

CANADIAN POWDER DIFFRACTION WORKSHOP - 2025

Latest innovations in Bruker XRD

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July 31th, 2025

Agenda

01 Introduction

02 Beyond powder – D6 Phaser

03 Hard Radiation – PDF and Batteries

04 Engineering Specials

01 - Introduction

Bruker X-ray Diffraction



- **D6 PHASER**
All-in-one
benchtop XRD



- **D8 ENDEAVOR**
XRD for process and
quality control



- **D8 ADVANCE**
Versatile and future-proof
XRD solution



- **D8 DISCOVER**
All-purpose cutting edge
XRD solution

02 – Beyond Powder – D6 Phaser

D6 PHASER

The Benchtop Platform



+ POWERFUL

- 600W or 1200W with internal cooling
- Time-tested goniometer
- Multimode LYNXEYE XE-T detector

+ VERSATILE

- Reflection and transmission
- Varied stages
- Source, stage, optics exchange

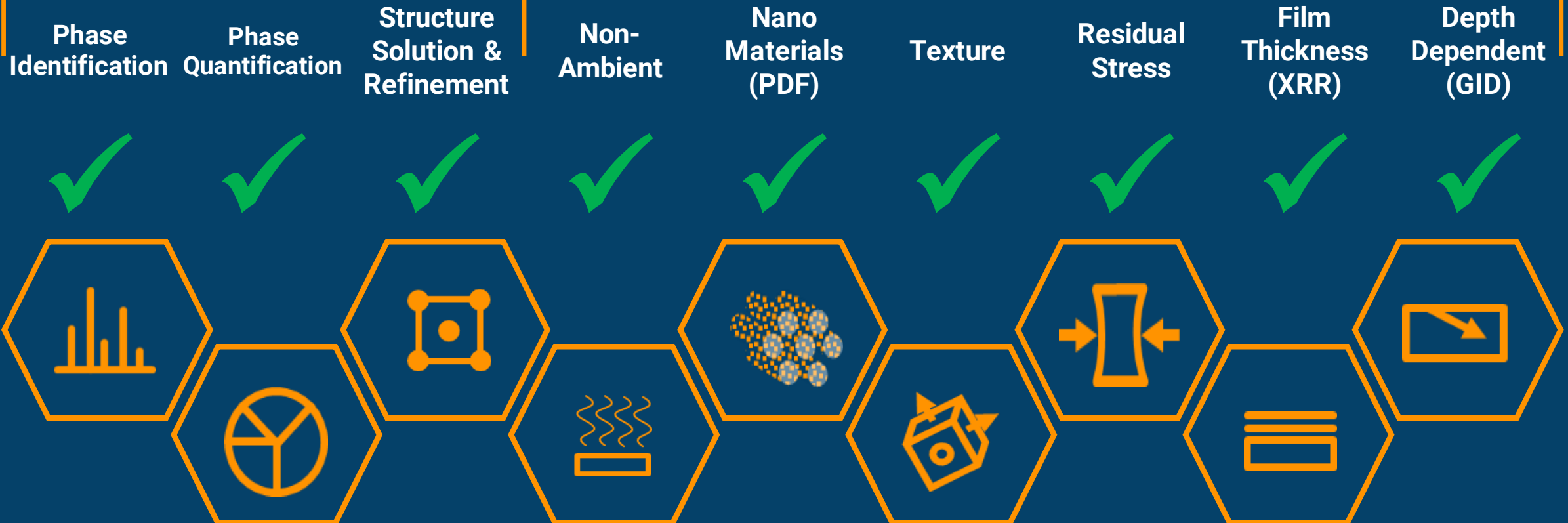
+ ACCESSIBLE

- Motorized beam optics
- Full Bruker software suite
- Integrated components

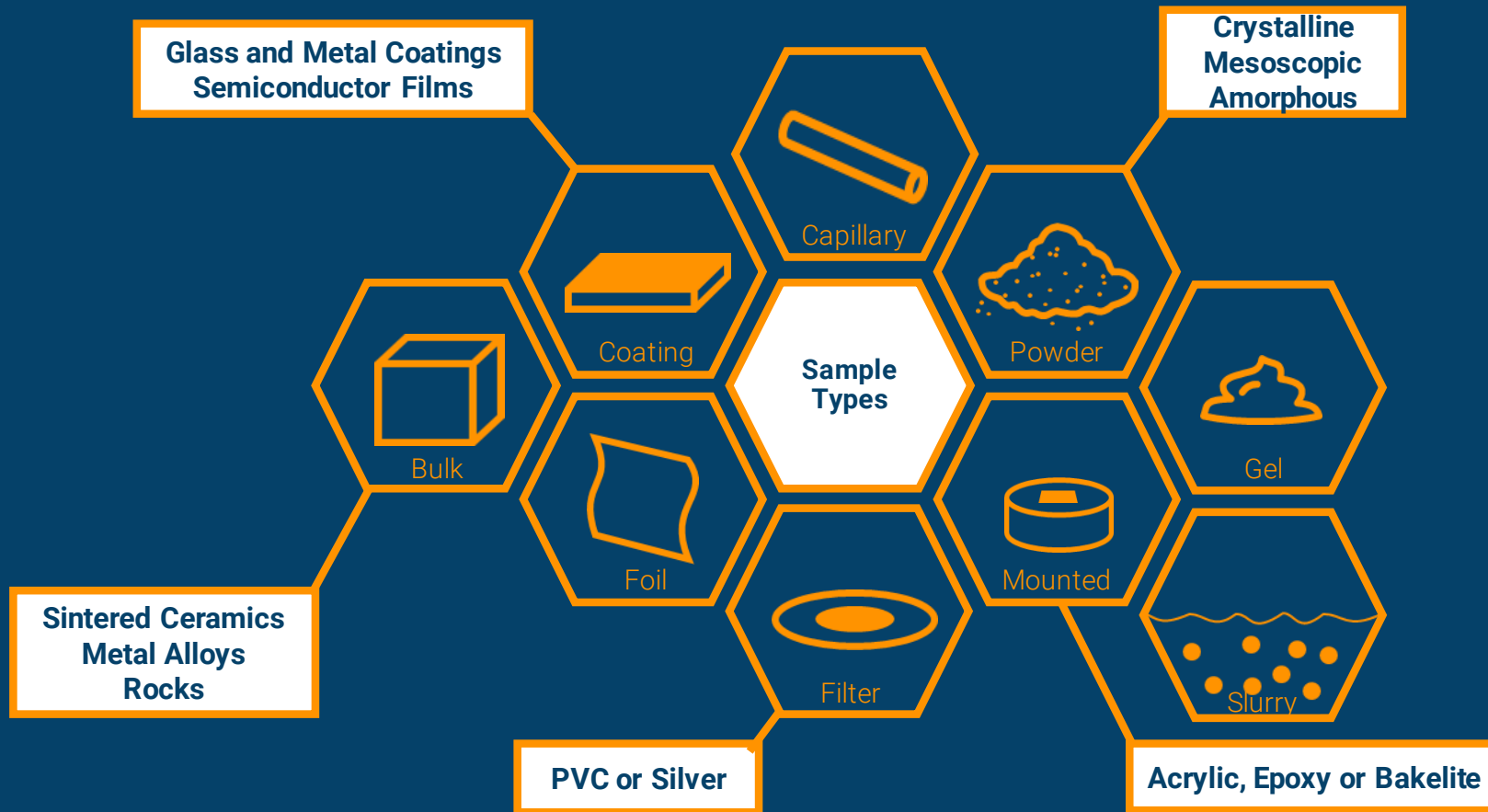
What information can the D6 PHASER provide?

Materials Research Methods

Traditional Powder Methods



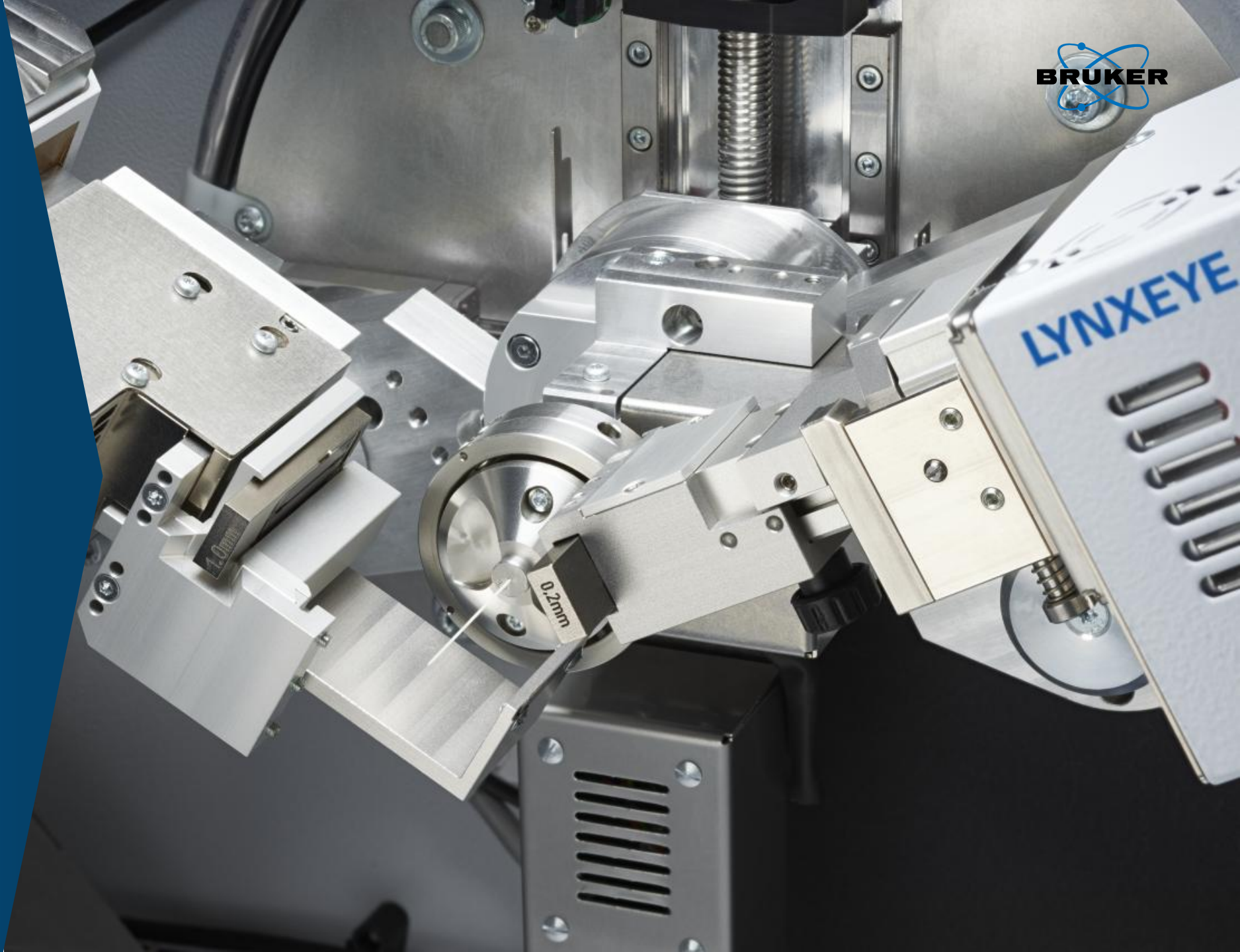
D6 Phaser – sample types



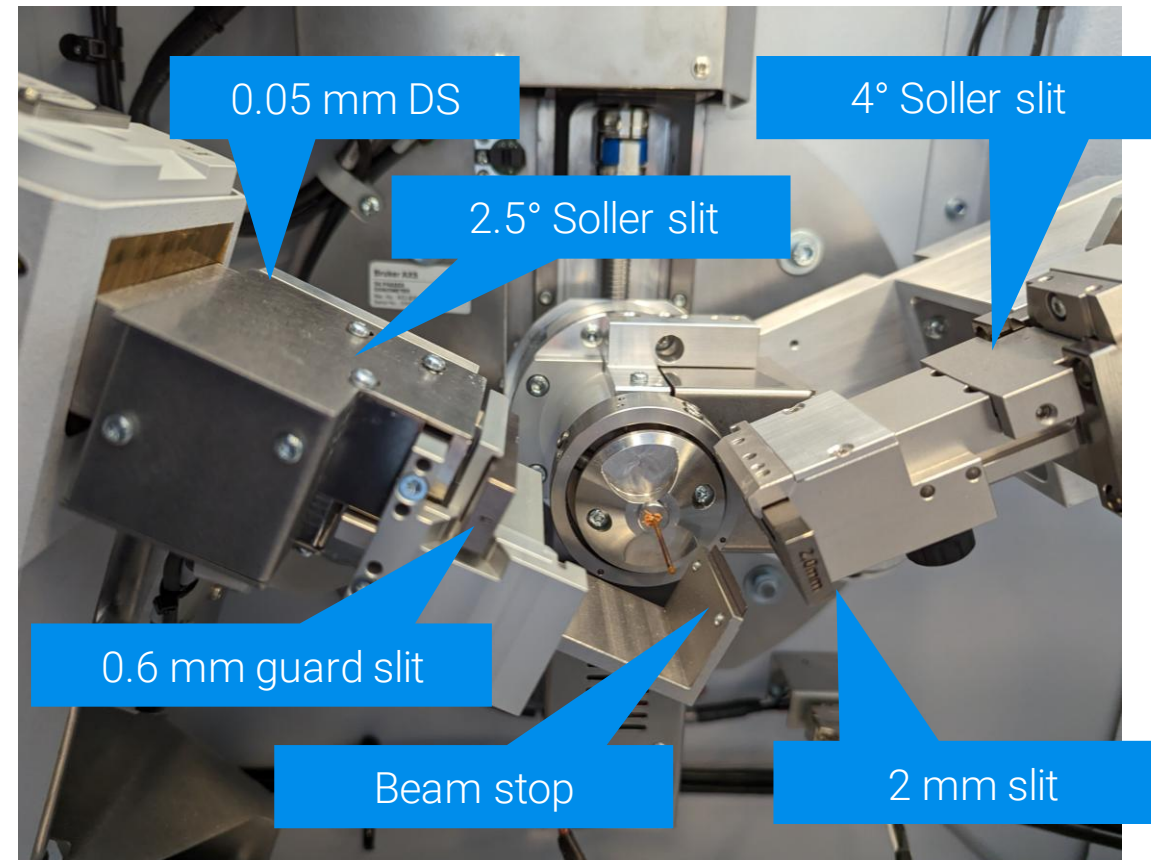
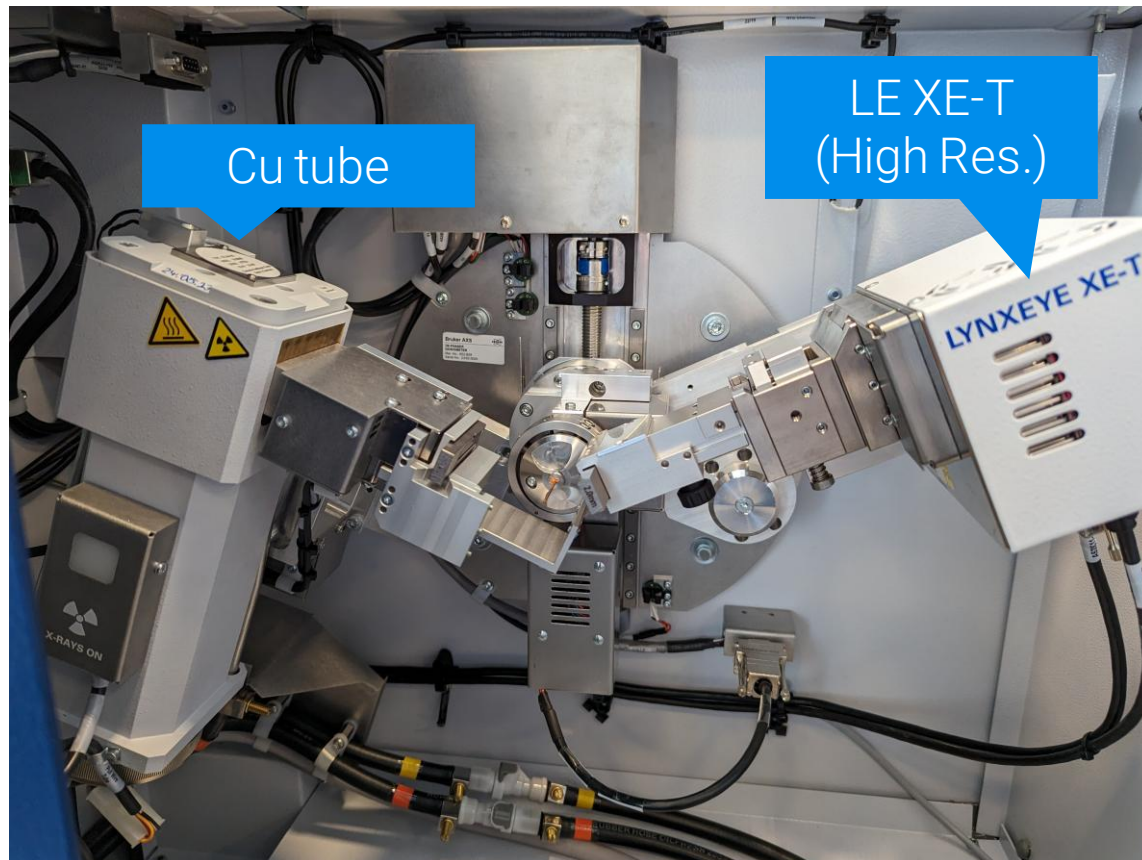


Capillary Diffraction

Generator 1200W
X-ray Tube Cu
Incident Optic Advanced Optic with Axial Soller
Sample Stage Capillary Stage
Air Scatter Control Guard Slit Holder with Beam Stop
Diffracted Beam Optic Telescopic Slit Shaft
Detector LYNXEYE XE-T



