

# SmartLab

Automated multipurpose X-ray diffractometer

Rigaku's flagship X-ray diffractometer



**Rigaku**

Leading With Innovation

# Leading With Innovation

## Next generation Rigaku SmartLab<sup>®</sup> intelligent multipurpose X-ray diffractometer

A highly versatile automated X-ray diffraction (XRD) system, the newest SmartLab diffractometer offers continued refinement of the ease-of-use features that enabled the original SmartLab diffractometer to receive the coveted R&D 100 Award, such as automatic alignment, component recognition, Cross Beam Optics and a 2D detector. SmartLab began as the flagship model from Rigaku in 2006 and new leading-edge, advanced technologies have been continuously introduced over the years. This newest addition to the SmartLab series of high-resolution X-ray diffraction analyzers is engineered to provide the best performance in all X-ray diffraction or scattering applications by offering not only breakthrough hardware, but also advanced "User Guidance" functionality within the new SmartLab Studio II software, to establish a new industry standard for multipurpose X-ray diffractometers.

Key features and benefits of the new SmartLab include:

- Highest flux X-ray source: PhotonMax
- HyPix-3000 high energy resolution 2D detector
- New CBO family, with fully automated beam switchable CBO-Auto and high-resolution micro area CBO- $\mu$
- Various *operando* measurements with the new SmartLab Studio II



# High performance X-ray source

## PhotonMax

The new generation rotating anode, PhotonMax, reduces the X-ray generator's environmental footprint, although it still produces 9 kW of high-power X-rays. The flux generated by the PhotonMax is about 5 times greater than a conventional sealed X-ray tube loaded at 1.8 kW. This enables you to see the fine details of your sample within a reasonable measurement time. The PhotonMax has a newly designed anode that provides more than 3 times longer lifetime than its predecessor. This maximizes the uptime of the instrument and minimizes the cost of ownership while reducing the environmental footprint.

### Specifications

Output	9 kW
Focus	Fine focus
Target material	Cu, Co, Cr, Mo
Long lifetime target is adopted.	





A new mechanical seal has been introduced to substantially improve the lifetime of the target.



# Leading-edge hybrid pixel array detector

## HyPix-3000\*

Multidimensional semiconductor detector

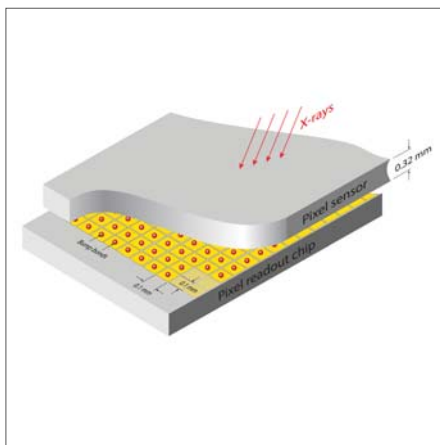


- Supports 0D, 1D and 2D measurement modes
- Excellent energy resolution to suppress XRF
- Keeps background noise to an absolute minimum
- Wide dynamic range
- Shutterless measurement
- Maintenance free

Active area	2,984 mm <sup>2</sup> (77.5×38.5 mm)
Pixel size	100 μm × 100 μm
Number of pixels	775 × 385 = 298,375 pixels
Global count rate	>2.9 × 10 <sup>11</sup> (>1×10 <sup>6</sup> cps/pixel)
Efficiencies	Cr, Co, Cu: ~99% Mo: ~38%
Energy resolution	40% better than previous type



Fully compatible with 5-axis goniometer design



Hybrid pixel array detector (HPAD) design



Shutterless high-speed *in-situ* measurement

\*This product was jointly developed by Department of Measurement and Electronics, AGH University of Science and Technology (Poland) and Rigaku Corporation.

Achieve excellent energy resolution

X-ray fluorescence background emitted by samples makes it difficult to detect minute peaks or scattering signals from amorphous components. It may also impede the correct calculation of the intensity of any detected peaks.

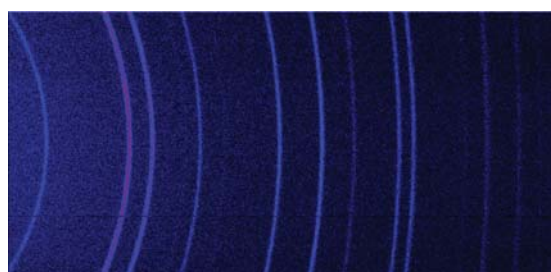
Unnecessary X-ray fluorescence can be removed using a monochromator placed between the sample and the detector. For two-dimensional (2D) measurement intended to detect Debye rings, however, no optical system can be inserted between the sample and the detector.

For the removal of X-ray fluorescence during 2D measurement, a mode in which the energy resolution of the detector can be used to disable counting of unnecessary X-ray fluorescence is available.

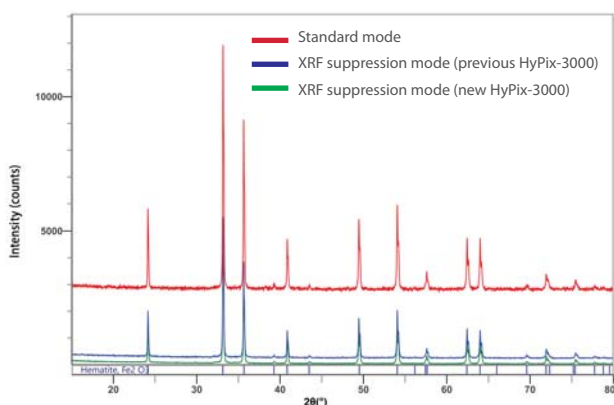
The excellent energy resolution greatly contributes to background suppression.



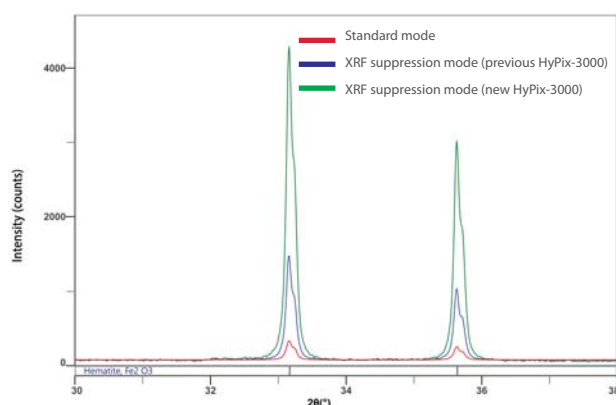
2D diffraction image measured in standard mode



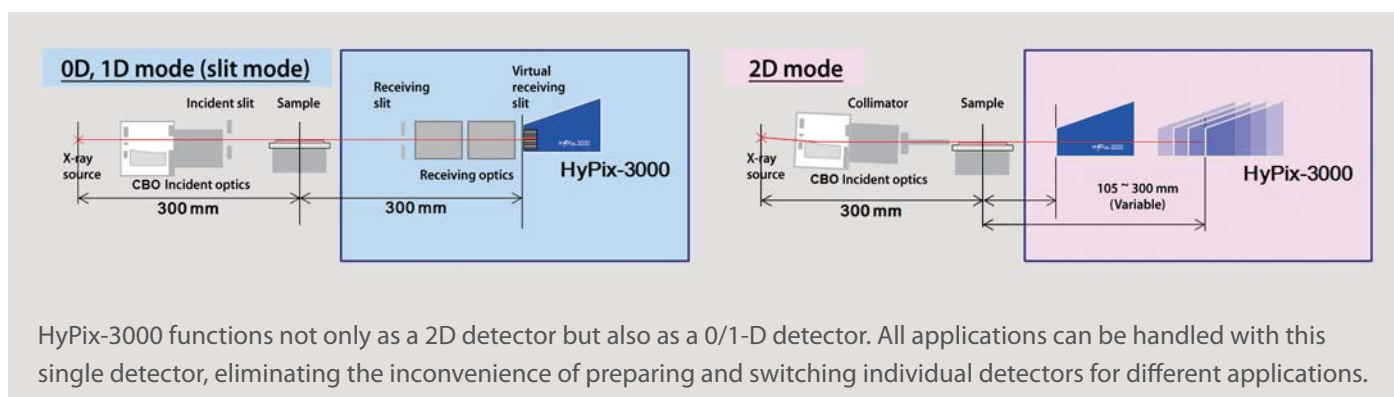
2D diffraction image measured in XRF suppression mode



1D diffraction pattern obtained by standard mode, XRF suppression mode (previous HyPix-3000) and XRF suppression mode (new HyPix-3000)




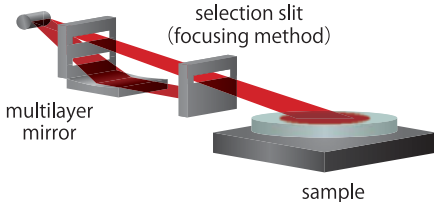
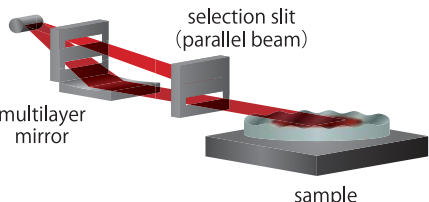

