

# Jack of All Trades, Master of All: The New D8 ADVANCE Plus with EIGER2 R 500K



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# D8 ADVANCE Plus

The Perfect Balance of Simplicity and Sophistication



## Effortless Equipment Setup

- No Touch Optics
- Bayonet-Mounted Stages
- Multi-Mode Detector Technology

## Intuitive Instrument Control

- Guided Method Development with WIZARD
- Full Control with COMMANDER
- Unrestricted Access to Diagnostics with TOOLS

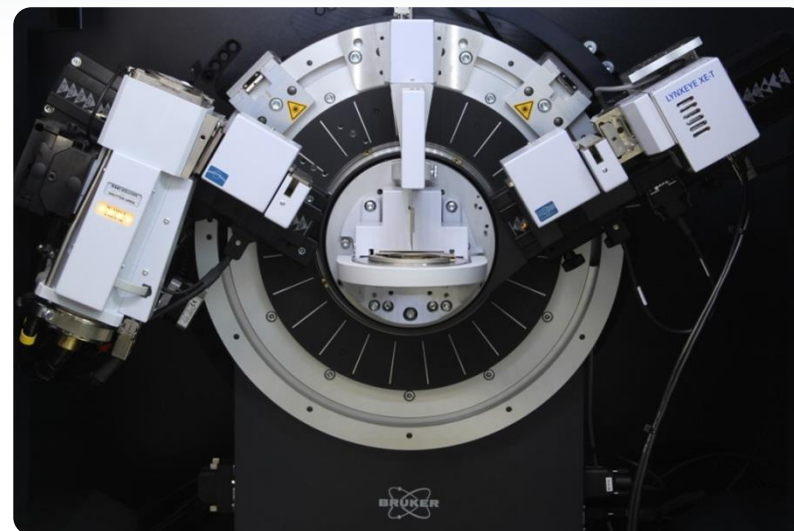
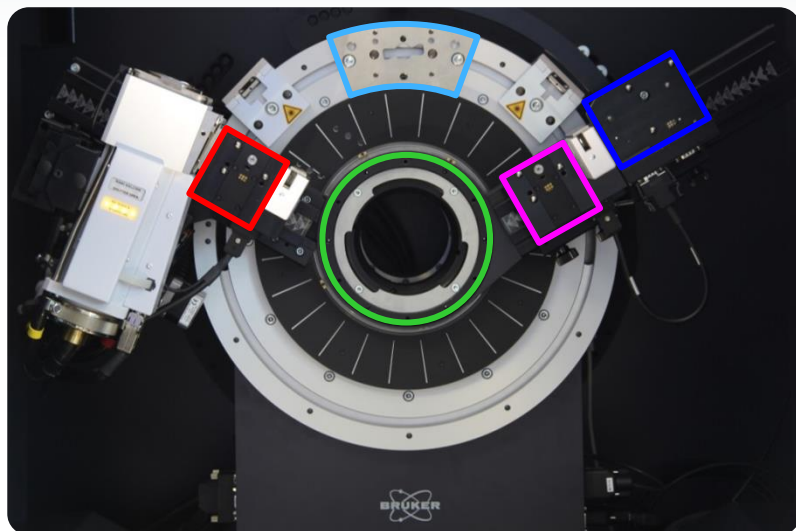
## Advanced Analysis Techniques

- Rapid Results with Industry Standard Methods
- Dig Deeper with the Latest Analytical Techniques

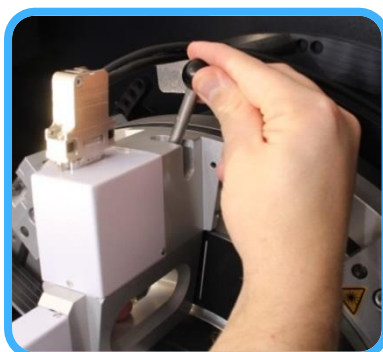
Finish Line

# D8 ADVANCE Plus

## Fundamentally Flexible



**Primary Optic  
Bench**



**Goniometer Accessory  
Mount\***



**Sample Stage  
Bayonet**



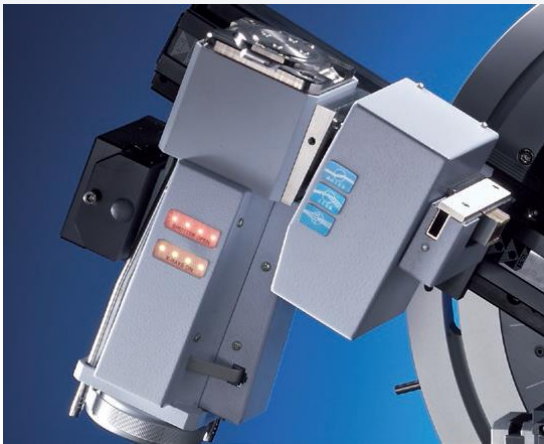
**Secondary Optic  
Bench\***



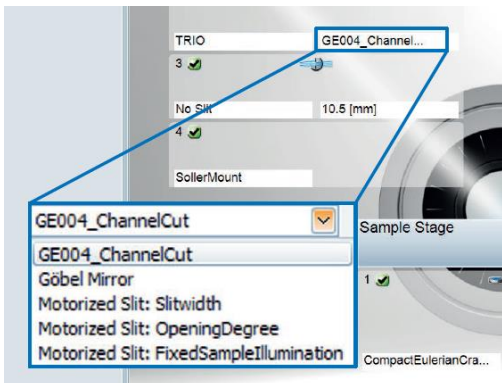
**Universal Detector  
Mount**

# TRIO

## Triple Beampath Primary Optics



- Beampath selection by one click  
No further realignment required

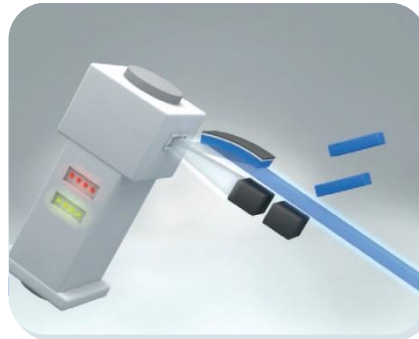


View in DIFFRACT.DAVINCI



### 1. Motorized divergence slit

Bragg-Brentano geometry  
Powder XRD  
Bragg2D



### 2. Goebel mirror

Parallel Beam geometry  
X-ray Reflectometry  
Grazing Incidence XRD  
Residual Stress  
Texture  
Microdiffraction



### 3. Goebel mirror + Ge(004) monochromator

Highly parallel  $K\alpha_1$  geometry  
High Resolution XRD  
Reciprocal Space Mapping  
X-ray Reflectometry (Thick Layers)

# POLYCAP

## High Intensity Point Focus Beam



### **TWIST.TUBE**

- Fast switching between line focus (for TRIO) and point focus (for POLYCAP)
- Automatic focus direction recognition



### **POLYCAP SNAP-LOCK optic**

- Same optic for all wavelengths
- Parallel beam optic (divergence  $\sim 0.25^\circ$  in both directions)
- Beam diameter: 4 mm



### **UBC Magnetic Collimators**

- Tool-free and alignment-free exchange
- Excellent reproducibility with remounting
- Sizes available (mm): 0.1, 0.3, 0.5, 1.0, 2.0

Primary Applications: Microdiffraction, In-Plane GID

# D8 ADVANCE Plus

## Bayonet-Mounted Sample Stages



**Rotation Stage** ✓

**CAPILLARY**

**COMPACT UMC**

**COMPACT CRADLE+** ✓

**Non Ambient** ✓

**Motorized Motion**

Phi Rotation

Phi Rotation

XYZ

Phi, Chi, Z  
Manual X,Y

Various

**Geometry**

Reflection  
Transmission

Transmission

Reflection

Reflection

Various

**Applications**

- Powder Diffraction
- GID
- XRR

- Powder Diffraction
- Structure Solution
- PDF
- SAXS

- Powder Diffraction
- Well Plates
- XY Mapping
- GID
- XRR

- Powder Diffraction
- Pseudo-Gandolfi
- HRXRD
- XRR
- IPGID

- Heating/ Cooling
- Humidity
- Reactive Gases
- Tension
- Electrochemical

# The New **EIGER2 R 500K** Ergonomic Design for Dynamic Detection



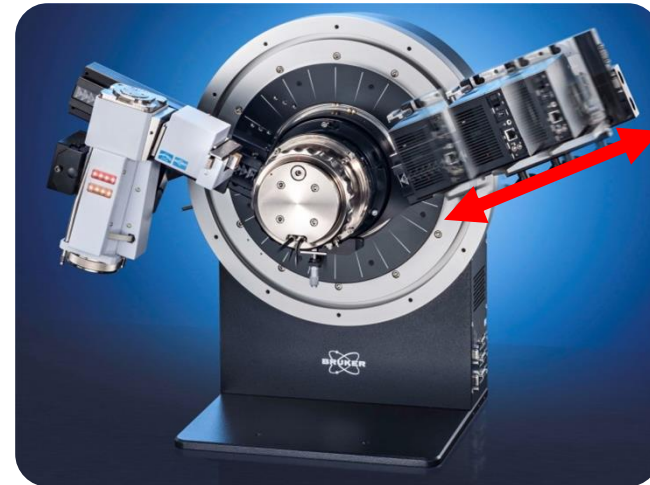
## Ergonomic 0°/90° rotation

- Optimize for  $2\theta$  or  $\gamma$  coverage
- Tool-free switching
- Component recognition for rotation

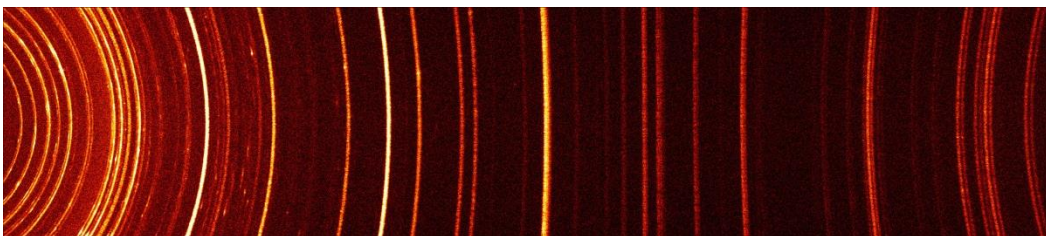
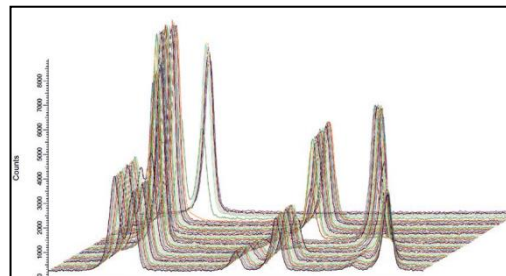
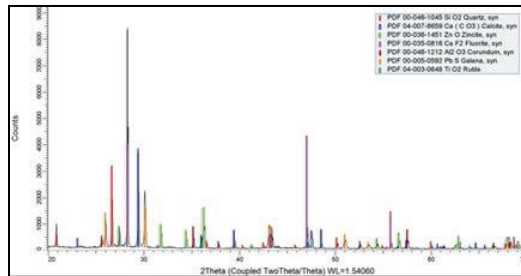
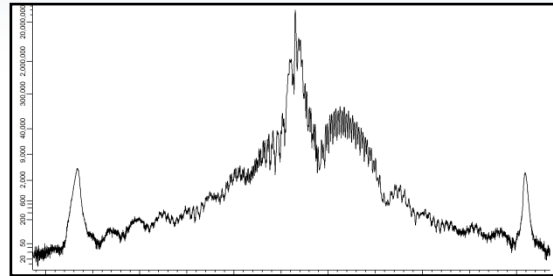
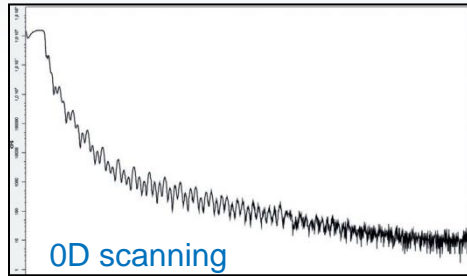


## Continuously Variable Sample-to-Detector Distance

- 100 mm to 350 mm (D8 Advance)
- Balance coverage and resolution
- Tool-free distance adjustment
- No alignment or calibration



# The New **EIGER2 R 500K** A Multitude of Measurement Modes

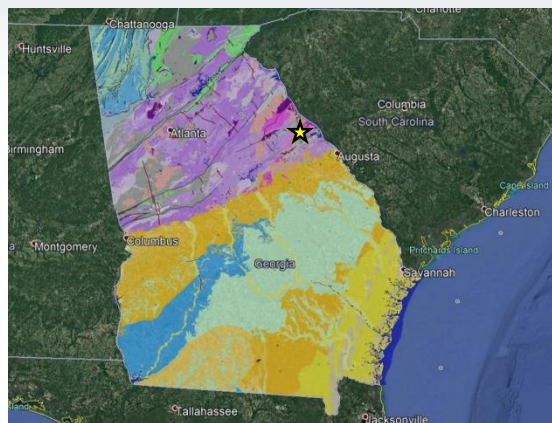


Mode	Type	EIGER2 R 500K
0D	Step Scan	✓
	Continuous Scan	✓
1D	Snap-Shot	✓
	Step Scan	✓
	Continuous Scan	✓
	Advanced Scan	✓
2D	Snap-Shot	✓
	Step Scan	✓
	Continuous Scan	✓
	Advanced Scan	✓