D6 PHASER with Capillary Stage Instrument Configuration

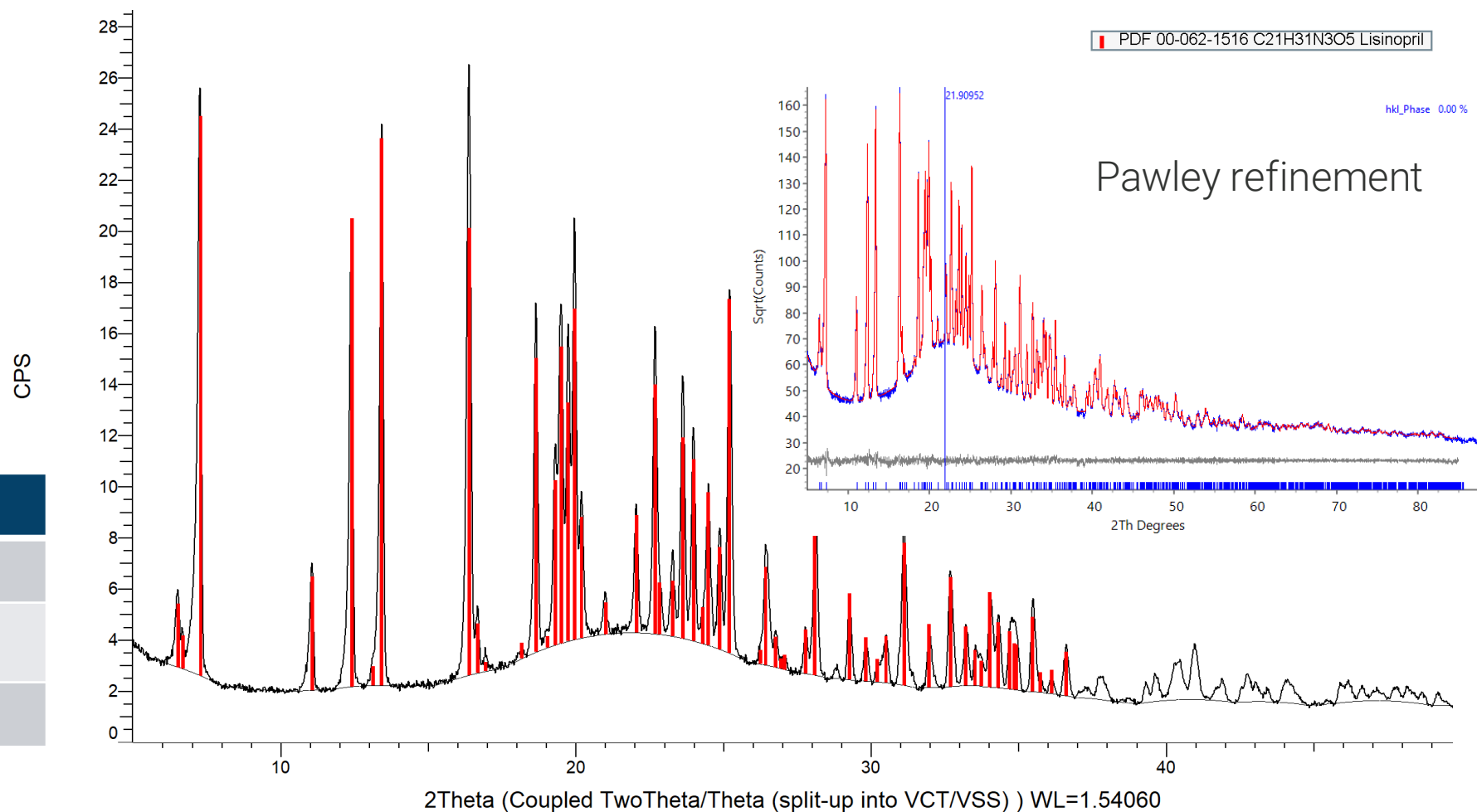


D6 PHASER in Capillary Geometry Organic Sample

- Lisinopril ($C_{21}H_{31}N_3O_5$) in 0.5mm capillary

Measurement Conditions

Scan range	5 – 120 deg 2th
Time/step	0.3 – 1.5 s
Step size	0.021 deg
Total time	8 x 90 min = 12 h

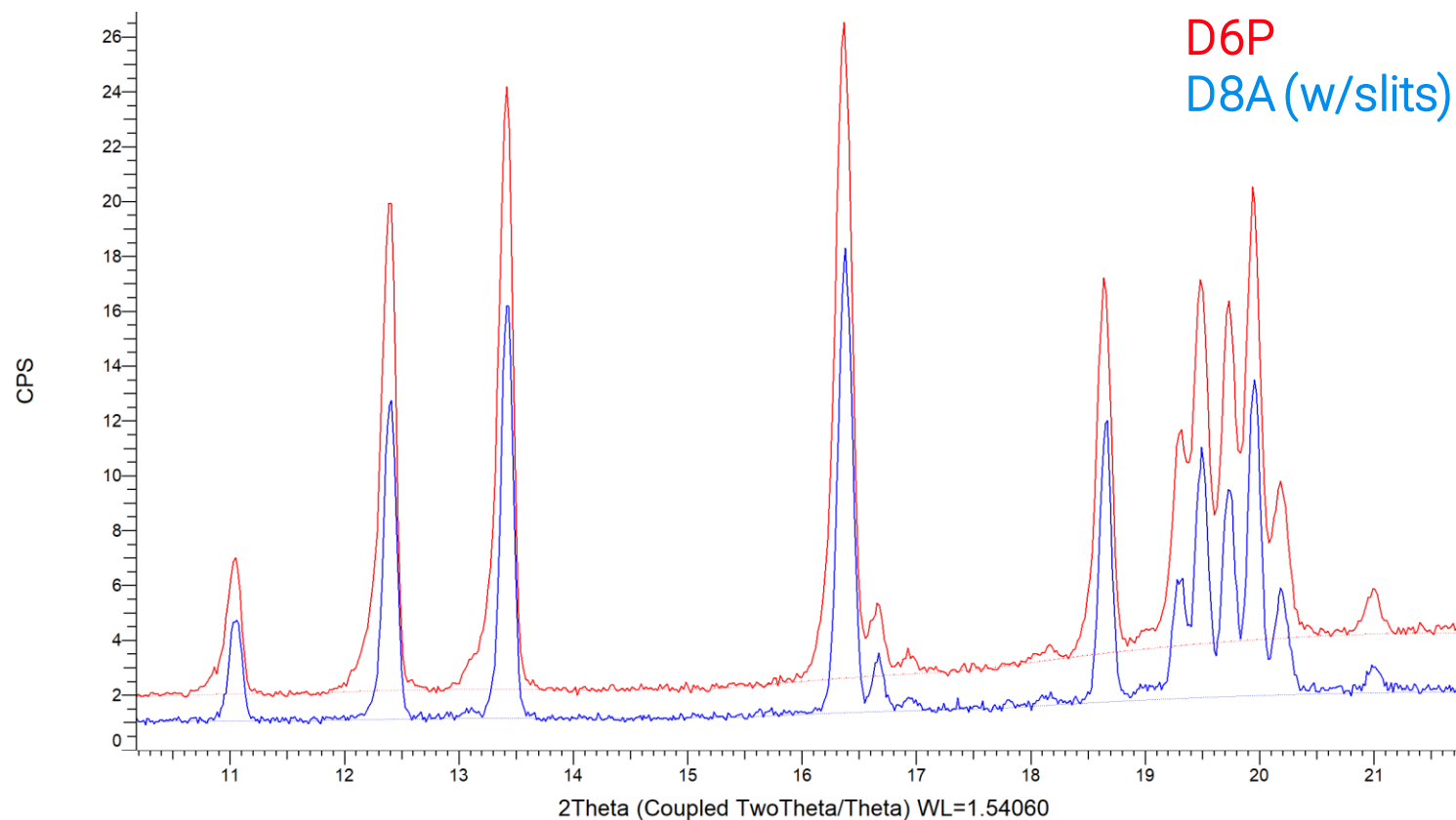


D6 PHASER in Capillary Geometry Organic Sample

- Lisinopril ($C_{21}H_{31}N_3O_5$) in 0.5mm capillary
- Divergent beam geometry

Measurement Conditions

Scan range	5 – 120 deg 2th
Time/step	0.3 – 1.5 s
Step size	0.021 deg
Total time	8 x 90 min = 12 h



D6 PHASER in Capillary Geometry

Organic Sample

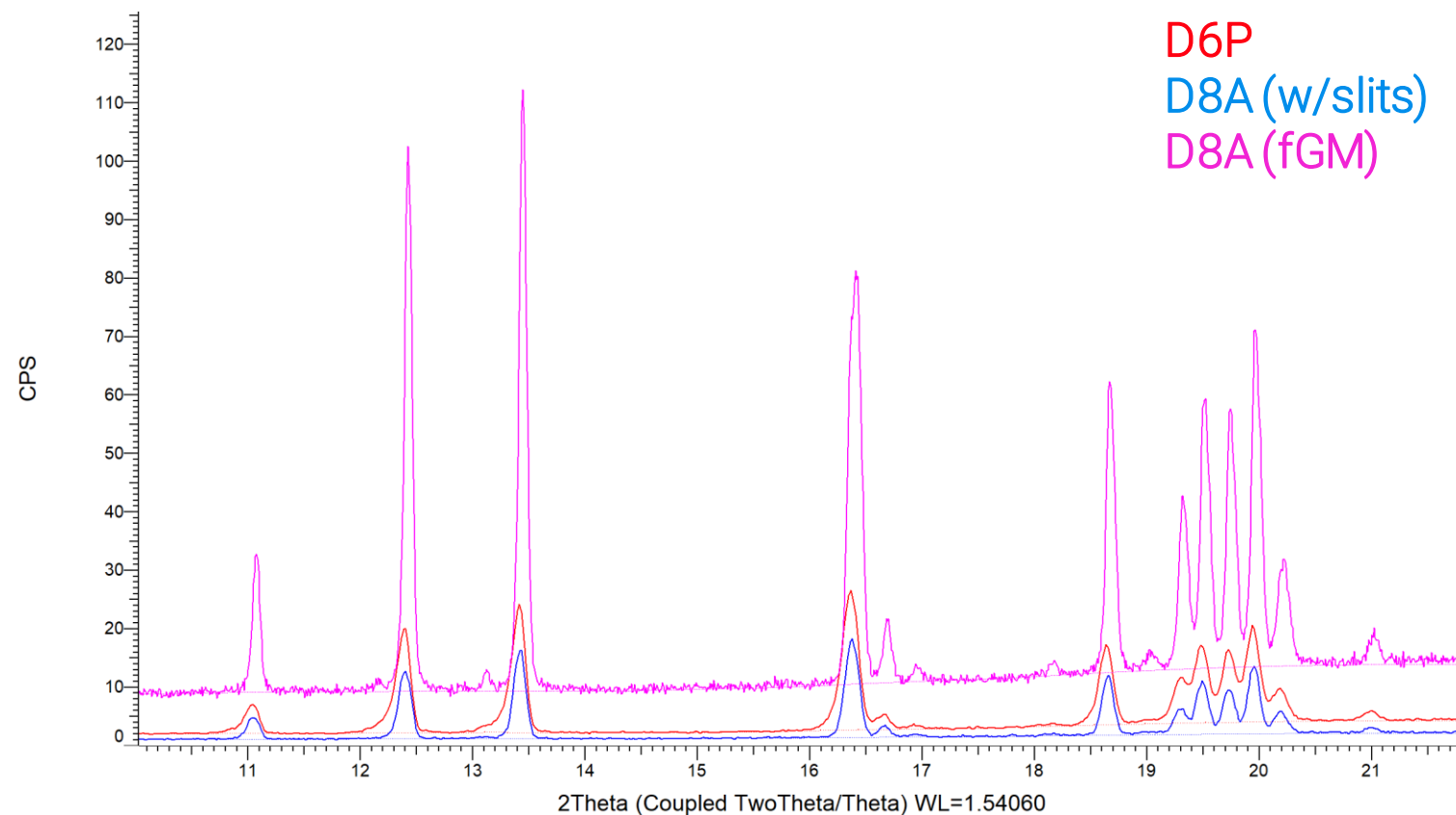
- Lisinopril ($C_{21}H_{31}N_3O_5$) in 0.5mm capillary
- Divergent beam geometry

Measurement Conditions

Scan range 5 – 120 deg 2th

Time/step 0.3 – 1.5 s
Step size 0.021 deg

Total time 8 x 90 min = 12 h



D6 PHASER - THE BENCHTOP PLATFORM



Grazing Incidence Diffraction & X-ray Reflectometry

Generator 1200W

X-ray Tube Cu

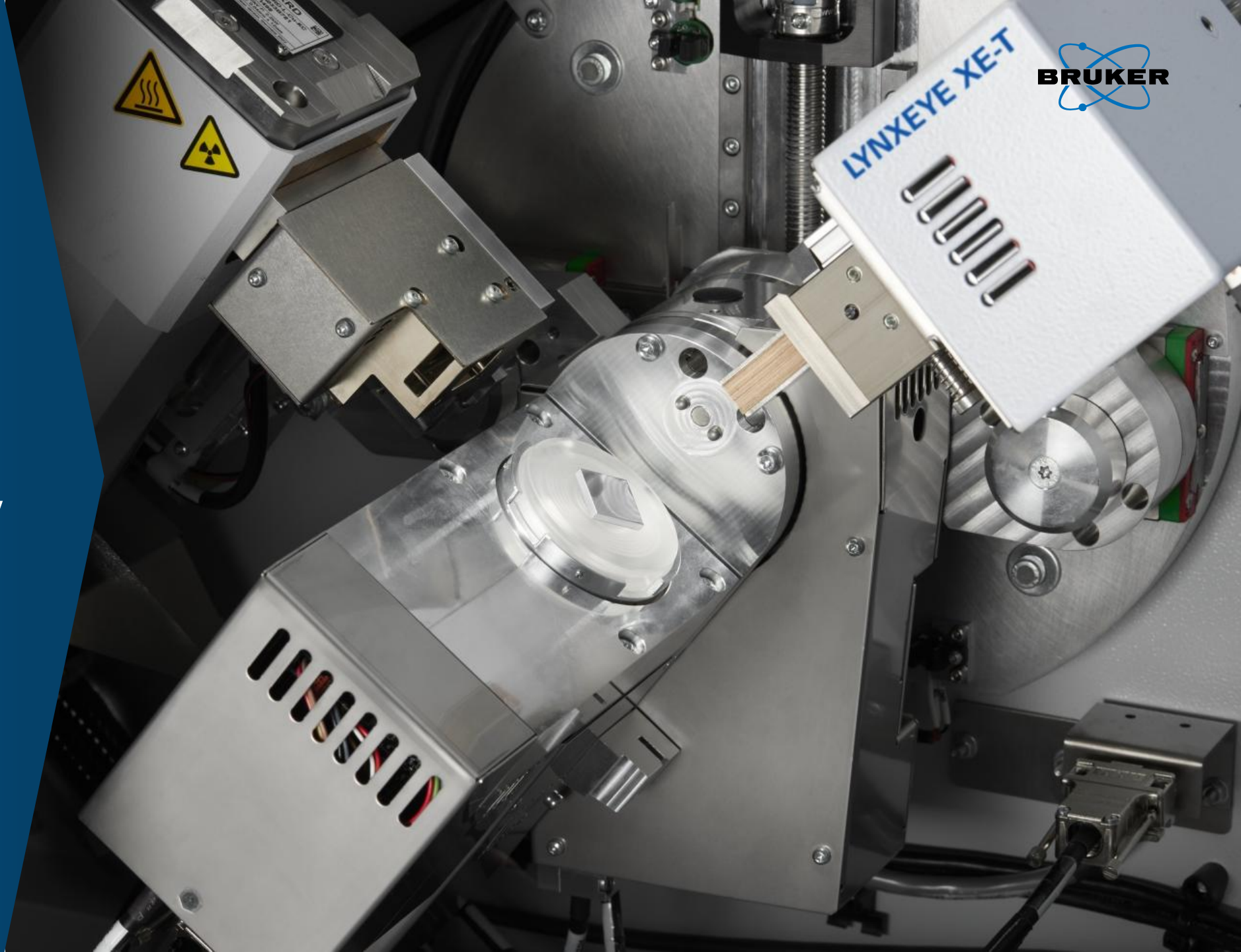
Incident Optic Advanced Optic with Axial Soller

Sample Stage Universal Stage with Phi Attachment

Air Scatter Control None

Diffracted Beam Optic Equatorial Soller (GID) or
Telescopic Slit Shaft (XRR)

Detector LYNXEYE XE-T



THIN FILM ANALYSIS

Components for GID



Primary side advanced optics

- Motorized variable divergence slit
- Axial Soller collimator
- Second pluggable slit or Cu absorber



Universal stage

- Decoupling of the incident and diffracted beam angle
- Motorized Z height for to accommodate various samples

