Ultima IV

X-RAY DIFFRACTOMETER

Rigaku

"Perhaps the greatest challenge facing X-ray diffractometer users today is how to minimize time and effort spent on reconfiguring the system for different applications." *Wade Adams, Ph.D., Director, Richard E. Smalley Institute for Nanoscale Science & Technology, Rice University*

Instrument downtime associated with reconfiguration reduces analytical flexibility by introducing a barrier between the desire and the opportunity of the user to make diverse measurements.

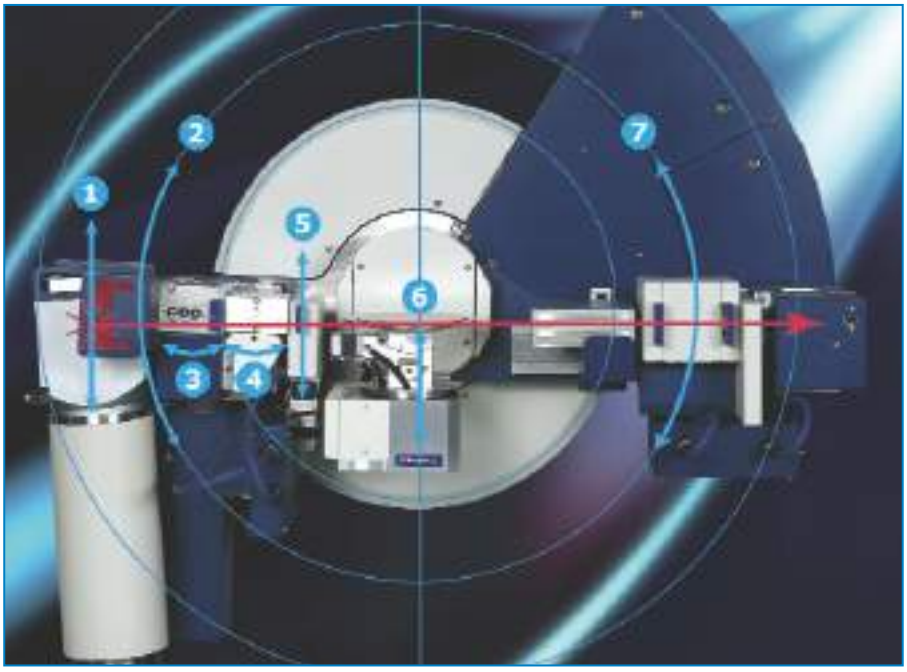
Time spent reconfiguring the diffractometer is time lost for sample measurement and data analysis.

Time and effort required for instrument reconfiguration is greatly impacted by the ease in which the system can be aligned.

What if your diffractometer aligned itself?

The Rigaku Ultima IV offers complete, fully automatic alignment of the entire system, including X-ray source, optics, goniometer, and sample stages.

Whether full system alignment at the time of installation or routine sample alignment before a measurement, Rigaku's automatic alignment functions reduce time and improve productivity.



1. Source height
2. Source angle
3. Cross Beam Optic
4. Crystal optics
5. Slit height
6. Sample surface
7. Detector angle