# Phase ID of Geological Samples

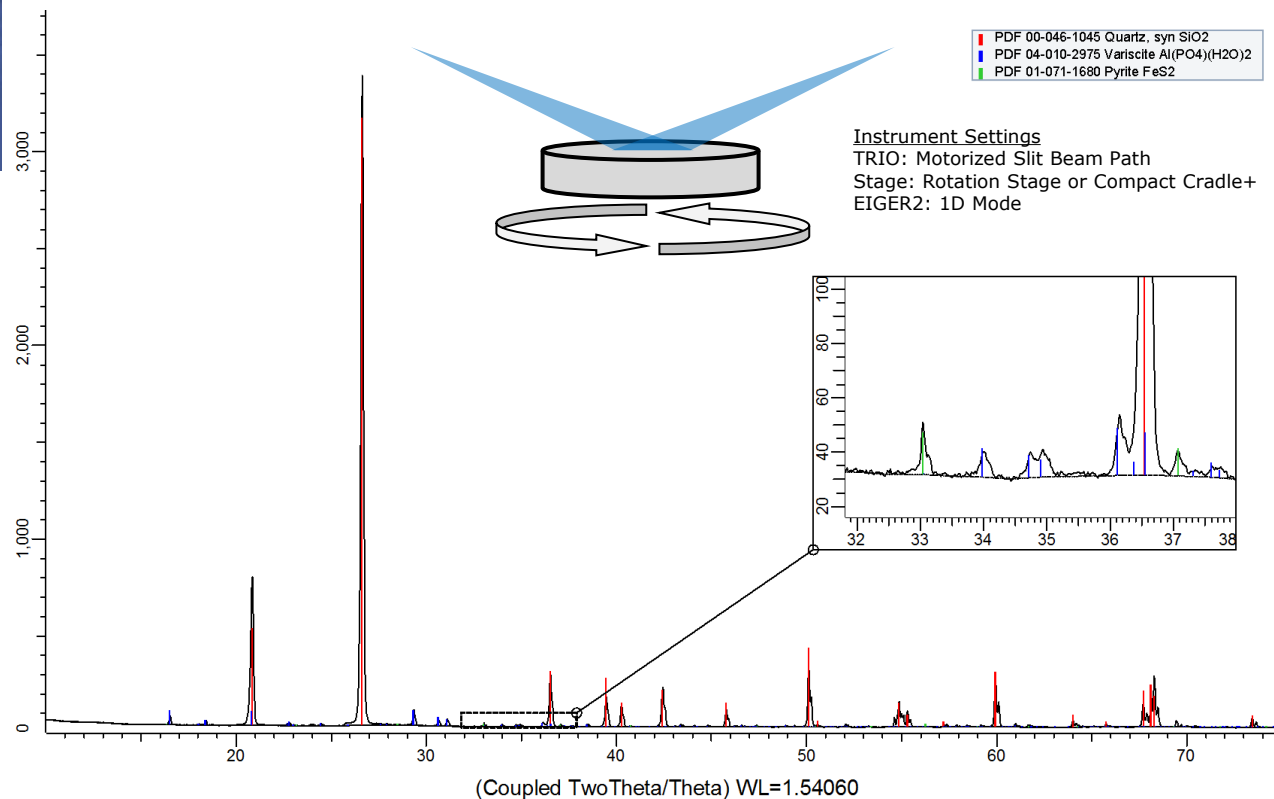
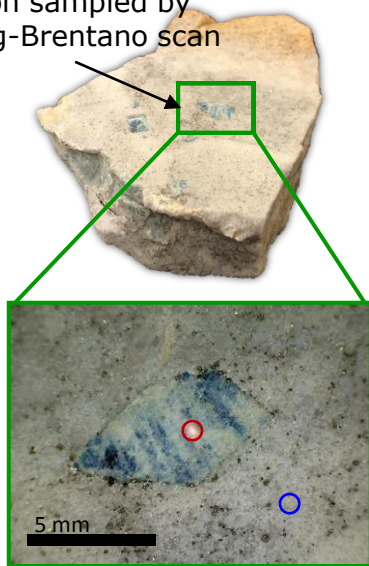
## Bragg-Brentano Powder Diffraction



Graves Mountain is a NE/SW striking monadnock located in eastern Georgia, USA (Figure 1). Part of the Carolina Slate Belt, the low to medium grade metavolcanics of Graves Mountain were mined for kyanite to be used in high-alumina refractory products. In addition to kyanite, the locale is known to mineral collectors for its world-class twinned rutile crystals, iridescent hematite coatings on quartz, lazulite, pyrite, and pyrophyllite.

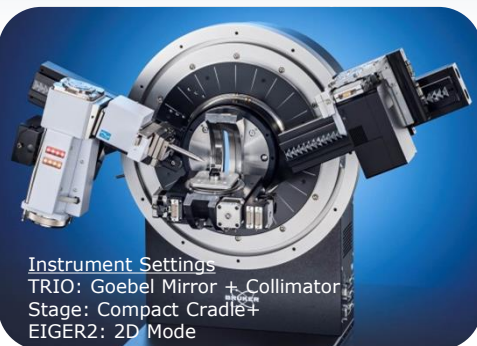
Reference: Hurst, Vernon J., 1959, *The Geology and Mineralogy of Graves Mountain, Georgia: Atlanta, GA, Bulletin Number 68, Georgia Geological Survey, 33 p.*

Region sampled by Bragg-Brentano scan

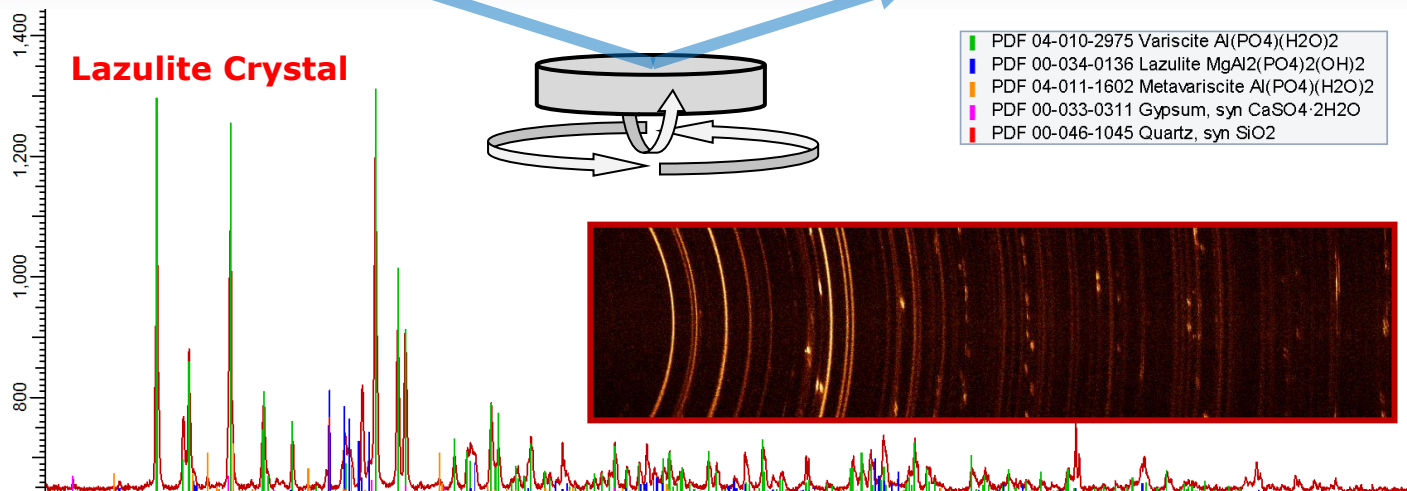


# Phase ID of Geological Samples

## Microdiffraction and Pseudo-Gandolfi

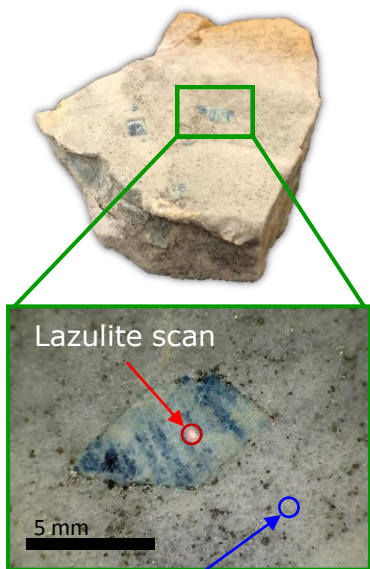
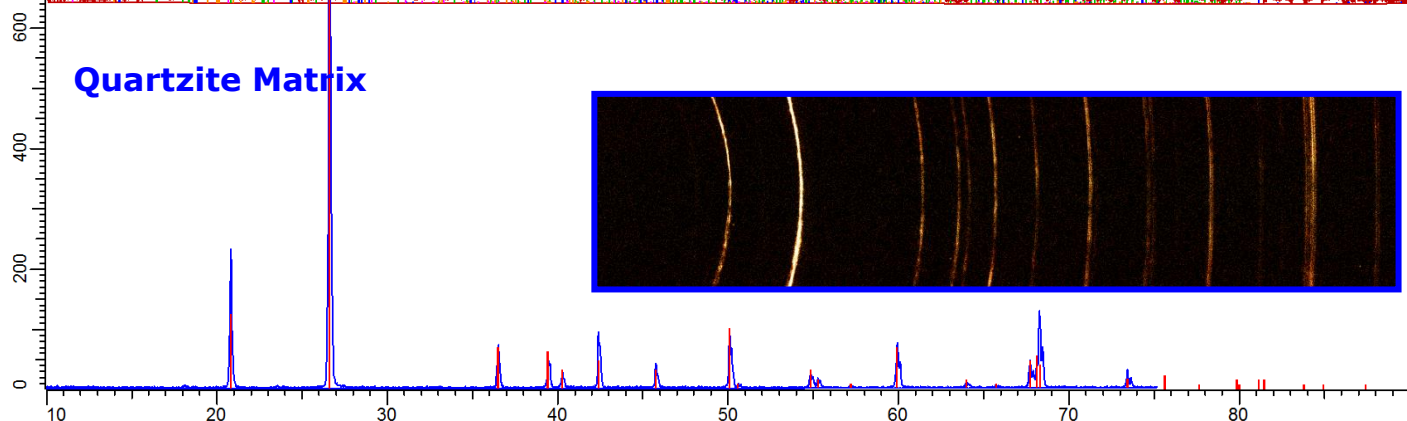


**Lazulite Crystal**



- PDF 04-010-2975 Variscite  $\text{Al}(\text{PO}_4)(\text{H}_2\text{O})_2$
- PDF 00-034-0136 Lazulite  $\text{MgAl}_2(\text{PO}_4)_2(\text{OH})_2$
- PDF 04-011-1602 Metavariscite  $\text{Al}(\text{PO}_4)(\text{H}_2\text{O})_2$
- PDF 00-033-0311 Gypsum, syn  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
- PDF 00-046-1045 Quartz, syn  $\text{SiO}_2$