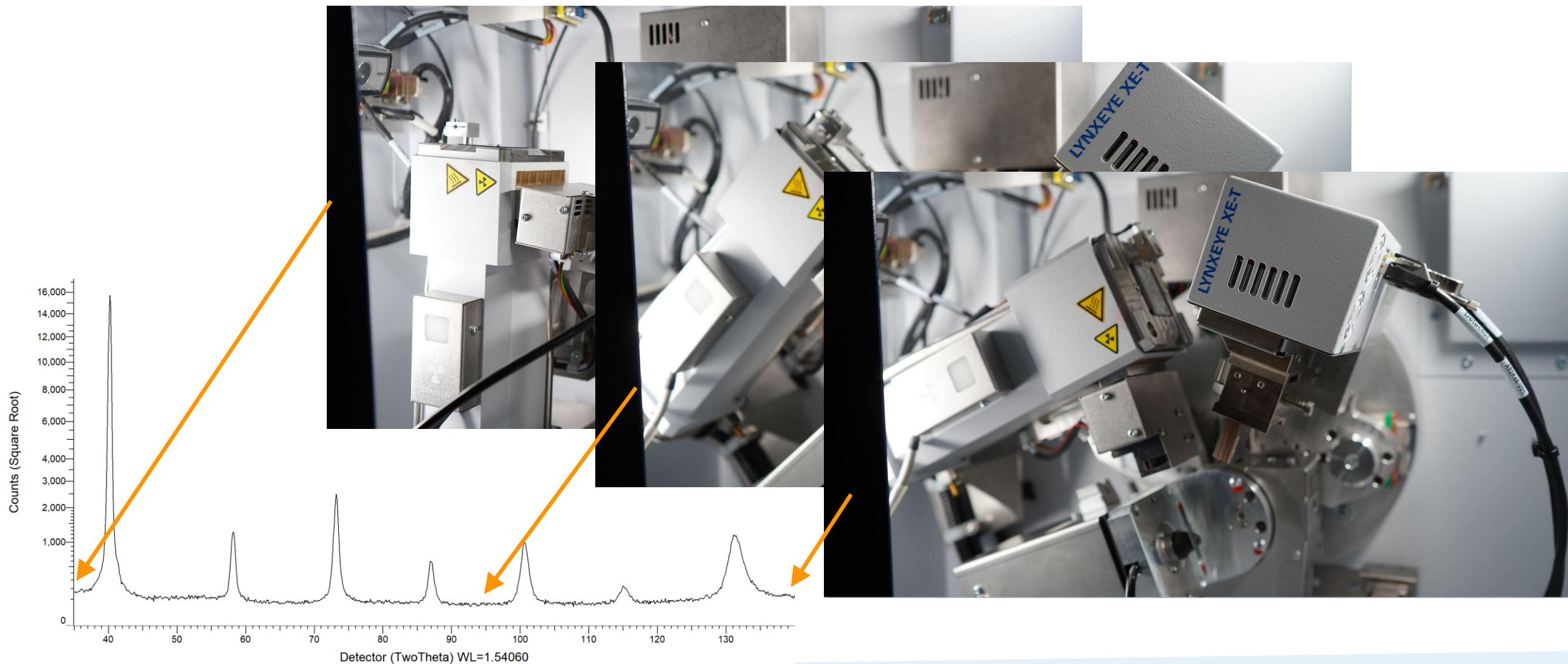


Equatorial Soller collimator

- Reproducible positioning with two fixing screws
- Extra slot for Cu absorber

THIN FILM ANALYSIS

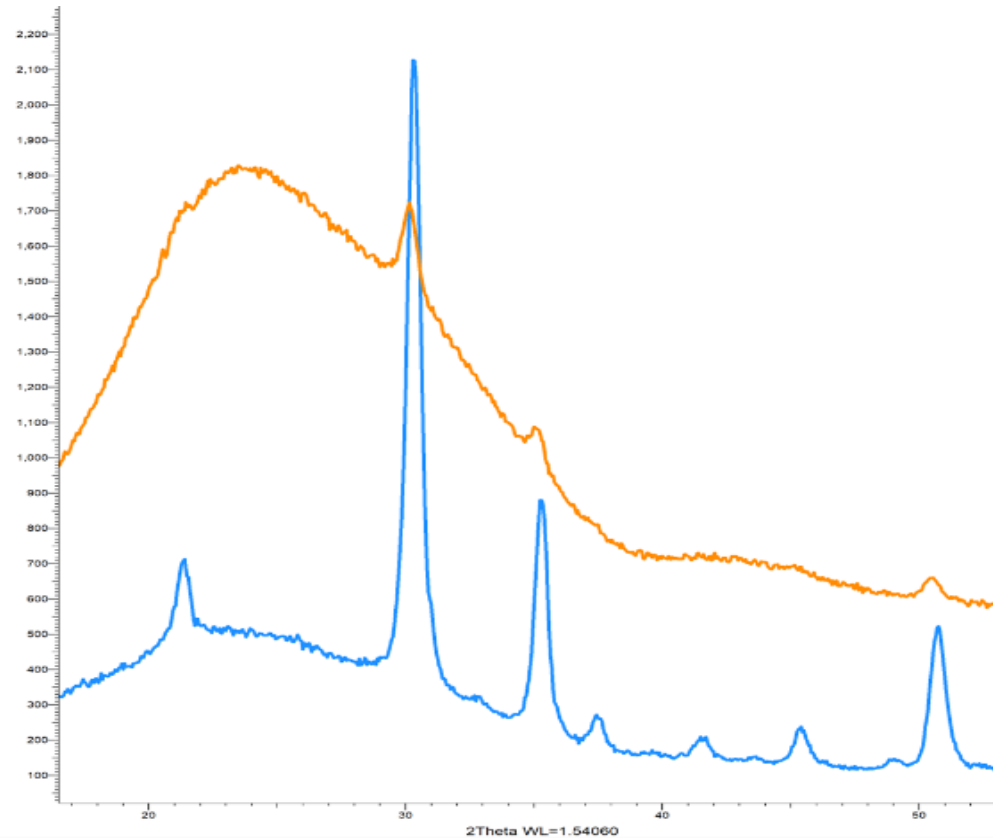
GID data aquisition



Materials Research

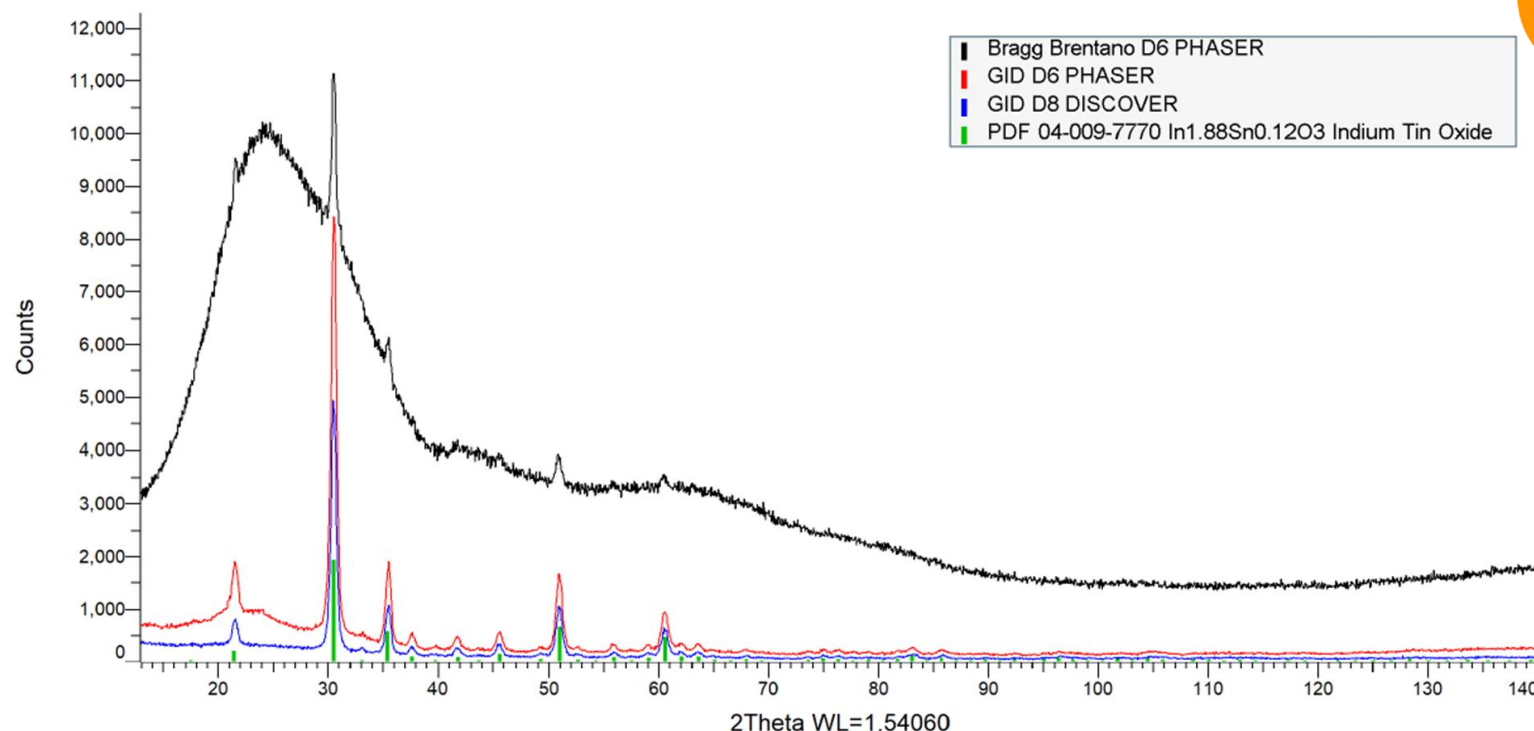
Grazing Incidence Diffraction (GID)

GID vs **Symmetric Scan**
ITO thin film on glass



VERSATILE. Thin-Film Analysis

Grating Incidence Diffraction (GID) for Phase Identification



The same sample (20 nm ITO on glass) was measured with the D6 PHASER (red, 50 μm slit) and the D8 DISCOVER (blue, GM with 0.1 mm slit) for separating the thin-film signal from the intense bulk material signal (black).



Both instruments efficiently separate the thin-film signal from the glass background

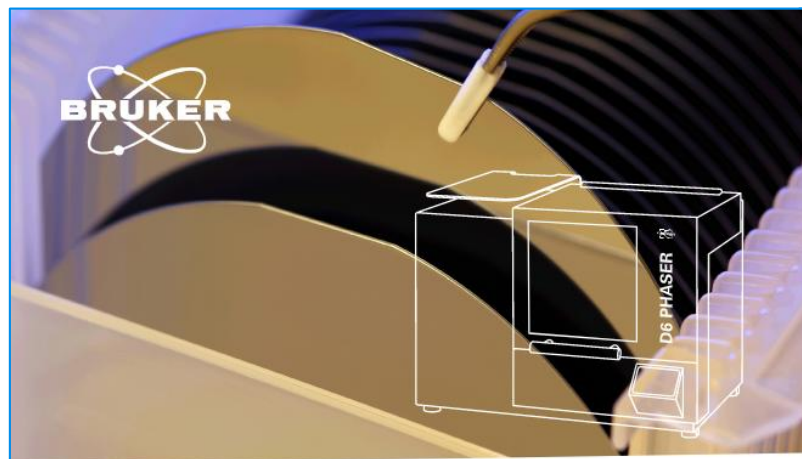
- Intensity of the D6 PHASER is about 2x stronger than for the floor standing unit
- The separation is better for the D8 DISCOVER due to the superior parallelization of the beam for a Göbel mirror compared to double-slit collimation

THIN FILM ANALYSIS

Grazing Incidence Diffraction



- 20 nm Indium Tin Oxide (ITO) layer on glass
- Comparison of symmetric and asymmetric diffraction geometry
- Control of the angle of incidence to minimize the glass substrate signal
- 20 nm W layer on silicon
- GID measurement up to high angle to enable residual stress evaluation
- Demonstration of the need to account for residual stress to properly refine GID data



X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD

Grazing Incidence Diffraction (GID)

Application Report 46

Grazing incidence diffraction (GID) is a method for studying polycrystalline thin films. These samples usually show very weak diffraction signal arising from the thin film in Bragg-Brentano symmetric diffraction because the X-rays pass through the thin film into the underlying substrate, which dominates the resulting signal due to its larger scattering volume.

If, however, instead of the divergent beam path of the Bragg-Brentano geometry, a thin parallel beam is directed at the sample surface so that it penetrates only the thin film but not the underlying substrate, the resulting signal is dominant. By optimizing the angle of incidence of the parallel beam, it is possible to decouple the scattering signal of the layer from that of the substrate (Figure 1).

Double slit collimation is used to produce a parallel beam in the D6 PHASER. The line focus of the tube combined with a tightly closed primary divergence slit is sufficient to collimate a suitably parallel beam. Alternatively, a combination of a tightly closed variable primary slit and a second pluggable slit can be used. By using the universal stage (with or without the optional sample rotation (Figure 2)), the angle of incidence of the parallel beam with respect to the sample surface can be held fixed while a detector scan is performed. An equatorial Soller collimator is used on the secondary side. It limits the angular resolution and the flux at the detector. The resulting data can be used for phase identification, as well as residual stress analysis and its depth variation controlled by the angle of incidence.

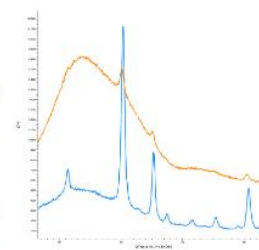


Figure 1
DIFFRAC.EVA view of a 20 nm indium tin oxide (ITO) layer deposited on a glass substrate. The symmetric theta/theta scan (orange) shows a signal dominated by the glass substrate, while the ITO signal is nicely emphasized in the 0.3° fixed angle GID scan (blue).

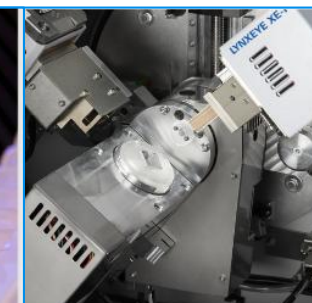


Figure 2
D6 PHASER equipped for GID with the variable divergence slit module, universal stage and a secondary side equatorial Soller collimator mounted on the LYNXEYE XE-T detector.

In asymmetric geometries such as GID, unlike symmetric geometries, the diffraction planes do not have the same orientation during data acquisition. The normal to the planes is close to the surface normal at low diffraction angles and approaches the in-plane direction at high diffraction angles. This allows analysis of the lattice deformation due to the strain exerted on the film by the substrate. Note that the observation and accuracy of the residual stress depends strongly on the ability of the diffractometer to acquire data up to high angles.

Figure 3 shows the evaluation of a GID scan performed on a 20 nm tungsten film and acquired in 40 min up to 140°. The thickness of the layer was also accurately measured with the D6 PHASER, as reported elsewhere (see Application Report 45). Tungsten is being considered as a replacement for copper as a metallization material in semiconductor devices with film thicknesses of less than tens of nanometers. Depending on the growth conditions, such confinement can result in significant residual stresses that can lead to undesirable effects (e.g., delamination). It is therefore critical to estimate the stress, which in some cases can be more than a tenth of a GPa.

The evaluation is based on a decomposition of the entire powder pattern using a cubic 1m-3m space group phase and additional angular shifts related to (i) the refraction effect of X-rays at the sample surface and (ii) lattice strain inducing an angular dependent peak shift expressed by elastic theory (multiple $\sin^2\psi$ method). The refinement is performed in DIFFRAC.TOPAS and shows a significant compressive residual stress in the tungsten layer of -2.4 GPa. The D6 PHASER and DIFFRAC.TOPAS are a great combination to go beyond qualitative phase analysis and provide quantitative structural information in thin film analysis.

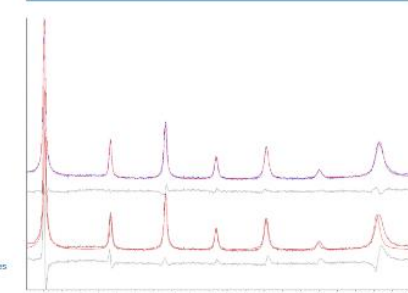


Figure 3
GID data from a 20 nm tungsten layer refined with DIFFRAC.TOPAS. The addition of residual stress in the model for the W phase (top pattern) significantly improves the fit quality as observed on the difference plots.

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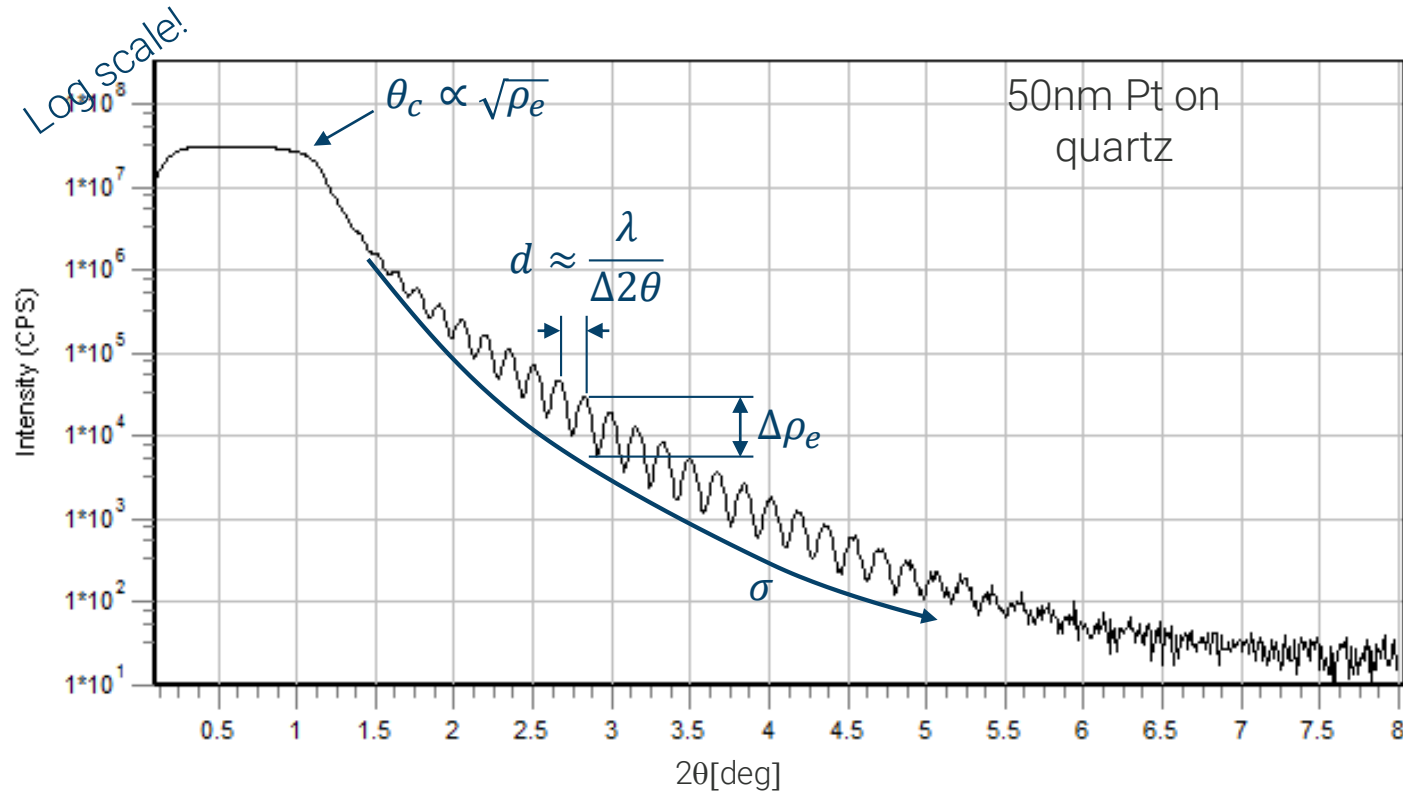


Online information
bruker.com/d6phaser



THIN FILM ANALYSIS

XRR Principle



- Layer thickness < 1 nm to > 200 nm
- Material density < 1-2%
- Roughness of surfaces and **buried interfaces** < 3-5 nm

THIN FILM ANALYSIS

Components for XRR

Primary side advanced optics

- **Motorized** variable divergence slit closed to 0.04 deg
- Typically no axial Soller slit
- 0,2 mm or 0,1 mm or no Cu absorber

Universal stage with **vacuum finger**

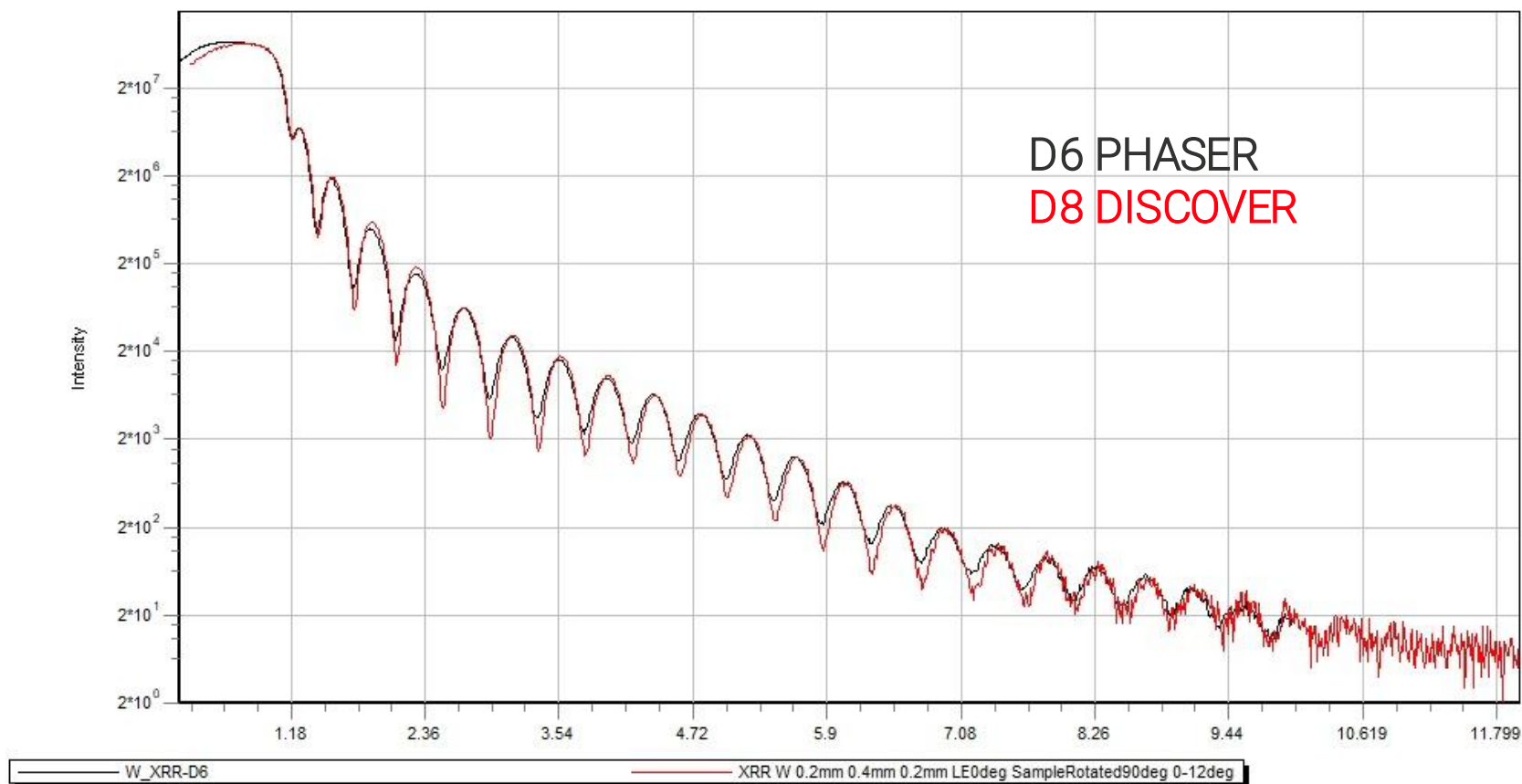
Telescopic slit shaft

- 0,1 mm antiscatter slit
- Typically no axial Soller slit
- **LYNXEYE** in **0D mode**, 1 channel