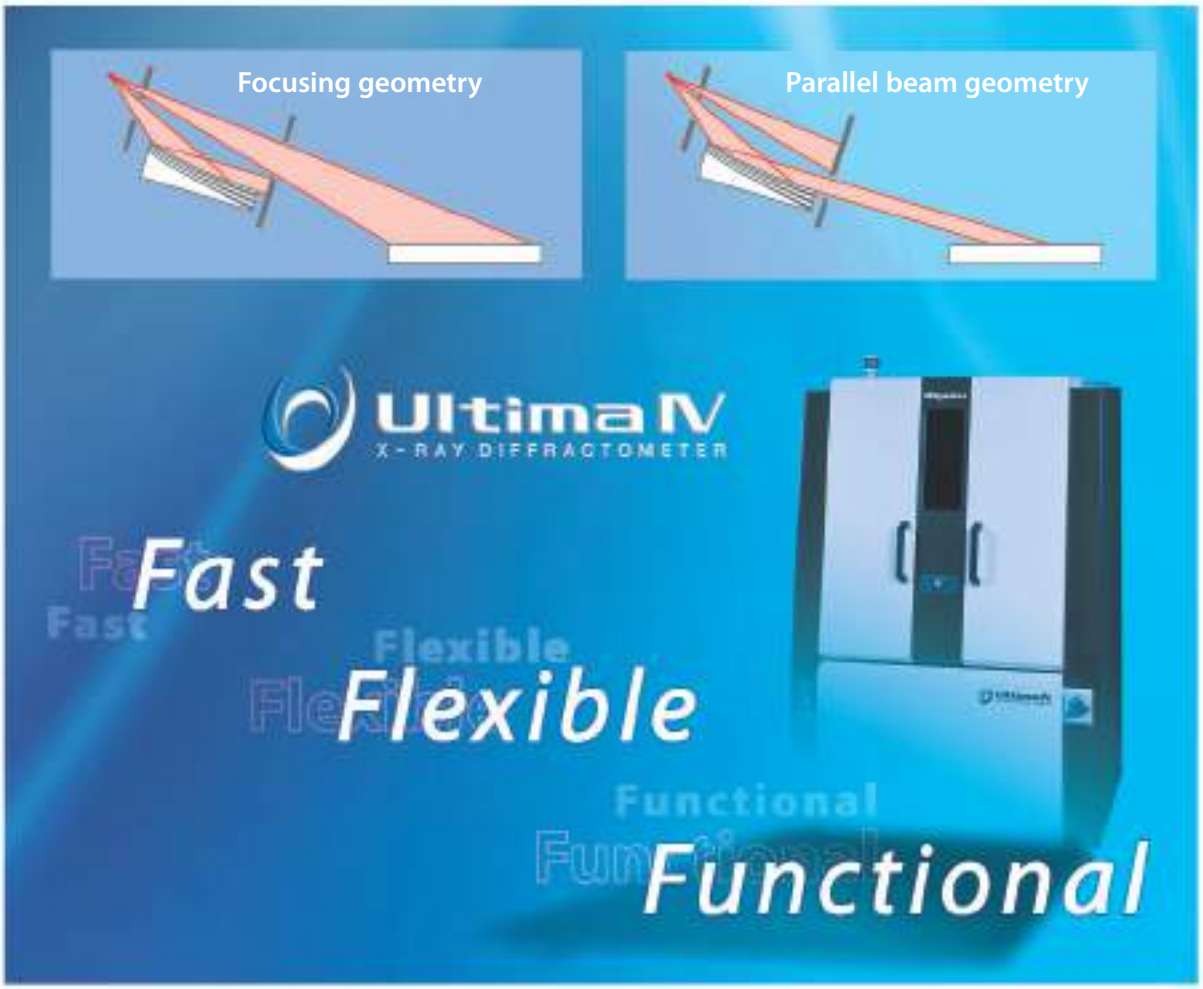
Start the automatic alignment procedure and have a cup of coffee* as the source height, source angle, Cross Beam Optic (CBO), crystal optics, slit height, sample surface, and detector angle are automatically aligned and optimized.

* Coffee not included

What if you could select focusing or parallel beam geometries without having to reconfigure the diffractometer?

Rigaku's patented Cross Beam Optics (CBO) design eliminates the need to switch between focusing (divergent beam), and mirror (parallel beam) geometries by having both sets of optics permanently mounted on the instrument, pre-aligned, and user selectable.

CBO technology eliminates time spent switching geometries, enables everyday users to run both sets of experiments without the need to reconfigure the system, and reduces wear and possible optic damage associated with the recurrent switching process.

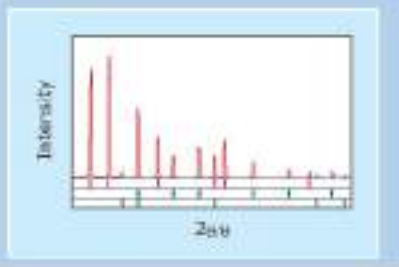


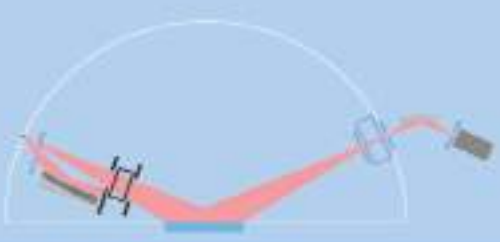
Automatic alignment and CBO for increased productivity and improved results

Ultima IV configurations

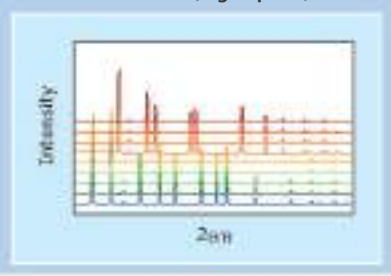
CBO technology, automatic alignment, and a flexible modular design combine to provide simple, fast reconfiguration for diverse applications.