**Quartzite Matrix**



Lazulite scan

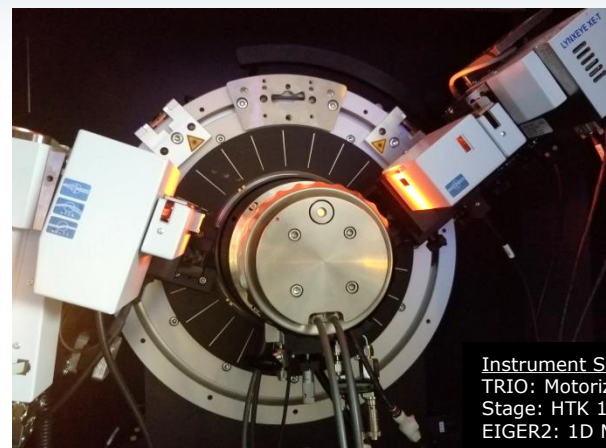
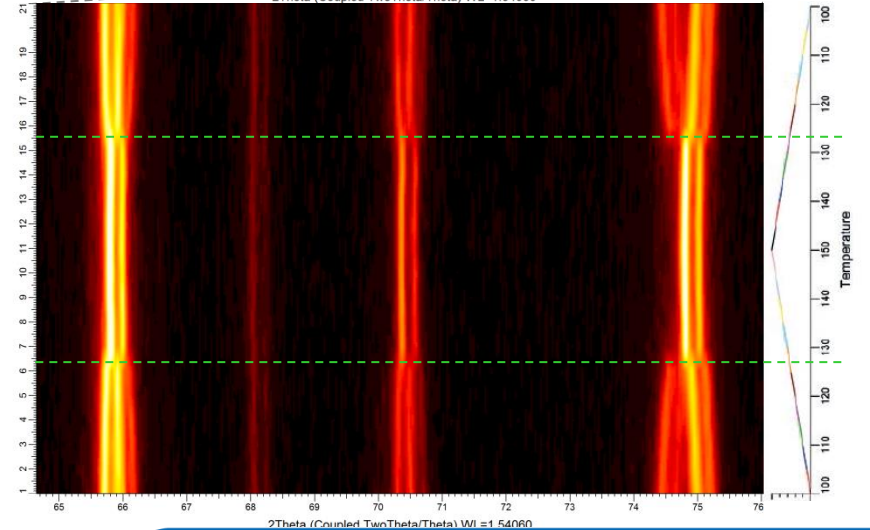
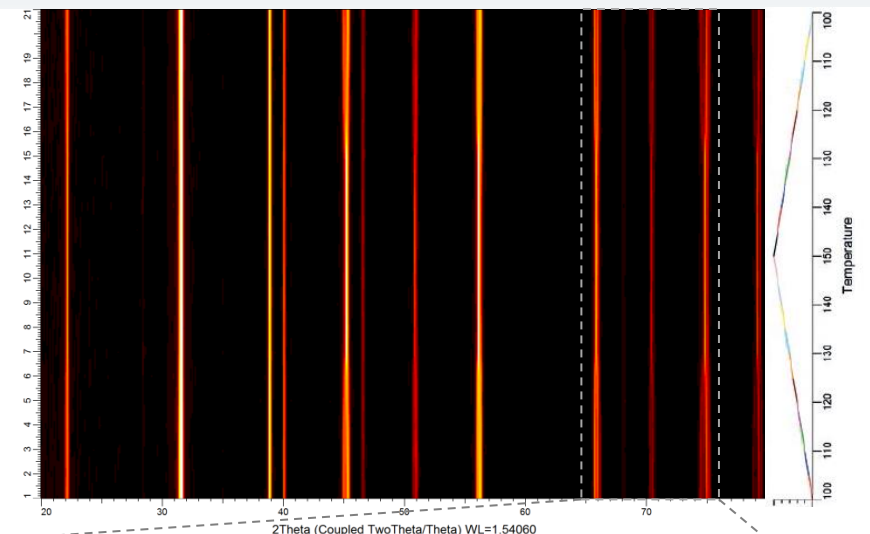
5 mm

Quartzite scan

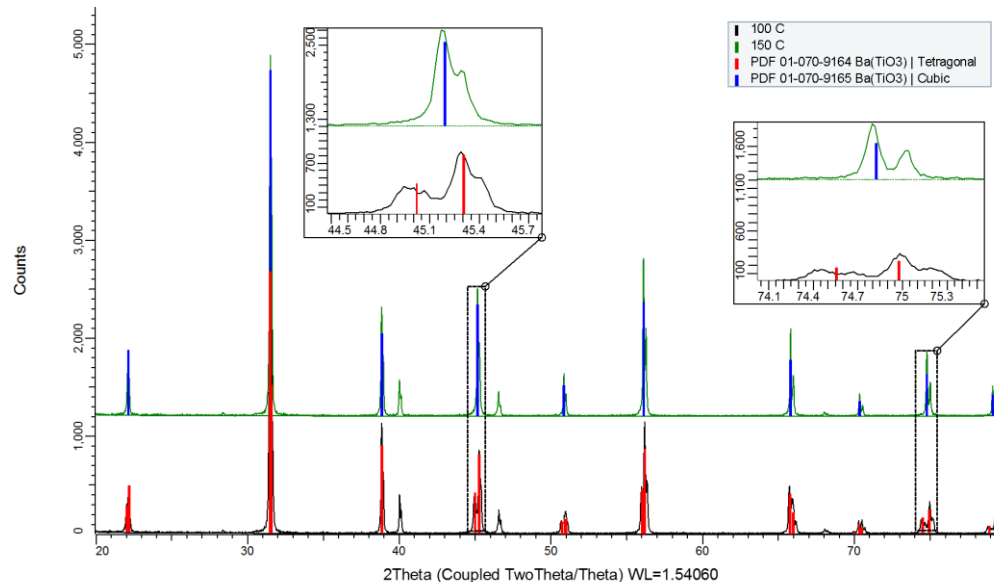
With Pseudo-Gandolfi technique it is possible to identify mineral phases without first crushing/grinding rock, many of which were invisible to a standard Bragg-Brentano scan.

# Non-Ambient Diffraction

## Cubic to Tetragonal Transition of BaTiO<sub>3</sub> Powder

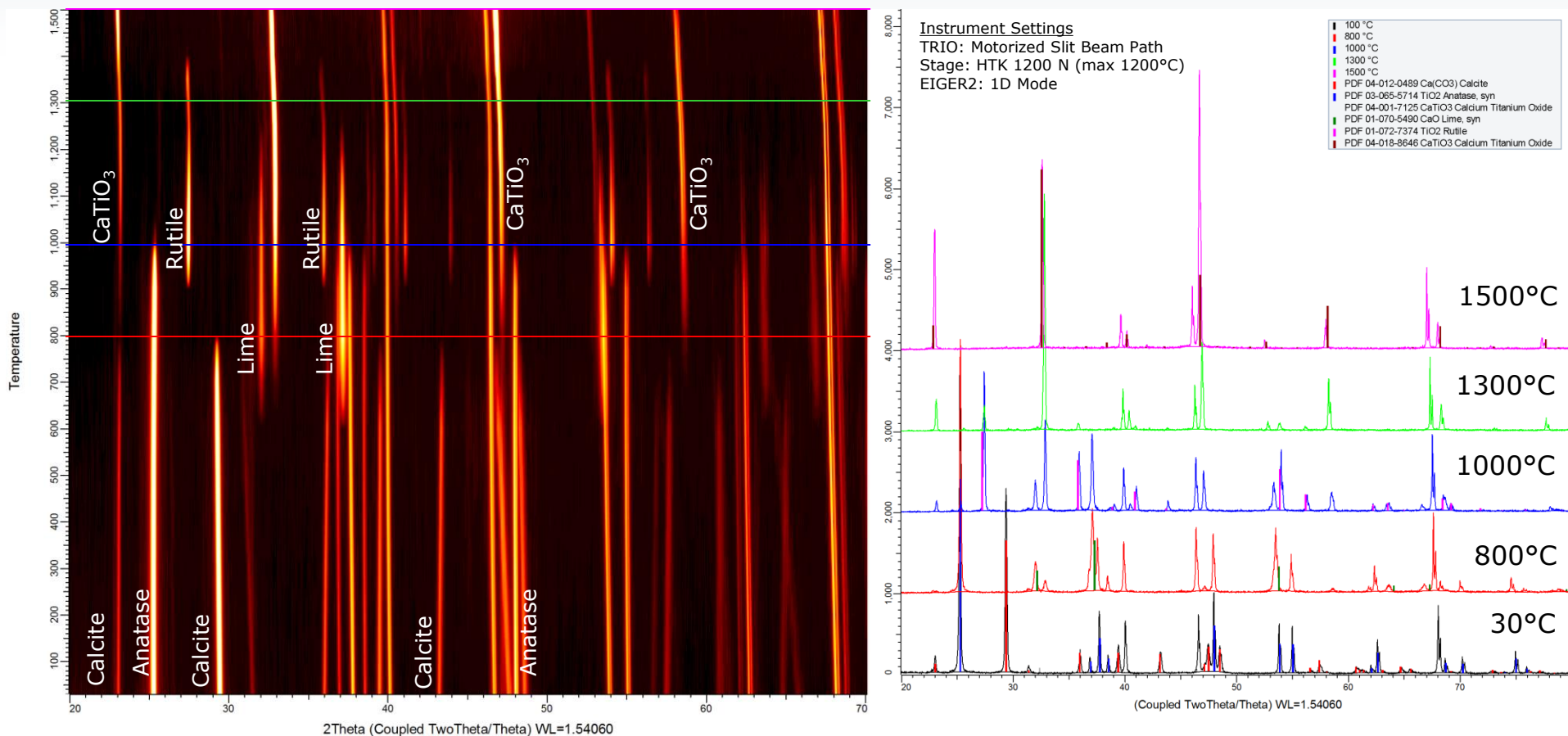


**Instrument Settings**  
 TRIO: Motorized Slit Beam Path  
 Stage: HTK 1200 N  
 EIGER2: 1D Mode



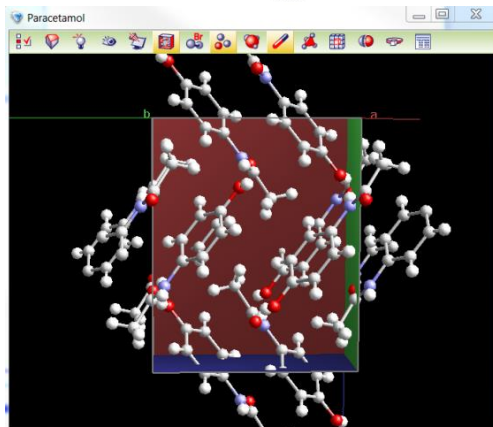
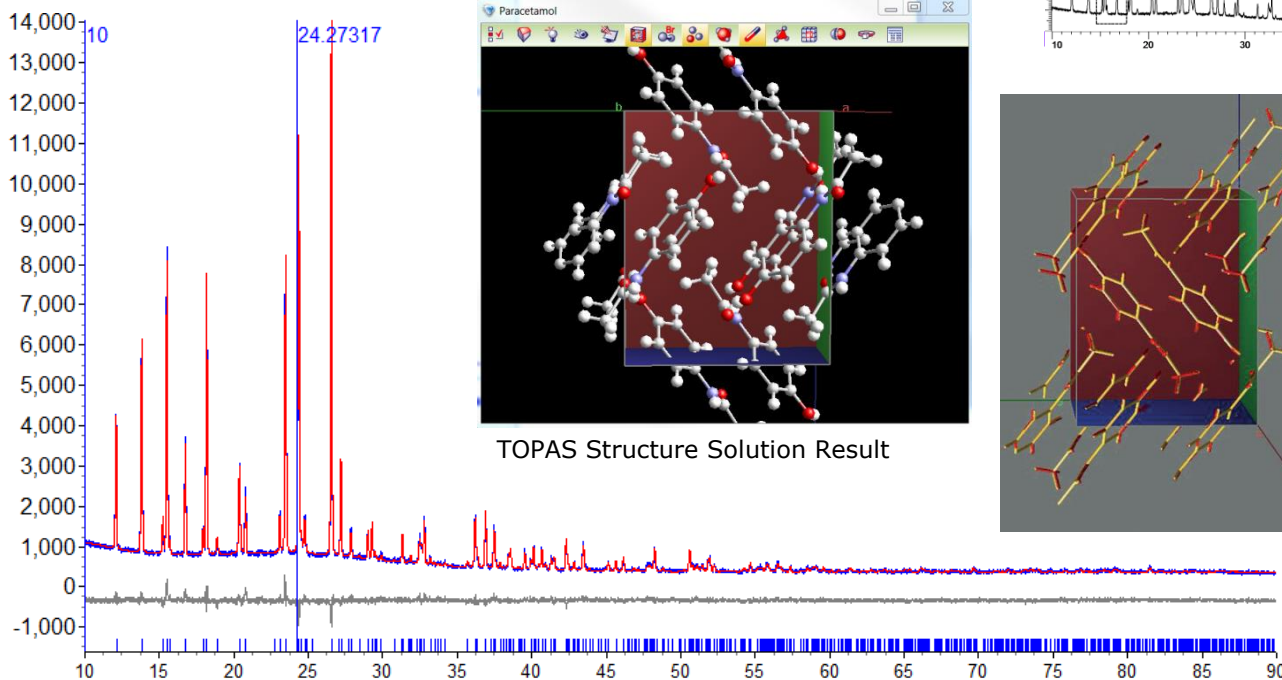
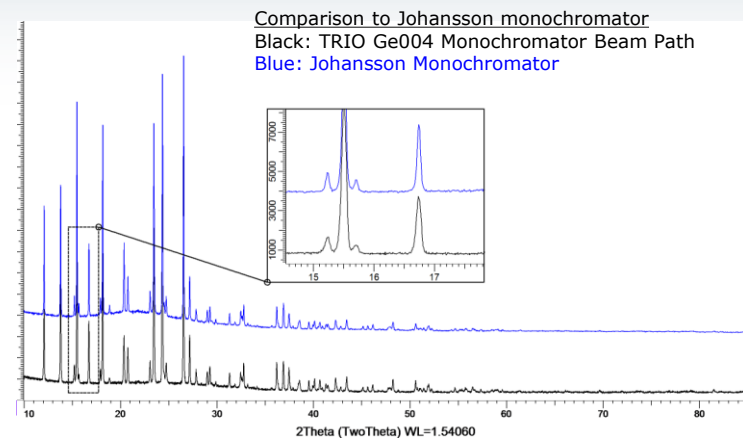
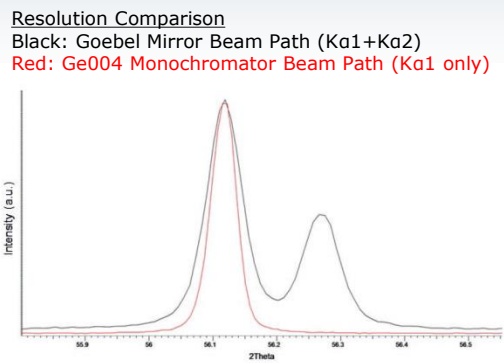
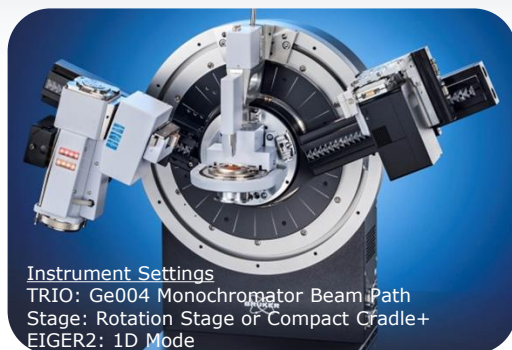
# Non-Ambient Diffraction