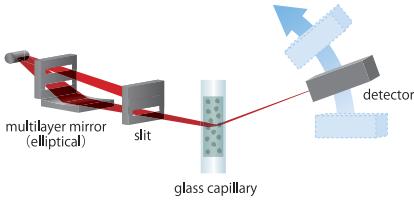

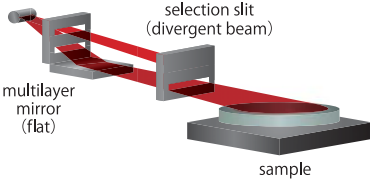

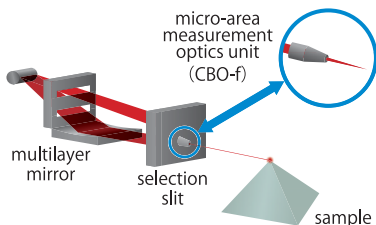

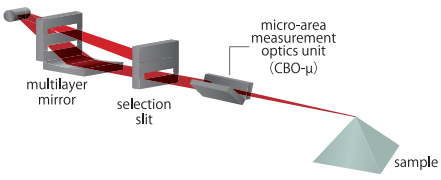
A graph of the same data as in the left figure normalized by the background noise intensities



HyPix-3000 functions not only as a 2D detector but also as a 0/1-D detector. All applications can be handled with this single detector, eliminating the inconvenience of preparing and switching individual detectors for different applications.

# Optical configurations for various applications

## CBO (Cross Beam Optics)

 <p>CBO</p>	 <p>selection slit (focusing method)</p> <p>multilayer mirror</p> <p>sample</p> <p><b>Divergent beam</b></p>	 <p>selection slit (parallel beam)</p> <p>multilayer mirror</p> <p>sample</p> <p><b>Parallel beam</b></p>
 <p>CBO-E</p>	 <p>multilayer mirror (elliptical)</p> <p>slit</p> <p>glass capillary</p> <p>detector</p> <p><b>Divergent beam/convergent beam</b></p>	  <p>selection slit (divergent beam)</p> <p>multilayer mirror (flat)</p> <p>sample</p> <p><b>CBO-α</b></p> <p><b>Divergent beam (High intensity, low background noise)</b></p>
 <p>CBO-f</p>	 <p>micro-area measurement optics unit (CBO-f)</p> <p>multilayer mirror</p> <p>selection slit</p> <p>sample</p> <p><b>Converges line beams to a minute point of <math>\approx 400 \mu\text{m}</math>. No need to change the X-ray tube focus.</b></p>	  <p>micro-area measurement optics unit (CBO-μ)</p> <p>multilayer mirror</p> <p>selection slit</p> <p>sample</p> <p><b>CBO-μ</b></p> <p><b>Converges line beams to a high-intensity and parallel beam of <math>\approx 100 \mu\text{m}</math>. No need to change the X-ray tube focus.</b></p>

## Detectors



1D semiconductor detector D/tex Ultra250/250HE	
Active area	384 mm <sup>2</sup> (19.2×20 mm)
Spatial resolution	75 μm
Global count rate	$2.5 \times 10^8$ ( $1 \times 10^6$ cps/pixel)
Efficiencies	Cr, Co, Cu: ~99% Mo: ~40%, ~70% (250 HE)



Multidimensional semiconductor detector HyPix-400*	
Active area	369 mm <sup>2</sup> (9.6×38.5 mm)
Pixel size	100 μm × 100 μm
Global count rate	$>3.7 \times 10^{10}$ cps ( $>1 \times 10^6$ cps/pixel)
Efficiencies	Cr, Co, Cu: ~99% Mo: ~38%

\*This product was jointly developed by Department of Measurement and Electronics, AGH University of Science and Technology (Poland) and Rigaku Corporation.



## CBO-Auto: Fully automatic switch between reflection and transmission optics and geometries



Reflection mode

The optimal measurement method depends on the type of sample or the application.

The Bragg-Brentano focusing (reflection mode) is the standard measurement method for generic powder samples. For samples with specific orientation or large grains (i.e., powder, solid, or films), the transmission method is the optimal approach.

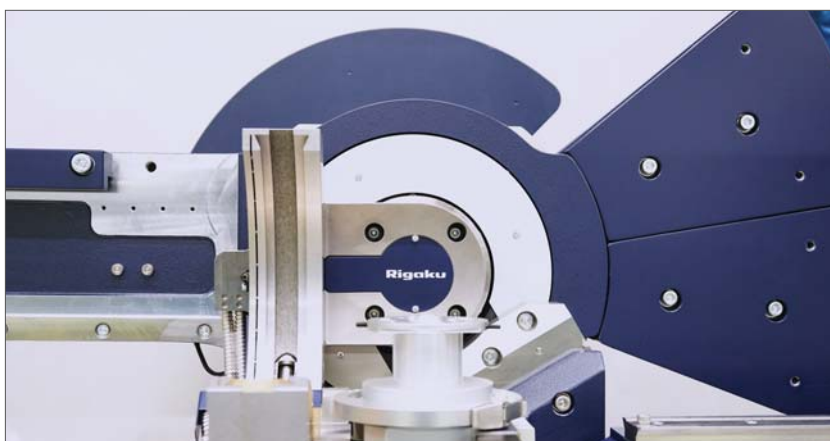
SmartLab provides fully automatic switching between the reflection and transmission methods.

CBO-Auto	
Ts axis	Automatic control
Optics	CBO-Auto (Cu) / CBO-Auto (Mo)
Sample stage	Reflection/transmission ASC-6



Transmission mode

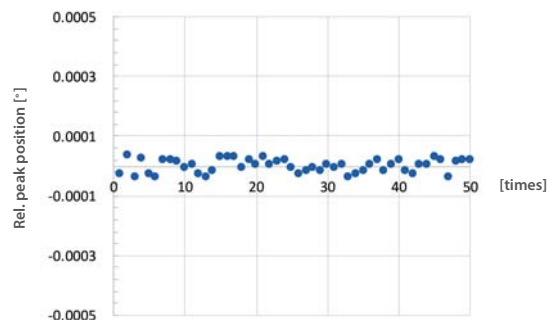
## High-precision goniometer with optical encoders



Encoder controlled high-precision goniometer

Type	Vertical goniometer with sample horizontal mount
Goniometer radius	300 mm (0D, 1D), 150 - 300 mm (2D)
Minimum step size	0.0001°

Reproducibility of the peak positions



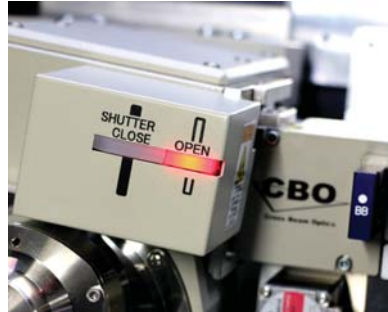
Peak position stability after 50 times repeat of  $2\theta$ - $\omega$  scan or  $004$  diffraction of silicon single crystal substrate. Distribution is within the range of reference accuracy  $\pm 0.00004^\circ$ .

# Designed for functionality and safety





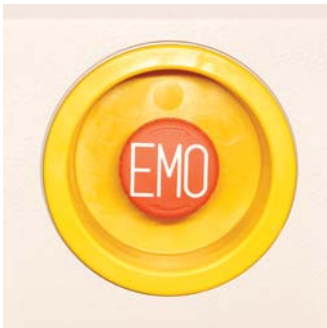
Shutter CLOSE lamp



Shutter OPEN lamp



X-rays on lamp



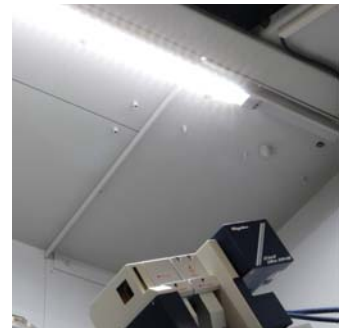
EMO button



Interlock



Main key



LED light



Error lamp



Door lock lamp



Generator on lamp



Power on lamp

- **Safety-friendly enclosure design**
  - Leakage X-ray dosage 0.1  $\mu\text{Sv}$  or less
  - Safe interlock mechanism mounted even in case of erroneous operation
- **Design based on ergonomics**
  - Easy access to sample position
  - Wide door opening improves accessibility to the inside of the device, which allows smooth changing of attachments
- **High visibility design**
  - Six wide-angle windows let users check the state of the sample from various angles
  - Indicator for easy confirmation of equipment status
  - An easily recognizable LED lamp system has been adopted

# SmartLab Studio II software suite

SmartLab Studio II is an integrated software platform with all functions from measurement to analysis.