Powder diffraction (focusing)

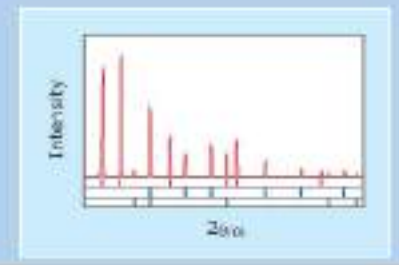


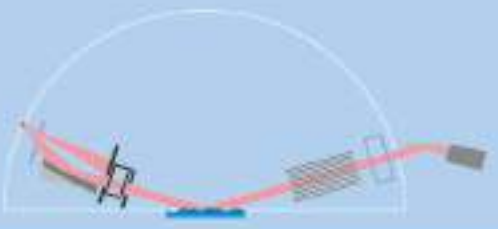


Powder diffraction (high-speed)

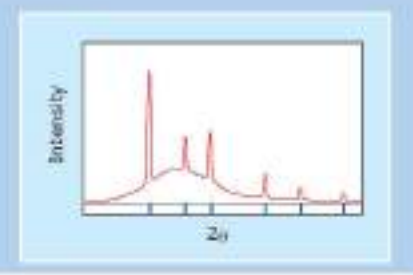


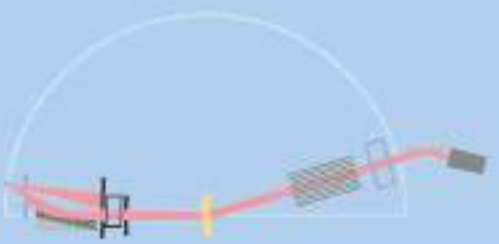
Powder diffraction (parallel beam)



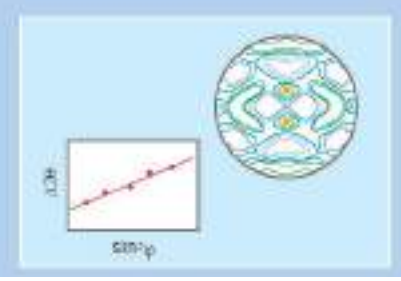


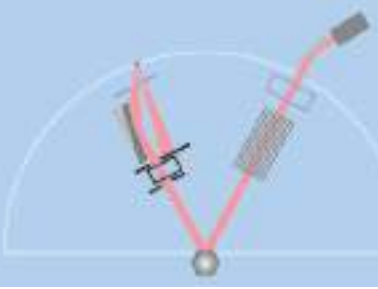
Transmission



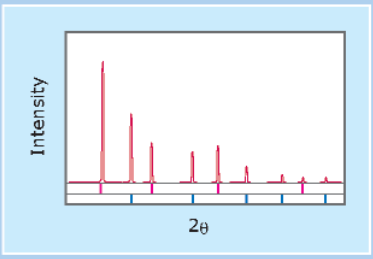


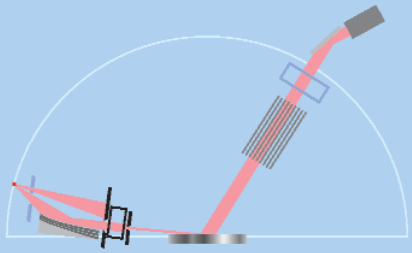
Stress and texture



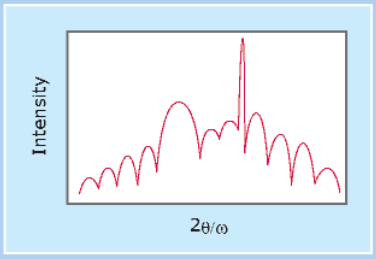


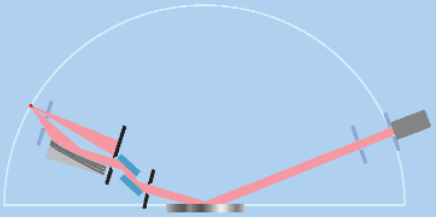
Thin film diffraction (glancing incidence)



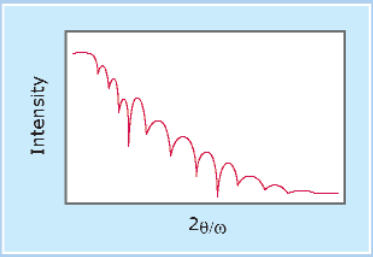


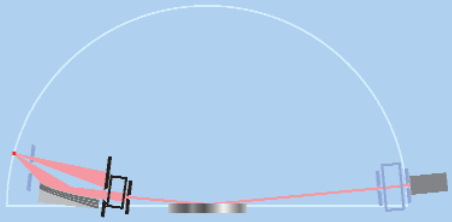
Thin film diffraction (high-resolution)