THIN FILM ANALYSIS

Reliable XRR data



THIN FILM ANALYSIS

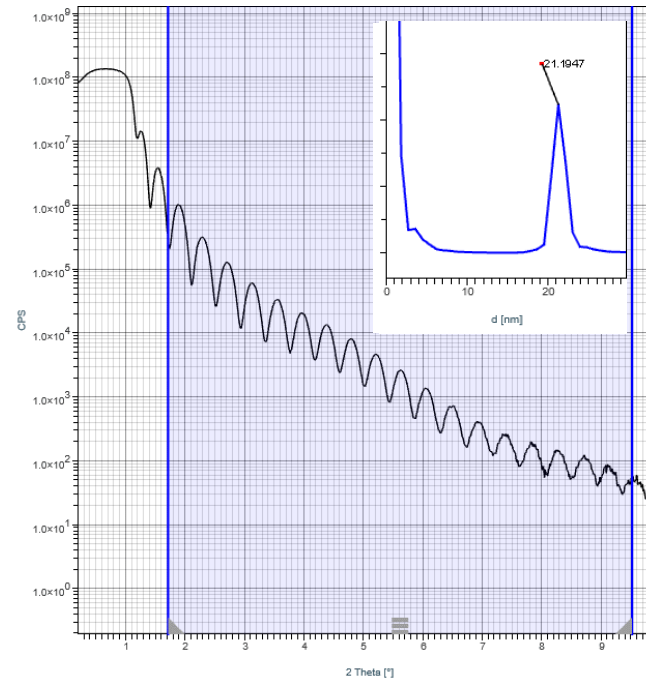
X-ray Reflectometry (XRR) Data

Measurement Geometry

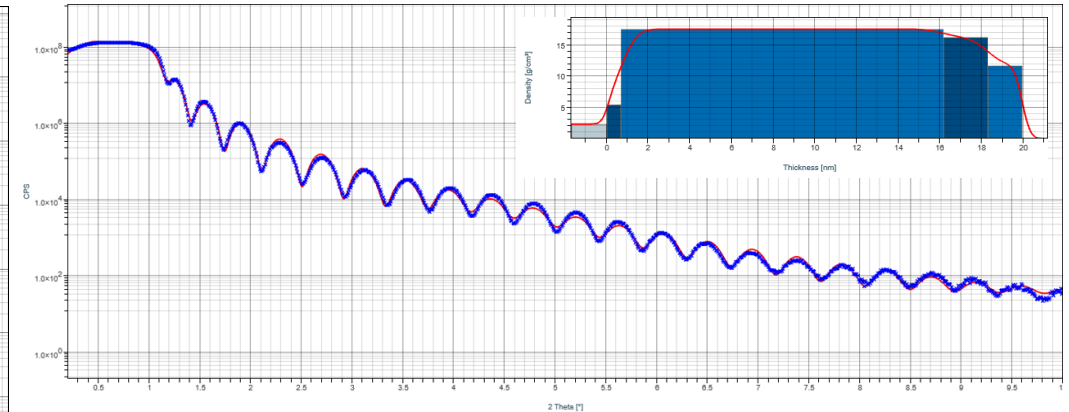
- Low incident and exit angle
- Sample height and rocking offset determined using the [Universal Stage](#)
- Scan collected from $\sim 0-10^\circ 2\theta$
- Multiple scans collected with different Cu absorbers






Analysis in DIFFRAC.XRR

- Data is imported and auto-merged into a single data set
- [One button FFT](#) analysis reveals film thickness
- [Fitting](#) allows determination of [thickness](#), [density](#) and [roughness](#)



[One button FFT](#) analysis of XRR data collected on a W film. The inset shows the result of the analysis.



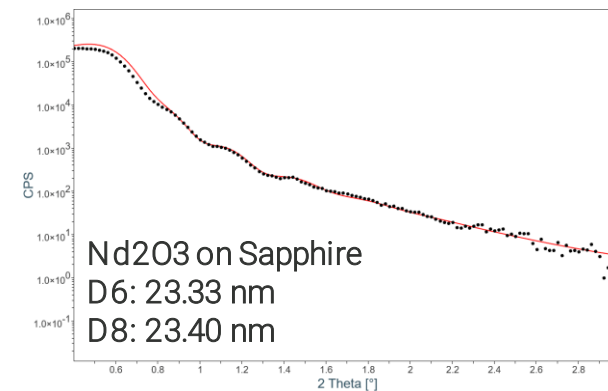
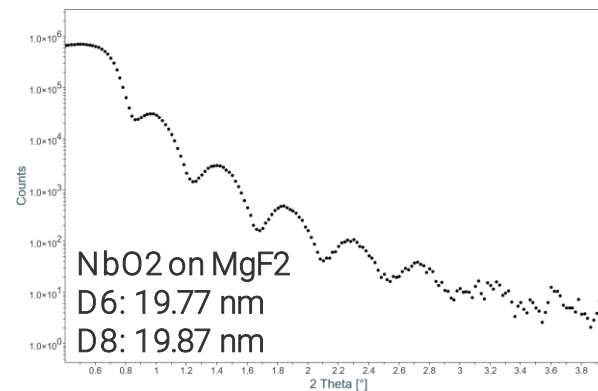
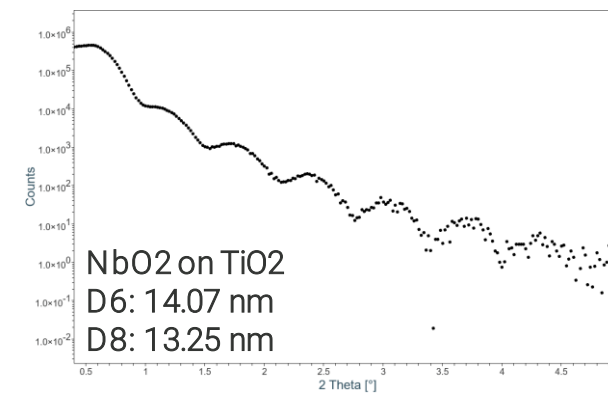
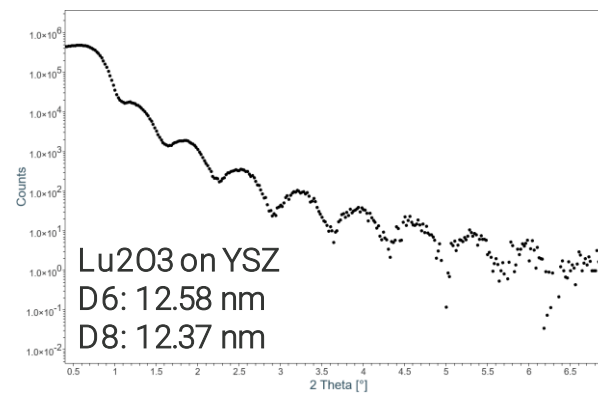
Material	Thickness [nm]	Density [g/cm ³]	Roughness [nm]	Color
W	1.6454	11.7224	0.3089	
W	2.1329	16.2913	0.6588	
W	15.4774	17.6108	0.6668	
SiO2	0.6875	5.4498	0.5957	
Si	∞	2.329	0.2564	

[Fitting](#) analysis of W film on Si. The resulting structure is seen in the inset while the table contains the [thickness](#), [density](#) and [roughness](#) of the film sub-regions.

D6 PHASER – Case Study

Dedicated film thickness analysis

- High end thin film growth lab specializing in functional oxides, typically 10-30 nm thickness.
- Extensive experience with XRD, looking for simple solution to check film thickness.
- Push button method including sample alignment and measurement.
- Measurement time of 5 minutes.



THIN FILM ANALYSIS

X-Ray Reflectometry - XRR

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD X-ray Reflectometry

Application Report 45

X-rays have a wavelength on the order of a fraction of a nanometer, making them very useful for the interferential analysis of films in the sub-nanometer to 100 - nanometer range. In an X-ray Reflectometry (XRR) experiment the x-ray beam illuminates the sample surface at a very low angle of incidence. At angles near zero, the beam is totally reflected by the surface resulting in a plateau of signal. From a material-specific angle, the incident beam penetrates the surface. This value is known as the critical angle and is proportional to the $\sqrt{\rho}$ of the material. Once the beam penetrates, internal layer-interfaces are illuminated, resulting in additional critical angle effects and reflections. The internal reflections as well as the surface reflection produce an interference pattern in the specular signal. Fringe spacing is related to the thickness of layers in the coating while signal persistence is related to the roughness of the interfacial layers.

In this application note we demonstrate XRR analysis with the D6 PHASER on a thin tungsten film.

For XRR, the recommended configuration is the advanced primary optic to create a thin, parallel beam, and the universal stage for adjustment of the sample surface normal and positioning in the center of the instrument. Samples can be placed on the spring stage holder or held fast with the vacuum finger. Any LYNXEYE Family detector used in high count rate mode is sufficient for analysis. The telescopic slit shaft allows positioning of a slit close to the sample, increasing resolution while also lowering background.

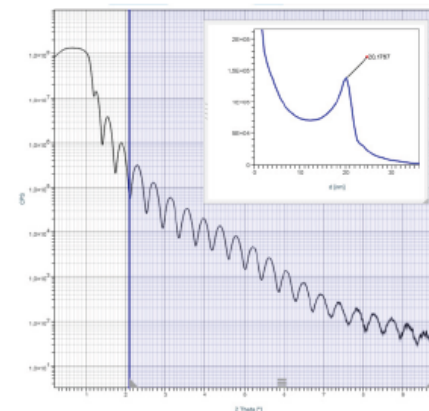


Figure 1
FFT analysis in DIFFRAC.XRR of the W thin film.

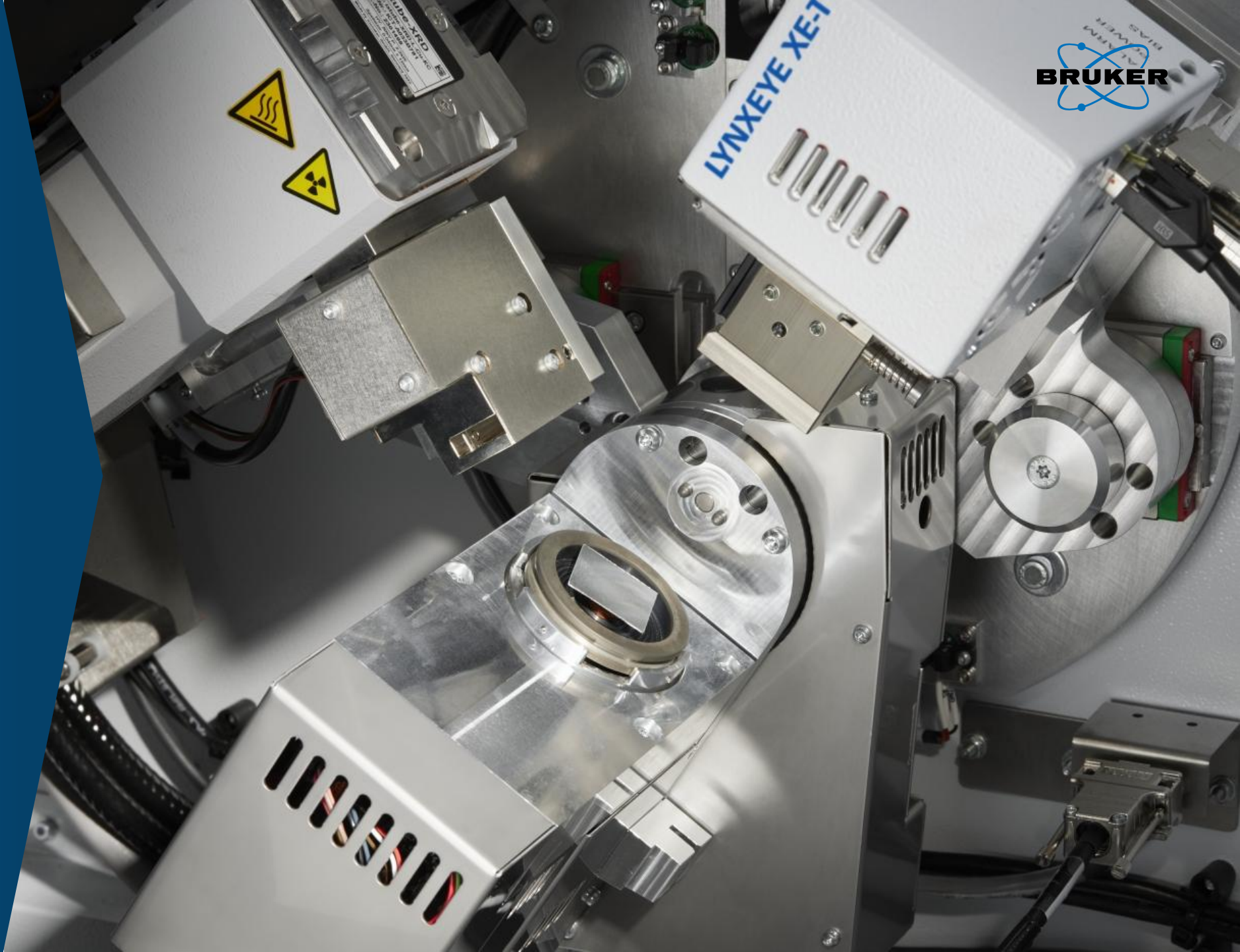
D6 PHASER - THE BENCHTOP PLATFORM



Residual Stress & Texture Analysis



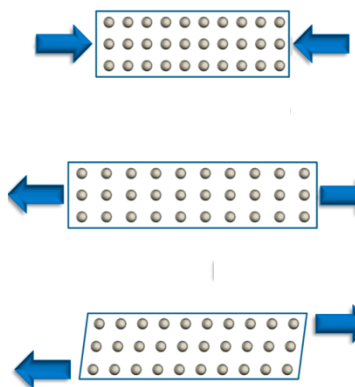
Generator 1200W
X-ray Tube Cu or Cr
Incident Optic Advanced Optic with Axial Soller
Sample Stage Universal Stage with Phi Attachment
Air Scatter Control None
Diffracted Beam Optic Axial Soller
Detector LYNXEYE XE-T



Residual Stress

What is it?

- Mechanical Stress



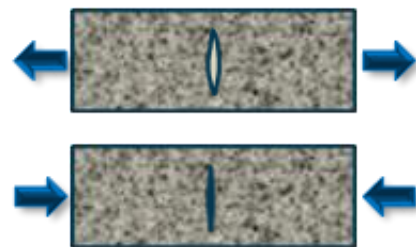
- Compressive

- Tensile

- Shear

- Residual Stress

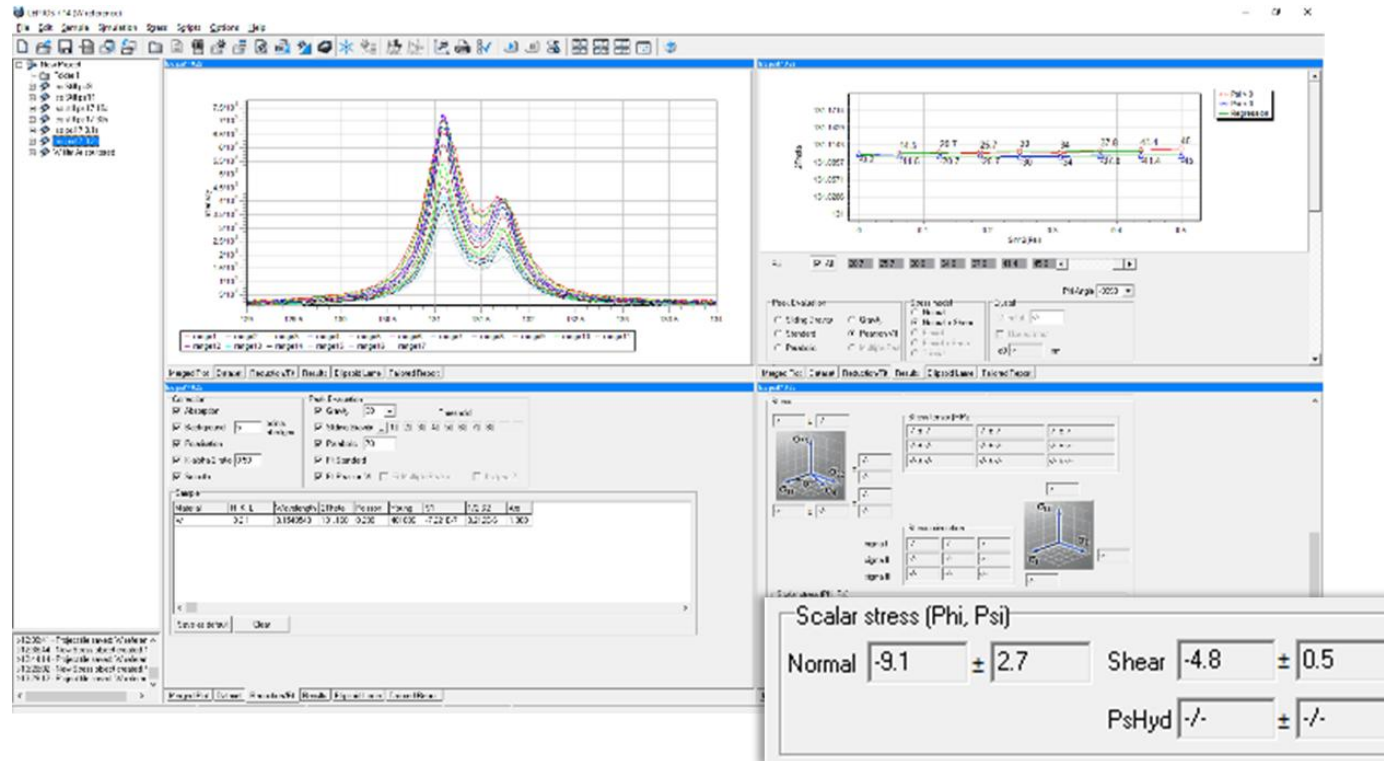
Residual stresses are stresses that remain in a solid material after the original cause of the stresses has been removed.



