SmartLab

Advanced Applications

X-ray diffraction (XRD) analysis with advanced Guidance expert system software

Rigaku

Leading With Innovation

Multipurpose XRD system with intelligent guidance

State-of-the art XRD

Modern X-ray diffractometers are expected to support multiple applications; e.g., powder diffraction, small angle X-ray scattering, residual stress and mapping, to name a few. However, with the increase in complexity and sophistication that accompanies a multipurpose instrument comes the risk of a decrease in usability. How do you know for certain that you or your fellow researcher is selecting the best optics for each application? When switching between complex configurations, how can you be absolutely certain that your instrument remains aligned and that the data that you measure is of the utmost quality?

SmartLab series diffractometers answer these questions in three ways. First, the instrument recognizes the specific optical components that are currently mounted on the instrument and checks the configuration against the type of measurement that you have selected. If the current configuration is not the best one for your intended measurement, the software suggests how you should change the hardware configuration for the type of application selected.

Second, after the proper hardware components have been added to the instrument, the instrument can perform an automatic alignment if needed—a unique feature of Rigaku and the only true way to know that your instrument is ready to collect the high-quality data that your research demands.

And third, new SmartLab Studio II software provides an integrated modular X-ray diffraction suite for the innovative SmartLab systems. Covering the full spectrum of operations required for X-ray diffraction analysis, including measurement, analysis, data visualization and reporting, SmartLab Studio II software was engineered from the ground up with ease-of-use in mind. The novel Guidance "expert system" ensures that even novice users are able to quickly master advanced measurements.

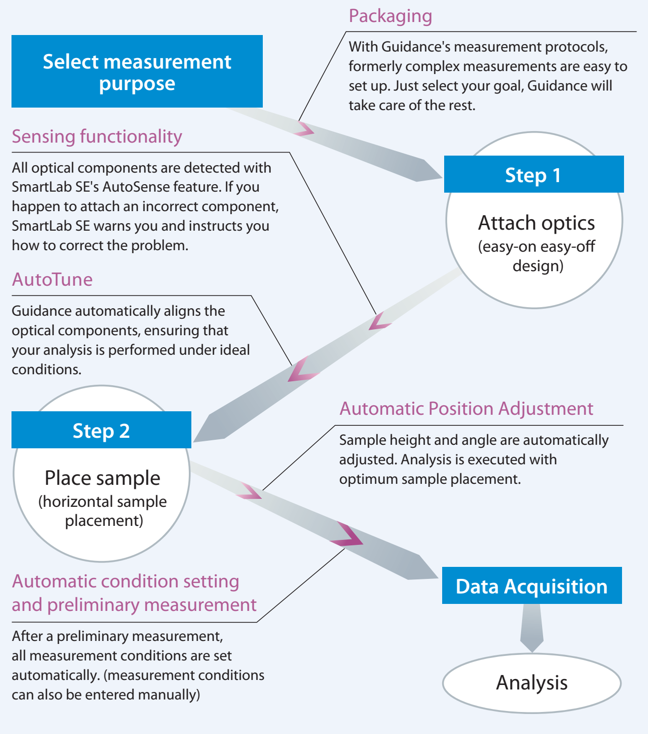
SmartLab

New SmartLab is the most novel XRD available today. The system incorporates a high-resolution θ/θ closed loop goniometer drive system, cross beam optics (CBO), an in-plane diffraction arm, and an optional 9 kW high-brightness X-ray source. With the addition of Rigaku's proprietary in-plane attachment, it is possible to evaluate structures perpendicular to the sample surface and perform depth profiling without substrate information. SmartLab Studio II's pole figure measurement function enables you to obtain perfect pole figures.

SmartLab SE

A highly versatile multipurpose X-ray powder diffractometer with built-in intelligent guidance, SmartLab SE offers continued refinement of the ease-of-use features that enabled the original SmartLab to receive the R&D 100 Award in 2006: automatic alignment, component recognition, cross beam optics and an advanced photon counting hybrid pixel array detector (HPAD).

Measurement flow



Guidance expert system

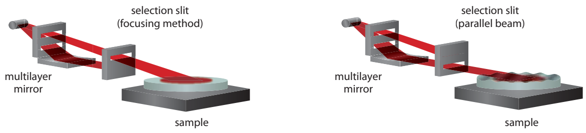
Guidance is an expert system within SmartLab Studio II that suggests the optimal hardware configuration and settings for specific application measurements. The software will determine which optics are most appropriate for a given application, determine the instrument settings and execute the measurement, offering a completely automated measurement sequence. Since SmartLab has built-in component recognition, Guidance will not only tell you how you should configure SmartLab for a given measurement, it will also warn you if you have not configured it properly. Expert advice coupled with hardware that will confirm the correct configuration is the foundation of the SmartLab system.

Cross Beam Optics: CBO (patented)

CBO is an optical selection unit that enables switching between two geometries by simply changing slits. For SmartLab, four types of CBO units are available:

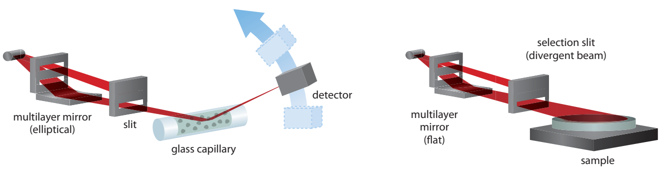
■ Bragg-Brentano focusing and Parallel beam (CBO)

The Bragg-Brentano method (left) is used for general powder XRD measurements. The Parallel Beam method (right) deploys a beam that has been parallelized by a multilayer parabolic mirror and is used for SAXS, thin films, rough surface sample measurements, etc.



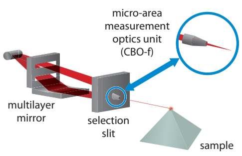
■ Convergent beam (CBO-E) and Divergent beam (CBO-α)

For CBO-E (left), an elliptic multilayer mirror converges the beam on the detection surface, providing high-angle resolution data in transmission geometry. For CBO-α (right), the beam is monochromatized to Kα-radiation with a plane multilayer mirror, which delivers higher P/B ratio at an equivalent intensity level compared to the classical focusing methods.



■ Micro area method (CBO-f)*

For CBO-f, the beam is monochromatized to Kα-radiation with a multilayer mirror, which delivers higher P/B ratio at an equivalent intensity level compared to classical methods.

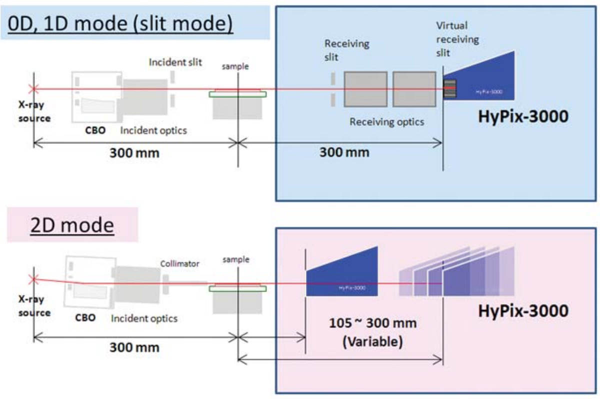


* not available on the SmartLab SE

HyPix series detectors

The Rigaku HyPix-3000 and HyPix-400 are next-generation two-dimensional (2D) hybrid pixel array detectors (HPAD). With a large active area of 3000 mm^2 (or 400 mm^2) and a small pixel size of 100 µm^2, HyPix-3000 delivers rapid data collection with high spatial resolution. These single photon counting X-ray detectors deliver count rates >10^6 cps/pixel, a fast readout speed and essentially no noise.

HyPix series serve as multi-dimensional detectors. Ranging from 0D measurement using a point detector and 1D measurement using a linear detector to 2D measurement using an area detector, they can deliver the full range of measurements without changing detectors.



Featuring a double-threshold (window) discriminator, HyPix-3000 has three readout modes that can be selected based on the desired measurement. "Differential" mode can be used to suppress fluorescence from elements in a sample or background derived from cosmic rays. "31-bit" mode is used for experiments in which a very wide dynamic range is needed. "Zero dead time" mode makes it possible to perform extremely fast data collection. HyPix-3000 was designed for optimal flexibility and minimal maintenance.