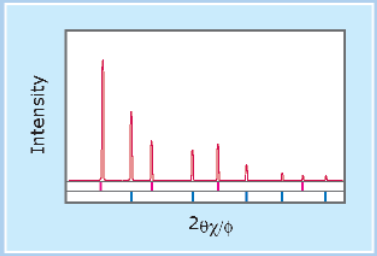


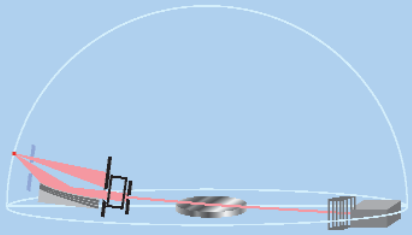
X-ray reflectometry



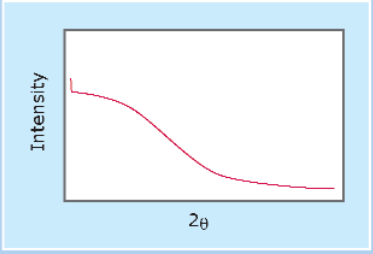


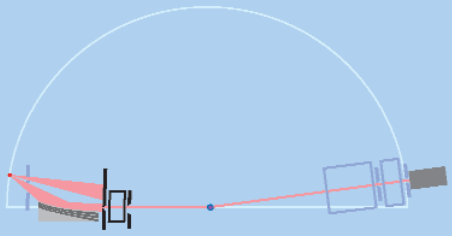
Thin film diffraction (in-plane)





Small angle X-ray scattering





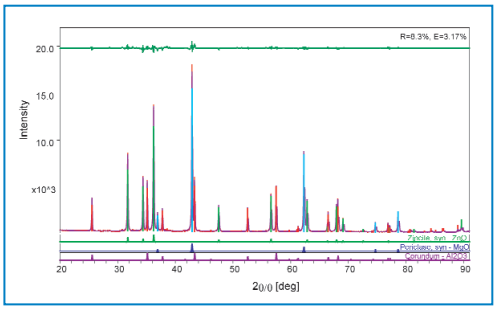
Rigaku

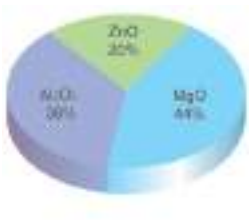
CBO technology provides unmatched flexibility and performance for powder diffraction measurements.

Traditional powder diffraction measurements use the Bragg-Brentano focusing geometry to provide high-intensity, high-resolution measurements of well powdered samples. For many bulk samples with surface irregularities, the use of parallel beam geometry is preferred. The on-demand availability of either geometry, fundamental to CBO, offers users complete flexibility without the need for reconfiguration.

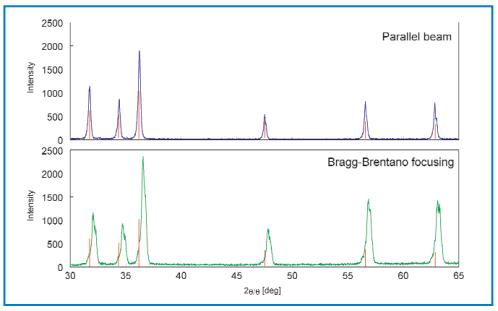
Supported powder diffraction applications include:

- **Phase identification**
- **Quantitative analysis**
- **Percent crystallinity**
- **Crystallite size/strain analysis**
- **Precise lattice parameter determination**
- **Rietveld refinement**

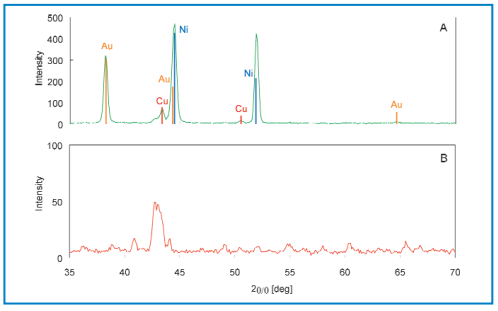




Accurate quantitative analysis using the Rietveld method is an example of an advanced X-ray solution made simple by the Ultima IV system. In this example, focusing geometry is used to obtain high-resolution peak profiles from a well prepared three phase mixture of ZnO, MgO, and Al_2O_3.



In the case where sample surface quality is poor, one touch selection of parallel beam geometry, made possible by CBO, enables accurate data to be collected under adverse conditions. In this example, the coarse grains of a photocatalyst cause no problems in data acquisition for the Ultima IV configured with CBO.



A seamlessly integrated XYZ positioning stage and magnified CCD camera system allow X-ray diffraction data to be easily collected from different points on a sample surface. In this example, the small area measurement system positioning capability and the intense parallel beam provided by CBO combine to make measurements from two different locations on a printed circuit board simple and fast.