- **Tensile** residual stress
Opens crack and increases crack propagation
- **Compressive** residual stress
Closes crack and slows crack propagation

Residual Stress

Stress in Iso-inclination Mode



Same setup of the measurements in WIZARD and evaluation in LEPTOS as for the D8s

Intensity and resolution are no limiting factors of a D6 PHASER over D8s for Stress analysis

The capability of the D6 for stress analysis is proven by showing norm compliance with EN15305

A stress-free tungsten sample is shown to have stress below 31 MPa

Residual Stress Analysis



- Stress-free sample (W-Powder)
Results according EN 15305
- W Thin Film
(one sputtered under Ar-atmosphere the other under Kr-atmosphere)
- Residual Stress Analysis using the Iso-Inclination method is fully implemented



X-RAY DIFFRACTION D6 PHASER – Benchtop Residual Stress Analysis

Application Report 40

The D6 PHASER is a multipurpose benchtop diffractometer that is uniquely suited for modern materials research characterization. In this report, we present the capabilities of this system in a reflection diffraction configuration for residual stress analysis.

Residual stress is the localized stress that remains in a material after it has undergone processes such as welding, casting, forming, machining, or thin film deposition. The analysis of residual stresses is important to understand how these stresses affect the performance and lifetime of a component. Additionally, the residual stress can be used to identify specific material properties and failure mechanisms which can be used in the design of components and parts.

Tungsten layers are components of thin-film transistors in TFT-LCD screens. They are used when large screen formats, high image definition, and optimized contrast are required. Tungsten is also used in microelectronics, for example for creating layers in frequency filters. Other applications for tungsten includes diffusion barriers made of tungsten-nitride, conductor tracks in microelectronic components as well as reactively sputtered transparent layers made from tungsten oxide for OLED displays and for use in electrochemistry. In this study the residual stress of a tungsten layer created by PVD under Ar atmosphere and one sputtered at Kr atmosphere is analyzed. The thickness of the films is in the range of 200nm.



Figure 1
Universal stage with spring sample holder



The $\{321\}$ reflection of tungsten was chosen due to its high angle, $131^\circ 2\theta$ for Cu radiation, resulting in high sensitivity to d-spacing changes. For the ISO-inclination method 17 psi steps were chosen between -45° and $+45^\circ$ with a constant step size in $\sin^2\psi$.

The Cu source was operated at 40kV/30mA, while the divergence was controlled with a 0.2mm slit and a 2.5° axial Soller. The LYNXEYE XE-T detector was used in high count rate mode with a 5° detector opening. Additionally, a 0.2mm Ni filter was positioned in the beam path. The Universal stage was selected to mount the sample. The measurement was planned using the WIZARD plugin of DIFFRACT. MEASUREMENT.

The diffractometer is verified for residual stress measurements based on the EN15305 by measuring a stress-free sample. According to this norm the equipment is certified if a stress-free tungsten specimen gives a normal stress smaller than $\pm 31\text{MPa}$ with an uncertainty of $\pm 31\text{MPa}$ and a shear stress smaller than $\pm 15.6\text{MPa}$ with uncertainty of 15.6MPa .

The stress-free sample was found to exhibit a normal stress of $-9.1 \pm 2.7\text{MPa}$ with a shear stress of $-4.8 \pm 0.5\text{MPa}$. The film deposited in a krypton atmosphere exhibits a strong compressive stress of -1.3GPa with a minimal shear stress of -8.1MPa while the film deposited under an Ar atmosphere pushed the stress to -2.0GPa with shear stress of -20MPa .

Figure 2
Measurement geometry for residual stress measurements in the D6 PHASER. Shown is the optional phi attachment which enables biaxial stress measurements.

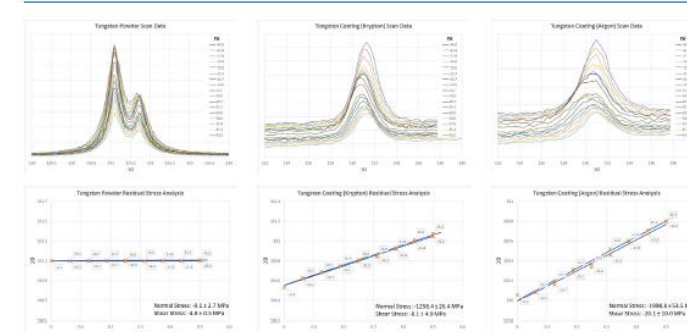


Figure 3
Raw scan data (Top) and $\sin^2\psi$ analysis (Bottom) of stress-free tungsten powder (Left) and two tungsten coatings deposited under krypton (Middle) and argon (Right) atmospheres.

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Online information
bruker.com/d6phaser

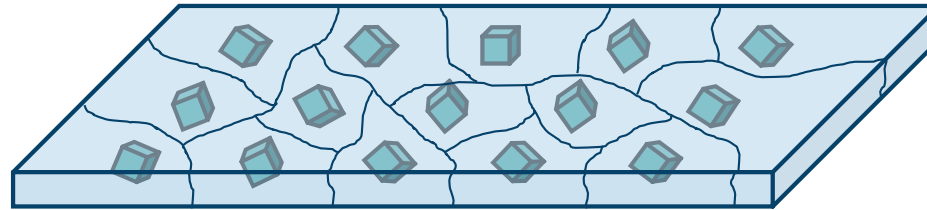


Texture Analysis

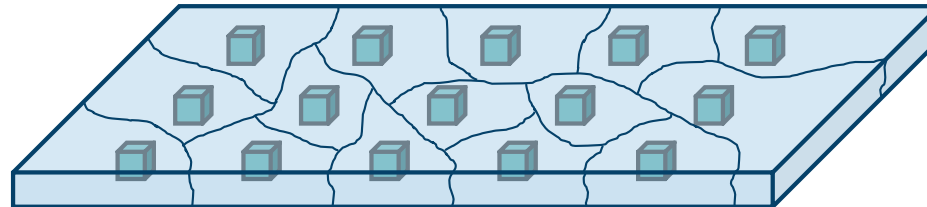
What is Crystallographic Texture?

- Texture deals with grain orientations (Crystallite orientations) in a polycrystalline material
- Polycrystalline material consists of a large number of small crystallites
- Each of these crystallites has a specific orientation of the crystal lattice.

- **Non-textured** sample
(crystallites are completely random, like in an ideal powder)



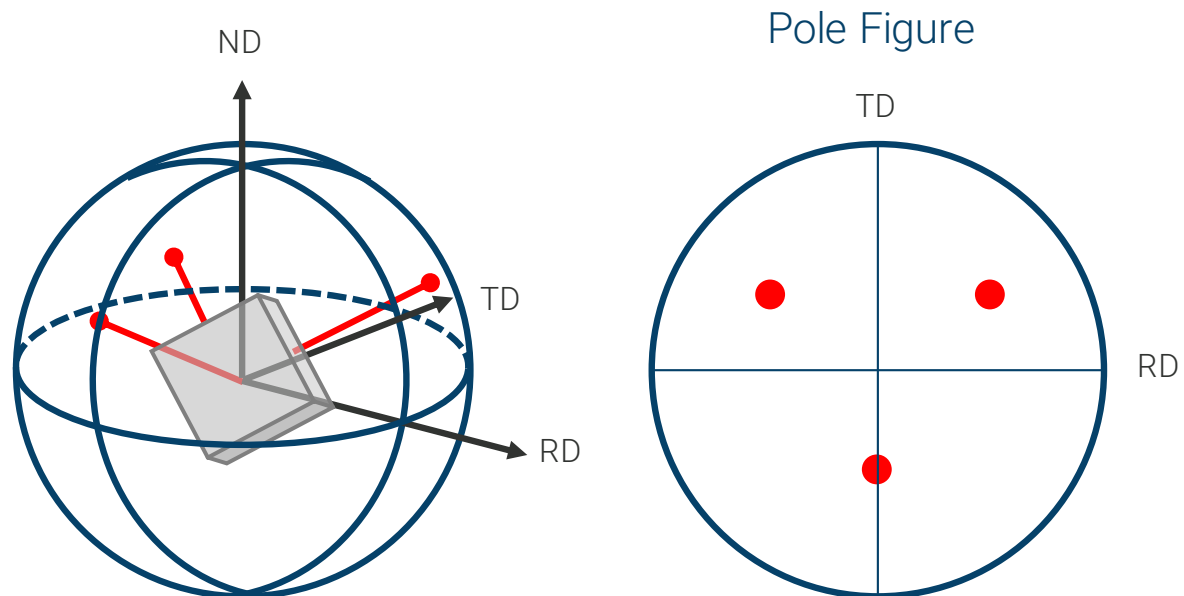
- **Strong textured** sample
(crystallites arranged like a single crystal)



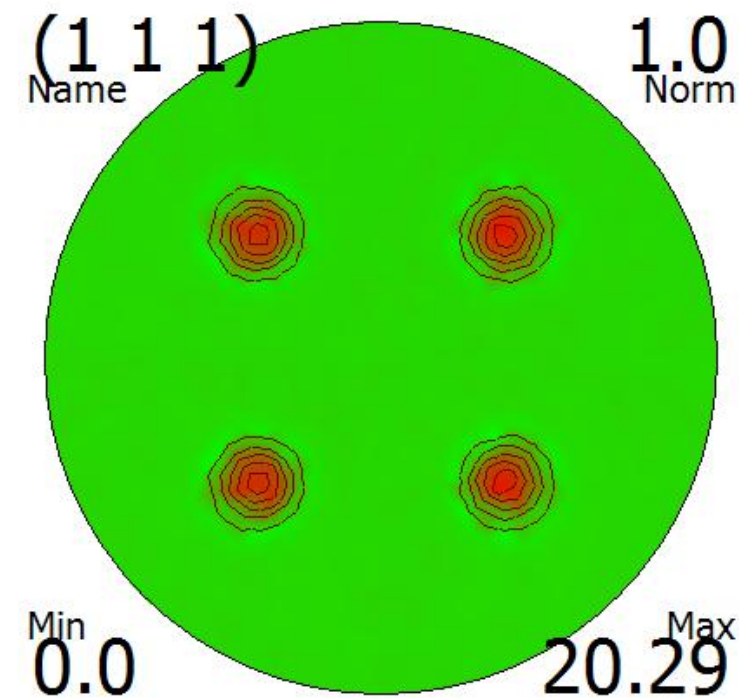
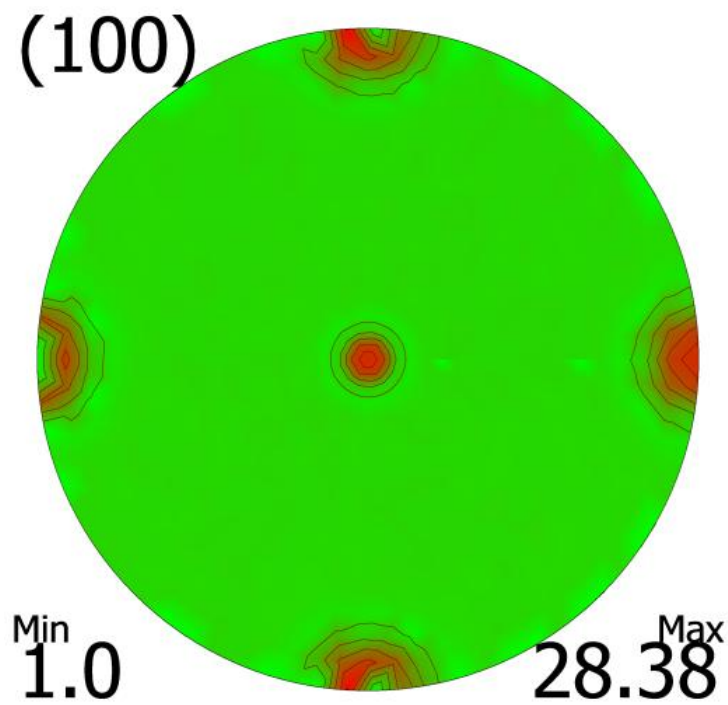
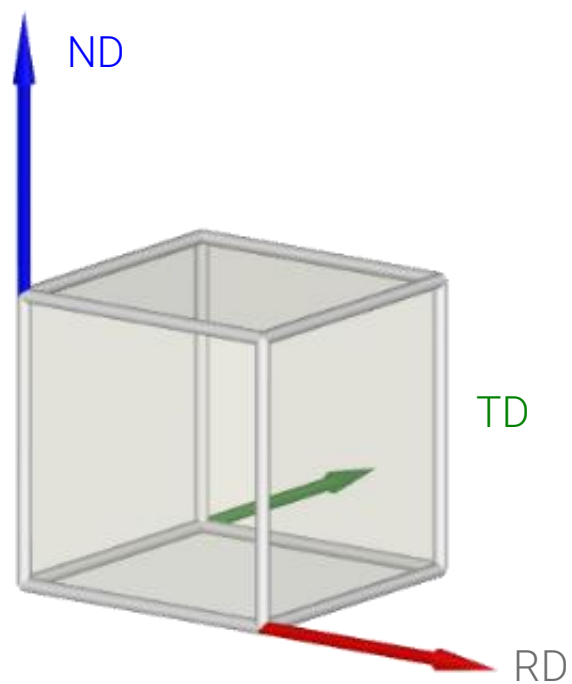
Definition

Pole Figure

- If we consider a lattice plane, the orientation of the plane can be given by its normal vector.
- If we draw a sphere with the center on the plane, then the intersection of the normal line and the sphere is the pole.



Pole Figure Example



TEXTURE ANALYSIS with D6

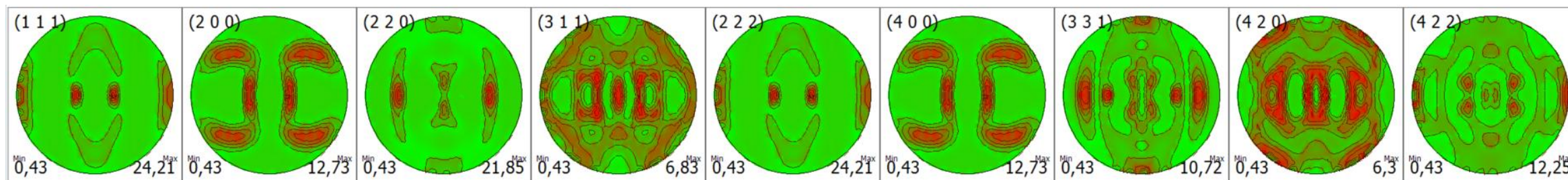
Rolled Al Sheet Metal



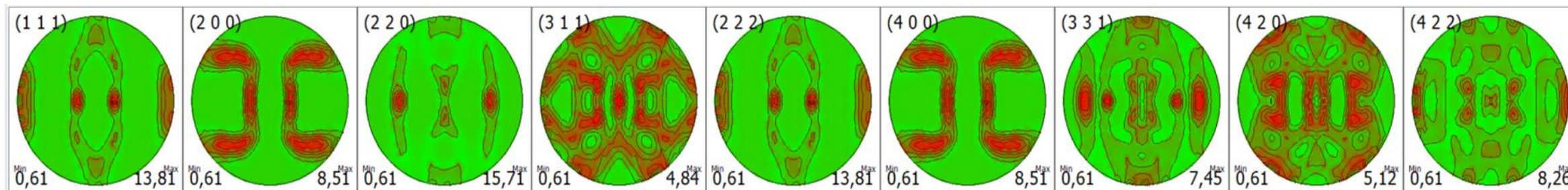
- Texture Analysis using high angle reflections yield to same texture results compared to traditional data collection (with cradle)
- D6 requires flat samples
 - Beam size can be controlled with fixed sample illumination
 - Beam height can be controlled with height limiting slits
- Large sample averaging due to larger beam size compared to point focus texture.
- Constant sample illumination (even at high tilt angles)

Recalculated Pole Figures

D6 Phaser



D8 ADVANCE / D8 DISCOVER



Texture Analysis



- Rolled Al sheet metal
- Short data collection on high angle Pole Figures
- Short interpretation on the results
- Texture Analysis can be performed using the Iso-Inclination method



X-RAY DIFFRACTION D6 PHASER – Benchtop Texture Analysis

Application Report 41

In addition to its atomic structure, the macroscopic morphology of a material significantly contributes to its properties. One of these properties is the degree of orientation of the crystallites which make up the sample. When the crystallites are strongly aligned, mechanical and electrical properties can be amplified. But at the same time, this alignment can ease fracture propagation leading to premature failure. The correct orientation, or texture, in a sample is essential in engineering it for a particular application.

As X-ray diffraction is sensitive to a material's atomic structure, it can be used to measure its degree of orientation. A perfect powder exhibits a completely random, or isotropic, texture. Most metal samples exhibit some degree of orientation due to mechanical deformation during processing. Coatings can exhibit exceptionally high degrees of orientation, even to the point of being a perfect single crystal.

To measure texture, a system capable of tilting the sample relative to the bisecting "Bragg" condition, when the incident and diffracted angle are equal, is required. In addition, the sample must then be rotated with intensity being collected as a function of rotation angle. This is accomplished in the D6 PHASER with the universal stage and phi rotation module. This stage can accommodate samples mounted in standard 51 mm holders or samples mounted in standard metallographic polishing mounts. In this application report, we present the results from a measurement collected on a piece of aluminum.

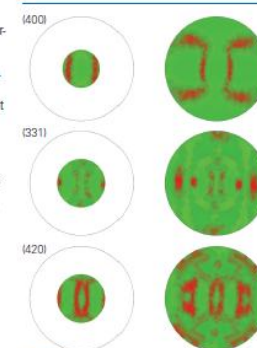


Figure 1
As measured and recalculated (400), (331) and (420) pole figures for the Al sample



Figure 2
Universal stage equipped with phi attachment

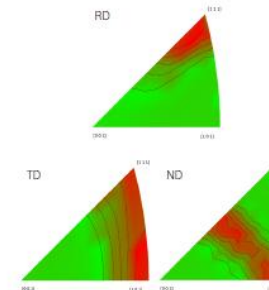


Figure 3
Inverse pole figures for the Al sample

Measurement planning was carried out in the WIZARD. Using the graphic user interface, a series of phi scans were carried out for 3 λ reflections; (400), (331) and (420). The 3 pole figures were collected in approximately 30 minutes.

The data was then imported into DIFFRAC.TEXTURE where the phi scans were converted into pole figures, shown in Figure 1. This process occurs automatically using metadata from the measurements. Corrections for absorption related to the iso-inclination method are applied automatically. Auto-Indexing of the pole figures was carried out using the included materials database.

Fitting of the pole figures was accomplished with the component method. In this case, a single elliptical component with a monoclinic process symmetry was employed. Figure 1 shows the resulting recalculated pole figures. By having access to all 3 pole figures and using crystallographic symmetry, the peripheral areas are filled in. Figure 3 shows the resulting recalculated pole figures for the RD, TD and ND directions. A strong (111) texture is observed in rolling direction, with a (101) to (112) fiber observed in the normal direction and (111) to (101) fiber in the transverse direction. For a more comprehensive picture, the orientation distribution function, ODF, is shown in Figure 4.