## Reaction of $\text{CaCO}_3$ and $\text{TiO}_2$ to form $\text{CaTiO}_3$

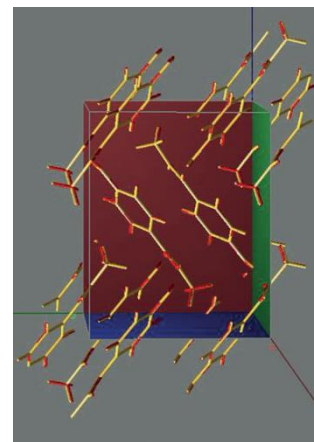


# Powder Diffraction with K $\alpha$ 1 only

## Structure of Paracetamol



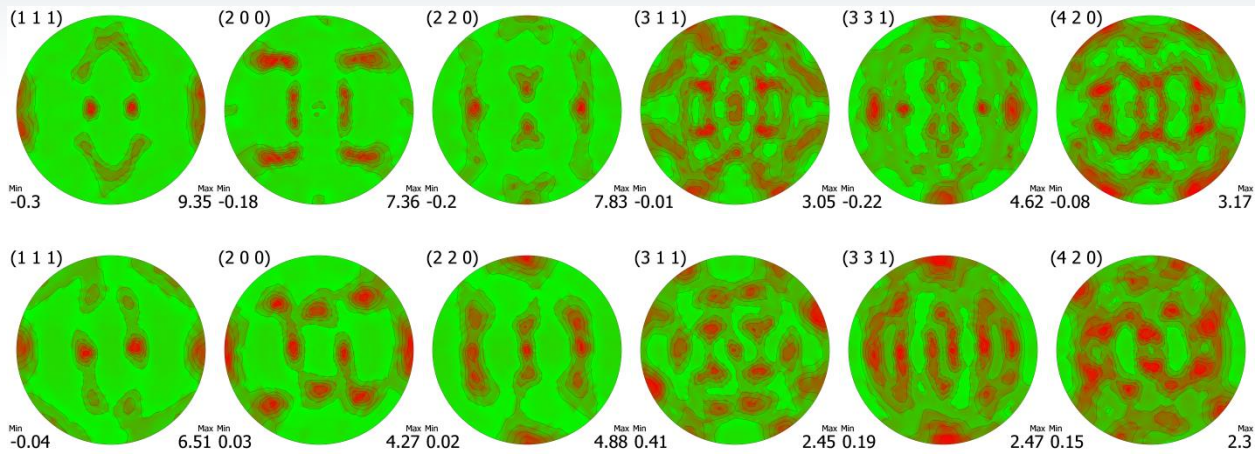
TOPAS Structure Solution Result



Perfect Agreement with Single Crystal Experiment  
Single Crystal Data (Yellow)  
Powder Diffraction Data (Red)

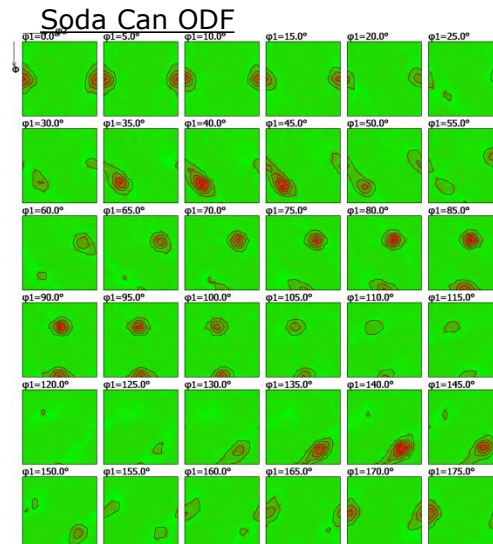
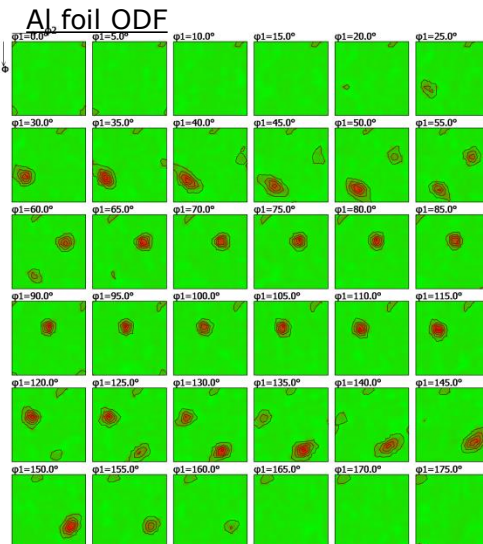
# Pole Figures and Texture Analysis

## Rolled Al Foil vs. Rolled and Drawn Al Can



A pole figure maps the intensity of a single HKL reflection as a function of the angle of the diffraction vector relative to the surface normal.

The orientation distribution (ODF) of crystallites is usually plotted as intensity in a cubic volume, where each axis of the cube corresponds to one of the Euler angles.



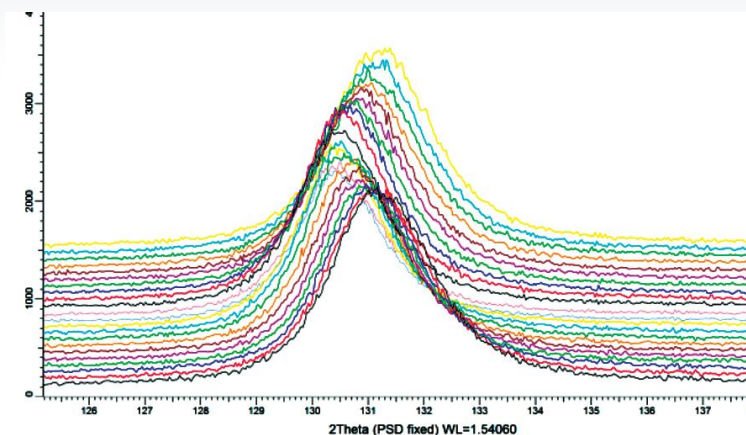
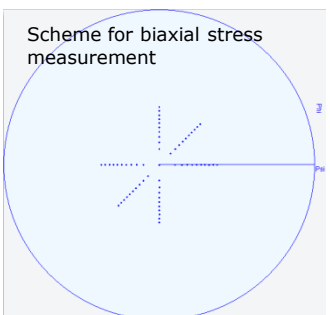
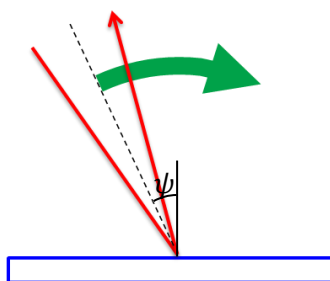
- Al Foil shows three distinct texture components in the ODF
- Soda can ODF shows the same three components, but with less broadening since it was not rolled as thin
- Soda can also shows a fourth component not present in the Al foil, due to the drawing process

# Residual Stress in 1D

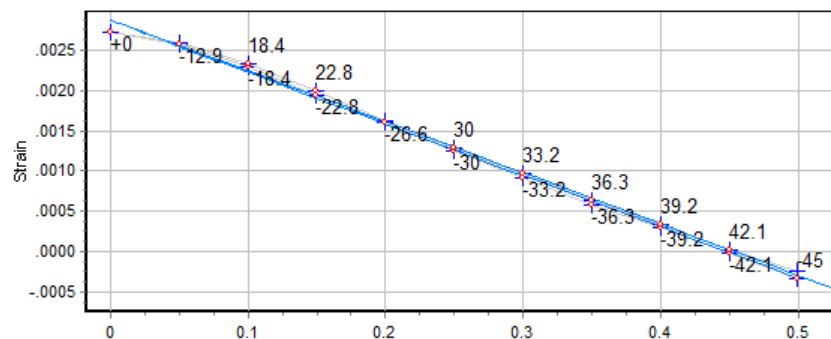
## Polycrystalline Ta Coating Deposited at High Temp.



$\text{Sin}^2(\psi)$  Method (1D)



The classic  $\text{sin}^2(\psi)$  technique determines the stress of a material by the shift of a high-angle diffraction peak as a function of the tilt angle  $\psi$  of the diffraction vector away from the sample normal.  
 Iso-inclination mode:  $\psi$  tilt is in the diffraction plane  
 Side-inclination mode:  $\psi$  tilt is perpendicular to the diffraction plane

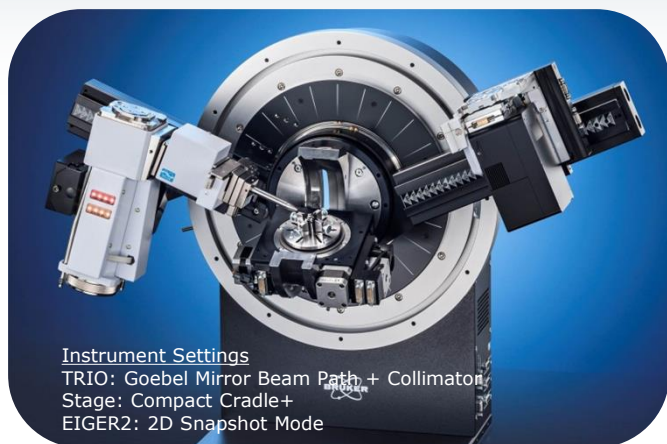


- Measured in iso-inclination mode using a line-focus beam and EIGER2 in 1D snapshot mode, resulting in extremely short measurement times ( $\sim 10$  s per scan)
- Suitable for large, flat samples
- This stress result represents an average value over a large surface area

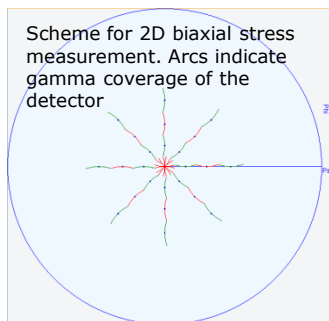
Stress tensor (MPa)		
$-1995.3 \pm 23.8$	$65.5 \pm 20.5$	$-3.8 \pm 5.3$
$65.5 \pm 20.5$	$-2008.6 \pm 23.8$	$6.3 \pm 5.3$
$-3.8 \pm 5.3$	$6.3 \pm 5.3$	$0.0 \pm 0.0$

# Residual Stress in 2D

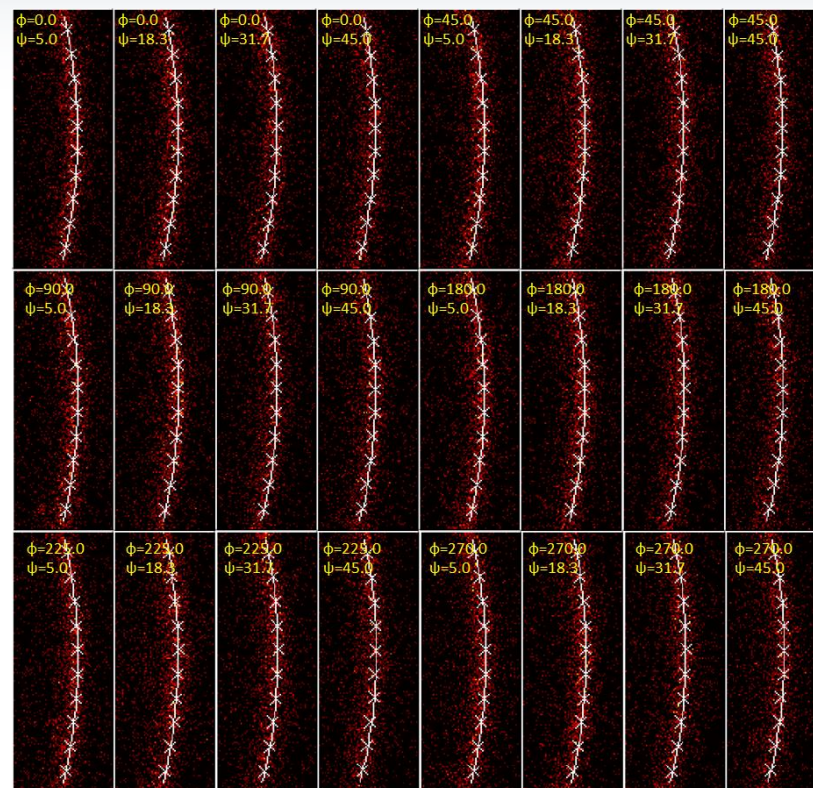
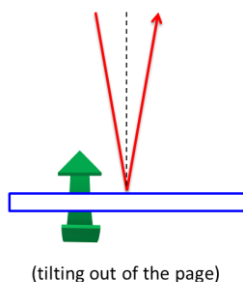
## Polycrystalline Ta Coating Deposited at High Temp.