SmartLab
X-RAY DIFFRACTOMETER

High speed *in-situ* measurement

HyPix-3000 for much faster measurements

The world's fastest multidimensional X-ray detector allows monitoring of fast chemical reactions. Using the "zero dead time" mode makes it possible to perform extremely fast data collection. Employing true shutterless operation, *in situ* and time-resolved measurement can be easily performed. In the example below, Figure 1 shows coarse phase changes—with respect to temperature rising over time—as observed by a conventional XRD. Figure 2 illustrates the benefit of rapid sequential measurement achieved with SmartLab technology. By setting the conditions as shown in Figure 3, 2D image data was saved every 0.1 sec during the 10-second exposure (100 total images).

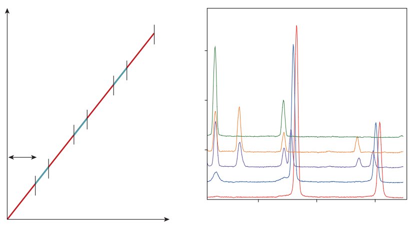


Figure 1. Conventional XRD displaying coarse changes over time

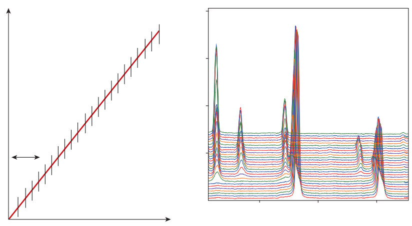


Figure 2. SmartLab can follow fast phase transitions with ease with the HyPix-3000 detector

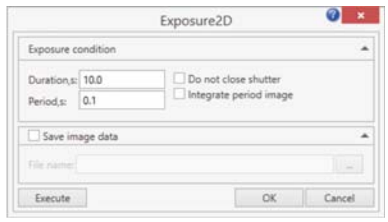
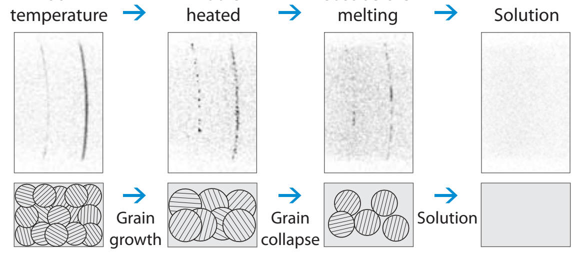


Figure 3. Setting for high-speed data collection

In-situ aluminum metal melting example

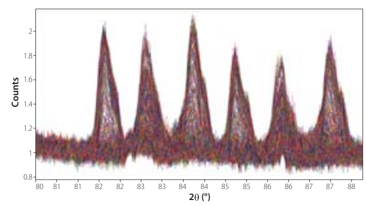
In this example, a sample of solid aluminum alloy was measured as a function of temperature until it was completely melted. The graphics at right show the results of a series of high-speed (0.5 sec/profile). exposure measurements. The alloy was subject to a steep temperature ramp of 300°/min. The data show grain growth and grain collapse during the melting process.



Operando battery charge/discharge example

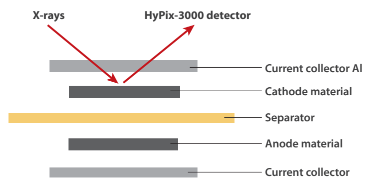
Using a battery cell attachment, *operando* charging and discharging of the positive electrode material $LiMn_{1.5}Ni_{0.5}O_4$ was examined. Evaluation of the stability of electrode materials is very important in the development of fast cycling Li-ion batteries.

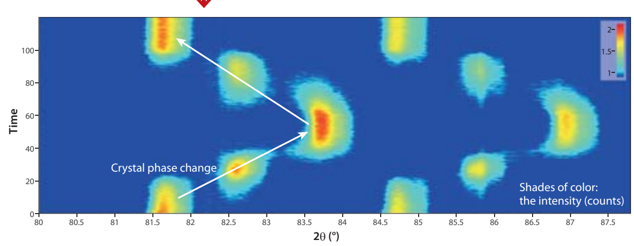
Multiple high-speed 1D profiles, acquired over time, are shown (top). The lower figure was generated with the Data Visualization plugin and clearly shows phase stability within the charge/discharge cycle.

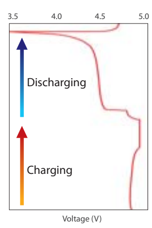


Multiple writing of the 1D XRD profile

Battery cell attachment







Horizontal axis is 2θ and the vertical axis is the elapsed time

Digital I/O to and from external equipment

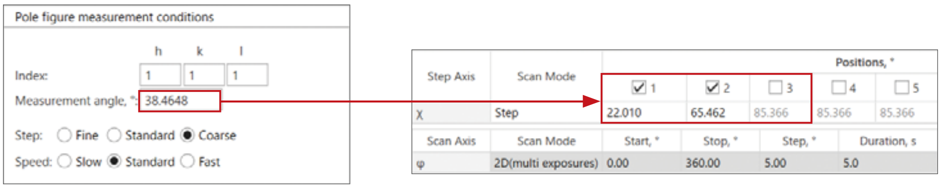
Input/output of digital signals from/to external controllers have been enabled. Digital signals can be used as triggers to start measurement and/or as output signals after the measurement is finished.

Pole figure measurement and orientation evaluation

2D mode HyPix-3000 for rapid data collection

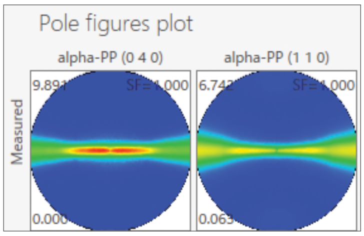
Standard universal Z stage with αβ attachment

Crystallographic texture (preferred orientation) can be introduced into materials during the fabrication process. Since texture can affect a material's properties by introducing structural anisotropy, it is desirable to measure a material's texture. Pole figure determination is the typical measurement used to determine preferred orientation and is made by recording the intensity of a given Bragg reflection as a function of rotation and tilt of a sample. HyPix-3000 delivers high-speed diffraction images that contain texture information in the form of systematic intensity variations along Debye rings associated with a given Bragg refection *hkl*, as well as correlations of these variations between different reflections that appear in the same image. With SmartLab Studio II, just enter the diffraction angle to be measured and appropriate measurement conditions are set automatically (patent pending).



Orientation coefficients of polymeric films

Based on the obtained complete pole figures, orientation coefficients can be calculated with the SmartLab Studio II Texture plugin. This is an excellent method to assess films with different physical properties or performance characteristics obtained by different manufacturing conditions, such as degree of stretching, uniaxial-/biaxial-stretching and stretching order.



Pole figures plot

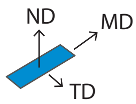
Orientation function

Phase: alpha-PP

Direction	ND	RD	TD	Fnd	Frd	Ftd
{0 4 0}	0.570	0.029	0.401	0.356	-0.456	0.101
{1 1 0}	0.420	0.077	0.503	0.130	-0.384	0.254
a-axis	0.400	0.081	0.506	0.100	-0.379	0.259
b-axis	0.570	0.029	0.401	0.356	-0.456	0.101
c-axis	0.024	0.889	0.087	-0.464	0.834	-0.370

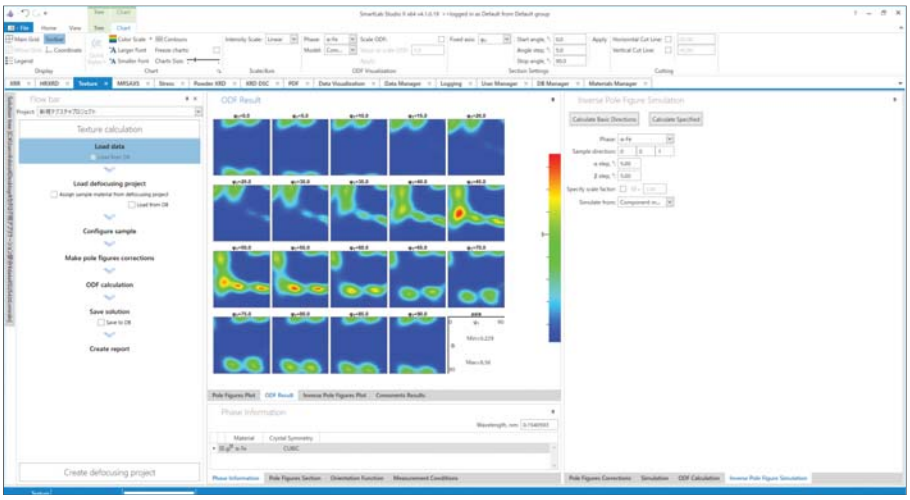
Orientation function

Orientation coefficients indicate the direction normal to sample surface of the material of interest AND in which direction the crystal axis face: ND, MD, and TD.

