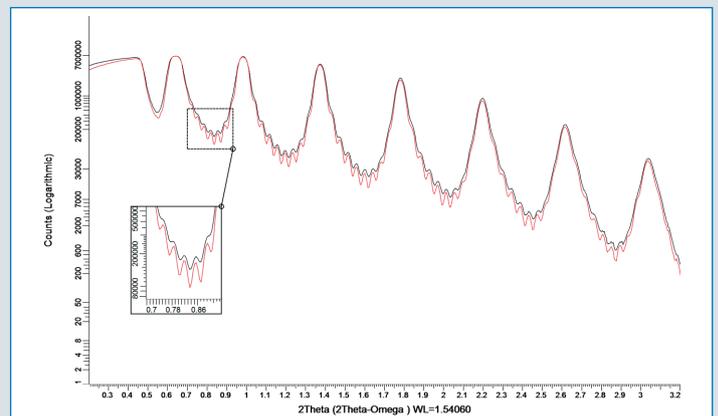
X-Ray Reflectivity (XRR) uses grazing incidence to look at the interference pattern of X-rays scattered in a specular geometry from interfaces in a layered thin-film sample to determine film thickness, interface roughness, and electron density. This application utilizes a high intensity parallel beam from the TRIO (Göbel Mirror), an optional knife edge collimator (KEC) over the sample to define the beam dimension and reduce diffuse scatter, and slits from the TWIN (slits) to define the specular beam entering the LYNXEYE XE-T detector.



This example shows the XRR curve from a nominal 50 nm $\text{Si}_{0.85}\text{Ge}_{0.15}$ epitaxial layer on single crystal Si substrate with a 25 nm Silicon capping layer. Note that reflectivity data is collected over 7 orders of magnitude in intensity. The XRR data is consistent with nominal values, but the low frequency oscillations seen in the data is indicative of a thin 3 nm Silicon Oxide layer that has formed at the surface.

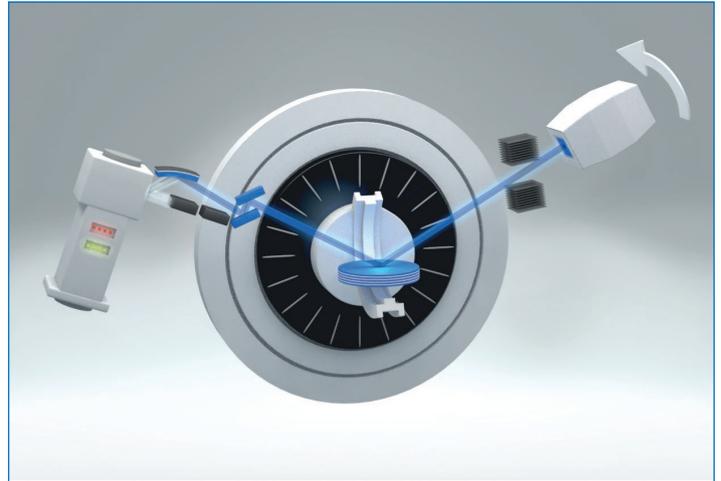


As the film thickness increases, the XRR interference fringes get closer together. Using the highly parallel, $\text{K}\alpha_1$ beam produced by the high resolution mode of the TRIO (Göbel Mirror + Monochromator) allows the resolution to resolve thicker films. Here a Ni/C superlattice is analyzed using the Göbel Mirror only (black) and the Göbel Mirror + Monochromator (red). Both datasets show the superlattice reflections (large peaks) and total thickness fringes (smaller peaks) although the total thickness fringes are better resolved with the monochromator.

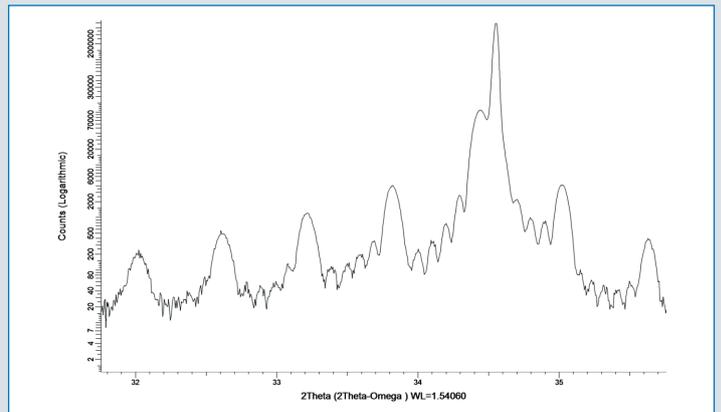


High Resolution XRD (HRXRD)