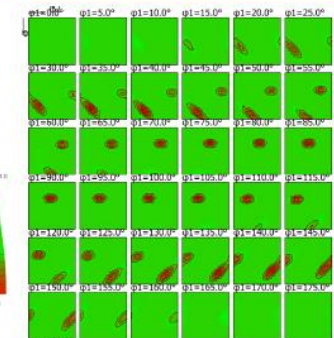


Figure 4
Orientation Distribution Function (ODF) for the Al sample

Bruker AXS
info.baxs@bruker.com

bruker.com

Worldwide offices
bruker.com/baxs-offices



Online information
bruker.com/d6phaser

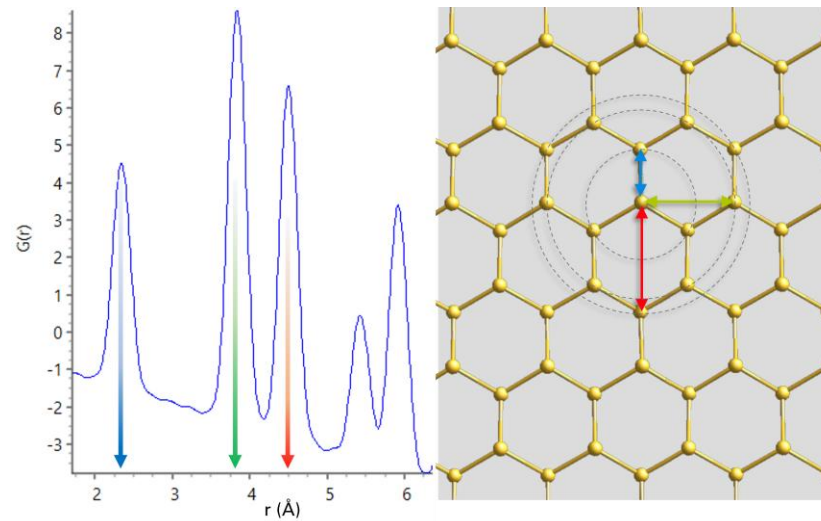


03 – Hard Radiation – PDF and Batteries

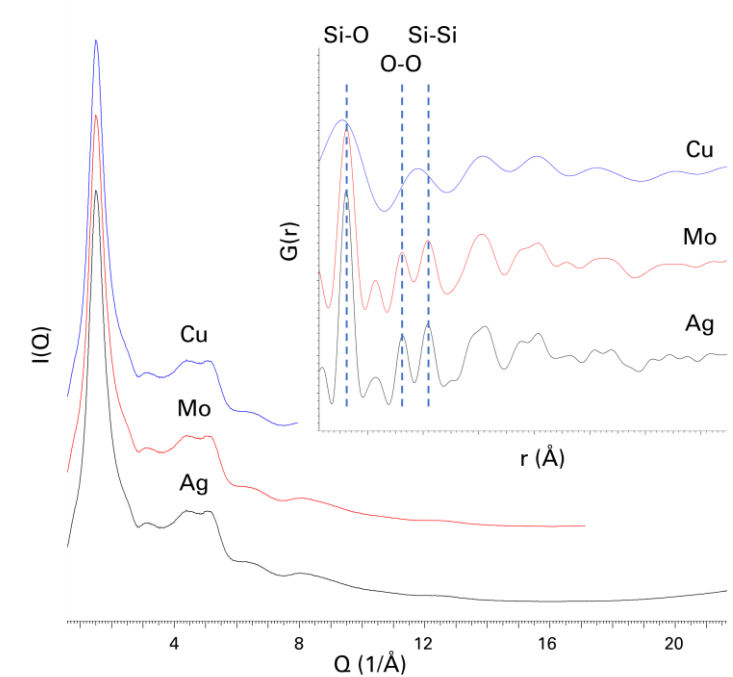
What is PDF Analysis?



- PDF analysis is an analytical technique that can provide **local structural information** from:
 - Amorphous materials
 - Nanocrystalline materials
 - Disordered materials
- Requires high energy X-rays (**Mo or Ag source**)

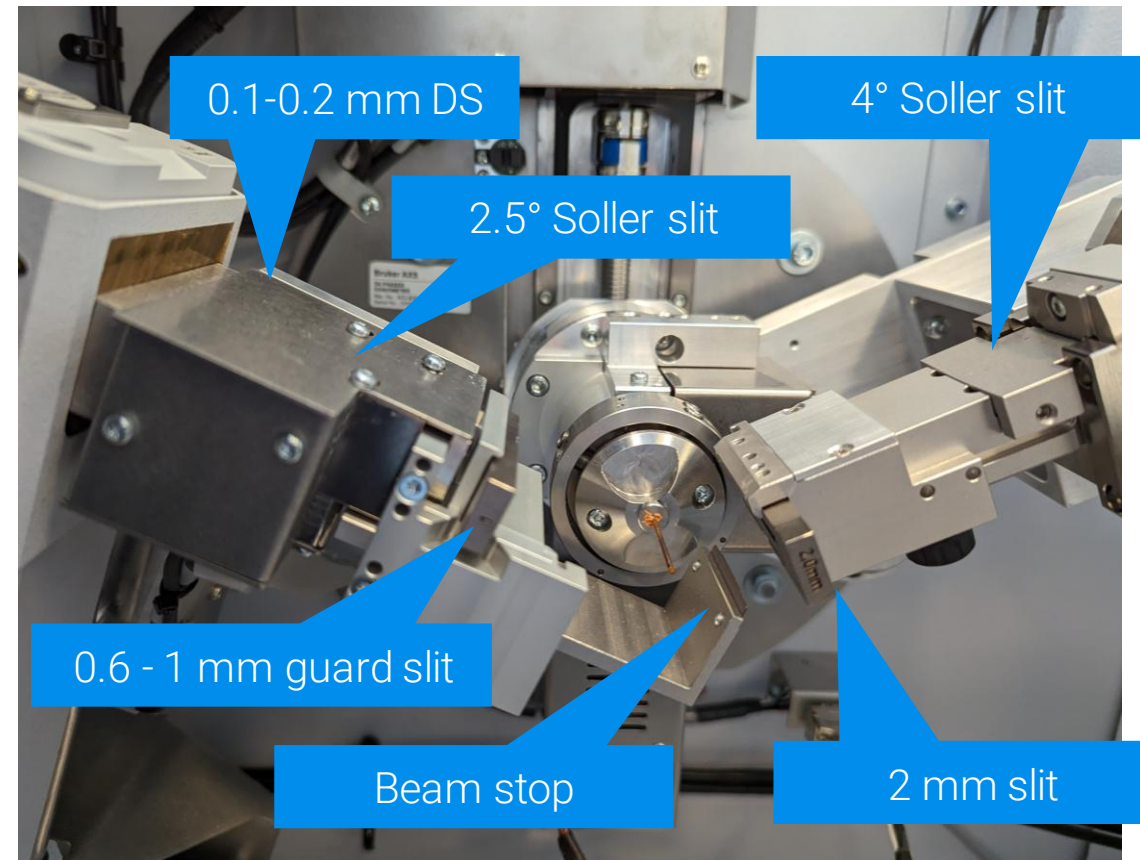
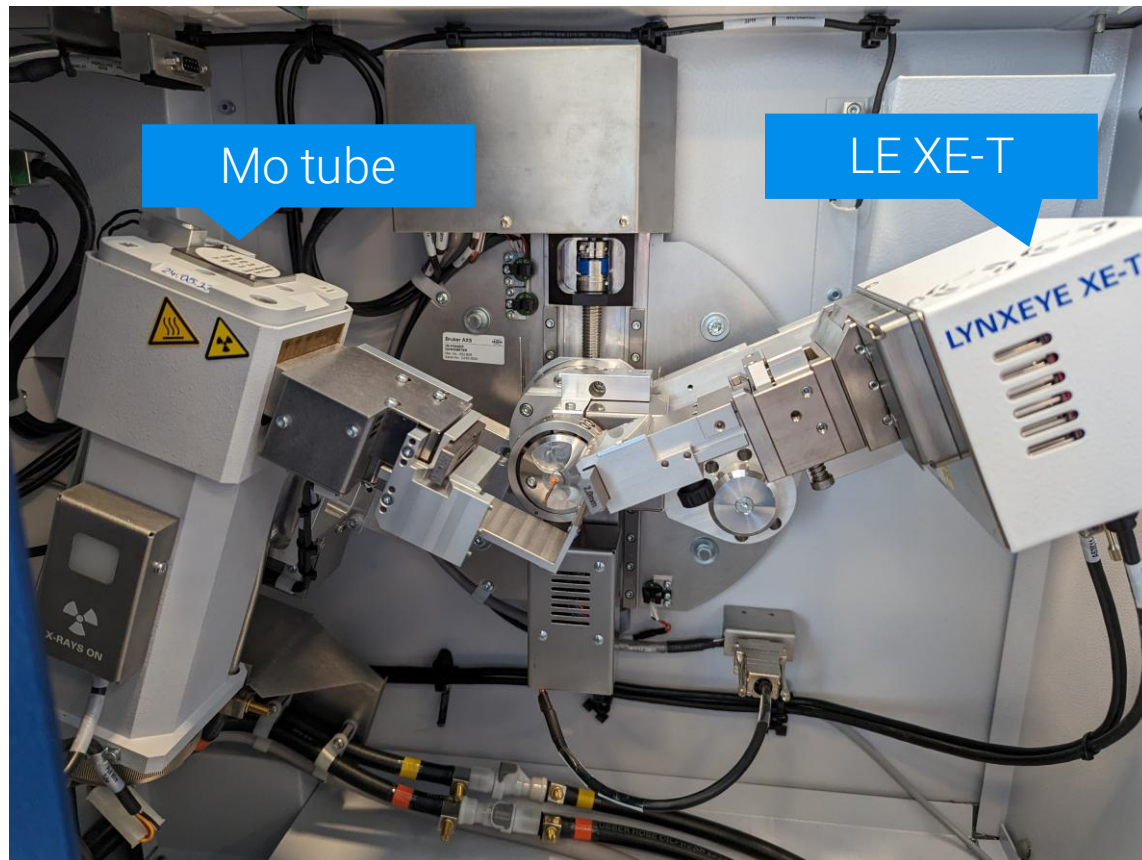


Schematic representation of a PDF



Accessible Q-range using different X-ray anodes, highlighting importance of high energy sources for PDF analysis

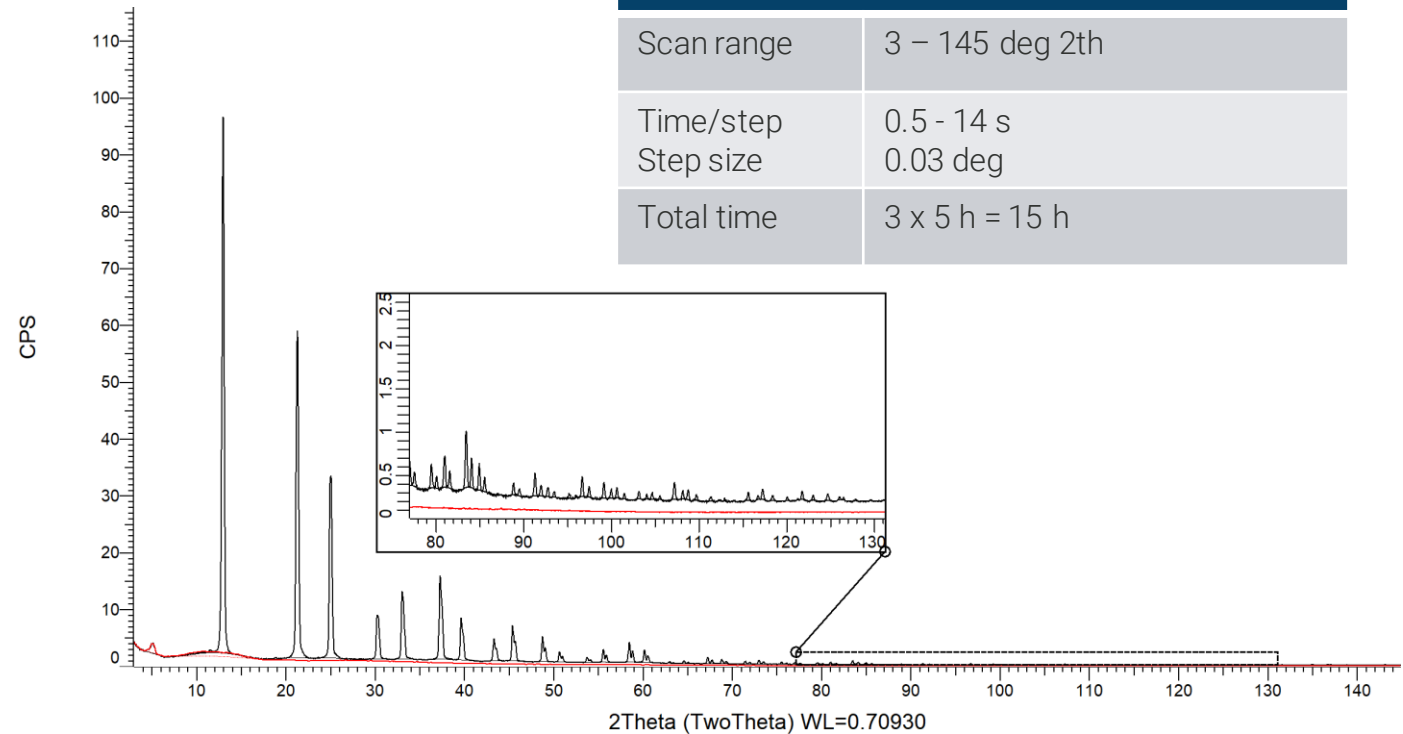
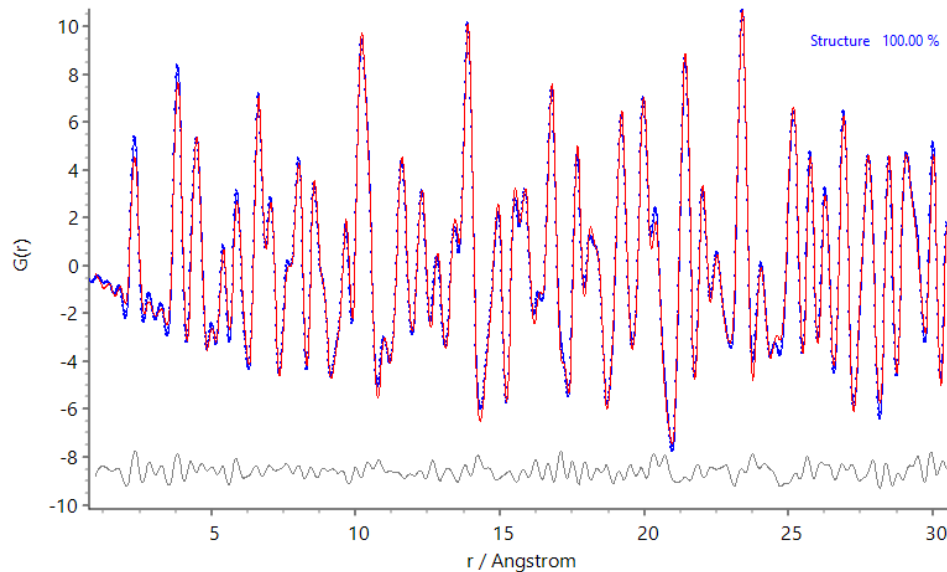
D6 PHASER with Capillary Stage Instrument Configuration



D6 PHASER with Capillary Stage

PDF Analysis – Mo tube

- Si powder in 0.5mm capillary
 - Standard material, good for validating instrument setup
- PDF processing using DIFFRAC.EVA V7



Measurement Conditions

Scan range	3 – 145 deg 2th
Time/step	0.5 - 14 s
Step size	0.03 deg
Total time	3 x 5 h = 15 h

D6 PHASER with Capillary Stage

PDF Analysis – Mo tube

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD Total Scattering Analysis

Application Report 44

The D6 PHASER is a multipurpose benchtop diffractometer that is uniquely suited for modern materials research characterization. In this report, we present the capabilities of this system in capillary geometry for total scattering analysis.

Total scattering, also known as pair distribution function (PDF) analysis, demands high intensities and low background to observe weak diffuse scattering features in the diffraction pattern. The D6 PHASER diffractometer, equipped with a Mo source, 1200 W of power, and compact goniometer radius, combined with the excellent energy resolution of the LYNXEYE XE-T detector, offers a unique analytical solution for total scattering analysis in a benchtop instrument.

Low instrument backgrounds are not achieved through the detector electronics alone: careful removal of air scattering and other parasitic scattering features is also key to high quality PDF data. The D6 PHASER uses the guard slit holder with beam stop and telescopic detector optics shaft together with the capillary stage to ensure the lowest possible instrument backgrounds.

Fixing and aligning the capillary to the goniometer center can also feel like a hassle. The D6 PHASER addresses this through the use of high precision magnetic mounting and alignment-free capillary holders,

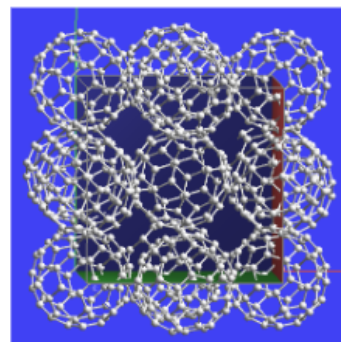
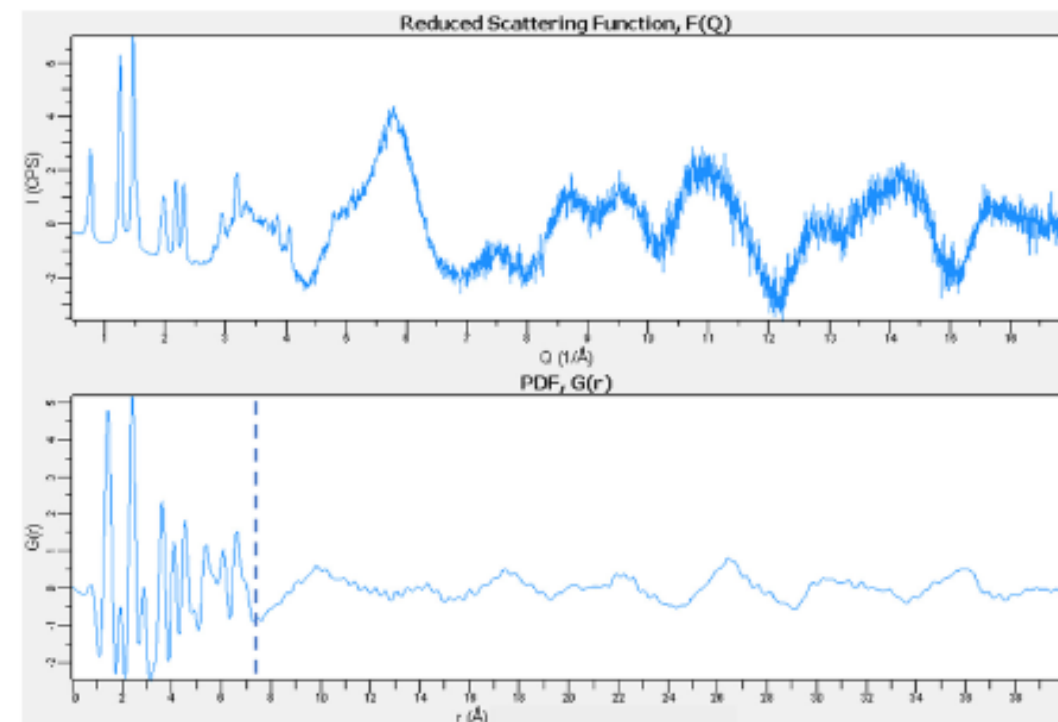


Figure 1
C₆₀ molecules, decorating the lattice points of a face-centered cubic (fcc) lattice.