Selection of sample type,  
Auto selection of measurement program

Optics change guidance/  
(Auto optics adjustment)

## Typical applications

<b>Powder</b>		<ul style="list-style-type: none"> <li>Phase identification</li> <li>Quantification</li> <li>Crystallite size and distortion</li> <li>Precise lattice parameter determination</li> <li>Percent crystallinity</li> <li>Indexing</li> <li>Structural determination</li> <li>Precise crystalline structure determination</li> </ul>	
<b>Stress</b>		<ul style="list-style-type: none"> <li><math>\text{Sin}^2\psi</math> method</li> <li>2D method</li> <li>Multiple-HKL method</li> </ul>	
<b>Small Angle Scattering (SAXS)</b>		<ul style="list-style-type: none"> <li>Grain size distribution</li> <li>Pore size distribution</li> <li>Long period</li> </ul>	

## Micro area measurement

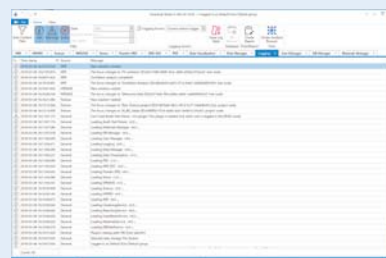
	<b>Specifications of beam size</b>	
Collimator optics	50 $\mu\text{m}$ to 1 mm	
CBO-f	400 $\mu\text{m}$	
CBO- $\mu$	100 $\mu\text{m}$	
No need to change X-ray tube focus		



Measurement, data processing, analysis

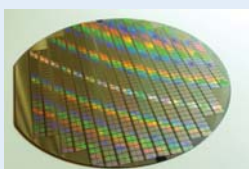


Reporting

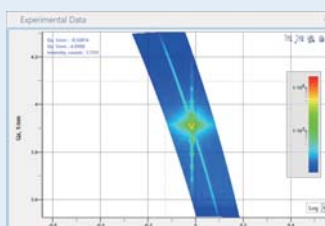


Control/analysis history view

## Thin Film



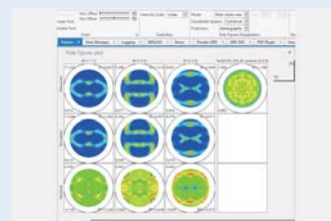
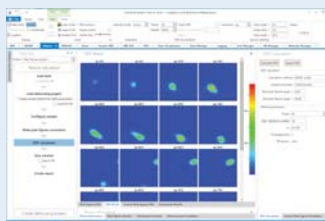
- Film thickness
- Density
- Roughness
- Composition



## Texture



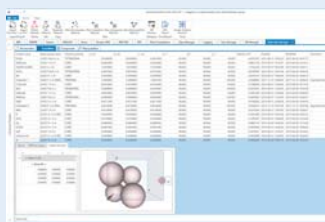
- Pole figure
- Stereographic projection
- ODF calculation
- Reverse pole figure



## Radial distribution PDF



- PDF calculation
- Simulation



## Large samples stage



### Specifications

Movable range	X-axis: -37~50 mm, Y-axis: -50~50 mm, Z-axis: -20~20 mm
Min step width of each axis	0.001 mm
Max sample weight	20 kg

# Advanced powder X-ray diffractometry

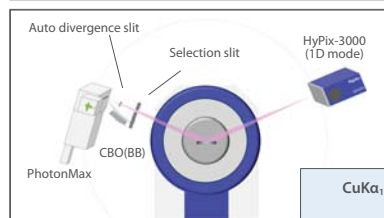


- Leading-edge 2D pattern direct qualitative analysis (2D-ID: 2D pattern phase identification).
- Seamless execution from qualitative analysis to Rietveld refinement.
- XRD data docking with DSC or other data.

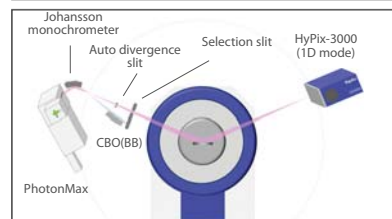
## SmartLab Studio II Rietveld refinement

- Automatic input of required initial crystal structure from qualitative analysis result.
- Fitting with WPPF (Whole Powder Pattern Fitting) method.
- Crystallite size distribution analysis using FP (Fundamental Parameter) method.
- Estimation of oxidation state of metal atoms using BVS (Bond valence sum) method.
- Unknown crystal structure analysis package including from indexing to structure determination is available.

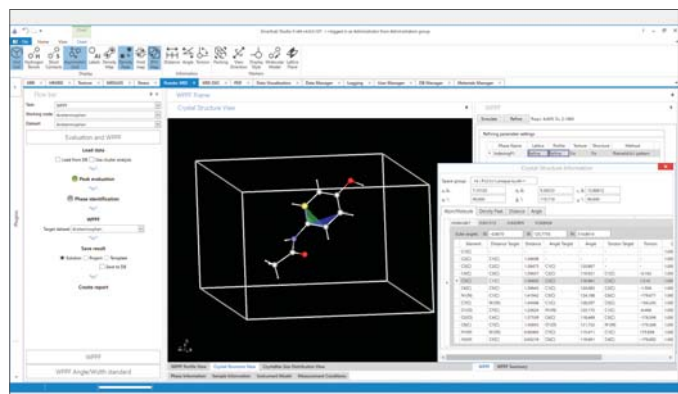
### Bragg-Brentano focusing optics



### CuKα, high resolution optical configuration with Johansson optics



Cement qualitative analysis and quantitative analysis using Rietveld refinement



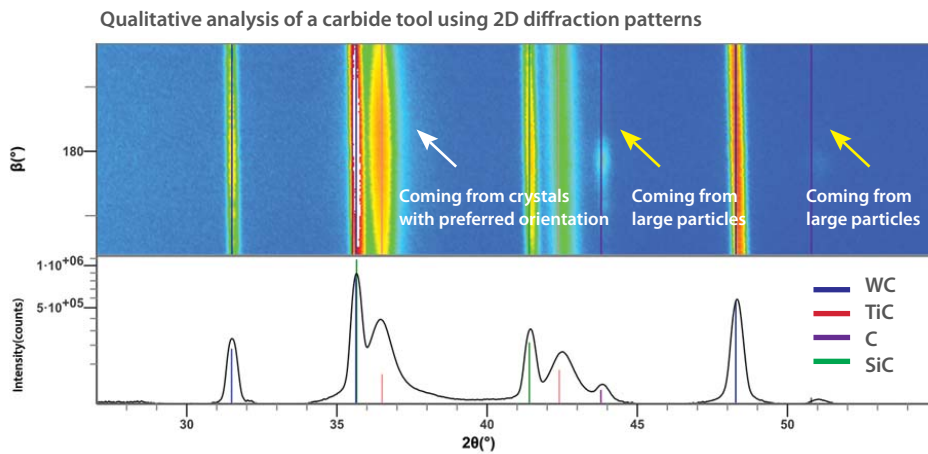
Organic powder precise crystalline structure determination

# Advantage of qualitative analysis from 2D diffraction patterns

For powder X-ray diffractometry, samples are generally crushed into grains small enough to obtain ideal diffraction patterns. However, the crushing process may cause crystal phase transition. In addition, there is often a need to measure bulk or thin film samples without crushing.

For powders with large crystal grains (coarse particles) or with preferred orientation, the conventional powder measurement method may produce unreliable peak intensities or no observable peak at all, which has been a barrier to ensure reliable qualitative analysis.

The HyPix-3000 2D detector can be used to obtain 2D patterns for powder diffraction. These 2D patterns include distinctive features implying coarse particles or preferred orientation. These patterns can be processed to enable qualitative analysis in which the 2D pattern information is added to the 1D patterns.



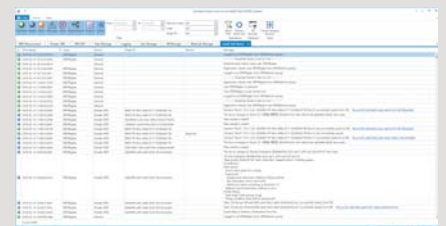
## Pharmaceutical solutions

Compliance to FDA 21 CFR Part 11

- High-level security
- Audit trail
- IQ/OQ/PQ
- Computer system validation



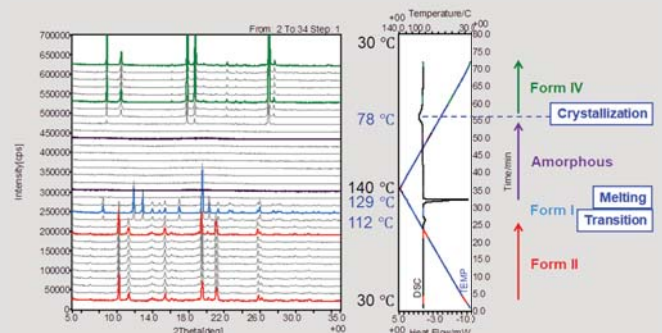
Validation document  
IQ/OQ/PQ



Electronic recording/signature  
(ER/ES) software



Unique XRD-DSC attachment



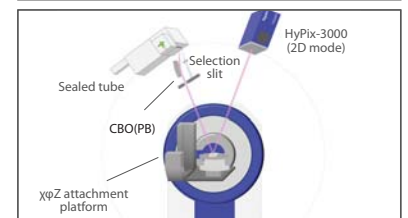
Simultaneous XRD-DSC measurement of tolbutamide  
(antidiabetic drug)

# Residual stress analysis



- Wavelength can be selected for the type of metal or ceramics (Cr, Co, Cu, Mo).
- Supports  $\sin^2\psi$ , Multiple HKL and 2D (triaxial stress) methods. Analyses of thin films or materials with shear stress are available.
- Using micro area optics allows measurement of micro area stress mapping.