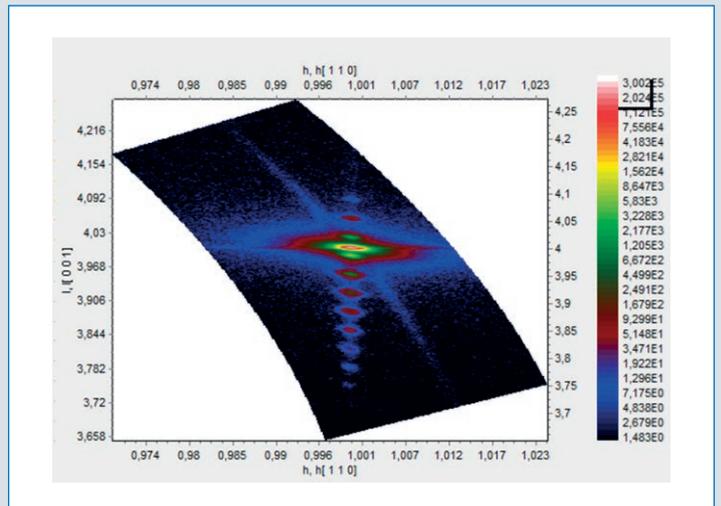
High Resolution XRD (HRXRD) is used to characterize the thickness, crystallographic structure, composition and the degree of strain/relaxation in thin, epitaxially grown films on substrates, such as SiGe and GaN on wafers. Because there are very small differences in lattice parameters between film and substrate, HRXRD requires a very parallel $K\alpha_1$ beam to clearly resolve the resulting peaks. This type of beam is achieved by using a TRIO (Göbel Mirror coupled with a Ge channel-cut monochromator) and the TWIN (slits). Typical measurements are rocking curves, where the sample is rocked or rotated around an out-of-plane Bragg angle, and reciprocal space maps (RSMs) in which a small region of reciprocal space is mapped to show, at a glance, the epitaxial relation between the film and substrate.



The HRXRD scans shown are from a typical LED sample which consists of 6 InGaN quantum wells on a GaN substrate. The data in this figure shows a $2\theta/\omega$ scan around the GaN (0002) peak which allows the determination of the superlattice thickness and indium concentration with great precision. The total amount of indium is an important parameter to monitor the progress of the growth process.

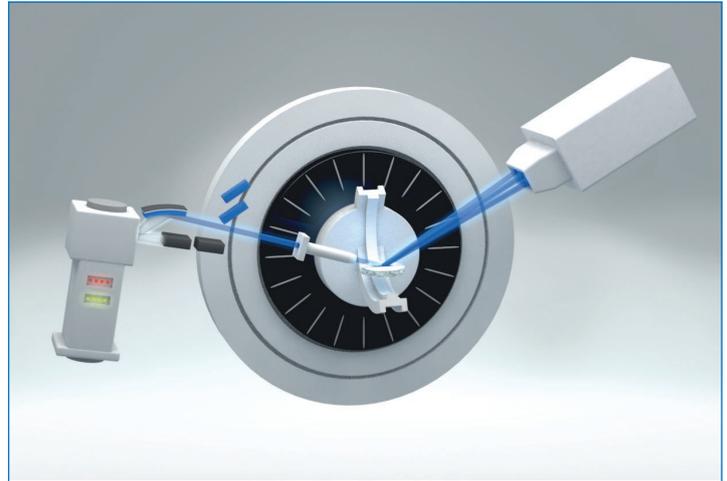


The RSM was collected with the LYNXEYE XE-T in 1D mode and represents 171 2θ scans looped over $\omega/2\theta$ relative conditions. The data is transformed to hkl and displayed as a map with DIFFRAC.LEPTOS. The orientation and position of the reciprocal lattice points indicates pseudomorphic, or coherent, growth of the film.

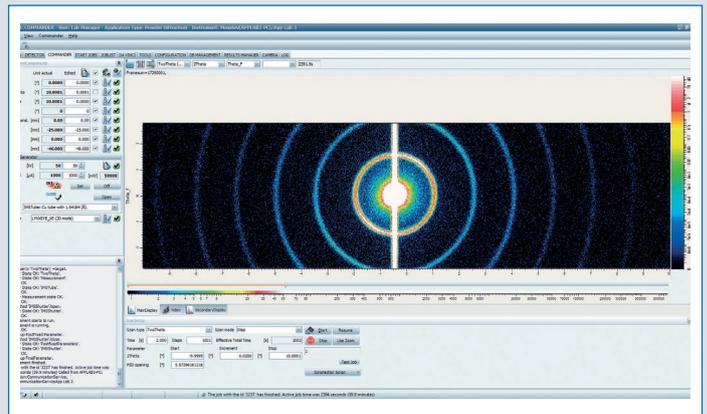


Debye Scherrer (2D)