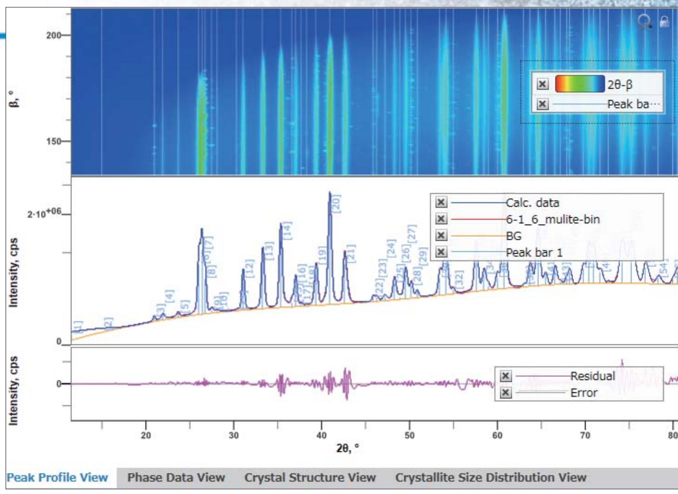


Crystal orientation distribution (ODF) analysis

By collecting data for several reflections and combining several pole figures, SmartLab Studio II's Texture plugin can arrive at the complete orientation distribution function (ODF) of the crystallites within a single polycrystalline phase that makes up a sample. ODF is a function of three independent angular variables that gives the probability of finding the corresponding unit cell (lattice) orientation. The plugin also allows recalculation of all pole figures and inverse pole figures.



Phase identification from 2D detector mode



Aligned 2D and 1D data for visual confirmation of grain information

Enhanced analysis for coarse particles and minor components

Employing a novel mathematical treatment (patent pending), the Powder XRD plugin for SmartLab Studio II has the ability to deliver enhanced analysis of two-dimensional (2D) diffraction data collected with SmartLab's HyPix series detectors.

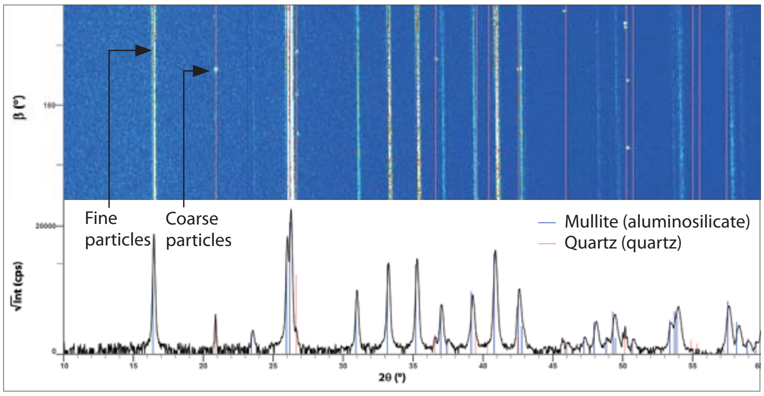
In the past, XRD software only attempted crystal phase identification after converting the 2D diffraction image to a one-dimensional profile. This can cause significant errors as it may be possible for unintended crystalline phase candidates to be listed in the search results on the basis of diffraction pattern groups that are not attributable to the same crystalline phase. While conventional "search/match" phase identification algorithms do not take into account the non-uniformity of 2D diffraction patterns, the new Powder XRD plugin fully analyzes all observed Debye-Scherrer ring and spot-shaped diffraction pattern data.

Grain sizes dramatically influence the observed 2D image, with fine grains appearing as rings and coarse grains as spots. In the classical approach, 2D data is collapsed to 1D prior to search/match and a great deal of information is therefore lost. Based on the difference in the intensity distribution along Debye rings, grouping (clustering) the Debye rings and weighting to each cluster improves search/match success. This new approach also estimates orientation coefficients of each phase for Rietvelt analysis, making this technique easier for novice users.

Identification of trace coarse particles

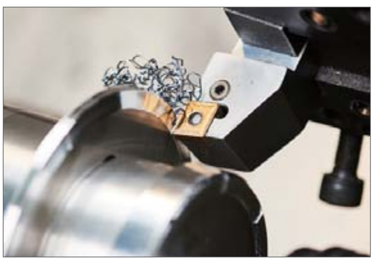
Peaks derived from coarse particles contained in trace amounts, which are difficult to identify by analyzing only one-dimensional profiles, have been identified as quartz.

Mineral powder

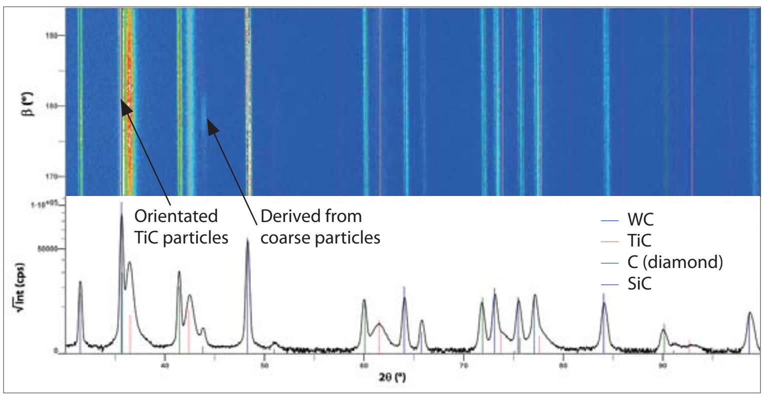


Crystal state confirmation

It is visually confirmed that TiC is oriented and C (diamond) exists as coarse particles.



Carbide tips



Real 2D: GI-WAXS measurements

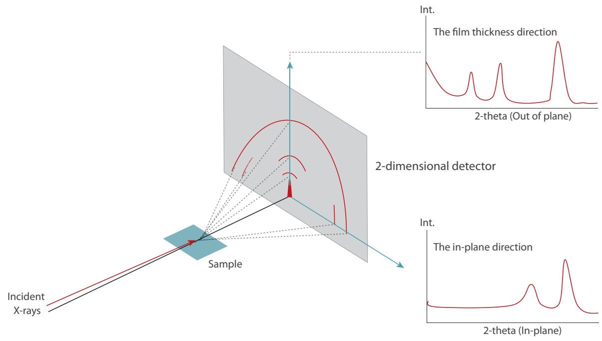
Functional thin film characterization

Development of new functional thin films, and the fabrication of functional devices using these materials, are the outgrowth of emerging demands for high-efficiency, energy-saving, lightweight devices. The smartphone is a typical example, where numerous functional thin film based devices are employed, such as display screens, backlighting, batteries, data storage devices, etc.

Characterization of functional thin films is necessary in terms of not only the phase identification of composing materials but also further crystallographic characterization of constituent crystals, such as their textures or orientation relationships with substrates, lattice distortions, film thicknesses etc., since these physical parameters are closely correlated with device performance. 2-dimensional (2D) detectors enable various kinds of XRD measurements to be performed in a remarkably short time, covering a wide range of reciprocal space and thus, enables certain measurements with laboratory equipment that have previously been performed only at synchrotron facilities.

SmartLab now support GI-WAXS measurement with a new optical system (patent pending) using an aperture slit. It is possible to capture the in-plane direction profile more clearly, independent of sample shape. The optical system using the aperture slit can be easily set up using the Guidance function.