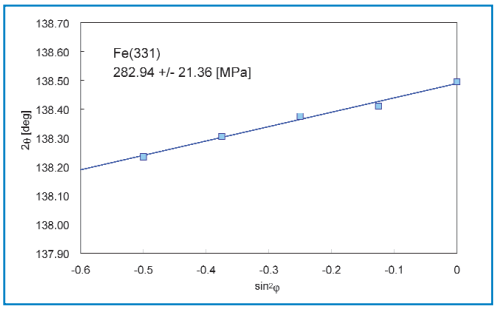
Automatic sample alignment and in-plane scanning combine for an easy-to-use, high-resolution stress and texture system.

Automated alignment makes the Ultima IV simple to use for stress and texture measurements. The on-demand in-plane scattering capability made possible by CBO offers two distinct advantages when compared to traditional pole figure measurements.

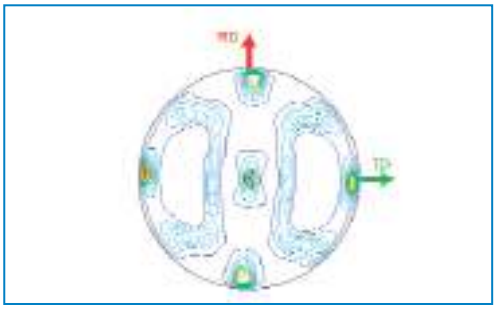
In-plane data collection can use a line focus beam, reducing data collection times and improving sampling errors in non-homogeneous materials. In-plane pole figure measurements remove the need to collect both reflection and transmission data sets for complete texture determination.

Supported stress and texture applications include:

- **Stress determination ($\sin^2\Psi$)**
- **Stress determination (Bi-axial)**
- **Conventional pole figures**
- **In-plane pole figures**
- **Inverse pole figures**
- **Transmission pole figures**
- **ODF (orientation distribution function) determination**



For residual stress measurements the determination of absolute peak positions is crucial. Since peak positions are strongly affected by sample displacement errors, CBO and automatic sample alignment are critical to both accurate results and ease of measurement. The figure to the left shows a traditional $\sin^2\Psi$ plot from an iron nut. This odd shaped sample is easily aligned and accurately measured using the Ultima IV configured with CBO.



On-demand in-plane scanning provides the ultimate in flexibility when collecting pole figure data. In this example, pole figure data collected from a Cu (111) plate using in-plane geometry extends out to the edge of the pole figure plot. It was not necessary to re-configure the system to point focus mode or re-mount the sample for transmission measurement to collect this "full", α = 0 to 90°, pole figure.

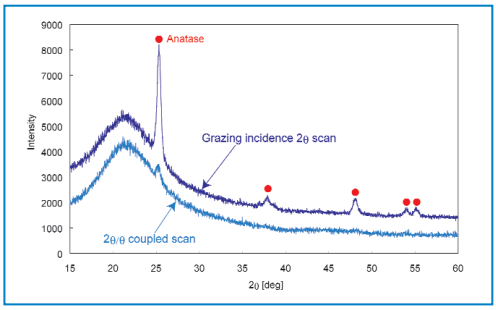
Rigaku

CBO and on-demand in-plane scanning combine to provide the world 's most comprehensive thin film diffraction system.

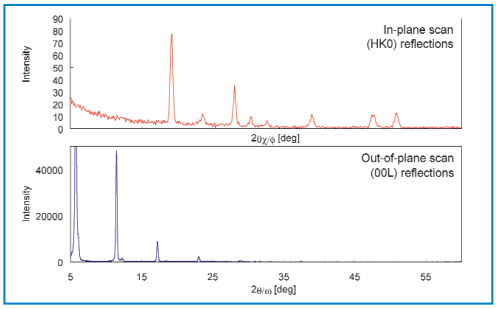
The ability to perform both in- and out-of-plane grazing incidence scans without the need for system reconfiguration revolutionizes thin film analysis. Layers as thin as 1 nm can be routinely measured with Rigaku's patented in-plane scanning goniometer. In-plane orientation and lattice parameters, difficult to measure with conventional asymmetric scanning, can be calculated directly from observed in-plane reflections.

Supported thin film applications include:

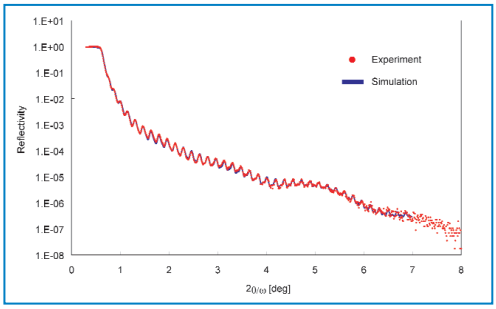
- **Phase identification**
- **Orientation/texture**
- **Crystal perfection**
- **Strain/stress**
- **Thickness**
- **Interface roughness**
- **Density**



The use of glancing incidence scanning is shown to be critical in this measurement of a photocatalyst thin film. CBO allows the measurement of thin films easily and in complement to other bulk materials and powders. The figure to the left shows the enhancement of the diffraction data in glancing incidence mode compared to a conventional $\theta/2\theta$ symmetric measurement. Diffraction peaks from the film are clearly visible in glancing incidence mode.