The 2D stress method extends the  $\sin^2(\psi)$  technique by including the deformation of the Debye ring at several points, rather than just at the center



**2D Method**



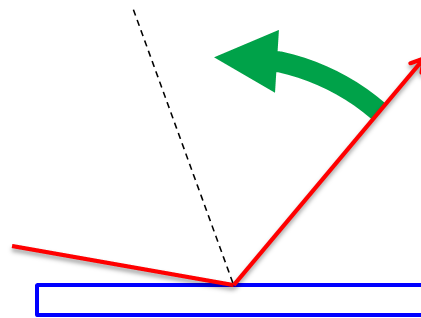
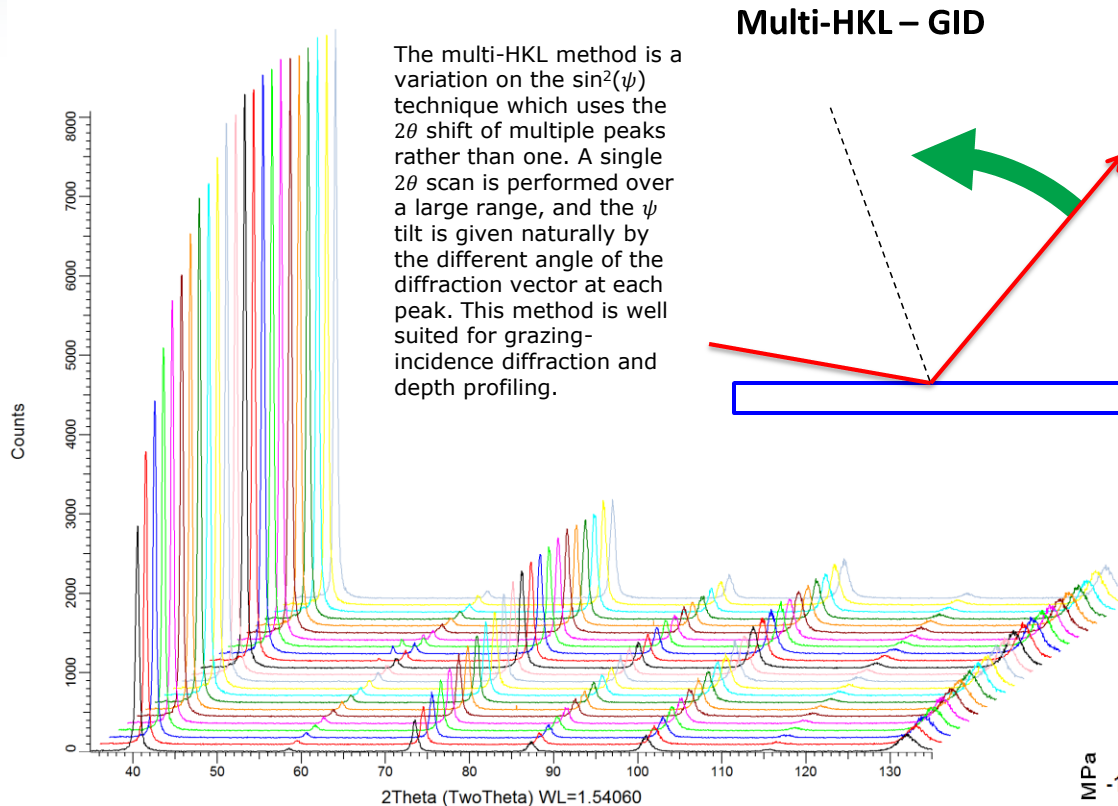
- Measured in side-inclination mode using a point-focus beam and EIGER2 in 2D snapshot mode
- Suitable for measuring small areas and features
- This stress result represents a local value
- Agreement between 1D and 2D methods suggests uniformity over the sample surface

Stress tensor (MPa)		
-2027.3 ± 19.2	10.3 ± 19.5	-1.6 ± 5.6
10.3 ± 19.5	-2027.7 ± 19.1	-0.1 ± 5.5
-1.6 ± 5.6	-0.1 ± 5.5	0.0 ± 0.0

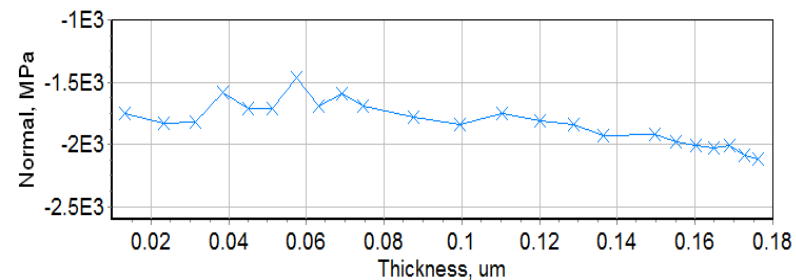


# Residual Stress Depth Profile

## Polycrystalline Ta Coating Deposited at High Temp.

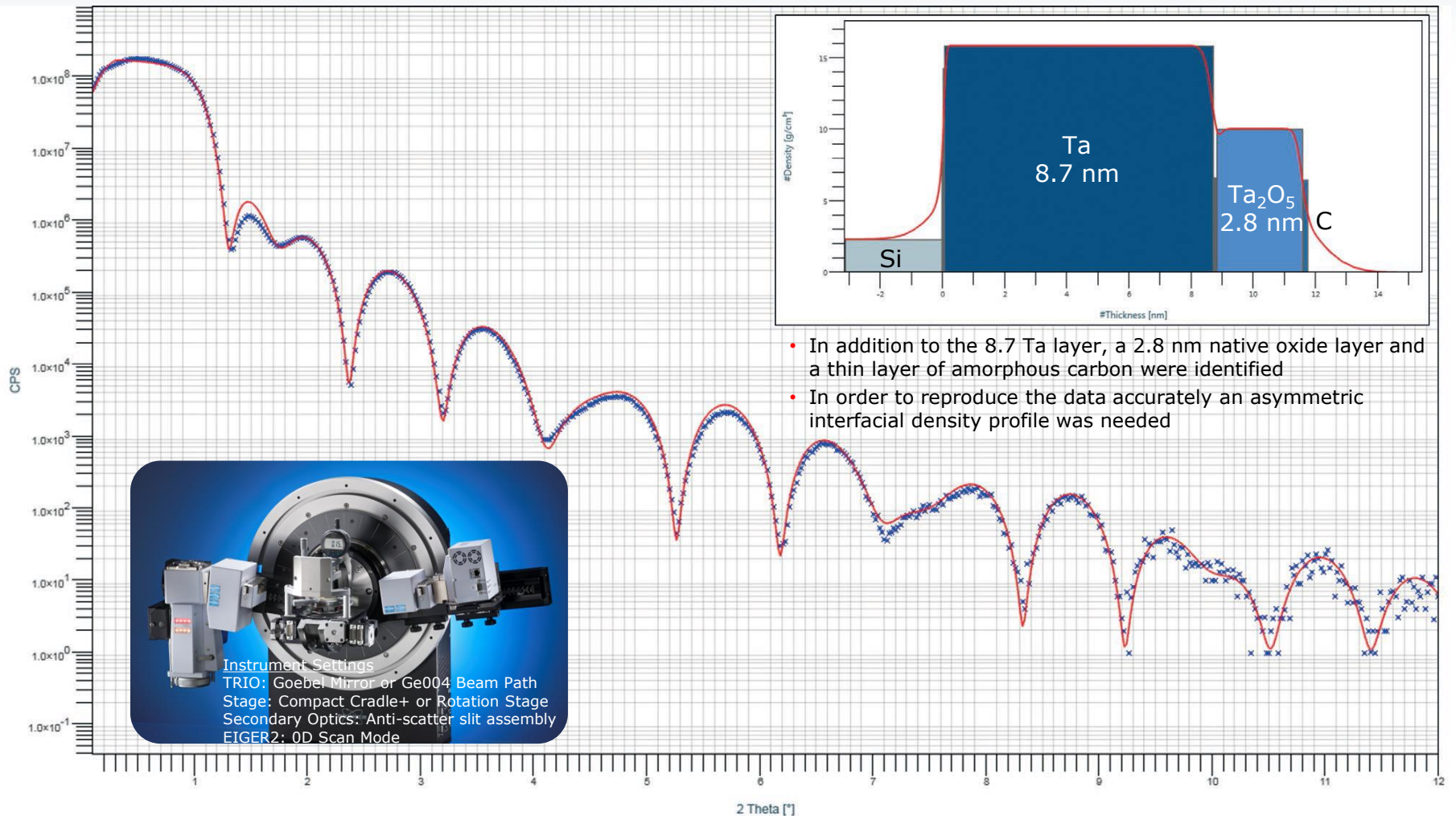


- Based on the GID depth profile the stress at the surface of the film is slightly lower than in the bulk
- The bulk value is in good agreement with the results of the 1D and 2D methods