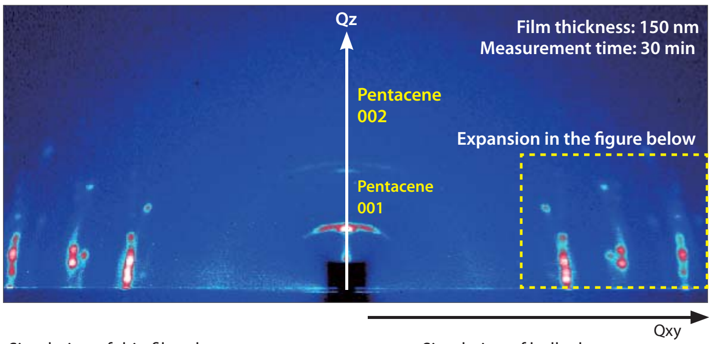
Standard χ cradle stage with 2D-SAXS/WAXS (reflection) attachment



Geometry of GI-XRD with HyPix series detector

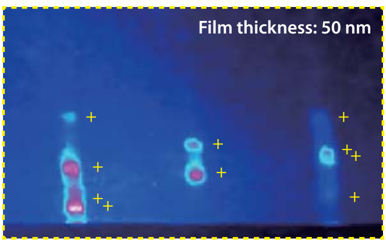
2D GI-WAXS measurements of different pentacene thin film thicknesses

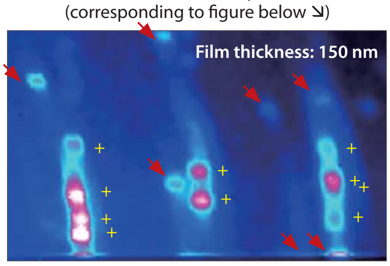
Pentacene, used as an organic electronics material, has a thin film phase observed when the film thickness is thin at the time of film formation and a bulk phase observed when the film thickness is thick. The GI-WAXS measurements of pentacene thin films with different thicknesses were used to identify crystalline phases at 50 nm and 150 nm.

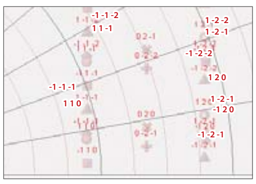


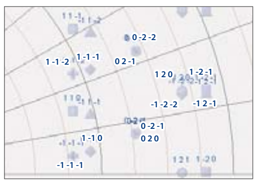
Simulation of thin film phase (corresponding to figure below +)

Simulation of bulk phase (corresponding to figure below ↘)









In the sample, with film thickness of 50 nm, only the peak attributable to the thin film phase was observed.

Samples with a film thickness of 150 nm were observed not only for the thin film phase but also for the bulk phase.

References S. Kobayashi and K. Inaba: Rigaku Journal (English version), 32 (2016), No. 2, 1-5.

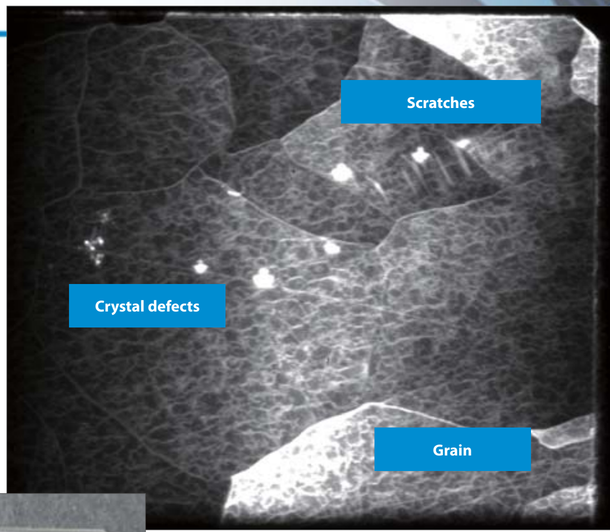
Crystal defects by X-ray reflection topography

High-speed, high-resolution imaging

SmartLab, equipped with the Xsight™ Micron FC detector, is now capable of performing fast, high-resolution X-ray topography on single crystal surfaces, providing visual images for quality assurance or quality control of crystal defects.

X-ray reflection topography is an imaging technique based on Bragg diffraction. Diffraction topographic images record the intensity profile of a beam of X-rays diffracted by a sample crystal. A topograph represents a two-dimensional spatial intensity map of reflected X-rays (i.e., the spatial fine structure of a Laue reflection). This intensity mapping reflects the distribution of scattering power inside the crystal, revealing the irregularities in a non-ideal crystal lattice.

A homogeneous sample (with a regular crystal lattice) would yield a homogeneous intensity distribution in the topograph (a "flat" image). Intensity modulations (topographic contrast) arise from irregularities in the crystal lattice, originating from various kinds of defects such as 1) voids and inclusions in the crystal, 2) phase boundaries, 3) defectives or amorphous inclusions, 4) cracks or surface scratches, 5) stacking faults, 6) dislocations 7) grain boundaries or domain walls, 8) growth striations, 9) crystal deformation and 10) strain fields.*



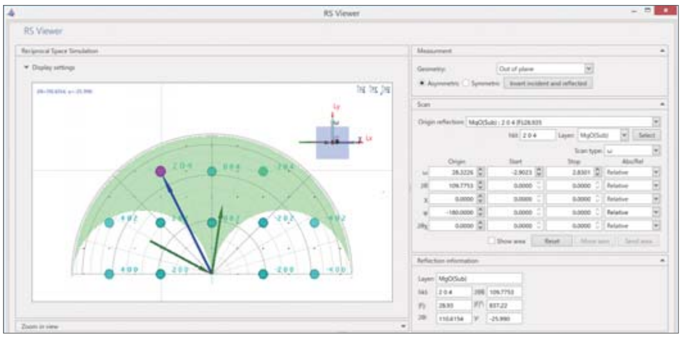
Topograph of magnesium oxide single crystal substrate (left). Grain and crystal defects are clearly visible.

Sample size: 10 × 10 mm
g = 024
Incident angle: 28.3 °
2θ: 109.8°
Exposure time 120 sec

* https://en.wikipedia.org/wiki/Diffraction_topography

Measured reflection index and arrangement with RSViewer

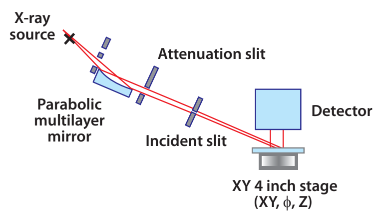
For X-ray topography, it is necessary to select the measurement plane that is appropriate for the single crystal material at the time of measurement. By using the patented RSViewer for SmartLab, which simulates reciprocal lattice points, it is possible to graphically select the optimum measurement for each substrate. Moreover, after selecting the index, SmartLab can automatically drive the goniometer to the desired measurement spot with just one click of the mouse and then begin the measurement.



Xsight Micron FC—X-ray CCD camera

Rigaku Xsight Micron FC is an ideal compact two-dimensional (2D) X-ray CCD camera optimized for use in applications as diverse as X-ray microscopy, X-ray topography, X-ray optics adjustment and metrology. The Xsight Micron FC camera is composed of a thin phosphor screen, fiber optics and a CCD camera. The CCD camera itself contains a full frame CCD image sensor with 3326 (H) x 2504 (V) pixels. Digital resolution is 16 bits with a pixel size of 5.4 x 5.4 um.