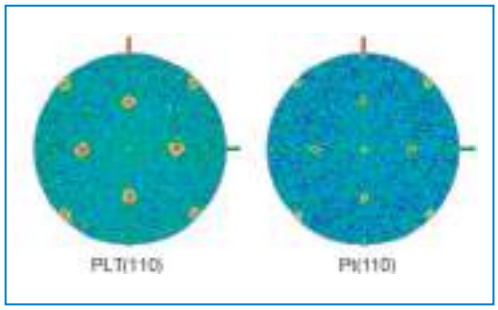


On-demand, combined in-plane and out-of-plane scanning provides a full picture of the texturing in thin films. In this example, in-plane (00L) and out-of-plane (HK0) scans were made on a 50 nm Si/pentacene thin film structure. The in-plane measurement done at a 0.18° glancing incident angle provides a unique look at the orientation of the film.

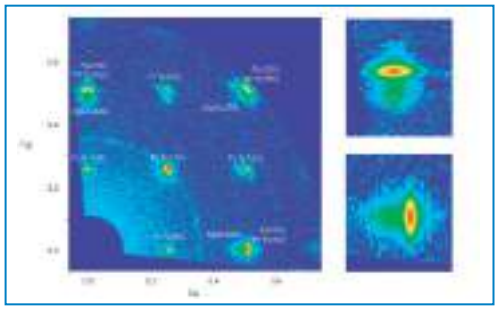


X-ray reflectivity (XRR) measurements are used to determine thin film thickness, surface roughness, interface roughness, and density in layered materials. Again, the one touch selection of parallel beam geometry provided by CBO, and automatic sample alignment critical to XRR, makes performing these measurements simple. The example to the left shows both the calculated and experimental collected XRR curves from a three layer stack of oxidized GaAs, GaAs, InGaAs on a GaAs substrate.

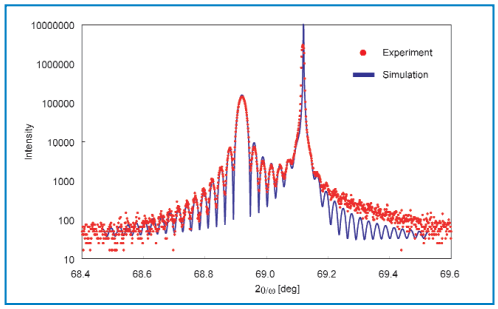
Analysis of thin film materials



For thin film materials, pole figure analysis can be used to determine orientation relationships between substrates and deposited materials. In this example, in-plane pole figures were collected on both the Pt substrate and (Pb, La)TiO_3/Pt/MgO PLT thin layer. The epitaxial relationship between the substrate and layer material is clearly shown. Critical to this measurement is the in-plane geometry, which allows full pole figures to be collected on both the substrate and the thin layer.



Perhaps the most useful measurement for the rigorous interpretation of thin film properties by X-ray diffraction is the reciprocal space map (RSM). RSMs show both the location and characteristics of reciprocal space points coming from epitaxial heterostructures. RSMs can be used to interpret orientation, perfection, strain, and relaxation in complex materials. In this example, the use of in-plane scanning allows information to be gathered in directions parallel to the thin film sample surface.



For high-resolution X-ray diffraction, the CBO module provides a high-quality, intense, parallel beam for further conditioning by the automatically aligned multiple crystal optics, proving optimal performance from a simple-to-use configuration. A high-resolution rocking curve (left) shows peak separation between the layer and substrate reflections, giving alloy composition.

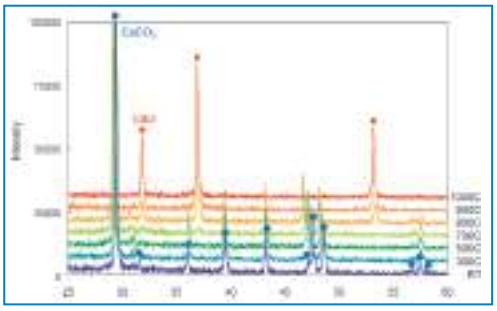
Rigaku

Rigaku non-ambient attachments are designed and manufactured by Rigaku, ensuring seamless integration and functionality.