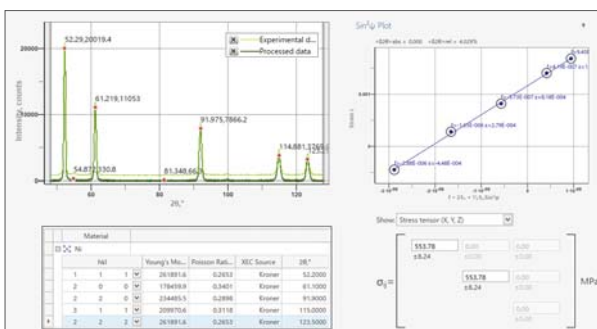
Residual Stress measurement optical configuration



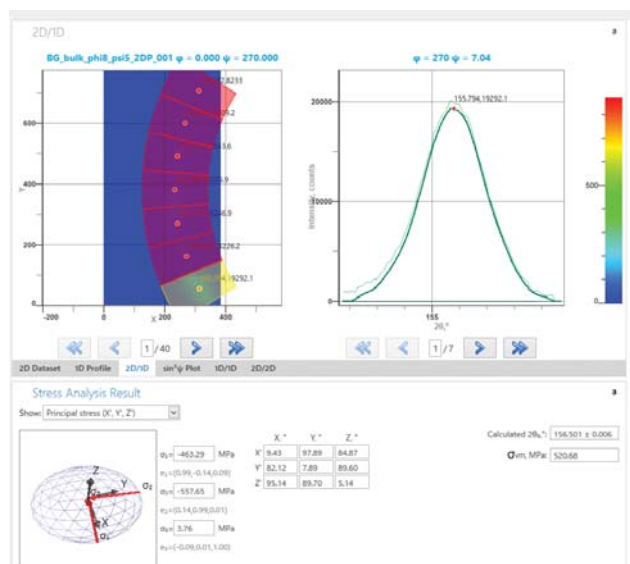
## $\sin^2\psi$ method: Ni plate stress measurements



## Multi HKL method: Ni thin film stress measurements

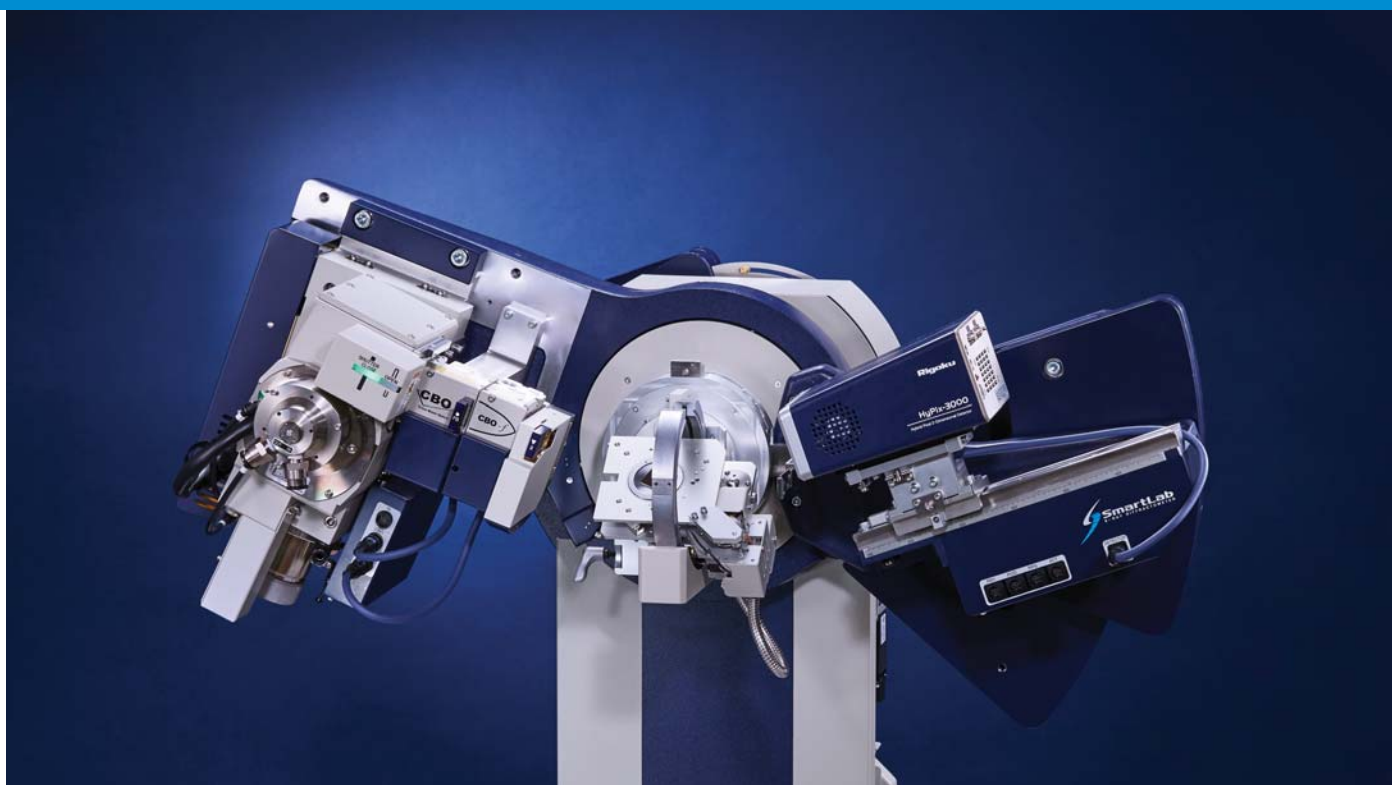


## Triaaxial stress: Shot peened steel stress measurements

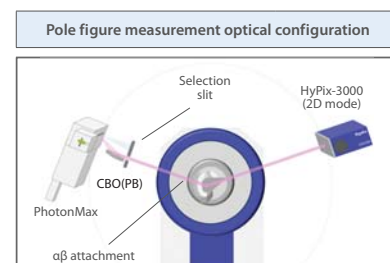




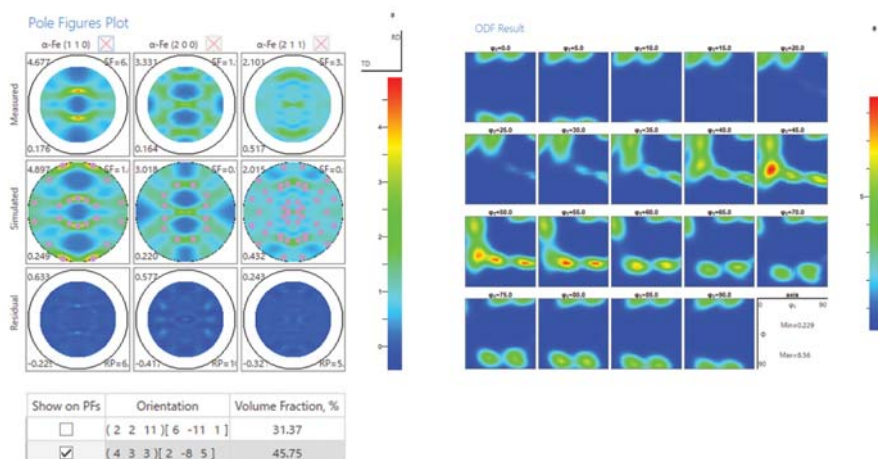
# Pole figure and ODF analysis



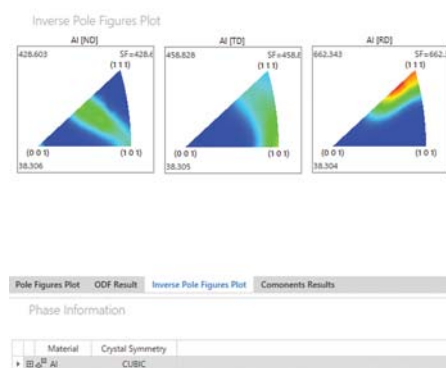
- In-plane or transmission measurement allows the acquisition of a whole pole figure.
- The  $\alpha$ - $\beta$  attachment allows  $\gamma$  rocking, alleviating the effect of large particles.
- With the use of a 2D detector, pole figures of two or more planes can be captured in a single measurement session.
- For analysis of complicated crystal texture, simulation analysis using not only pole acquisition but also ODF (Orientation Distribution Function) analysis allows calculation of the actual orientation distribution rate.
- Reverse pole figures can be used to determine the orientation of the lattice plane parallel to the sample surface.