Measurement Conditions

Scan range	3 – 145 deg 2 θ
Time/step	0.6 - 18 s
Step size	0.04 deg
Total time	3 x 5 h = 15 h

- C₆₀ powder in 1 mm Kapton capillary
 - Low absorbing, big capillary: ideal sample for D6P
- PDF processing using DIFFRAC.EVA V7



D8 Family – PDF analyses



1 X-ray Sources

Mo or Ag anodes deliver the short wavelengths required to achieve data with high Q_{max} for PDF analysis.



2 Primary Optics

Beam-path optics determine the instrument geometry and condition the beam for the best fit to the sample size.

Motorized Divergence Slits

- Straightforward solution for all wavelengths
- PDF data in reflection and transmission geometry

Focusing Goebel mirror

- Highest flux density, $K\alpha_{1,2}$ beam
- Fastest data collection in transmission

Johansson Monochromator

- Pure $K\alpha_1$ beam, no $K\alpha_2$ artifacts at higher r
- Best instrument resolution; minimal instrument dampening



X-RAY DIFFRACTION

Total Scattering Solutions PDF Analysis with the D8 Diffractometer Family

4 X-Ray Detectors

Large, efficient detectors reduce measurement time and can even eliminate unwanted fluorescence signal.



3 Sample Stage

The D8 accommodates a huge selection of ambient and non-ambient sample stages.

Reflection as well as foil transmission geometry:

- Rotating single sample holder, 9-positions sample changer, 90-positions sample changer, UMC150 HTS

Reflection geometry:

- Various XYZ stages and Eulerian cradles
- Non-ambient chambers covering the temperature range from -190 to 2300°C

Transmission geometry:

- Capillary stage (room temperature)
- Non-ambient capillary stages covering the temperature range from -190 to 1 000°C

D8 Advance with EIGER500K – PDF analyses



Lab Report XRD 90

PDF Analysis with D8 ADVANCE

- Rietveld and PDF refinements using Ag radiation and EIGER2 R 500K detector

This labreport describes a Pair Distribution Function (PDF) measurement of LiFePO_4 powder to investigate structural disorder using a D8 ADVANCE multipurpose diffractometer.

LiFePO_4 (LFP) is widely used as cathode material in commercial Li-ion batteries for its good cycling properties and excellent safety characteristics.

New synthesis routes are constantly developed to reduce the battery cost, and also to tune the structural properties since they are known to affect the electrochemical properties of LFP.

Combined PDF and Rietveld refinements are an effective tool to study local defects and help guide more effective synthesis routes.

Table 1. D8 ADVANCE configuration

Ag radiation (0.56 Å - 22.2 keV)

Focusing Goebel mirror, 1 mm exit slit, 4° primary Soller collimator

0.7 mm Kapton capillary

4° panoramic Soller collimator,
EIGER2 R 500K detector in 2 θ -optimized mode at 118 mm sample to detector distance

Variable counting time scan from 2 – 152° 2 θ
8 h total measurement time

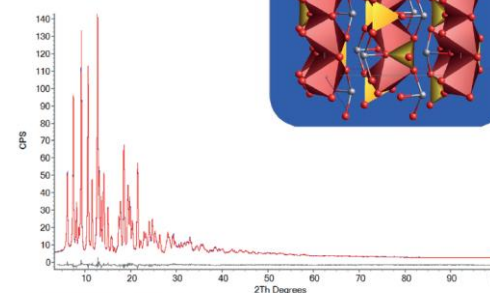


Figure 1: Rietveld structure refinement of LFP with DIFFRACTOPAS.

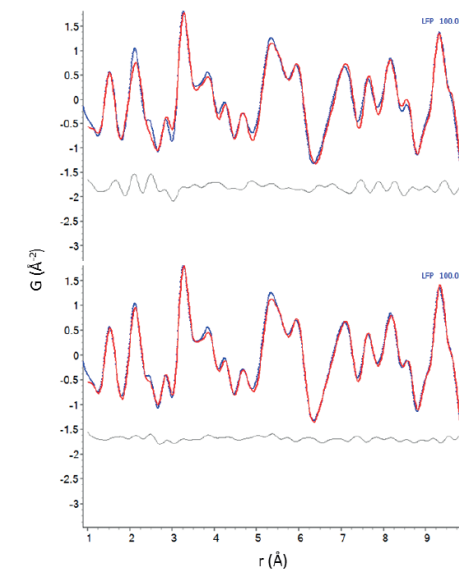
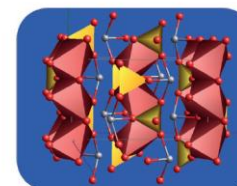


Figure 2: Structure refinements to the PDF data for LiFePO_4 using the average structure model (top) and the disordered model (bottom).

D8 Discover Plus with TXS – PDF analyses



Application Report XRD 31 D8 DISCOVER Plus

● PDF Analysis on Titanium Dioxide Nanoparticles

The D8 DISCOVER Plus equipped with the ATLAS™ goniometer and the high-efficiency turbo X-ray source (TXS-HE) is a diffraction solution designed to meet current and future analytical needs in research and production. In this report, its capabilities for the analysis of the pair distribution function (PDF) is presented.

TiO₂ is a widely used metal oxide due to its low cost, low toxicity and chemical inertness. The properties of TiO₂ are highly dependent upon, among others, the crystal structure and the particle size, especially for very small sizes < 10 nm. The conventional particle size analysis using Rietveld

structure analysis is limited to long range order, which obviously is not the case for nanoparticles.

The pair distribution function (PDF) describes the probability of finding two atoms separated by a distance r . The experimental PDF is determined directly from powder diffraction data making use of both Bragg and diffuse scattering intensities. The resulting reduced total scattering function $F(Q)$ finally is Fourier transformed. PDF data is displayed in real space and, in contrast to Rietveld analysis, allows for comparative structural investigations at small and intermediate distance scales.

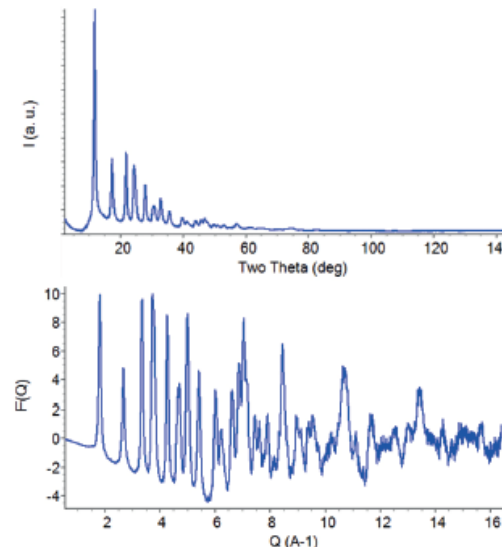
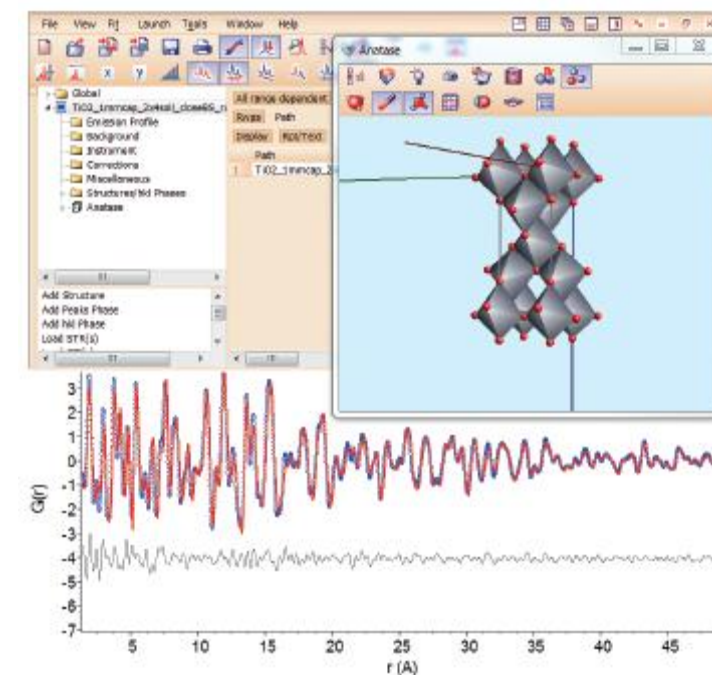


Figure 1: Raw diffraction data (top) and reduced structure function $F(Q)$ (bottom).



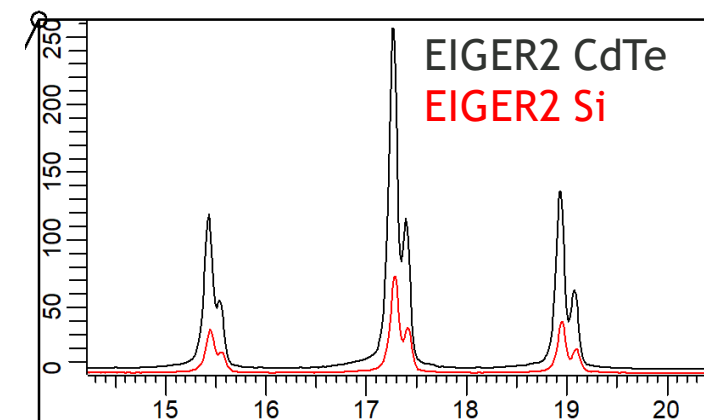
High Energy Solutions

EIGER2 R 500K CdTe for D8 DISCOVER

- Largest CdTe detector on the market for XRD lab systems
- **Fully integrated** for D8 DISCOVER, including all scanning modes
- Requires: water-cooling, dry air (included)



	EIGER2 R 500K Si	EIGER2 R 500K CdTe		
Sensor	450 μm Si	750 μm CdTe		
Active Area	2960 mm^2	2960 mm^2		
Efficiency (Mo)	47%	>99%		
Efficiency (Ag)	27%	>99%		



LaB₆ measured with Ag source
(~3.7x intensity compared to Si sensor)

Batteries – In Operando solutions



X-RAY DIFFRACTION

D6 PHASER – Operando XRD on a Benchtop

Application Report 49

Operando X-ray diffraction plays a crucial role in advancing our understanding of energy storage systems, aiming to enhance the efficiency and durability of batteries by providing valuable insights into dynamic processes such as phase transitions, lattice parameter variations, and other crystallographic transformations within the electrodes. In this report, we present the capabilities of the D6 PHASER for operando XRD analysis on a Li-ion battery pouch cell.

The pouch cell used in this experiment consisted of a single 139 μm thick layer of NMC622 ($\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$) coated on Al foil (15 μm), a separator (20 μm), and a graphite layer (46 μm) coated on Cu foil (10 μm), all packed in a polymer-Al composite bag.

The total thickness of the pouch cell was about 500 μm . Typically for diffraction experiments on pouch cells, shorter wavelength X-ray sources like a Mo tube that produce harder radiation are preferred to reduce the effects of X-ray absorption and to penetrate through the entire cell. However, for relatively thin pouch cells like this, a Cu source can also provide excellent results.

Two charge/discharge cycles were performed at a C/10 rate (10 h charge, 10 h discharge, 40 h total), controlled by a SP-50 potentiostat from BioLogic. The potentiostat is fully integrated into DIFFRAC.SUITE, meaning the entire experiment can be planned and controlled in DIFFRAC.WIZARD. Diffraction patterns between $15 - 62^\circ 2\theta$ were continuously collected during cycling.

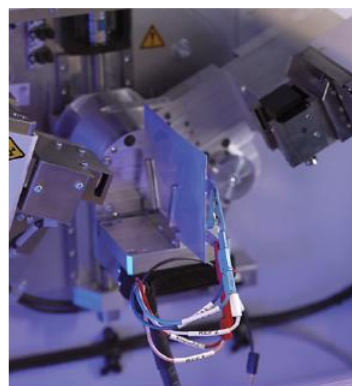


Figure 1
A pouch cell mounted in the D6 PHASER.



X-RAY DIFFRACTION

In Operando XRD of Battery Pouch Cells

Characterization of an NMC pouch cell with the EIGER2 R 500K

Pouch cells have become an industry standard battery design due to their efficient shape and lightweight construction. In operando measurements allow simultaneous monitoring of the cathode and anode for cycling effects which influence energy storage performance. This lab report describes in operando characterization of an NMC pouch cell using XRD with the D6 ADVANCE equipped with Mo radiation and the EIGER2 R 500K detector.

The pouch cell used in this experiment was made of a single NMC ($\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$) layer (67 μm) coated on Al foil (15 μm), a separator (40 μm), and graphite (82 μm) coated on Cu foil (9 μm). The electrodes were immersed in a LiPF_6 electrolyte solution, and packed in a polymer-Al composite bag.

The pouch cell was positioned in the center of the diffractometer, held by two clamps. The total thickness of the pouch cell was about 920 μm . Mo radiation rather than the more common Cu radiation was used for the transmission measurements to reduce the effects of X-ray absorption by the pouch cell. The thick Si sensor of the EIGER2 R 500K is well suited for wavelengths ranging from Cr to Mo, producing high signal while reducing the background by minimizing the effects of charge sharing.

Operando Battery Research

BioLogic SP-50 Single Channel Potentiostat

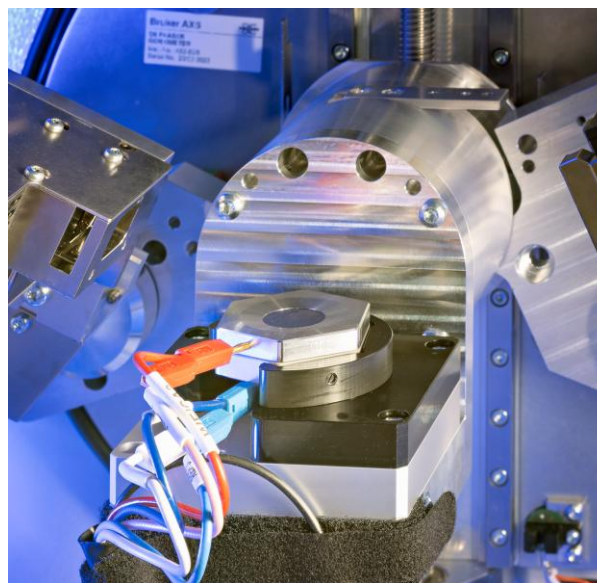
Fully integrated into DIFFRAC.SUITE

- ✓ Perform charge/discharge cycles synchronized with diffraction measurements.

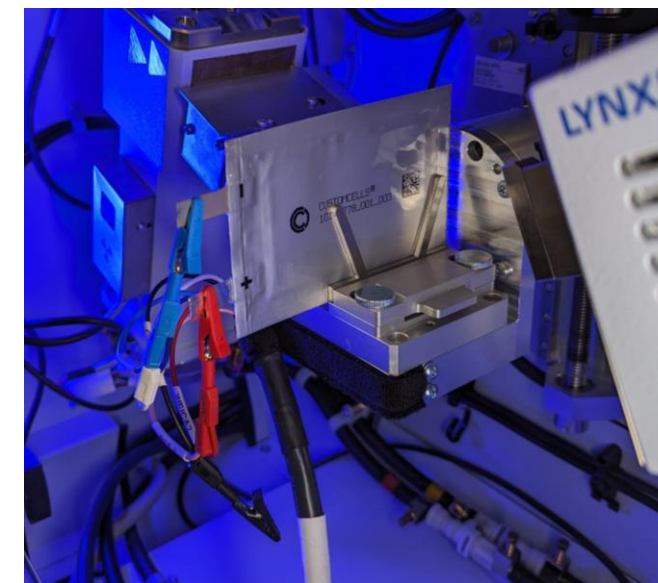


- Voltage Range: +/- 10V
- Current Range: +/- 800 mA

Two types of battery cell holder stage attachment:



- Reflection Battery Cell Holder

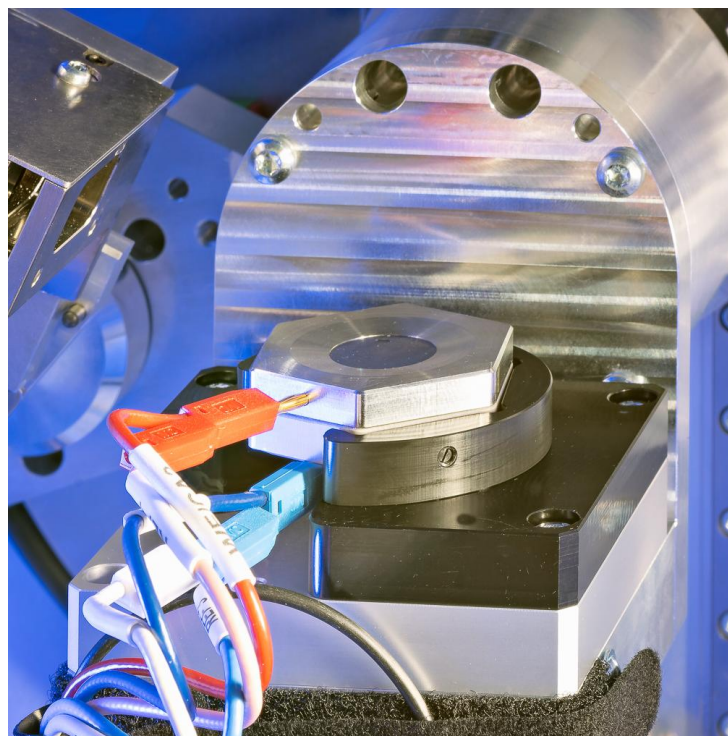


- Pouch Transmission Battery Cell Holder

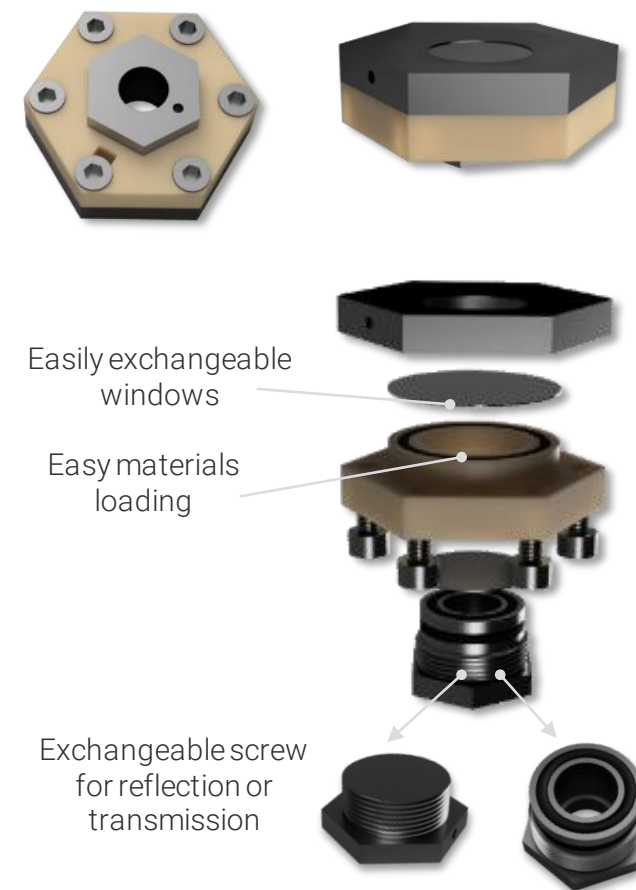
LeRiChe'S Battery Cell Type 2 – for *in situ* XRD