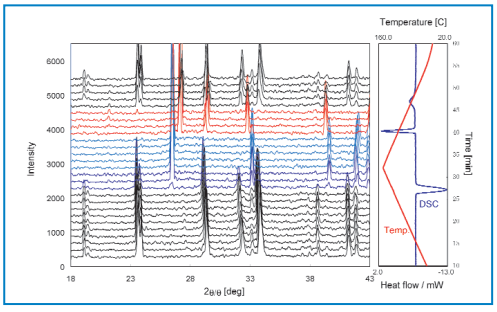
The ability to measure samples under specific non-ambient conditions is essential in the research and development of many advanced materials. The Ultima IV has a wide range of fully integrated non-ambient configurations, including the world's only commercially available combined XRD/DSC system.

Non-ambient capabilities include:

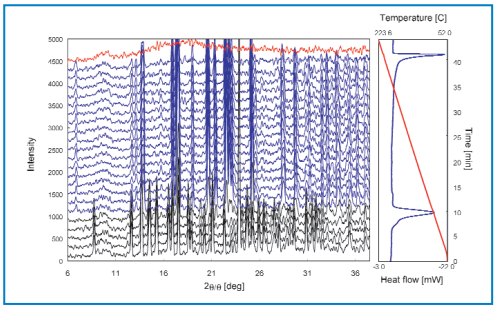
- **High temperature**
- **Low temperature**
- **Variable humidity**
- **Reactive gases**
- **Combined XRD/DSC**



Collecting XRD data at variable temperatures can provide structural information on materials under conditions similar to those in which the material will ultimately be used. In this example, the Ultima IV with CBO in focusing mode coupled with a fast position sensitive detector is used to collect high-speed (two minutes per scan) diffraction data from a $CaCO_3$ sample undergoing a thermally induced reaction to CaO. The transition to CaO can be seen to occur at approximately 800°C.



Perhaps the most unique non-ambient capability of the Ultima IV is the combined XRD and DSC configuration. The ability to measure XRD/DSC data from the same sample at the same time can be critical in the accurate study of heat-based transitions in many materials. The figure to the left shows the combined XRD/DSC data display in which multiple XRD patterns, the variable temperature curve, and the associated heat flow data can be observed. In this example, a sample of KNO_3 is observed as the material undergoes three distinct, reversible solid state transitions.



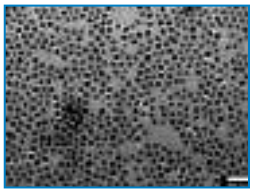
The Ultima IV combined XRD/DSC configuration can also operate under conditions of variable humidity. In this configuration, hydration reactions can be studied as a function of both variable temperature and variable humidity. In the figure to the left, XRD patterns at temperatures from 50 to 275°C were collected from a trihalose sample under constant humidity. As in the example above, the XRD patterns, temperature curve, and heat flow data are displayed. A crystalline anhydrous phase is directly generated under theses conditions, as indicated by the XRD data.

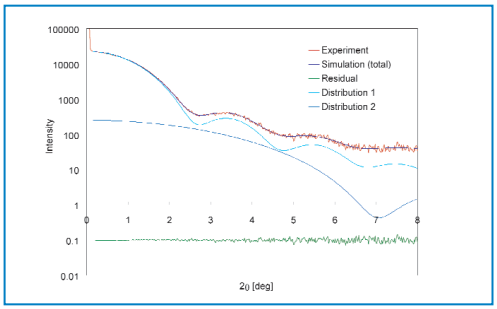
Small angle X-ray scattering (SAXS) measurements of nanomaterials is the fastest growing application in analytical X-ray analysis.

Rigaku's patented CBO SAXS design allows SAXS measurements to 0.1 in q (65 nm) to be performed routinely and in direct complement to other wide angle scattering and diffraction methods. Both solid and liquid samples can be measured in a wide range of SAXS geometries, including glancing incidence (GISAXS).

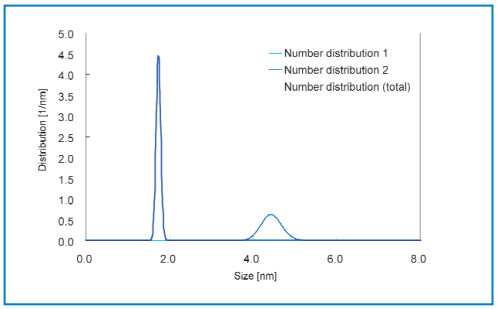
SAXS applications include:

- **Macromolecular structure and orientation in polymeric materials**
- **Particle size distributions of nanoparticles suspended in solution**
- **Particle/pore size distributions in deposited or bulk solid nanomaterials**
- **Molecular morphology and orientation in nanocomposites**

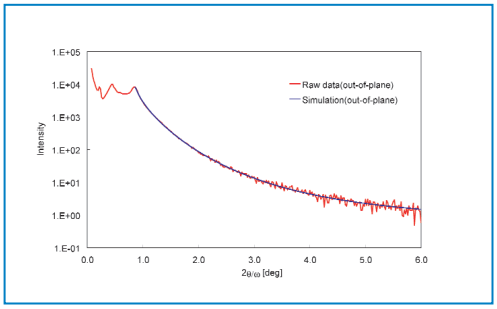




Transmission SAXS from nanoparticles suspended in solution is a powerful method for the determination of particle sizes and shapes. The requirement for accurate size and shape information is important in many of the emerging areas of nanomaterials research currently taking place throughout the world. The Ultima IV with CBO offers one touch switching from conventional wide angle scattering and diffraction geometries to the ultra-low angle performance needed for SAXS measurements.