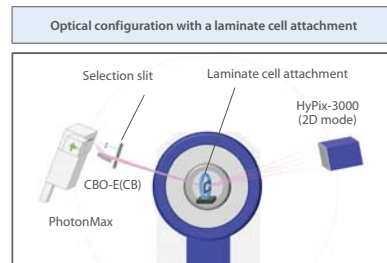
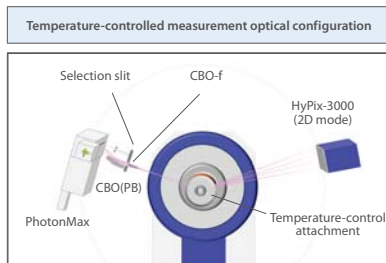
## SUS430 pole and ODF analyses



## Reverse pole figures of aluminum foils

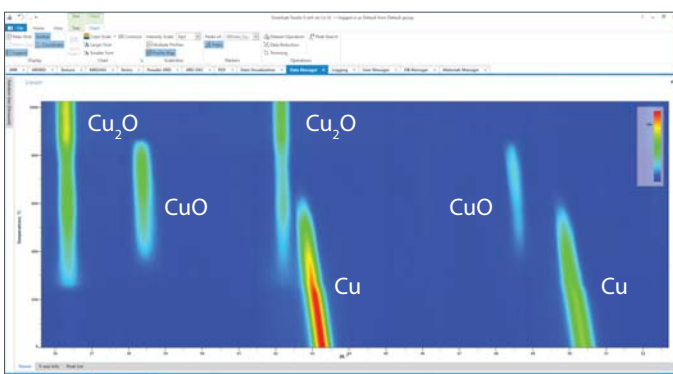


# in-situ/Operando measurements



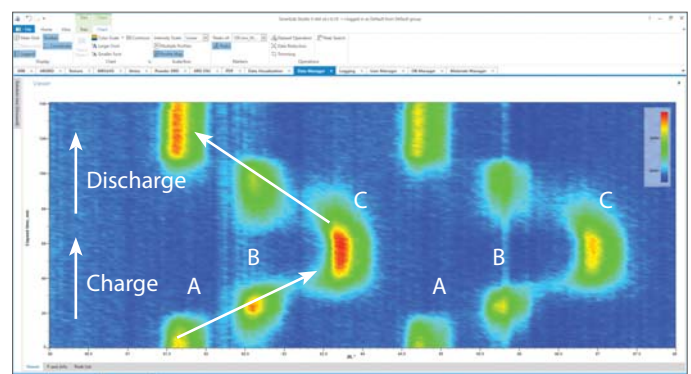
- HyPix-3000 detector measures 30° range of 2θ by single shot. It can repeat the shot every 0.1 s. minimum.
- The Reactor X achieves high-speed temperature increase with infrared heating. It can accept many different types of gas because the heater section is separated from the sample chamber.

**Cu powder *in-situ* high-speed temperature increase measurement (300°/min) 2θ-temperature-intensity**

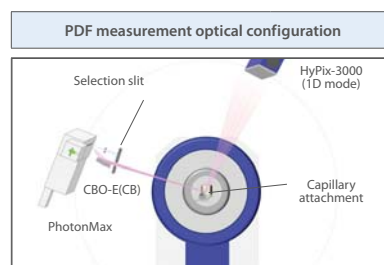
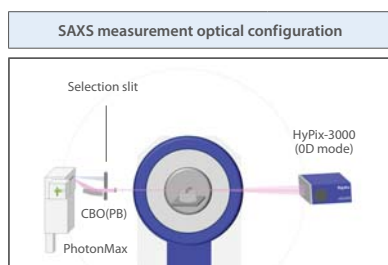
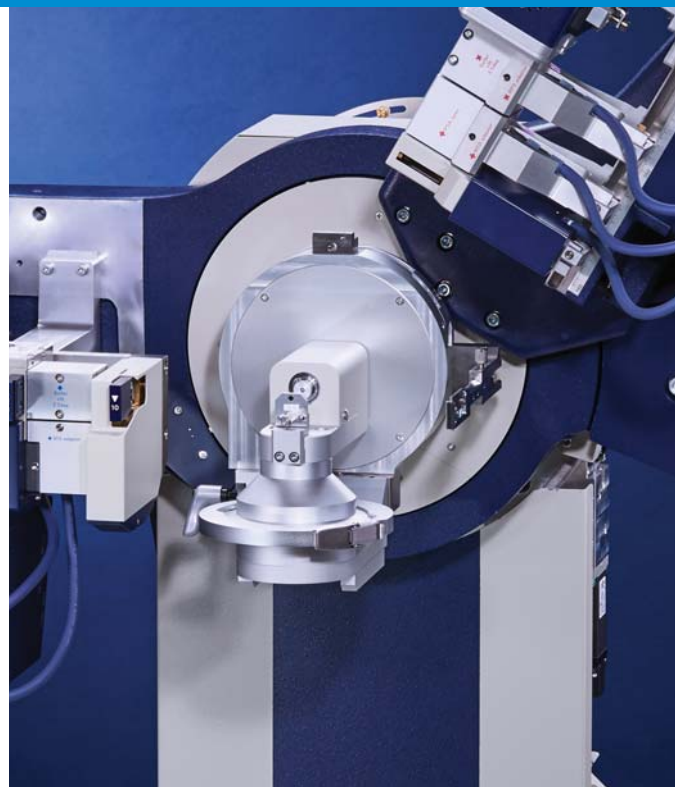


- A battery cell attachment for evaluation of charging/ discharging, indispensable for battery material assessment, and a laminated cell attachment are available.
- Bulk data obtained from *in-situ*/operando measurement can be processed as a single unit by software. In addition, effective visual 3D diagrams can easily be created by adding a time axis to the angle/intensity axis.

**Li(Ni,Mn)O<sub>4</sub> operando charge/discharge measurement 2θ-time-intensity**



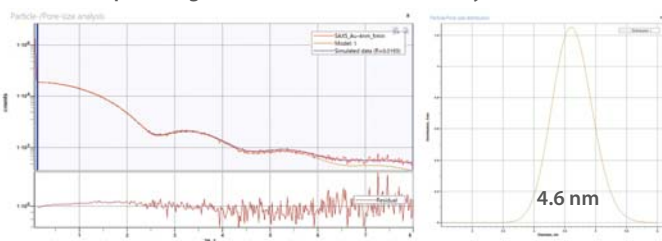
# Nanomaterials analysis



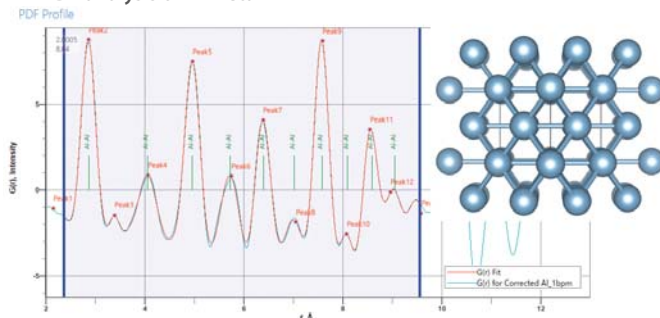
- SAXS can be used to analyze the grain size of a material (minimum 100 nm) and its distribution as well as the periodic structure. Furthermore, USAXS (Ultra SAXS) is also available, achieving measurement of minimum grain size of 1000 nm.

- PDF is an approach to derive real space information by inverse Fourier transformation of reciprocal space data. It can analyze interatomic distances, coordination number and periodicity irrespective of crystallinity of samples.