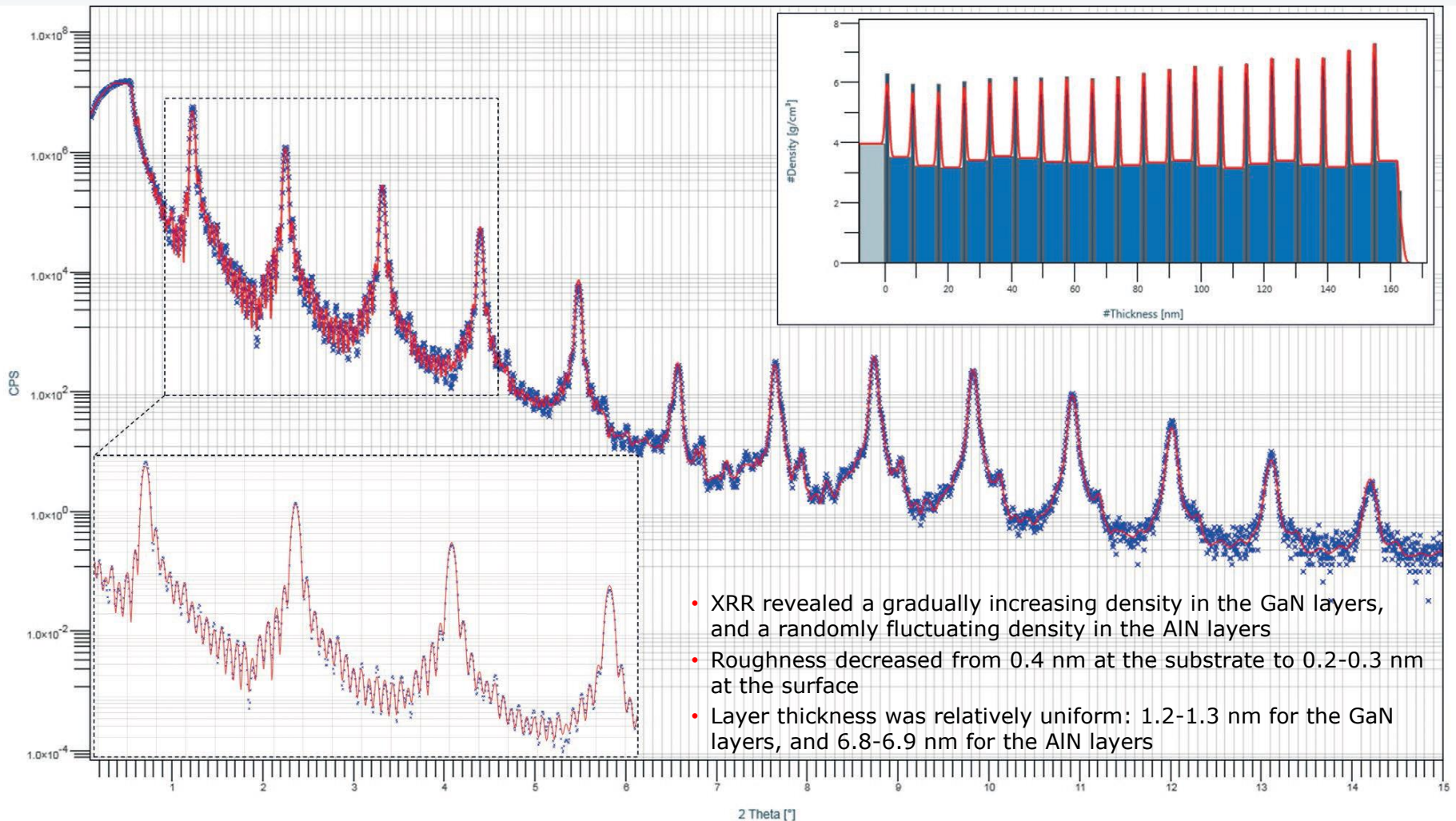


# X-ray Reflectometry

## Thin Ta Coating on Si

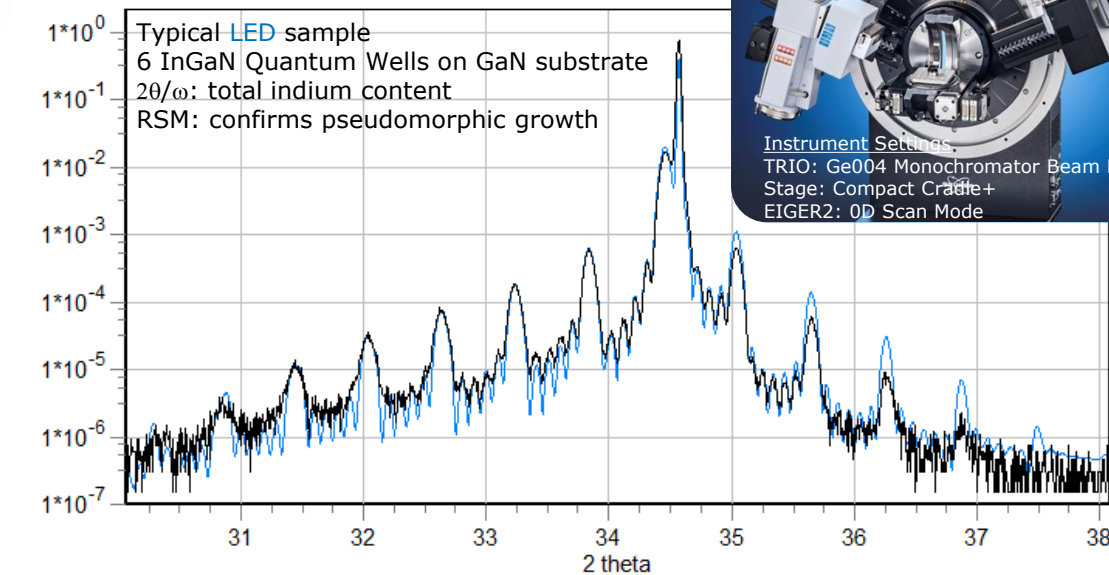
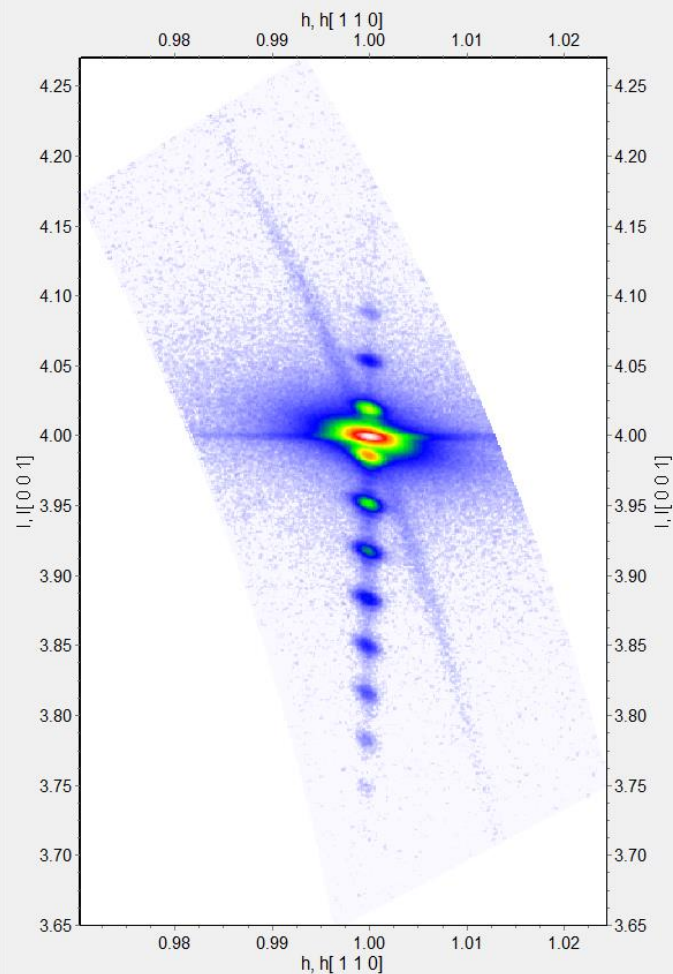


# X-ray Reflectometry GaN/AlN Superlattice on Si



# High-Resolution X-ray Diffraction

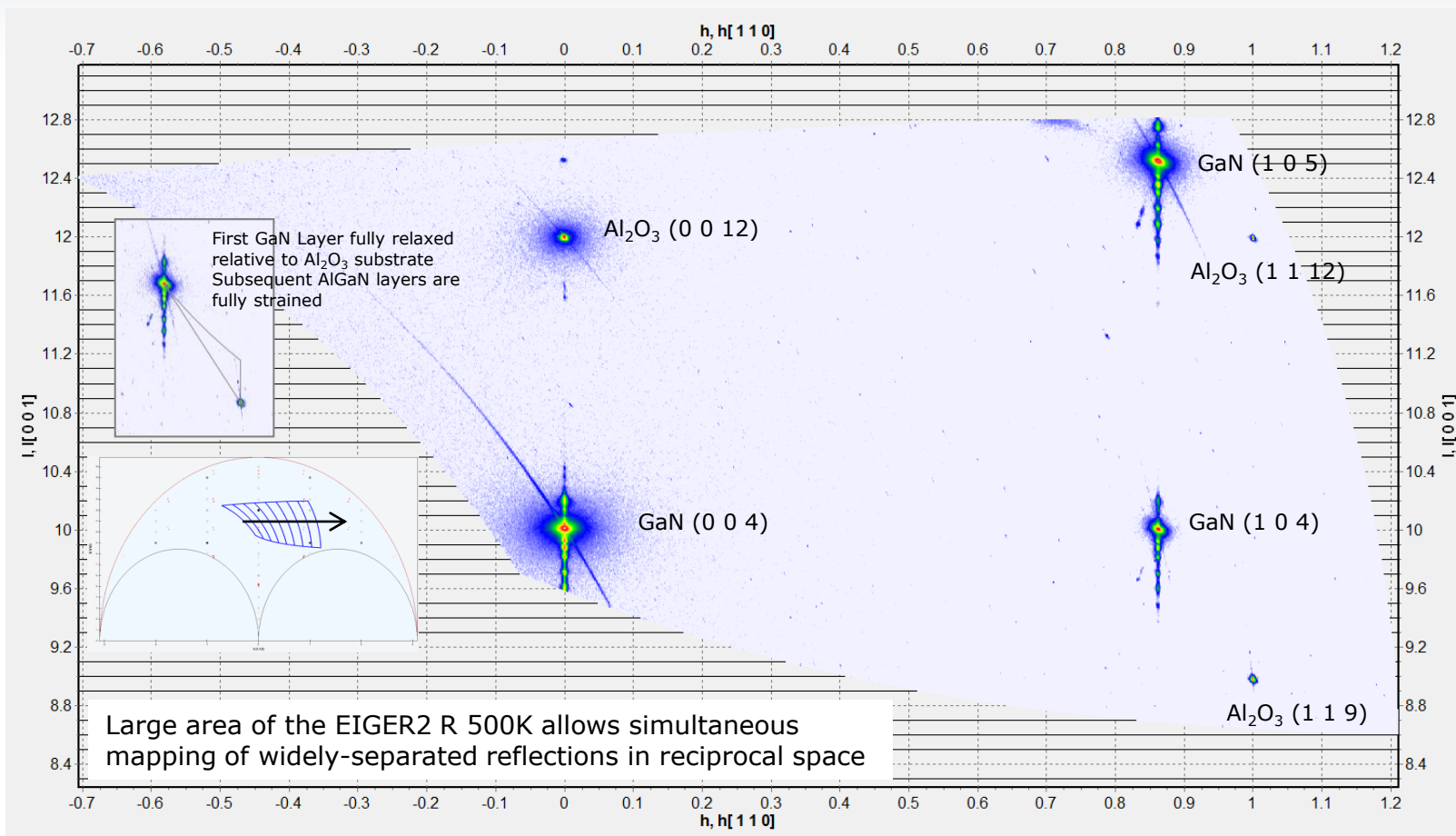
## Characterization of an LED multilayer structure



N	R	Material	Thickness (nm)	Conc In (%)	SL thickness (nm)
1	6	GaN	13.1425	0.0000	15.3383
2	6	Ga(1-x)In(x)N	2.1957	18.8660	
SUB	1	GaN	0.0000	0.0000	0.0000

# Large-Area Reciprocal Space Mapping

## AlGaN LED on Sapphire



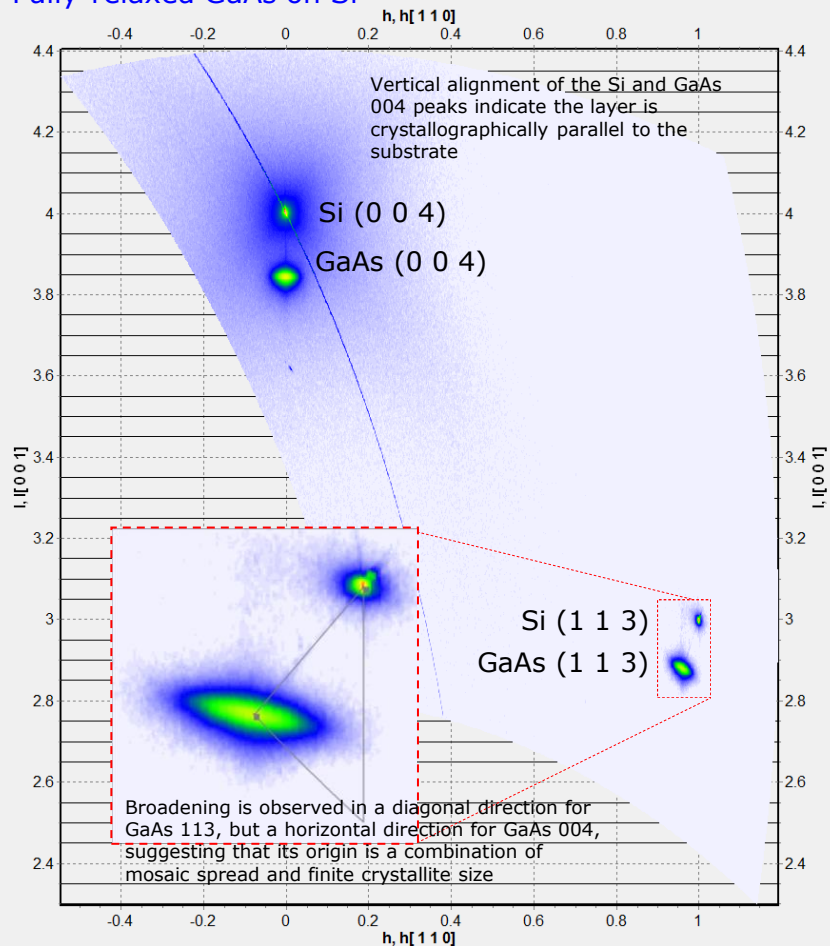
### Instrument Settings

TRIO: Ge004 Monochromator Beam Path  
 Stage: Compact Cradle+  
 EIGER2: 1D Snapshot Mode

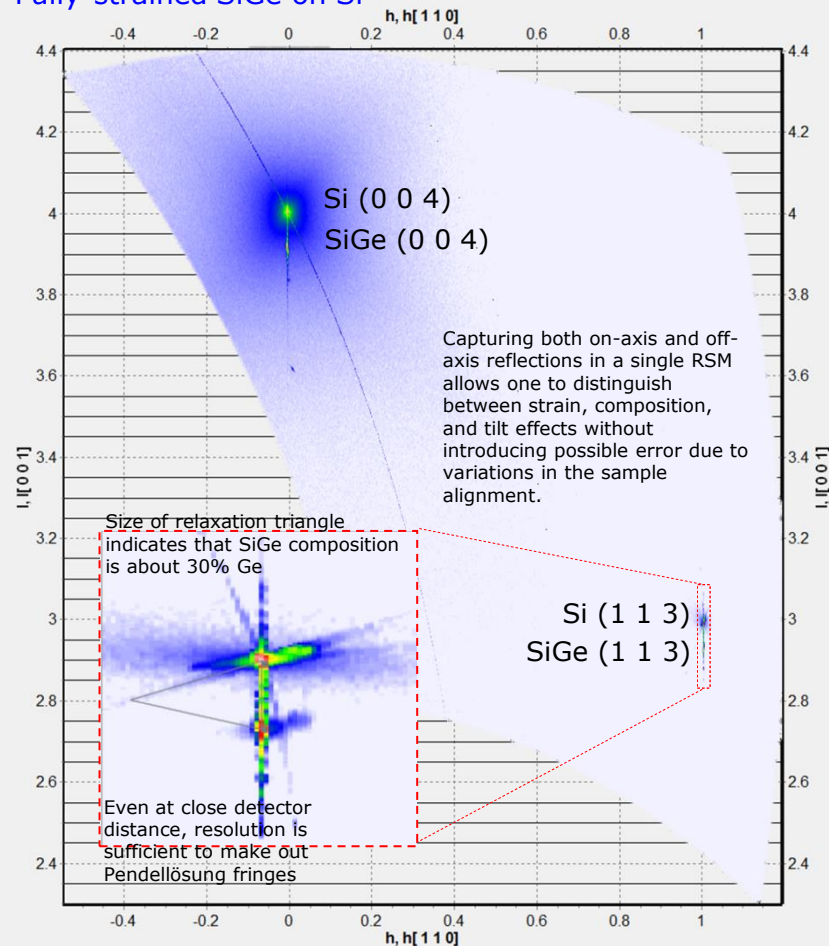
# Large-Area Reciprocal Space Mapping Single Layer Epitaxial Films