Xsight Micron FC

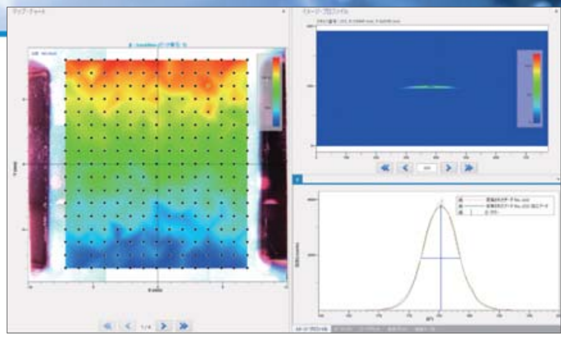


Optical schematic for topography measurement

Data visualization

Multi-data analysis

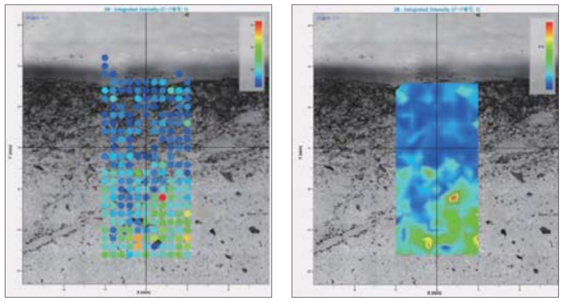
Data visualization is viewed by 21st century scientists as the modern equivalent of visual communication. It involves the creation and study of visual representations of data with the primary goal of communicating information clearly and efficiently via statistical graphics, plots and informational graphics. The intent is to make complex data more accessible, understandable and usable.



XY display of PLT thin film (left). 2D measurement data and 1D profile are shown at right.

Rigaku's Data Visualization plug-in is the data analysis and graphing software module within SmartLab Studio II. The plug-in works in conjunction with data collected with the other plug-in modules and offers an easy-to-use interface, combined with the ability to perform advanced visual customizations.

Data Visualization graphs and analysis results can also automatically update on data or parameter change, allowing the creation of visual animations as a function of another variable like time or temperature.



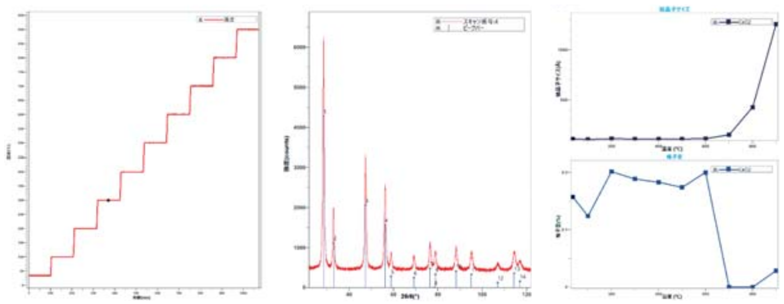
Images of serpentinite sample overlaid with mapping results. Dot display shown (left) and topo/contour display shown (right).

As an example (top right), with few steps, the measured 2D image data can be transformed into a 1D profile with mapping of the peak search results (peak parameters). By clicking any measurement point on the XY mapping, the two-dimensional image data of that point is displayed along with the converted 1D profile. An optical image of the sample may be overlaid with the XY mapping result (bottom right) so that the correlation between the crystal perfection and the sample position can be easily understood at a glance.

Phase change as a function of temperature

Physical quantities such as lattice constant, crystallite size, lattice distortion, etc. are calculated by the integrated powder X-ray analysis plugin (Powder XRD). For example, a physical quantity trend graph may be displayed along with a temperature graph and measurement profile. On the temperature graph, click on the temperature of interest and the corresponding measurement profile is displayed.

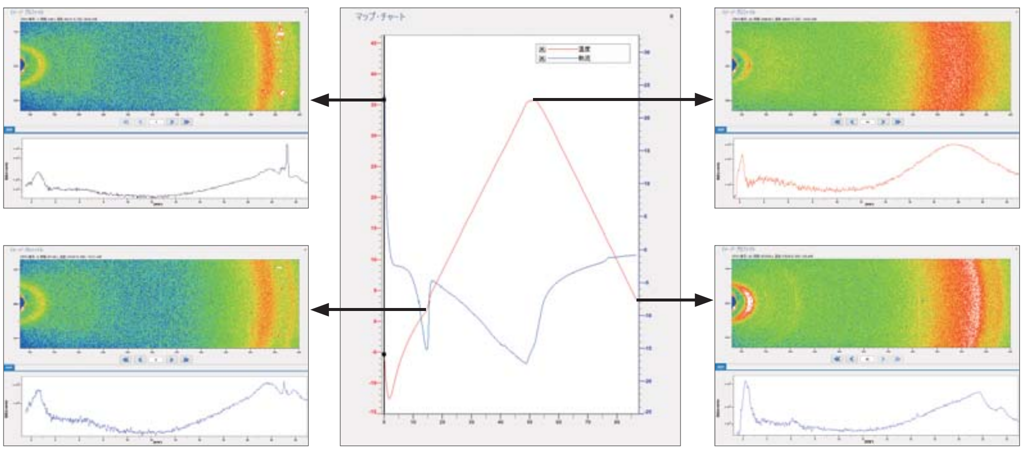
Sample: CeO_2 powder



Animation of *operando* measurements

Measurement points on the temperature graph, along with the corresponding two-dimensional image data and the converted one dimensional profile, can be displayed in a slide show. In this example, phase changes as a function of temperature are easy to understand.

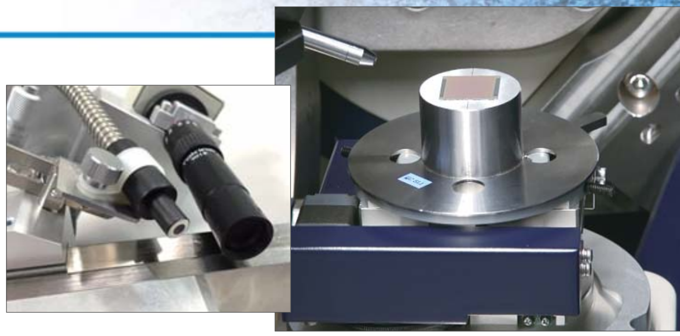
Sample: Fresh cream



High-precision micro analysis with XY mapping

XY stage and CCD camera

With the high-precision XY stage , CCD camera and dedicated light source, sample specimens may be observed with exceptional clarity. Specimen height and measurement points are easily adjusted. For multi-point measurements, the Data Visualization plugin can be employed for communicating the resulting complex information clearly and efficiently via statistical graphics, plots and informational graphics.



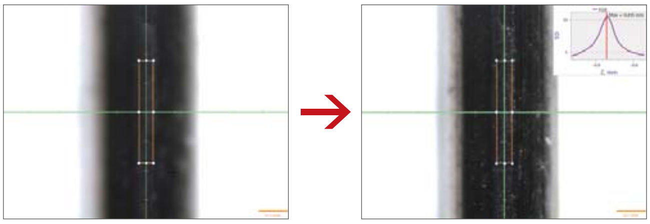
CCD camera and light source (left), XY head shown with sample (right).

Advanced functionality for ease of use

Autofocus function

In high-magnification mode, automatic height adjustment of a specimen can be performed by using camera focus. The area used for adjustment may be easily set using the camera image. This method may also be applied to specimens with irregularities. In the histogram chart (top right), the degree of focus is displayed as a numerical value.

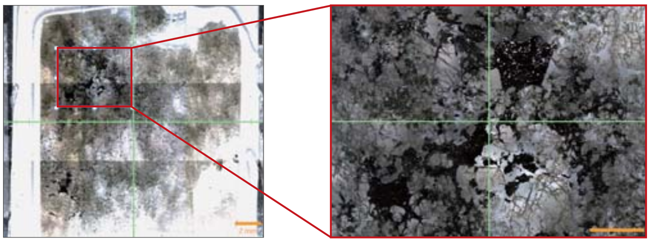
Sample: SUS wire (φ0.5 mm)



Snapshot function

With the snapshot function, multiple high-resolution images are stitched together to form a large composite image. In this way, it is possible to obtain a complete "overview image" of a sample, allowing easy inspection with high magnification. Measurement points may then be selected with high-resolution precision.

Sample: rock

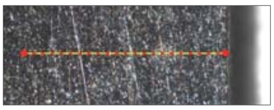


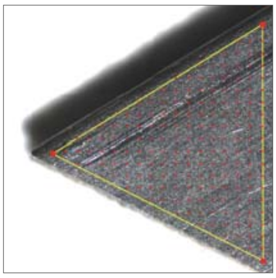
Low-magnification mode

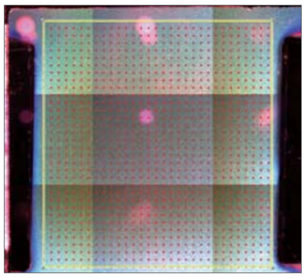
High-magnification mode

Multipoint mapping measurement by batch input

Multi-point mapping—in the shape of a line, triangle, or rectangular area—on an acquired snapshot may be easily set for a "mesh" measurement. The shape, size and shift of all measurement points are simple to input using mouse operations.