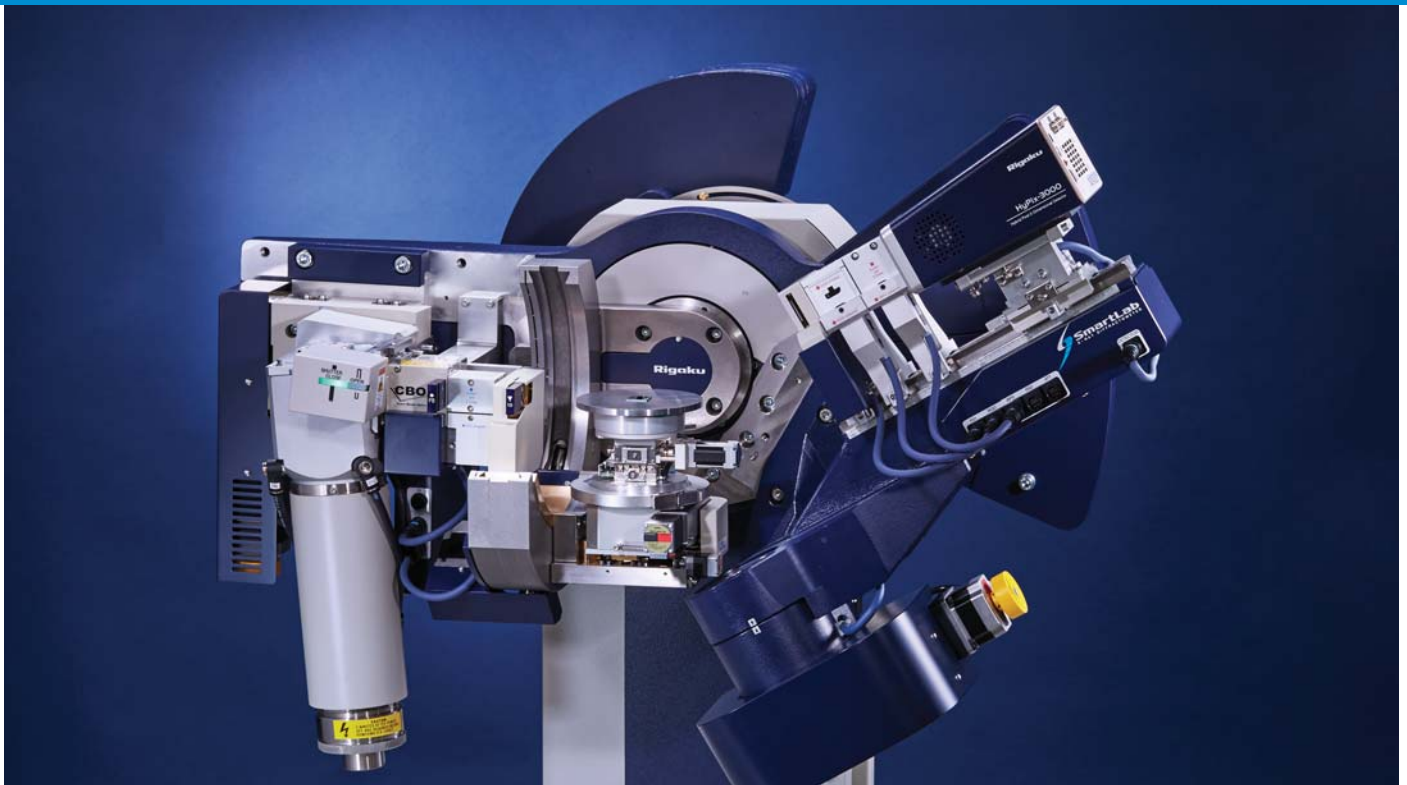
**Au nanoparticle grain size and distribution analysis**



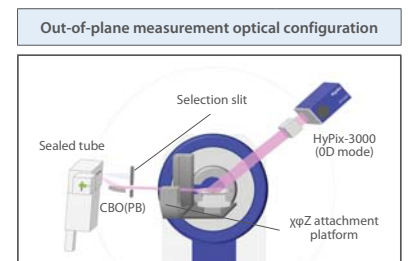
**PDF analysis of Al metal**



# Advanced thin film analysis



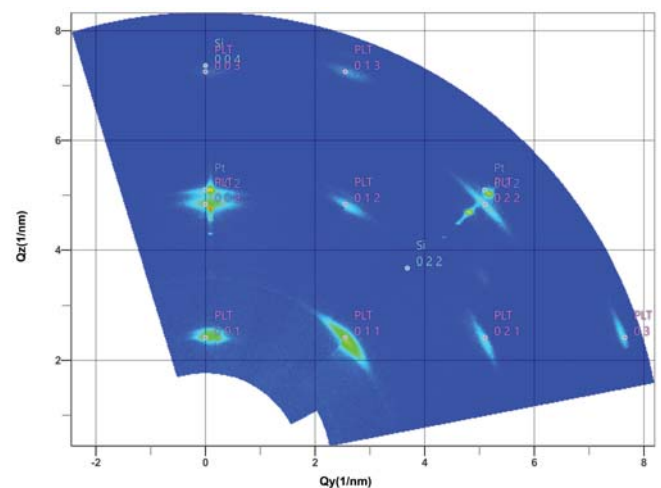
- A goniometer equipped with  $\chi$  and  $\phi$  axes as standard specifically designed for thin films is used to support the various measurements necessary for thin film analysis.
- Capable of obtaining thin film surface information by limiting the incidence angle to a micro angle.
- The  $\text{CuK}\alpha_1$  high-resolution parallel beam optics using Ge crystal monochromator allows evaluation of single-crystal thin films including epitaxial films.
- High-resolution rocking curve (HRRC) measurement allows analysis of thin film structure information.



## InGaN/GaN MQW thin film thickness analysis using HRRC

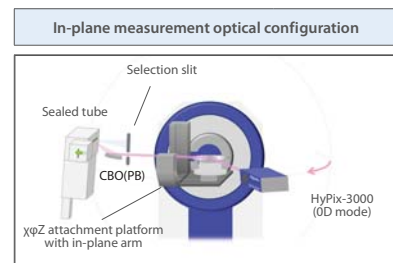


## High-speed wide reciprocal lattice map measurement of ferroelectric thin films using 2D detector

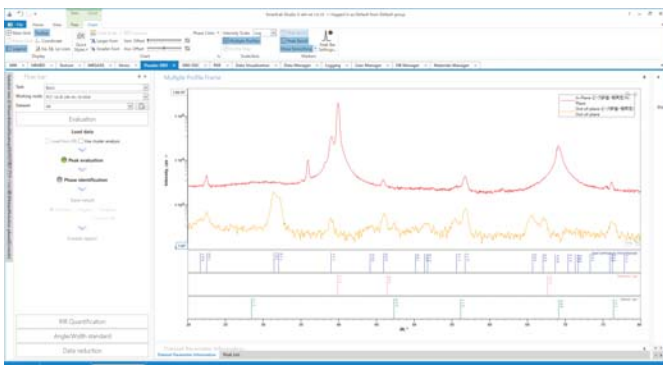




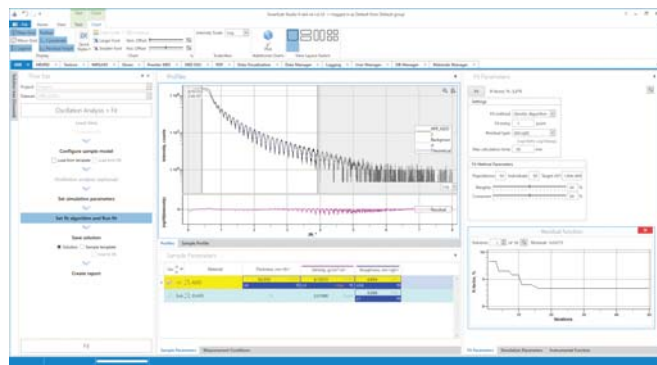
- Rigaku's original in-plane axes allow in-plane diffractometry with the X-ray incidence angle accurately controlled.
- Reciprocal lattice map measurement allows user to learn the orientation relationship between the substrate and the film as well as the crystalline state. The use of reciprocal lattice simulation allows user to easily determine the film condition.
- The 2D detector HyPix-3000 can be effectively used to obtain a wide reciprocal lattice map in a short time. The map is drawn in real time during measurement, resulting in a shorter analysis time.



#### Out-of-plane/in-plane measurement



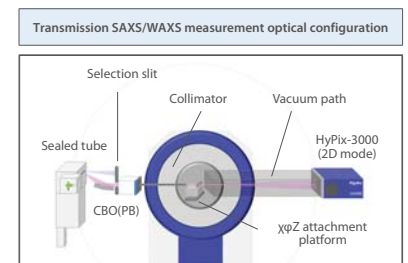
#### IGZO film thickness and density analysis using XRR