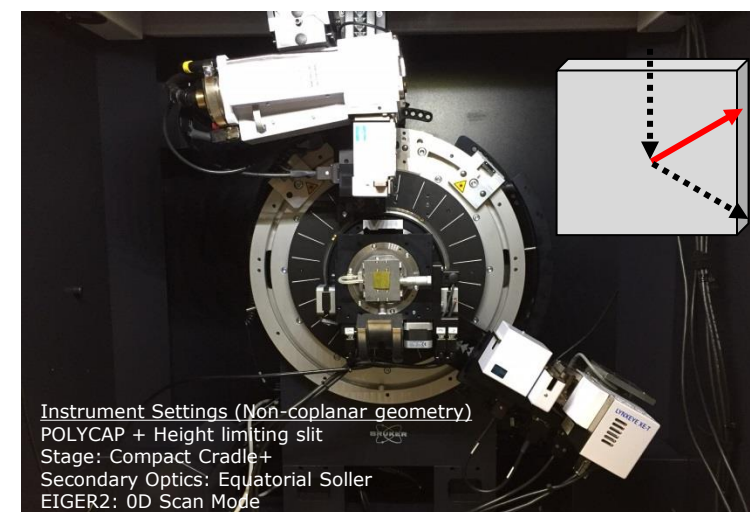
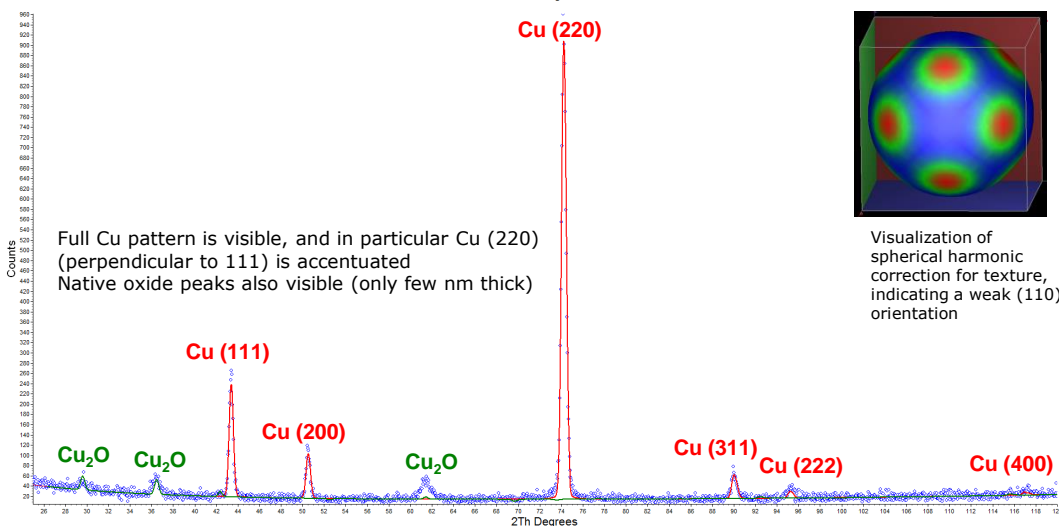
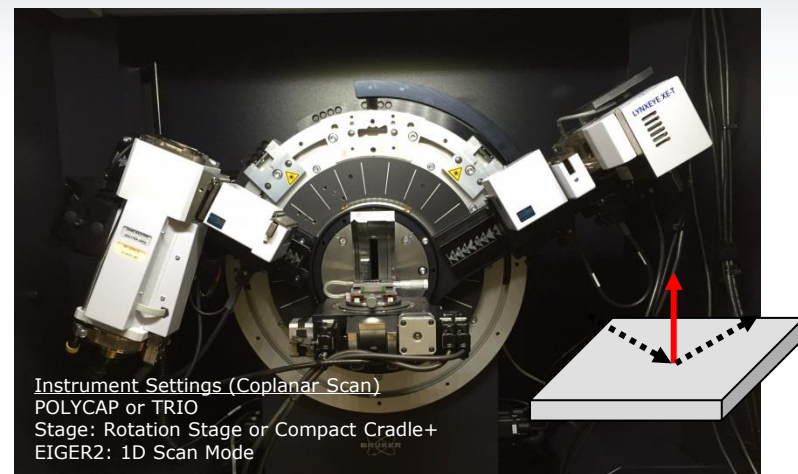
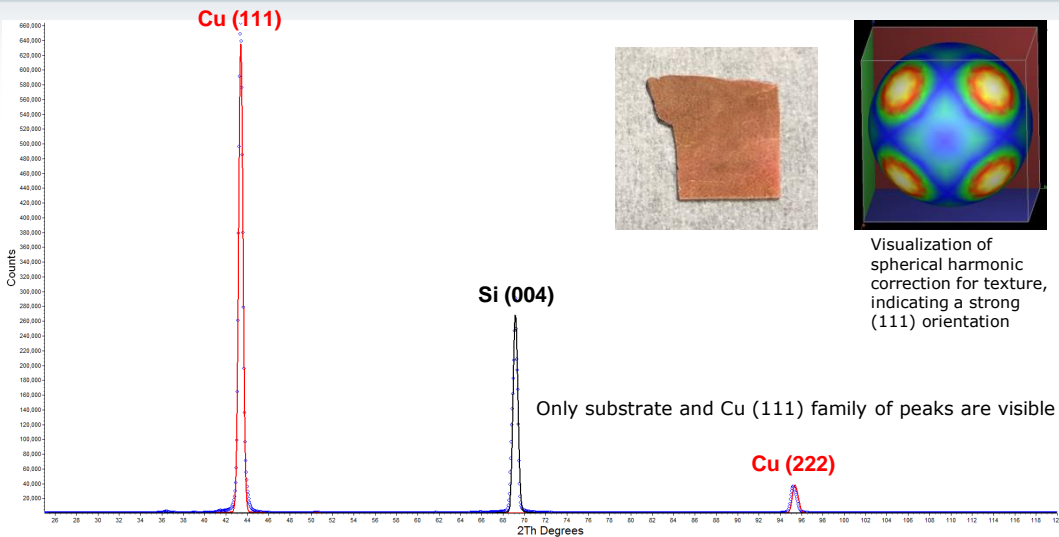
Fully-relaxed GaAs on Si



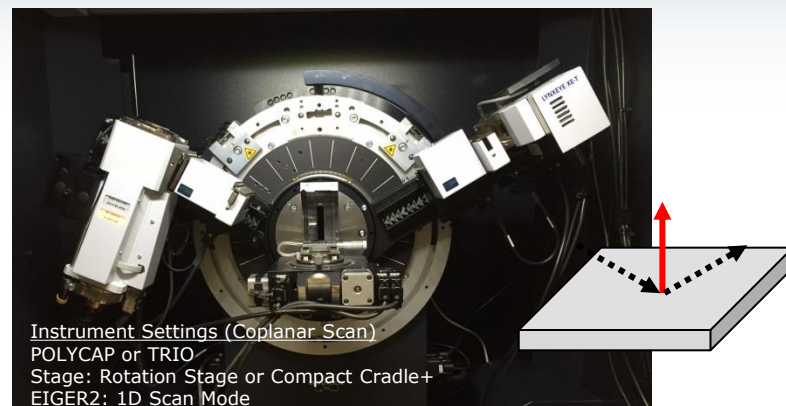
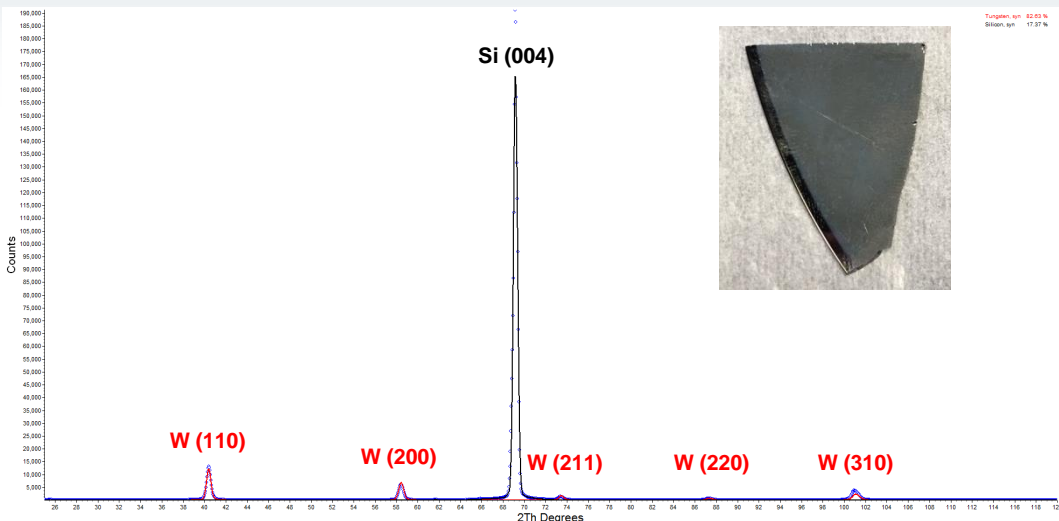
Fully-strained SiGe on Si



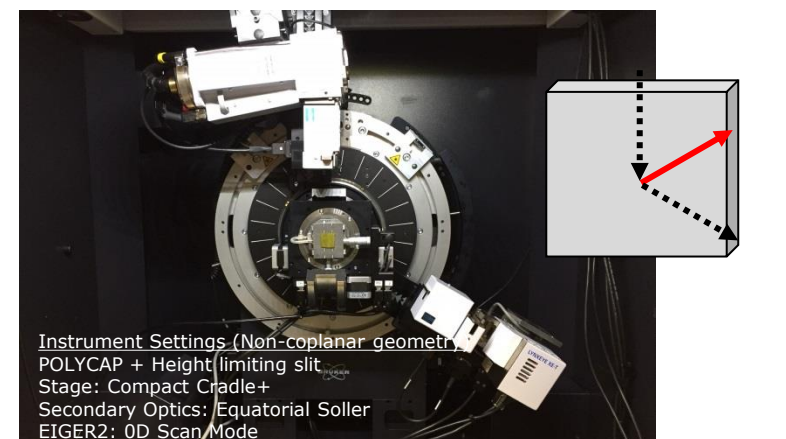
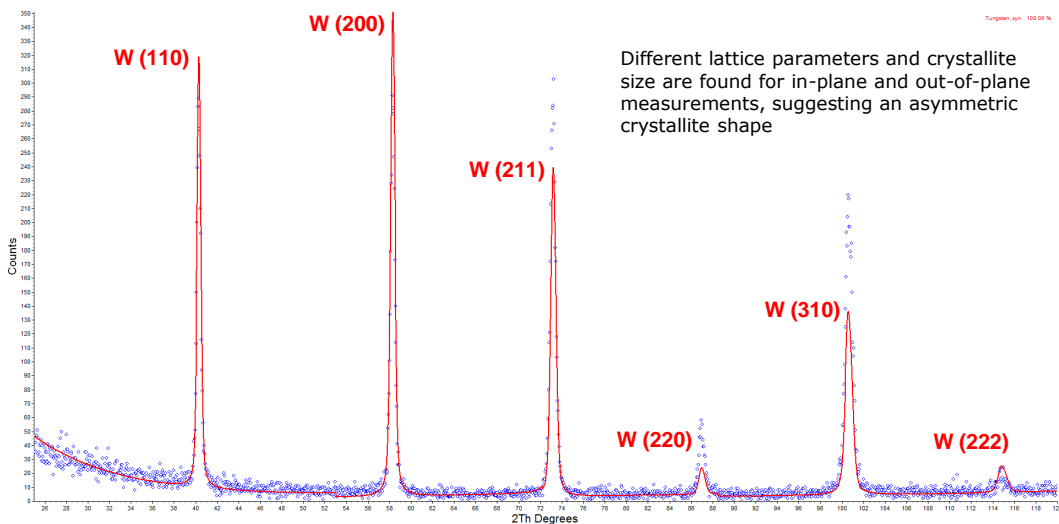
# In-plane Grazing Incidence Diffraction Fiber-Textured Cu Film on Si