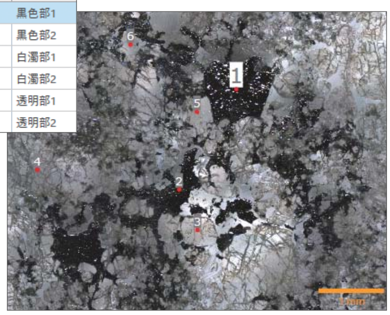




Point and click operation

When clicking on a desired measurement point within an acquired snapshot, the coordinates of that point are entered into a database for automatic multi-point measurement. In addition to moving a sample in the XYZ direction, it is possible to rotate the sample about the sample plane (φ axis) so as to better characterize odd shapes of foreign matter or other anomalies. In addition, information about the measurement points, such as those entered in the comments section, is reflected in the file name of the data … a useful feature.

No.	X(mm)	Y(mm)	Z(mm)	φ	コメント
1	-2.4074	4.4986	-1.81000	0.000	黒色部1
2	-3.2799	2.9691	-1.81000	30.000	黒色部2
3	-3.0000	2.3531	-1.81000	0.000	白濁部1
4	-5.4467	3.2731	-1.81000	0.000	白濁部2
5	-3.0080	4.1531	-1.81000	0.000	透明部1
6	-4.0235	5.1772	-1.81000	90.000	透明部2



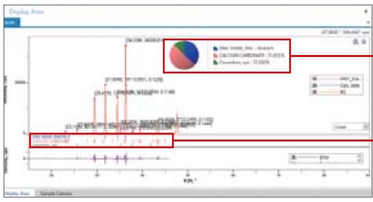
Real-time analysis

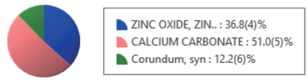
Save time and money

Display the search/match results, reciprocal space maps, and pole figures in real time at any point in a measurement. Before the measurement is completed, use "quick report of analysis result" to check whether the measurement is proceeding as expected. If a measurement is not correct, it may be easily canceled, saving time and money. Likewise, if the real-time result provides the necessary answers, the full measurement may also be cancelled to save time. Thus, real-time results can significantly increase workflow efficiency.

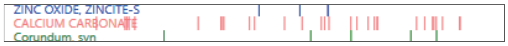
Qualitative and quantitative analysis in real time

While collecting powder diffraction data, search/match (qualitative) and quantification (Rietveld method) may be performed based on the data accumulated to the current time.*





Quick Rietvelt quantitative analysis

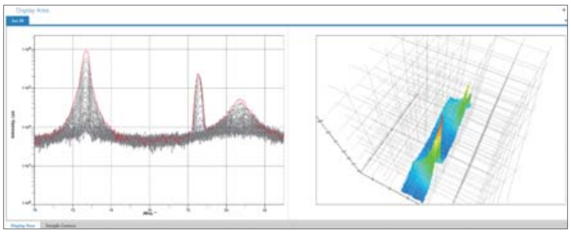


Quick search/match qualitative analysis

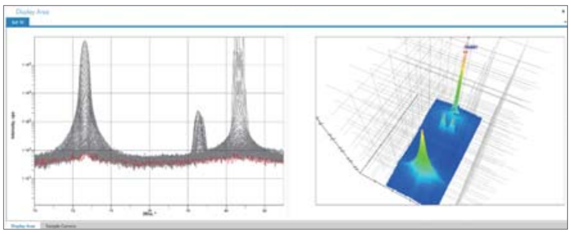
* patent pending

Display reciprocal space maps in real time

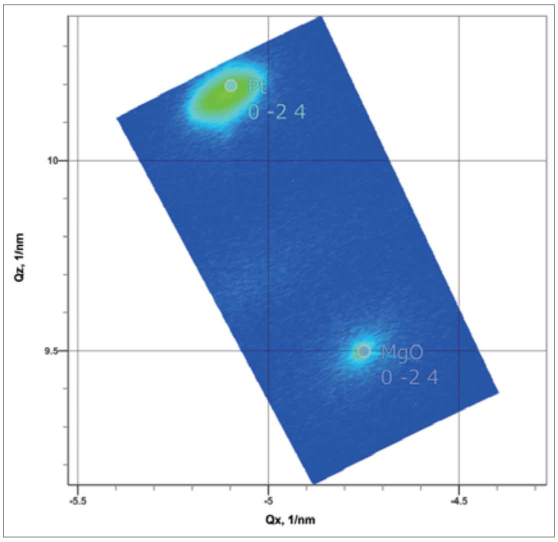
Reciprocal Space map is displayed in real time during measurements in the reciprocal space.



Early in the measurement, the left side of raw data is insufficient to judge the success of the measurement.



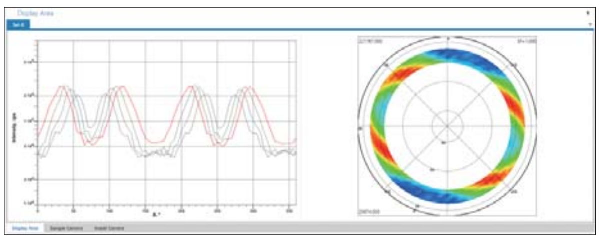
Later during the measurement, it is possible to display a reciprocal space map that provides quick results.



Moreover, mid-measurement results may be overlaid with simulated data to allow real time evaluations.

Display pole figures in real time

Using the scan profile obtained with the HyPix-3000, pole figures may be calculated and displayed during a measurement. This allows the preferred orientation to be known as soon as possible, which improves work efficiency.

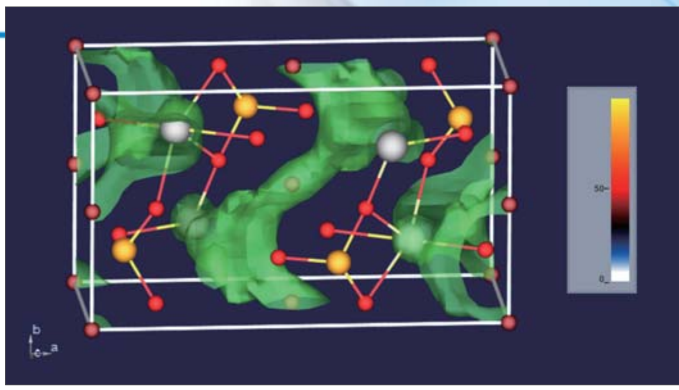


Ion conduction path obtained by BVS method

Visualize ion transfer channels

The relationship between crystal structure and ion conductivity provides insights for the development of the next generation of solid electrolytes based on advanced materials exhibiting rapid and selective lithium ion conduction. These materials will form the basis for the next generation of advanced batteries.

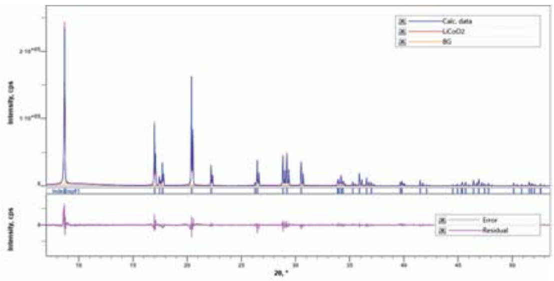
SmartLab delivers the ability to visualize ion transfer channels in electrolyte materials overlapped with a crystal structure view obtained by the Rietveld method. In this example (image at right), the ion transfer channels (green) are calculated using the bond valence sum (BVS) method. The ion channels are clearly defined relative to the crystal structure.