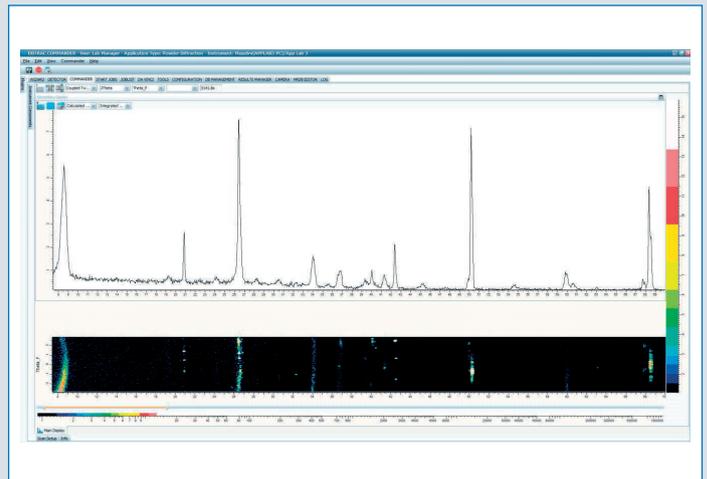
2D scattering data can be collected using Debye Scherrer geometry which is created with a parallel spot beam using the TRIO (Göbel Mirror) with a collimator, the TWIN (slit) to define the 2Theta angle and the LYNXEYE XE-T in 2D-mode can be scanned around angles of interest. The resulting 2D data shows sections of the Debye cones and the intensity along these cones contains additional information about the sample in including stress, texture, particle size, and phase type (single crystal, few crystal, or polycrystalline).



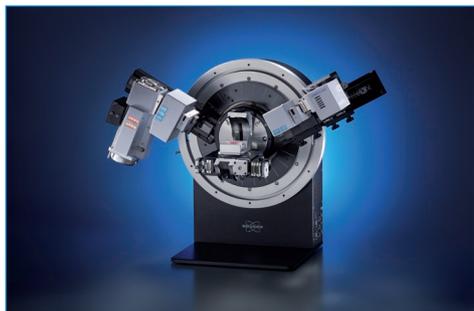
In the first example a silver behenate powder sample, a SAXS standard, is examined in the very low + and - 2Theta region. Large sections of the Debye rings corresponding to Bragg diffraction peaks can be seen. This data can be easily integrated to produce 1D scattering intensity vs 2Theta with even very low angle peaks clearly resolved and with very little peak shape asymmetry which can accompany typical 1D scans.



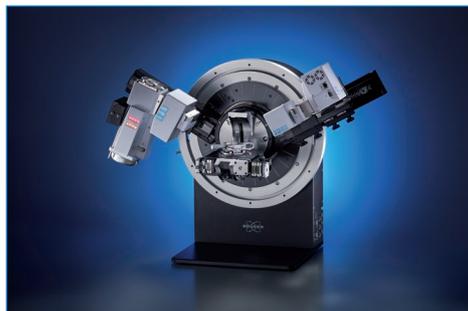
The second example is of a textured polycrystalline rock sample. Note the differences in the intensity distribution of the Debye Rings. This is indicative of 2 different phases; one that is fine grained and textured as indicated by the continuous, but non-uniform, intensity distribution along gamma and one that is large grained as shown by the spotty and non-uniform intensity. This information may give important insight into sample composition and morphology.



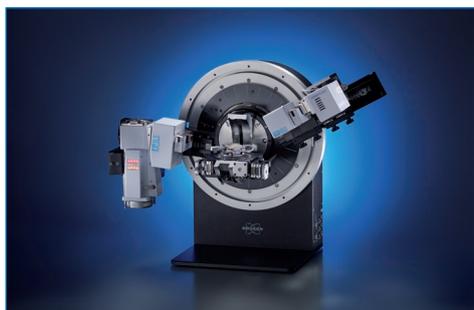
D8 ADVANCE Plus Configurations



Powder Diffraction (Bragg-Brentano)



High Resolution XRD (HRXRD)



Grazing Incidence Diffraction (GID)



X-Ray Reflectometry (XRR)

Table: Full list of configurations

Typical Sample	Scattering Geometry	TRIO	TWIN	LYNXEYE XE-T
Flat powder	Bragg-Brentano	Motorized Slit	Slit	1D
Uneven polycrystalline	Parallel Beam	Göbel Mirror	Soller Collimator	0D
Powder in capillary	Powder Transmission	Göbel Mirror	Slit	1D
Solid, powder	Small Angle X-Ray Scattering (SAXS)	Göbel Mirror	Slit	0D
Polycrystalline thin film	Grazing Incidence Diffraction (GID)	Göbel Mirror	Soller Collimator	0D
Thin film	X-Ray Reflectivity (XRR)	Göbel Mirror	Slit	0D
Epitaxial thin film	High-Resolution XRD (HRXRD)	Göbel Mirror + Monochromator	Slit	0D or 1D
Textured polycrystalline	Debye Scherrer	Göbel Mirror	Slit	2D

Secondary TWIN: EP 2 194 375 A1 patent; US 7,983,389 B2 patent

TRIO: patent pending

LYNXEYE, LYNXEYE XE and LYNXEYE XE-T turned 90°: US 7,263,161 B2 patent, EP 1 647 840 A2 patent and EP 1 510 811 B1 patent

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