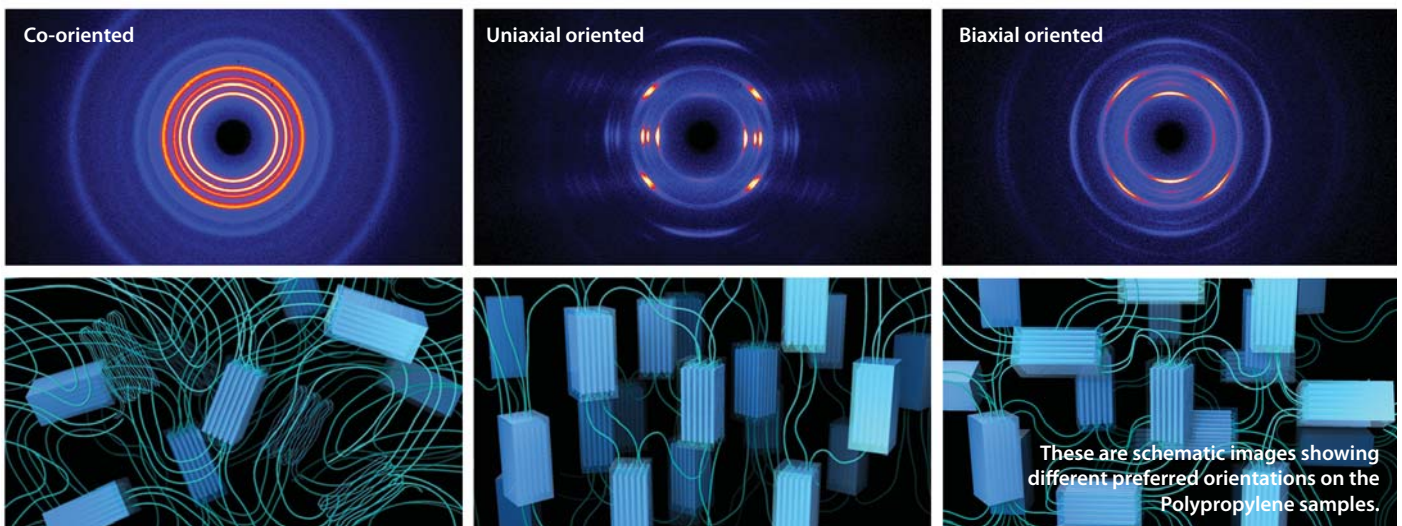
# 2D SAXS, WAXS and GI-SAXS



- Transmission 2D patterns of film materials can be measured. Circular measurement of Debye rings is available. This makes it easy to evaluate the oriented state and the degree of orientation.
- Transmission WAXS (Wide Angle X-ray Scattering) and SAXS (Small Angle X-ray Scattering) can be switched to obtain information including from long period structure to lattice level.
- With holders for different sample shapes, the equipment can accept powder, films and even textile samples, and provide mapping measurement within a sample. Measurement from the direction of the film cross section is also available.

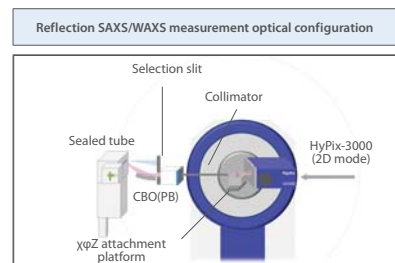


## Preferred orientation of Polypropylene observed y transmission 2D WAXS measurements

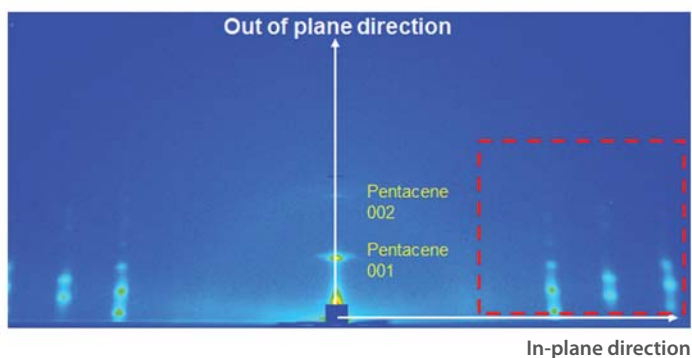




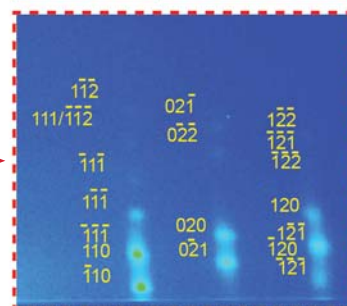
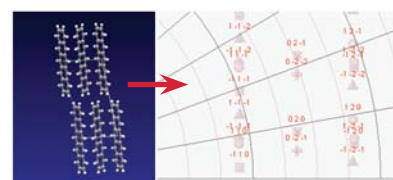
- The GI-SAXS/WAXS unit can be used for 2D measurement of thin film materials.
- A newly developed aperture slit (patent pending) allows even clearer profile capturing in the in-plane direction.
- Information in both the lamination and the in-plane directions can be captured in a single measurement session.
- The oriented state and crystallinity of organic thin layers can be evaluated in a short time.



Phase identification of Pentacene thin film with GI-WAXS



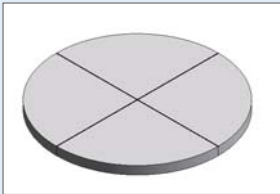
Thin-film phase



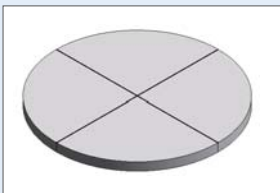
Thin-film phase is detected.

# Optional attachments and sample holders

## Sample plate



4" wafer sample plate



4" wafer sample plate (9 mm)



6" wafer sample plate



8" wafer sample plate



Height reference sample plate

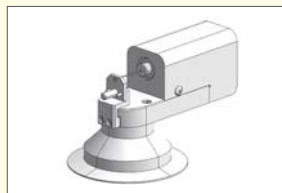


Transmission SAXS sample plate

## Attachment head



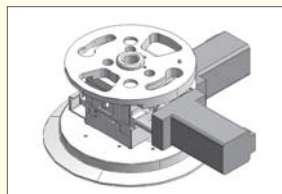
Battery cell ATT. head



Capillary spin ATT. head



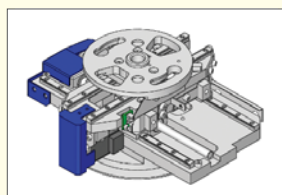
Standard ATT. head



RxRy-ATT. head



XY 20 mm-ATT. head



XY 4-inch ATT. head

## Attachment base



Standard ATT. base



ASC-6 ATT.



$\chi\phi$  ATT.



$\alpha\beta$  ATT.

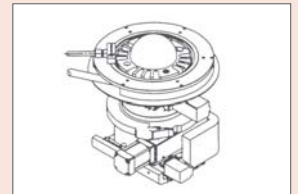


$\beta$  ATT. base

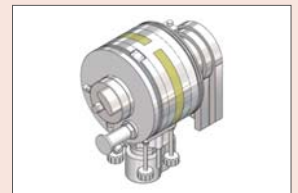


$\phi$  ATT. base

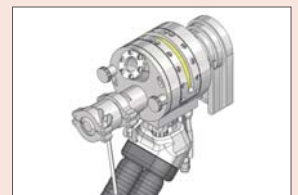
## Non-ambient chambers



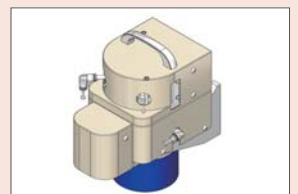
DHS 1100\*



HTK 1200N\*



TTK 600\*



DSC attachment



Reactor X (infrared heating high temperature attachment)



Multipurpose high temperature attachment

\*Products of Anton Paar. Many other Anton Paar attachments are available for SmartLab.

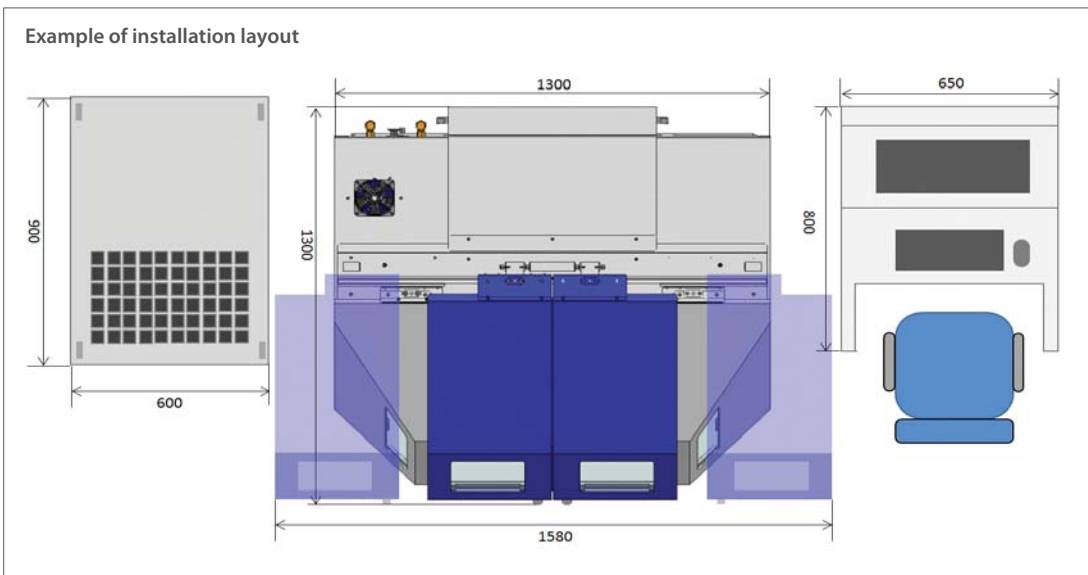
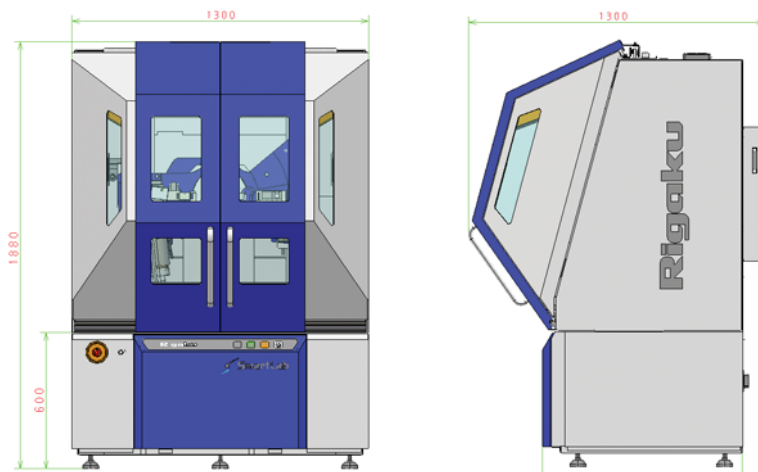