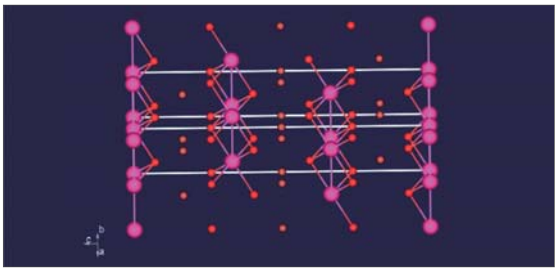
All images courtesy of Professor Nobuya Machida, Science & Engineering department, Konan University

Rietveld for crystal structure, BVS to display ion transfer channels

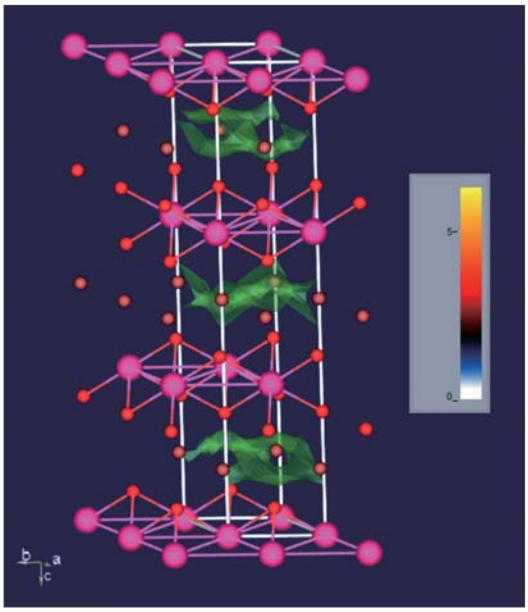
Crystal structure of the sample was determined by employing the Rietveld method using a crystal structure from a similar chemical compound. Li-ion channels were calculated using the BVS method.



Step 1. Profile fitting by Rietveld method.



Step 2. Refine the crystal structure by using similar chemical compound.



Step 3. Visualize ion transfer channels by BVS method.

What is the BVS method?

The bond valence sum (BVS) method is a popular technique in coordination chemistry to estimate the oxidation numbers of atoms in a crystal structure. It is calculated from interatomic distances and empirical bond valence parameters. The ion transfer pathways may be investigated by using BVS maps, which are the spatial distributions of BVS within the unit cell.

$$\mathrm{BVS} = \sum_{j=1}^{n} exp\left(\frac{r_0 - r_j}{0.37}\right)$$

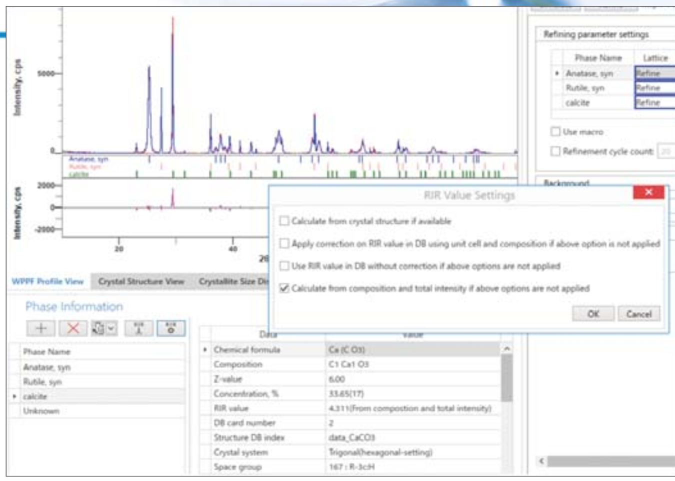
r_j is the observed bond length, r_0 is a tabulated parameter expressing the (ideal) bond length when the element j has exactly the valence of 1.

Direct Derivation (DD) quantitative method

New quantitative analysis method

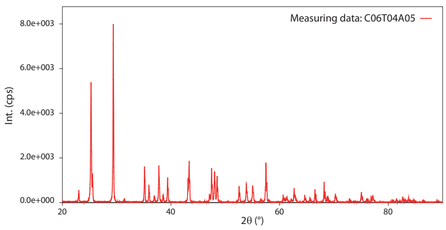
SmartLab now offers users a unique new quantitative method for XRD. No crystal structure data and no reference samples are required. Just input chemical formulas for known phases into the Direct Derivation (DD) method quantitative analysis application. In the conventional quantitative analysis method, reference sample measuring (calibration measuring, or RIR figure measuring) is required to analyze a "crystal structure unknown" sample, adding a scaled reference material into it.

SmartLab Studio II supports the new DD method as well as conventional methods, like Rietveld and RIR methods. The XRD Powder plugin allows each phase to have a different quantitative analysis method. For example, Rietveld may be used for a "known structure" phase while DD is employed for an "unknown structure but known chemical composition" phase.

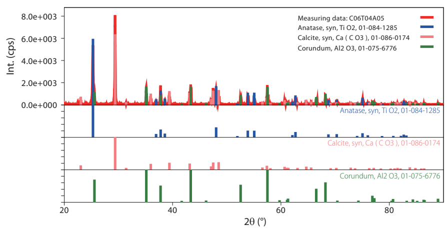


Quantitative analysis using known chemical compositions

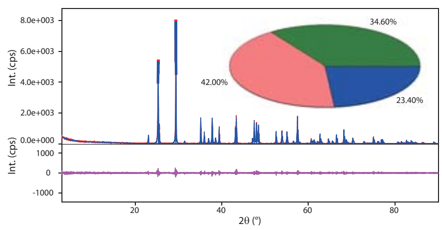
The integrated intensity of a diffraction line is proportional to the volume fraction of a relevant crystalline phase in an irradiated sample, and the volume fraction, when it is multiplied with the chemical formula weight, can be related to the weight fraction. The structure-factor-related quantity in the intensity formula, when it is summed in an adequate 2θ range, can be replaced with the sum of squared numbers of electrons belonging to composing atoms in the chemical formula. Unit-cell volumes and the number of chemical formula units are all cancelled out in the formula. Therefore, the formula requires only single-measurement integrated intensity datasets for the individual phases and their chemical compositions. No standard reference material, reference intensity ratio or crystal structure parameters are required.



Step 1. Data collection



Step 2. Qualitative analysis



Step 3. Whole Powder Pattern Fit (WWPF) – Pawley method

Superior to Rietveld method when compositions are known

The DD method was developed by Dr. Hideo Toraya, who serves as Vice President and Director for the X-ray Research Laboratory of Rigaku Corporation. The root-mean-square error (RMSE) is a frequently used measure of the differences between values (sample and population values) predicted by a model and the true value. An example of the power of the DD method is provided (right table) for a typical sample mixture of three phases.

	DD法	Rietveld	RIR
RMSE(%)	**1.41**	9.32	10.74

Example: **$BaSO_4$ (barite)** + Al_2O_3 + SiO_2 = **50.27** : 38.13 : 11.60

Bibliography:

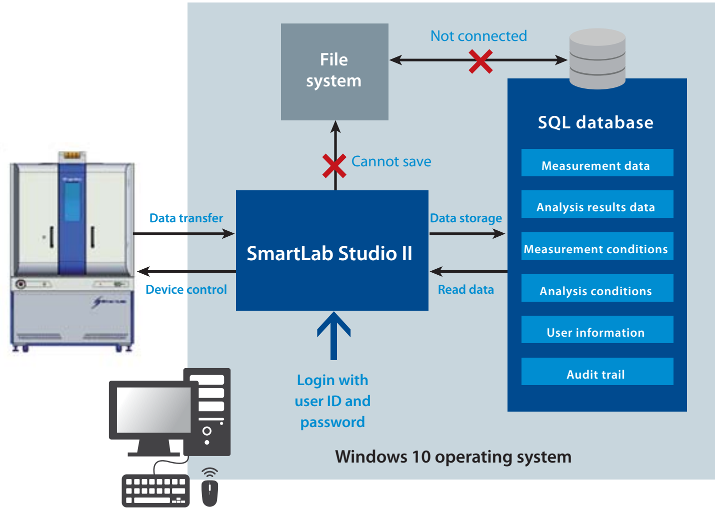
H. Toraya: J. Appl. Cryst . 49 (2016), 1508–1516.
H. Toraya: J. Appl. Cryst . 50 (2017), 820–829.