# In-plane Grazing Incidence Diffraction Polycrystalline W Film on Si



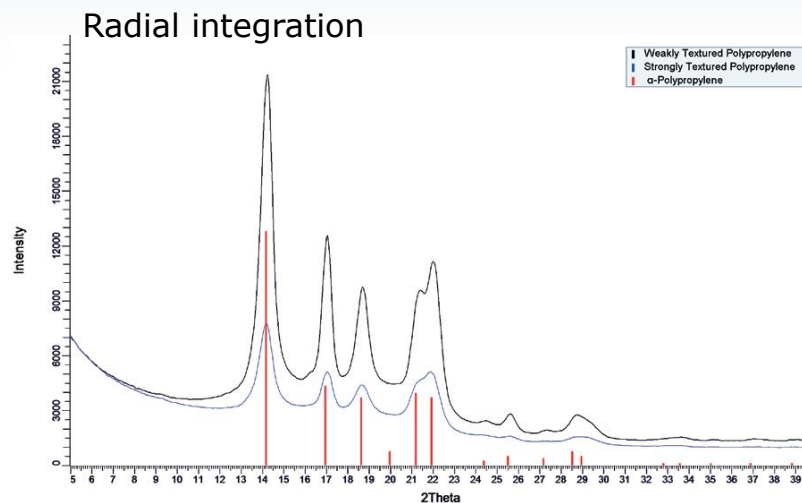
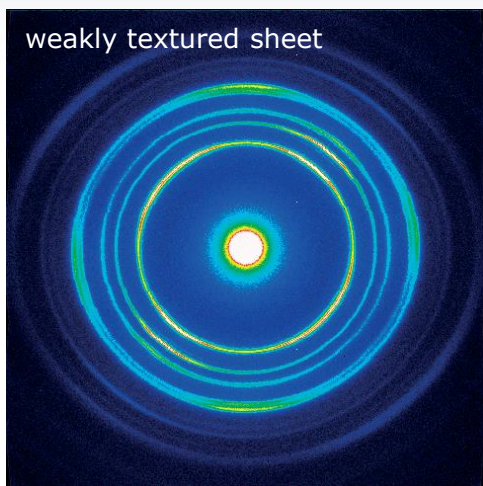
W Film	LP	Crys	Strain
Normal Scan	3.1565±0.0002	84.4±22.6	0.0008±0.0001



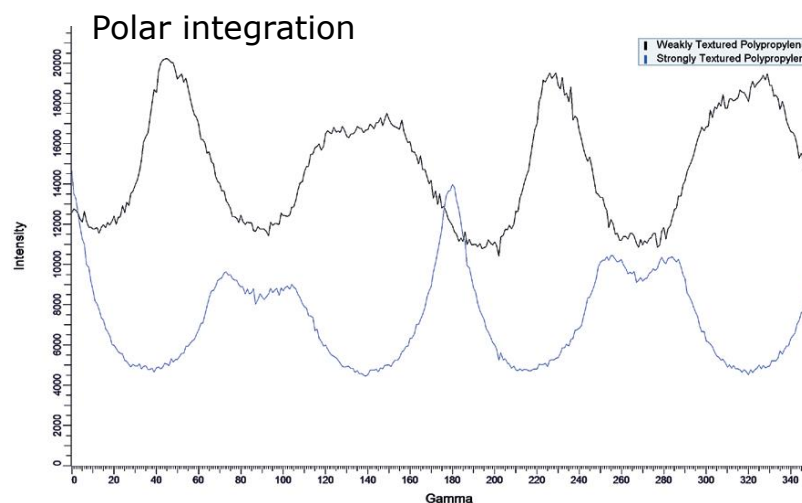
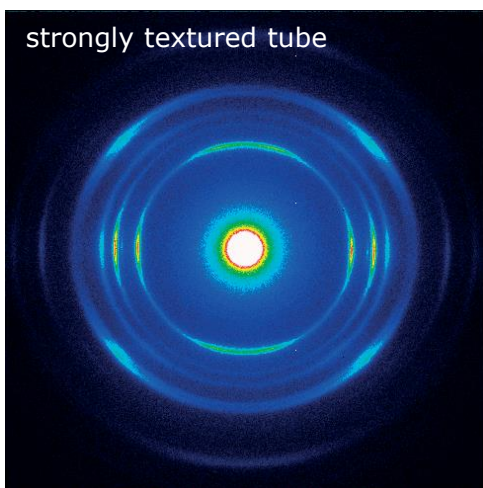
W Film	LP	Crys	Strain
IPGID Scan	3.1688±0.0003	53.1±16.8	0.0006±0.0002



# 2D Transmission Two Polypropylene Sheets



In addition to phase ID, radial integration contains information about microstrain and crystallite size



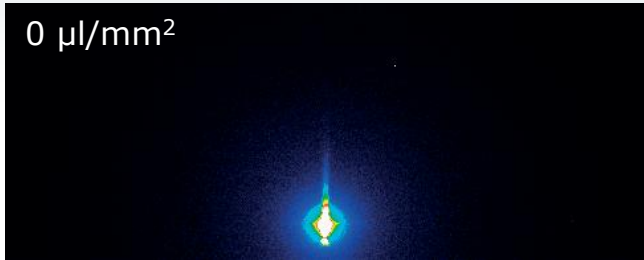
Polar integration gives information about the degree of orientation

Instrument Settings  
POLYCAP or TRIO Goebel Mirror + Collimator  
Stage: Compact Cradle+  
EIGER2: 2D Snapshot Mode

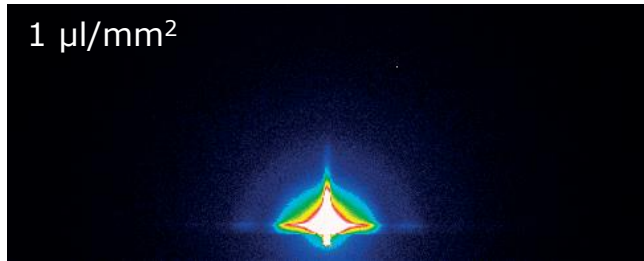
# Grazing-Incidence Small-Angle X-ray Scattering Ordering of Au Nanoparticles on a Surface



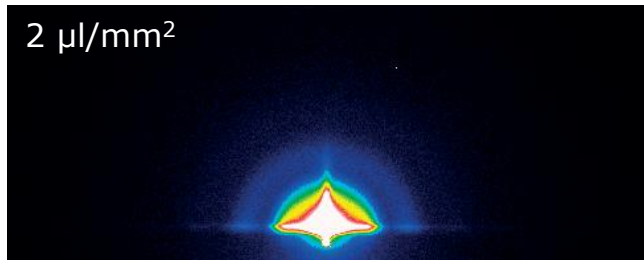
0  $\mu\text{l}/\text{mm}^2$



1  $\mu\text{l}/\text{mm}^2$

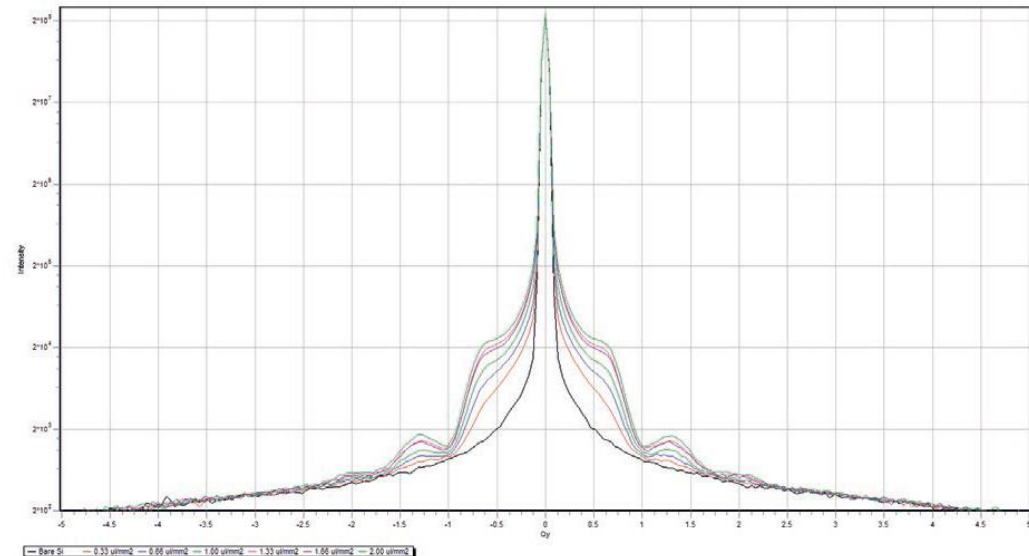
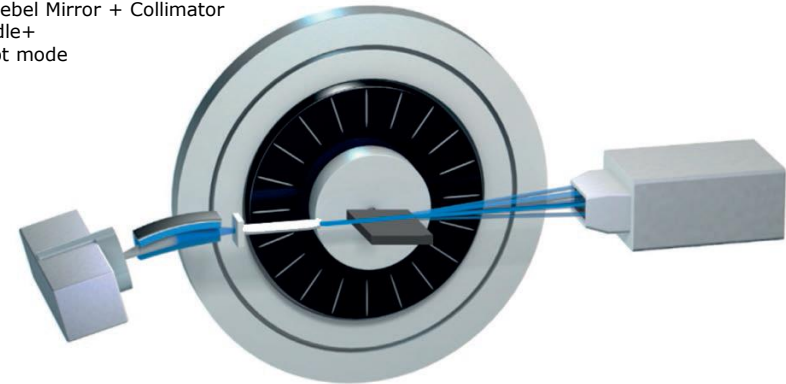


2  $\mu\text{l}/\text{mm}^2$



The isotropic scattering pattern indicates that the nanoparticles arrange in a 3D manner. As expected, higher order reflections become visible as the nanoparticle concentration is increased. First, second and third order reflections from the 10 nm Au nanoparticles are visible near the expected positions of  $Q = 0.62 \text{ nm}^{-1}$ ,  $Q = 1.26 \text{ nm}^{-1}$  and  $Q = 1.89 \text{ nm}^{-1}$  ( $Q=2\pi n/d$ )

Instrument Settings  
POLYCAP or TRIO Goebel Mirror + Collimator  
Stage: Compact Cradle+  
EIGER2: 2D Snapshot mode

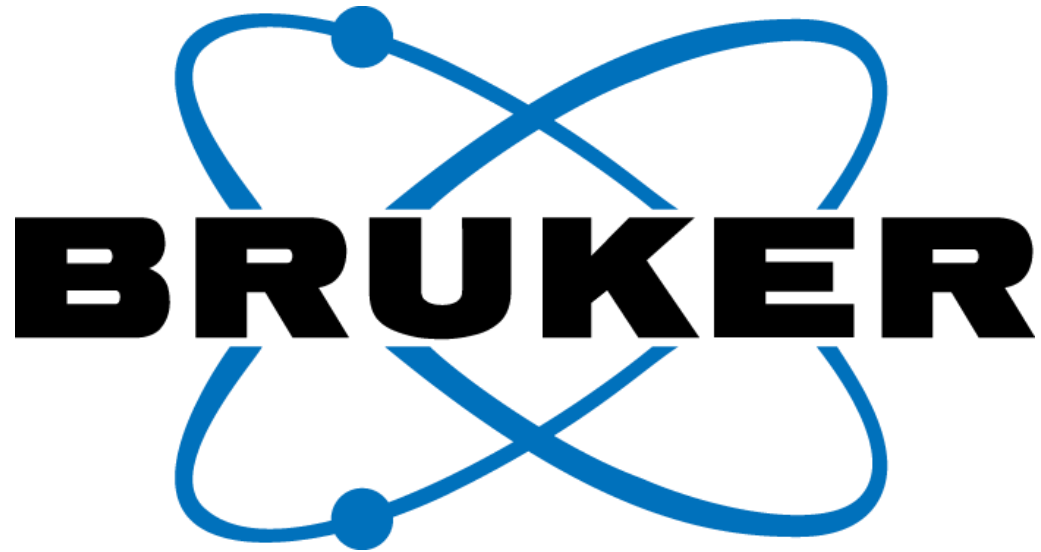


# D8 ADVANCE with TRIO and EIGER2 R 500K

## Applications Summary



- Powder Diffraction in Bragg-Brentano or Parallel Beam
  - Phase ID
  - Quantification
  - Indexing
  - Rietveld Refinement
  - Structure Solution
  - Bragg 2D
  - Non-Ambient Diffraction
  - Microdiffraction
  - Transmission
- Residual Stress Analysis
  - 2D Method
  - $\text{Sin}^2(\text{Psi})$  Method
  - Multi-HKL method
- Texture Analysis
  - Pole Figures
  - Orientation Distribution Function
    - Component Fitting
    - Harmonic Fitting
  - Inverse Pole Figures
- High-resolution XRD
  - Rocking Curves
  - On-Axis Coupled Scans
  - Reciprocal Space Mapping
- Grazing Incidence Diffraction
  - Phase ID
  - Depth Profiling
  - GI-SAXS
- In-Plane Grazing Incidence Diffraction
  - 2D Materials and ultra-thin films
  - Surface oxides
  - In-plane lattice parameters and crystallite size
  - Epitaxial films relationship to substrate
- X-ray Reflectometry
  - Thickness, density, roughness of single and multi-layered films
  - Crystalline, amorphous, or liquid layers



Innovation with Integrity