# Specifications

X-ray generation*		
X-ray generator	3 kW for sealed X-ray tube	9 kW for <i>PhotonMax</i> rotating anode
Tube voltage variable range	20 – 60 kV	20 – 45 kV
Tube current variable range	2 – 50 mA (option 60mA)	10 – 200 mA
Installation requirements		
Enclosure dimensions	1,300 x 1,300 x 1,880 mm, 51.2 x 51.2 x 74.0 inch (W x D x H)	
Weight (without any options)	~750 kg, ~1,653 lb for standard configuration	~850 kg, ~1,874 lb for standard configuration
Power supply	Three phases AC200 V, 50/60 Hz, 30 A or single phase AC220 – 230 V, 50/60 Hz, 40 A	Three phases AC200 V, 50/60 Hz, 60 A
Ground resistance	≤ 100 Ω	

\*The maximum rated values depend on the type of X-ray tube (target, focus). Please refer to the instruction manual of the X-ray tube for details.

## Dimensions (unit: mm)



# Technologies from Rigaku

## X-ray generators

In 1952, Rigaku was the first company in the world to commercialize a rotating anode X-ray generator. Today, Rigaku's product line ranges in output from 50 to 9000 W, and at our Yamanashi plant and US facilities we develop and produce everything from high-brightness, high-output types, to sealed tube microfocus X-ray generators. In the area of high-voltage generation power supplies, we develop and produce molded types more compact and stable than previous systems.



**MultiMax-9**  
Multipurpose rotating anode X-ray generator



**MicroMax-007HF**  
Microfocus rotating anode X-ray generator



**MicroMax-003**  
Multipurpose sealed tube X-ray generator



**FR-X**  
Ultrahigh-intensity microfocus rotating anode X-ray generator

## X-ray optics

At Rigaku Innovative Technologies, which became part of our group in 2000, we are developing and manufacturing X-ray spectroscopy and focusing components. A key part of high-precision, high-sensitivity X-ray analysis is focusing X-rays and making them monochromatic or parallel using optic elements fabricated with sophisticated technology for artificial multilayer stacked films. At our Osaka factory, we develop and manufacture analyzing crystals for wavelength dispersive X-ray fluorescence spectrometers. The CBO (Cross Beam Optics) series, which facilitates optical system switching, also broadens the possibilities of X-ray analysis.



**Confocal Mirror**  
Multilayer optics



**RX series**  
Multilayer optics



**CBO series**  
Cross Beam Optic units



**K $\alpha$  optics**



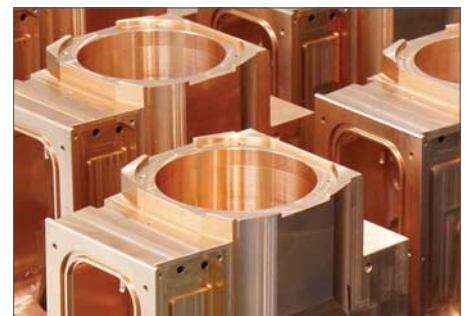
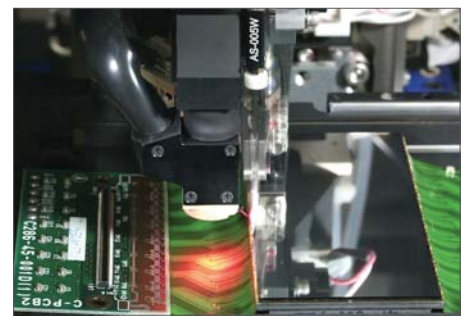
Yamanashi plant



Rigaku Innovative Technologies, Inc.

# X-ray detectors

Among Rigaku's X-ray detectors, the mainstay systems are direct-detection semiconductor detectors such as the 1-dimensional D/tex Ultra250/250HE series and 2-dimensional HyPix series, which combine high speed, low noise, high resolution and other features. Our Yamanashi factory is equipped with a cleanroom, a semiconductor process line and bonding equipment, and we produce detectors in-house. At Rigaku Innovative Technologies Europe s.r.o., established in 2008, we are also developing devices such as ultra-high-resolution CCDs. We are continuing our detector innovation in pursuit of greater convenience, such as simple switching between 0, 1 and 2 dimensions.



**HyPix-6000C/6000HE**  
Hybrid pixel array detector



**HyPix-3000**  
Hybrid pixel array detector



**HyPix-400**  
Hybrid pixel array detector



**D/tex Ultra250/250HE**  
High-resolution and high-speed  
1D silicon strip detector



**XTOP**  
High-sensitivity X-ray  
camera



**HR-XTOP**  
High-sensitivity,  
high-resolution X-ray camera



**XRM**  
High-resolution camera



**Management System**  
ISO 9001:2015  
ISO 14001:2004  
[www.tuv.com](http://www.tuv.com)  
ID 9105040952

Rigaku Yamanashi Plant has obtained the international quality system certificate according to ISO 9001 and ISO 14001 and is addressing continual improvement with the PDCA cycle to provide reliable products to customers.

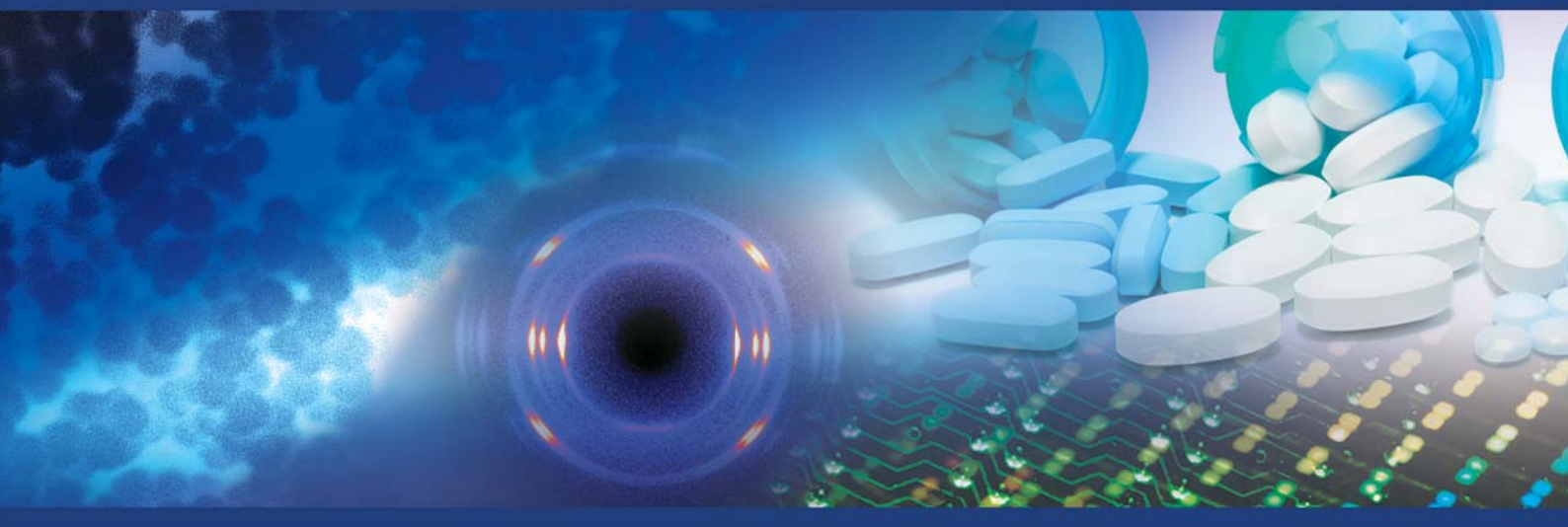


# SmartLab

Automated multipurpose X-ray diffractometer



[www.rigaku.com/en/products/xrd/smartlab](http://www.rigaku.com/en/products/xrd/smartlab)



**Rigaku Corporation and its Global Subsidiaries**  
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