Features

- Designed for XRD by battery experts
 - ✓ Lightweight, compact design
 - ✓ Easy to assemble and use
 - ✓ Reliable electrochemical performance
- Reflection geometry
- Different window materials available



Cell designed by LRCS, Univ. Amiens (France)



Full Integration of Potentiostat SP-50 from BioLogic

Features

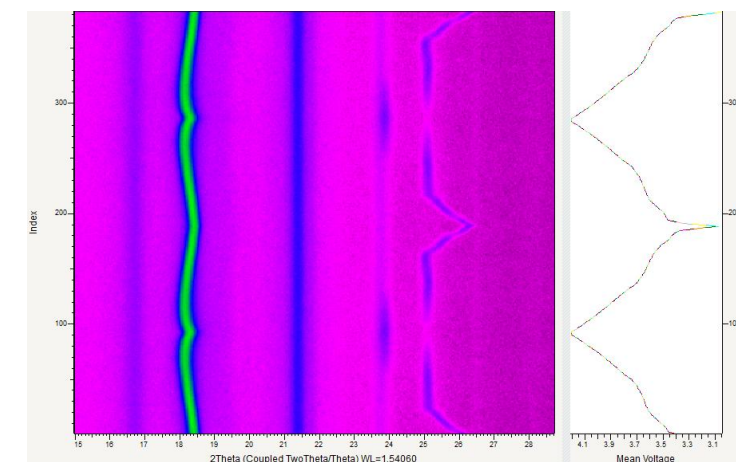
- Plan sequence of cycles and XRD measurements in the non-ambient tab of the WIZARD
- Measure at *defined* points of the battery's charge-discharge cycle
- Writing of the corresponding current & voltage parameters to the measurement result file (.brml)

Experiment Planning

Segment	Start time	Duration [s]	Operation
1	0:00:00.0	1.000	Prepare Experiment...
Initial State			
2	0:00:01.0	793.500	Open Circuit Voltage
- Measurement	Method#1 Coupled...	Start delay:	0.000
Charge			
3	0:13:14.5	3600.000	Constant Current
- Measurement	Method#2 PSD fixed	Start delay:	0.000
4	1:13:14.5	10.000	Constant Voltage
Discharge			
5	1:13:24.5	2500.000	Constant Current
- Measurement	Method#2 PSD fixed	Start delay:	0.000
6	1:55:04.5	10.000	Stop
→ end of segments			

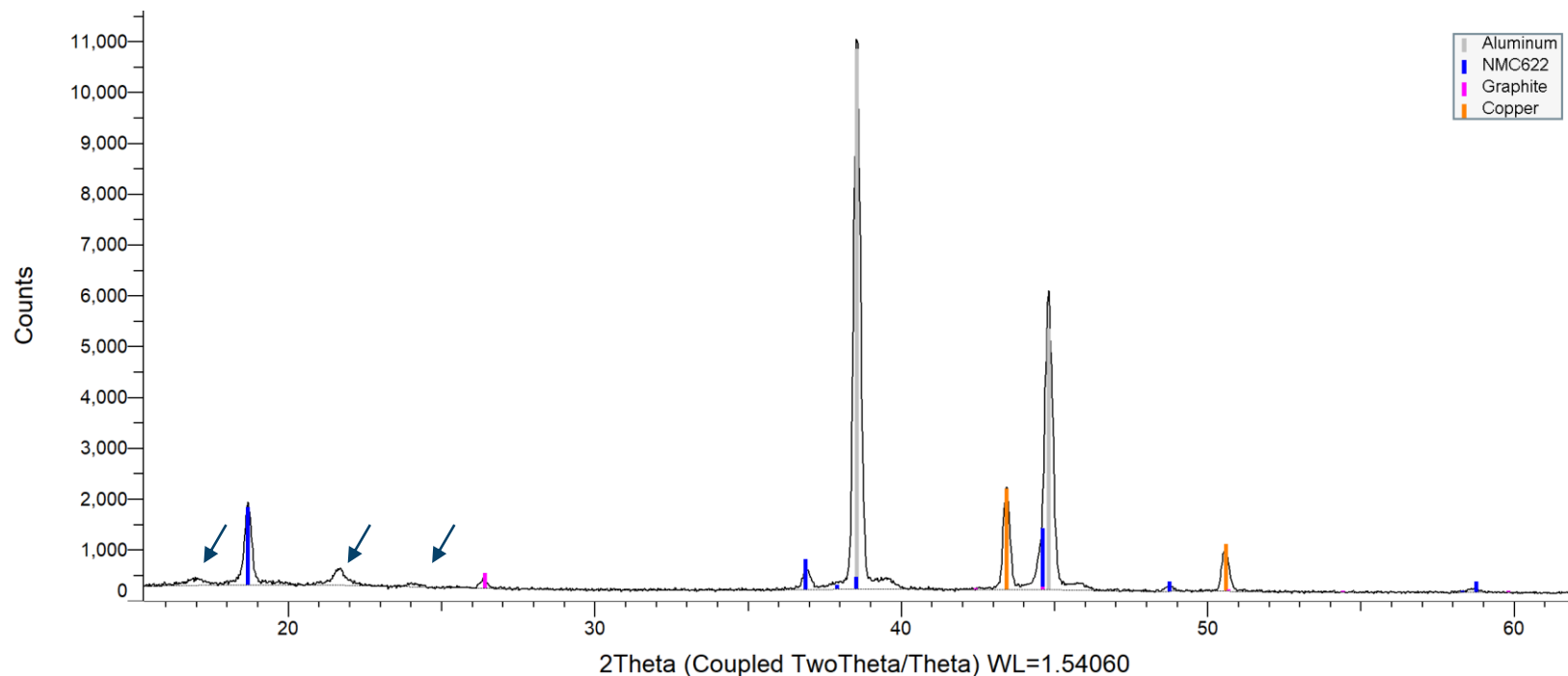
- Build modular experiments in WIZARD, combining XRD scans with different operation modes

Data Visualization



- Plot current/voltage parameters with 2D data view in DIFFRAC.EVA

Measurement Results and Phase ID



- High quality data in just **6 min** measurement time
- Discharged state: NMC622, graphite, Al, and Cu clearly identified
- Additional peaks from separator

Instrument Parameters

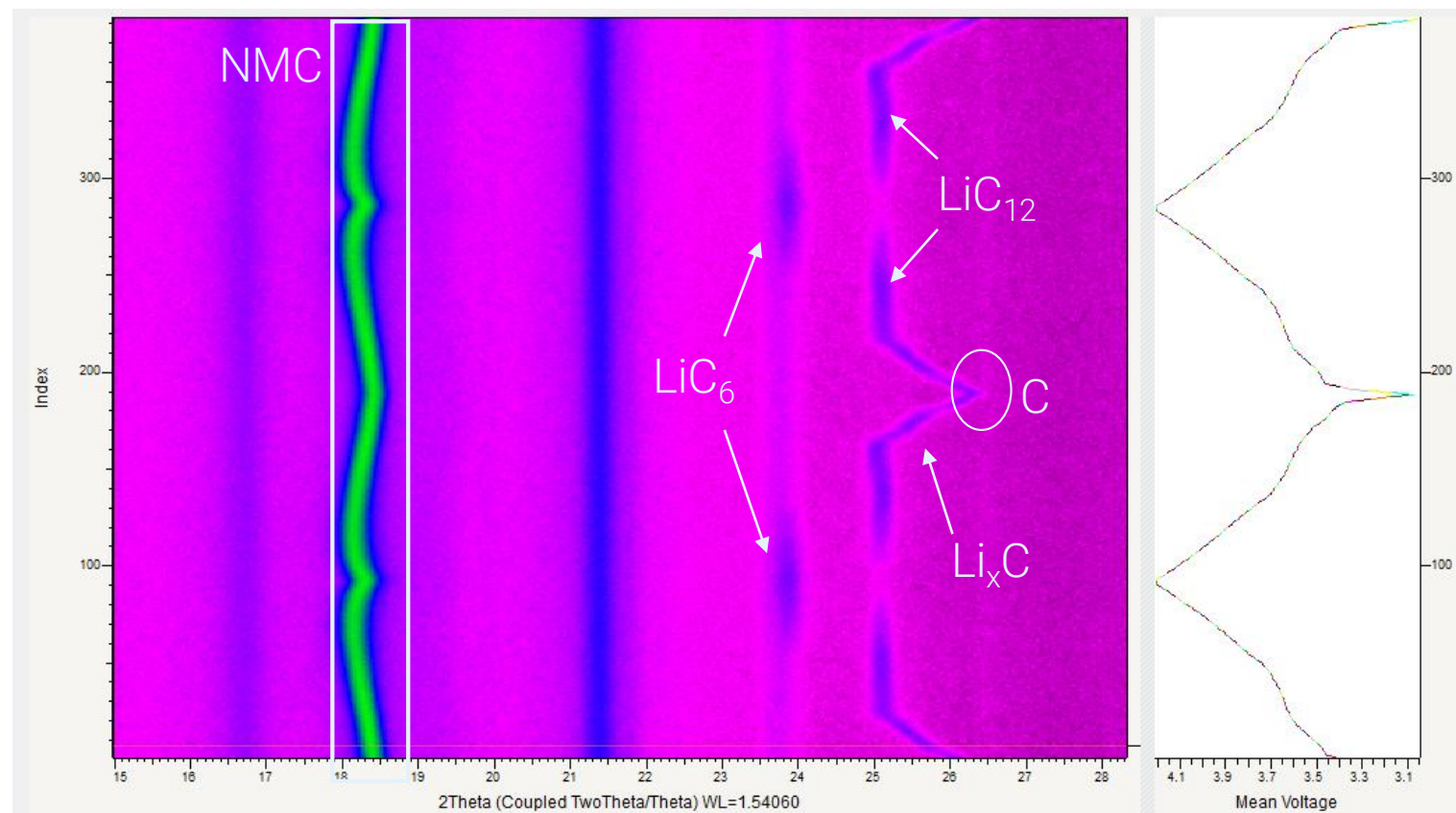
Source	Cu Sealed Tube (40 kV, 30 mA, 1200 W)
Prim. Optics	0.1 mm Fixed Slit 4° Soller Slit
Stage	Pouch Cell Holder
Sec. Optics	Telescopic Slit Shaft 4° Soller Slit
Detector	LYNXEYE XE-T (4.9°)

Measurement Parameters

Range	15 – 62°
Increment	0.03°
Time/Step	0.2 sec
Total Time	6 min per scan

In-Operando Data Visualization in DIFFRAC.EVA

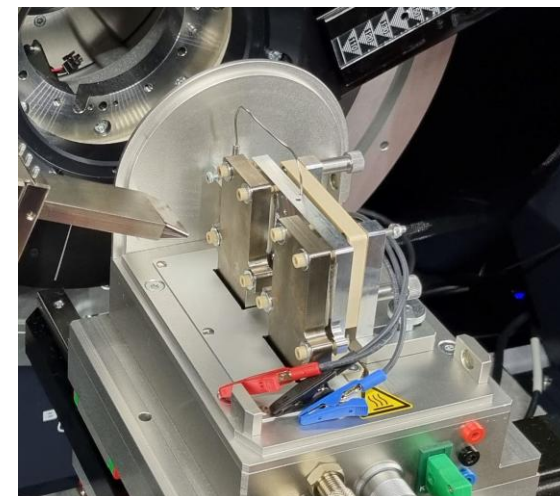
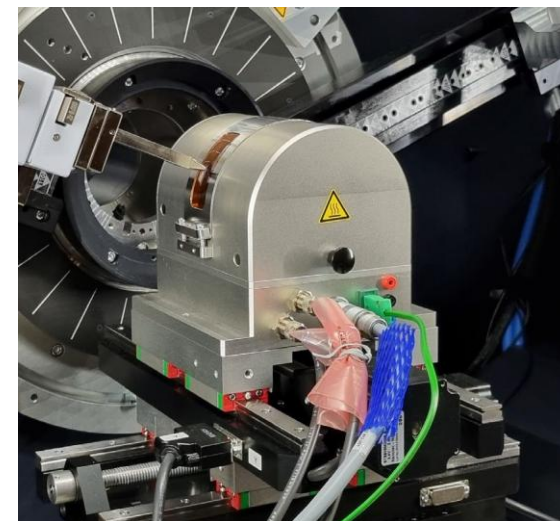
- 2 complete charge/discharge cycles using C/10 charge rate
- Visualize individual XRD datasets, or the complete experiment with **waterfall display** or **2D view**
- Plot current/voltage parameters alongside 2D view for easy identification of new species or transition points



Battery Solutions

Non-Ambient Operando Stage for Pouch Cells

- Available for D8 ADVANCE and D8 DISCOVER
 - Fits on [floor-standing UMC stages](#) for mapping on D8 DISCOVER
 - Fits on [modified Compact UMC stage](#) (extended Z-range to fit stage) for D8 ADVANCE and D8 DISCOVER
- Pouch cells up to Width 120 mm x Height 80 mm
 - Larger pouch cells can be mounted without cover
- Peltier heating/cooling, temperature range: -10°C to +80°C
 - Temperatures below RT require dry air (not included) to avoid condensation

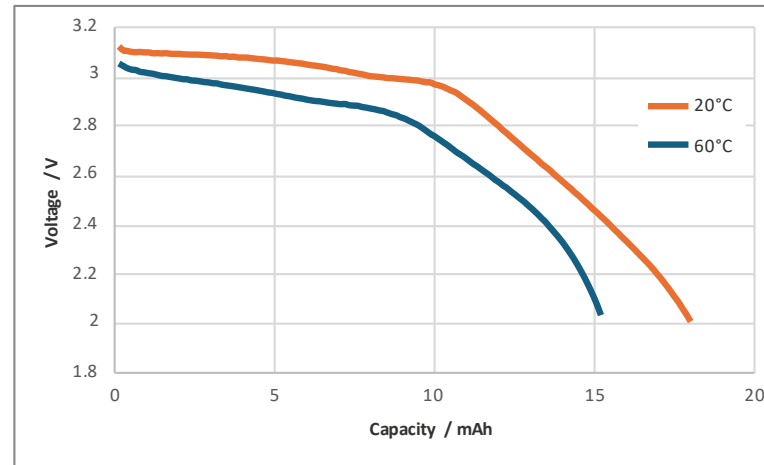


Non-Ambient Operando Stage for Pouch Cells

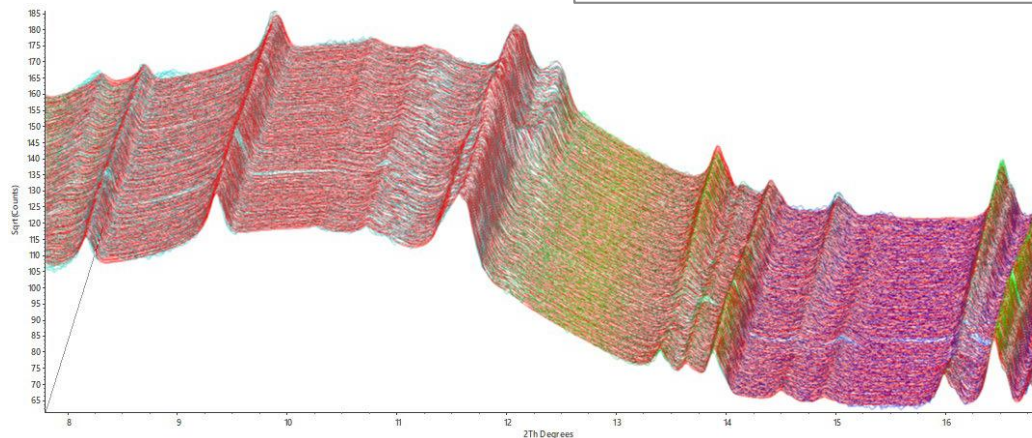
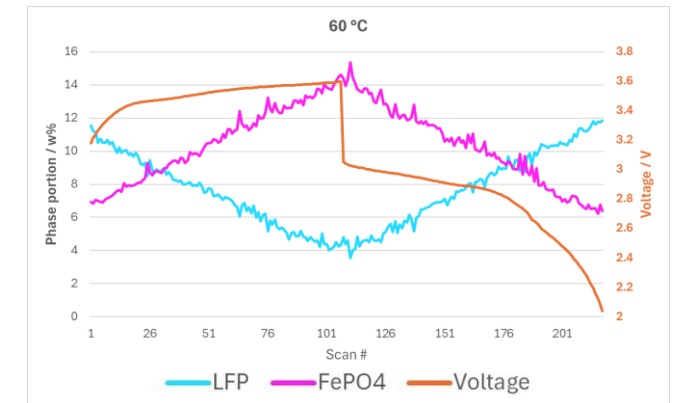
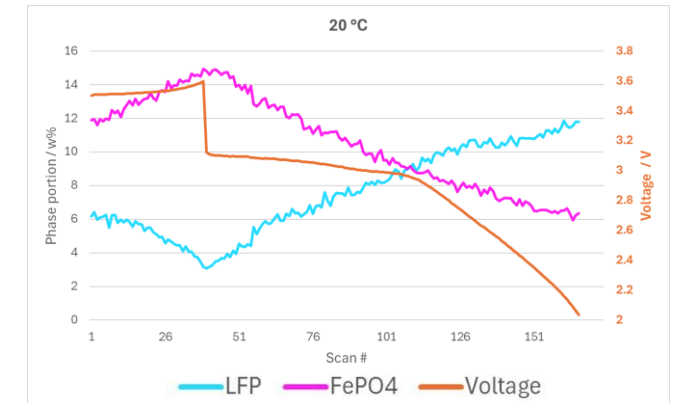
Example: LFP Pouch Cell Performance at Elevated Temperature

- Charge/discharge cycles performed at 20 °C and 60 °C
- Lower capacity observed at higher temperature

Capacity at 20 °C & 60 °C



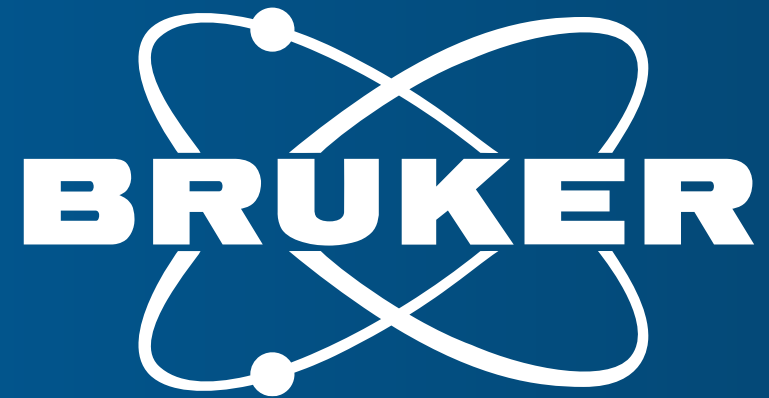
Quantification at 20 °C & 60 °C



Thank you!

Sid Pharasi

Siddharth.Pharasi@bruker.com



Innovation with Integrity