Pharmaceutical regulatory compliance

CSV and ER/ES compliance success

Computer systems validation (CVS) is the process of ensuring that any technology component (software and hardware) fulfills its purpose in accordance with regulatory guidelines. It is especially crucial in heavily regulated industries like biotech and pharma, as products from these sectors impact public health and safety.

21 CFR Part 11 established the US FDA regulations governing electronic records and electronic signatures (ER/ES). These mandates define the criteria under which electronic records and electronic signatures are considered trustworthy, reliable, and equivalent to paper records.



Robust security and validation protocols

Access to SmartLab Studio II is protected by a user account and password where stringent security policies may be set. In SmartLab Studio II, about 40 privileges may be configured relative to a user's hierarchy (group) by the administrator. In this way, routine users may be limited to only running macros with no possibility of instrument changes.

Data integrity management and audit trails

■ Data integrity management

In SmartLab Studio II's ER/ES mode, all measurement data and analysis results are managed in an SQL database. All data are assigned a unique ID and all processing of data is recorded in an audit trail along with the ID. Reanalysis of data cannot overwrite the original result, but rather the same ID is stored in a different revision number.

■ Audit trail viewer

Input and operations by users, such as the output of results and data associated with it, is recorded in the audit trail. Inspection of audit trails is only available to users with rights to the audit trail plug-in (Audit Trail Viewer). The viewer has filtering functions that are easily set up and display only when required for verification. For example, by clicking on an ID number in the database browser, the audit trail that is associated with the ID will be displayed. These functions are available for periodic validation and in support of regulatory agency inspections (for quick response times).

■ Electronic signature

Users with electronic signature privileges can add a digital signature to the data. When a digital signature is added to a report, the electronic signature information in the report (the signer, the signature date, signature reason, etc.) is displayed.

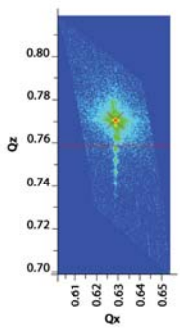
Customized support

Rigaku's expert support help achieves cost-effective compliance. A properly crafted and customized compliance engagement delivers value and is more cost effective than a reactive approach. In regulated industries, compliance with government and international standards requires documented verification that your systems are installed and functioning according to their manufacturer's intended use. Whether you need Installation Qualification (IQ), Operation Qualification (OQ), Standard Operating Procedures (SOP) or validation plan support, Rigaku services help provide your regulating agency with the necessary documentation as proof of compliance.*

* This service is optional. For more information, please contact customer support.

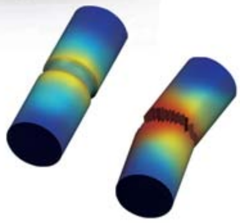
Multi-dimensional applications with SmartLab

1D/High-speed readout, High spatial resolution



High-Speed Reciprocal Space Map (High Resolution)

Lattice constant, strain and stress analyses of epitaxial films.

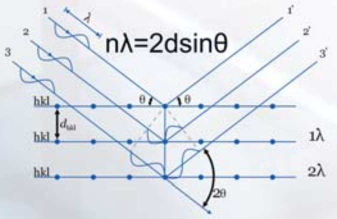


High-Speed Residual Stress (Fixed ψ_0)

High-speed residual stress and strain analyses of bulk samples.

High-Speed Powder XRD (Time Delay Integration)

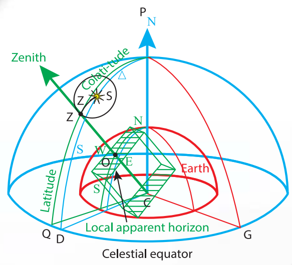
High-speed measurement for identification, quantitative analysis, crystallite quality of powders.



2D/Large active window with high sensitivity

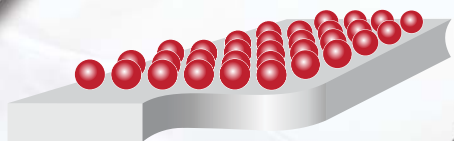
Pole Figure

Texture, orientation analyses of inorganic bulk samples.



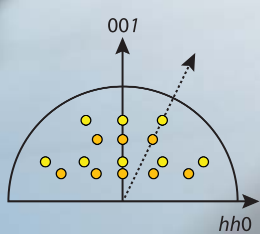
GI-SAXS

Characterization of quantum dot arrays and self-assembled organics.



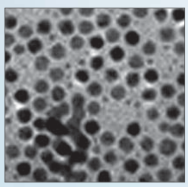
Wide Range Reciprocal Space Map

Epitaxial relationships, domain evaluation of epitaxial films.



***In situ* High-Temperature XRD**

Phase transition, reaction, dehydration analyses of organic compounds.



In-Plane XRD

Phase identification, crystal quality, and orientation analyses of bulk and thin films.

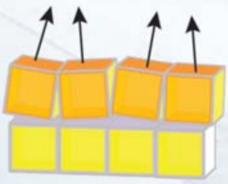
0D/High count rate, High accuracy photon counting

Reflectivity

Thickness, density, and roughness analyses of thin film samples.

High-Resolution Rocking Curve

Thickness, composition and mismatch evaluation of epitaxial films.



Rocking Curve

Mosaic spread analysis of samples with preferred orientation.

SAXS and U-SAXS

Particle size and distribution analyses of nano/sub micron scale particles.

SmartLab
Advanced Applications

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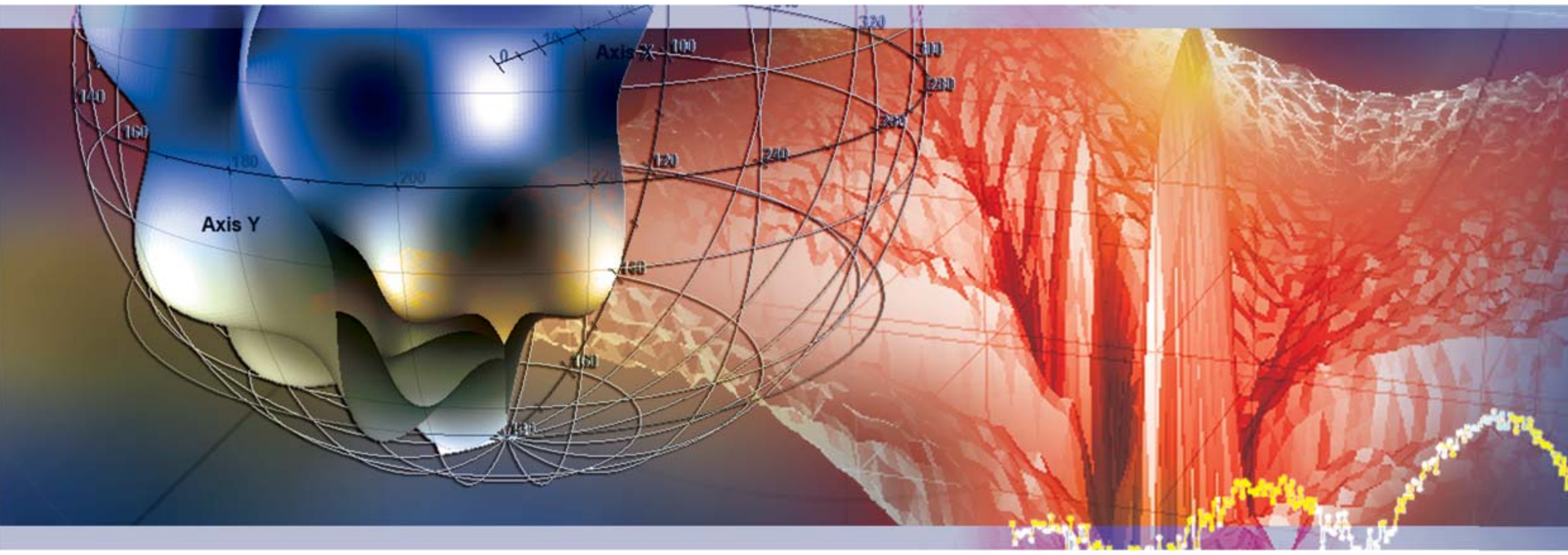

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