In this example, the experimental SAXS curve (above left) has been overlaid with the calculated SAXS curves from a bimodal distribution of particle sizes (left) modeled in NANO-Solver, Rigaku's powerful software solution for SAXS data processing. The agreement between the experimental and calculated curves shows that the proposed distribution model is in good agreement with what is actually contained in the sample, as observed by TEM.



In addition to transmission measurements, the CBO SAXS geometry with automatically aligned sample stage is capable of performing reflection SAXS. The figure to the left is a reflection SAXS measurement recorded from a 100 nm thin layer of Ni embedded C on Si. The figure shows output from NANO-Solver displaying the overlaid experimental and calculated reflection SAXS curves. The agreement of the calculated and experimental curves suggest a broad distribution of Ni particle sizes ranging from 2 to 10 nm.

Rigaku

www.Rigaku.com

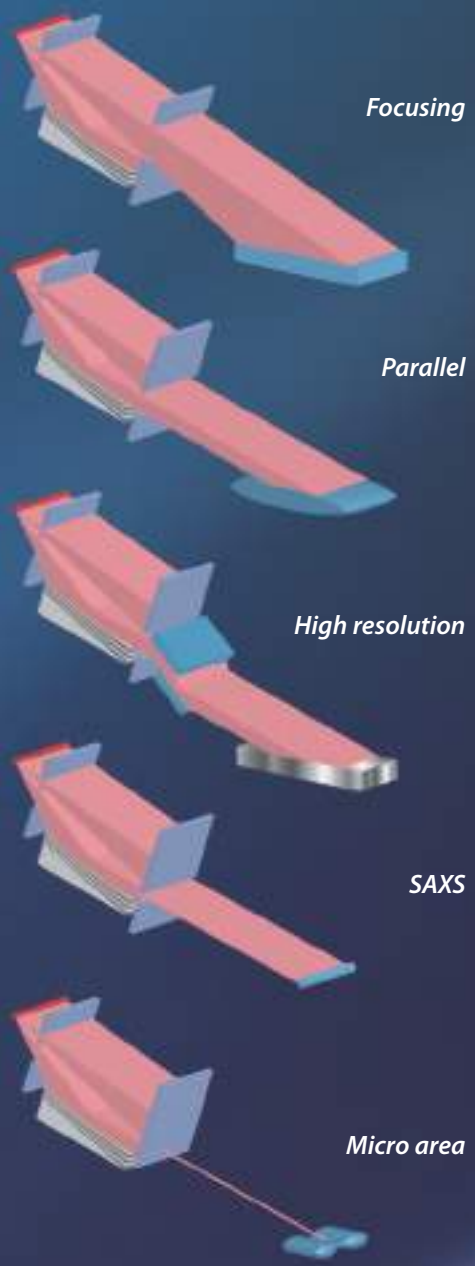
Ultima IV

Specifications

X-ray generator	Maximum rated output	3 kW		
	Rated tube voltage	20 - 60 kV		
	Rated tube current	2 - 60 mA		
	Target	Cu (others: optional)		
	Focus size	1 x 10 mm (others: optional)		
Goniometer	Scanning mode	θs/θd coupled or θs, θd independent		
	Goniometer radius	185 or 285 mm		
	2θ measuring range	-3 to 162° (maximum)		
	Minimum step size	0.0001°		
Optics	Divergence slit	Fixed or automatic variable		
	Scattering slit	Fixed or automatic variable		
	Receiving slit	Fixed or automatic variable		
	Optics alignment	Automatic alignment of tube height, goniometer, optics and detector		
	Monochromator	Dual position graphite diffracted beam monochromator for Cu (others: optional)		
Detector	Detector	Scintillation counter (others: optional)		
Dimensions (mm)	Height 1600	Width 1100	Depth 800	Sample height 1050

Rigaku Corporation
4-14-4, Sendagaya, Shibuya-ku
Tokyo 151-0051, Japan
Phone: 81-3-3479-0618
FAX: 81-3-3479-6112
E-mail: rinttyo@Rigaku.co.jp

Cross Beam Optics



SYS_BRO_UIV_01