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SmartLab™ Automatic X-Ray Diffractometer

1. Overview

SmartLab is the world's first high resolution diffractometer with horizontal sample mounting theta-theta geometry. Designed for the structural analysis of advanced materials and thin films, the system combines a uniquely flexible instrument geometry with a comprehensive knowledge-based software platform. SmartLab is exceptional in that it enables users with little or no expertise in diffraction methods to make advanced structural measurements quickly and easily.

Applications for the SmartLab exist across a wide range of disciplines, from basic research to process development, and materials production. Example areas of interest include: organic films, epitaxial films for semiconductor and optical devices, metal and alloy nano-particles, and magnetic films for next generation recording media.

2. Benefits

The Rigaku SmartLab was specifically designed to offer comprehensive structural analysis capabilities to a broader range of research disciplines. Whether for geologists, chemists, biologists, materials scientists and engineers, precise structural data are attainable—directly and non-destructively—across a wider range of applications than are possible with any other commercially available instrument.

SmartLab offers the new and patented Cross Beam Optics (CBO) described in Section 3 in detail and features an intelligent expert system based software interface. The former eliminates the requirement to execute a complicated manual reconfiguration in order to switch between different measurement arrangements. Having highly intelligent software means that SmartLab does not require a very high level of specific expertise in order to acquire and reduce data with appropriate hardware configurations and measurement conditions. Flexibility of the measurement configurations, coupled to intelligent software, provides a value paradigm that cannot be matched by competitive instruments that require detailed knowledge about X-ray measurements.

SmartLab is the only commercially available X-ray diffractometer to be equipped with a high resolution theta-theta goniometer for horizontal sample mounting. This has the benefit of pre-

venting either bending or bowing of the sample, and is thus ideal for measurements of large or liquid samples while making sample handling trivial.

3. System

SmartLab offers the complete range of X-ray diffraction measurements in one fully automated tool suitable for use by the nonspecialist. It combines a high resolution, high powered, horizontal sample mount X-ray diffractometer with an automated, knowledge based control system. Offering simplified automated operation, the system can address the full range of samples: bulk solids, liquids, powders, and thin films. In addition, it can analyze all crystalline forms: perfect, textured, polycrystalline, disordered, and amorphous materials. SmartLab offers the complete range of structural X-ray measurements: Powder X-ray Diffraction (PXRD), High Resolution X-Ray Diffraction (HRXRD), Grazing Incidence X-Ray Diffraction (GIXRD), X-Ray Reflectometry (XRR), and Small Angle X-Ray Scattering (SAXS).

The SmartLab Guidance^{TM} software is used to automate all processes, from the setting and aligning of the optics to sample measurements. Software is pre-programmed at the factory with various optical alignment, sample alignment, and data analysis applications packages.

These modular Guidance Packages (GPAKS) contain the techniques and know-how to make advanced measurements, such as film thickness, texture, and pore/particle size analyses. GPAKS suggests the best optical configuration for each application, checks the hardware settings, and run automatic alignment sequences. Customizations of GPAKS are also available for advanced users and their measurement conditions can be customized.

SmartLab contains a horizontal sample mount, theta/theta goniometer, with high resolution scanning both in the plane of the sample surface and perpendicular to the sample surface. This geometry provides users a simple, uniform, stress free mount for all sample types including liquids. The high resolution scanning in both parallel and orthogonal directions eliminates the user requirement of changing and realigning the instrument configuration when making in-plane and out-of-plane measurements.

SmartLab uses an available 9 kW rotating anode X-ray source. This high power source is optimal for many advanced materials. Ultra thin films and dilute solutions are two areas benefited by high intensity. The 9 kW rotating anode is available direct from the factory or may be added as a field upgradeable component.

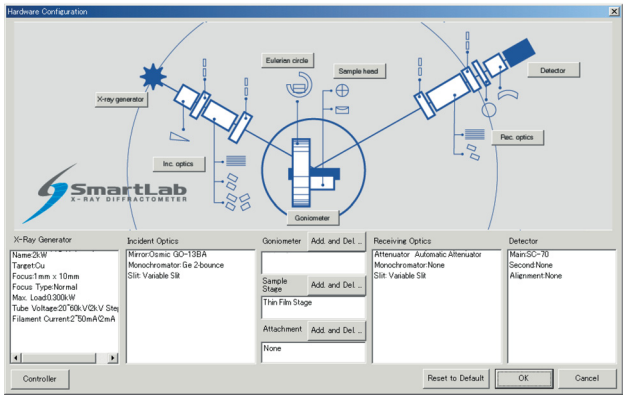

Fig. 1. The Guidance main status screen shows the system's current configuration and available optics.

SmartLab uses CBO as the foundation of a fully automated flexible optical system. CBO allows the SmartLab system to operate in both parallel beam and focusing geometries without reconfiguring the diffractometer system. Both geometries are permanently mounted, simultaneously aligned, and user selectable. SmartLab has completely automated control and sensing of all downstream monochromators and slit optics. Non-expert users benefit by the time saved and ease-of-use afforded by a dual geometry system and by the ability of the Guidance soft-

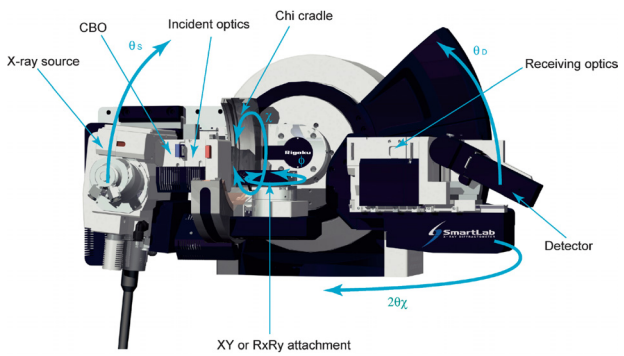

Fig. 2. The SmartLab horizontal sample mount goniometer. θ_D and $2\theta\chi$ are the orthogonal scanning directions.

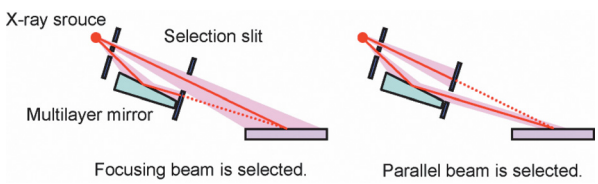

Fig. 3. The Rigaku's patented Cross Beam Optics.

ware to monitor and recommend optics configurations.

4. Applications

SmartLab has a wide range of applications, including: traditional PXRD, HRXRD, XRR, and SAXS. PXRD methods use signature diffraction patterns from powdered crystalline materials as fingerprints in the identification of crystalline composites in geology, materials science, and pharmaceutical applications. In contrast, HRXRD and XRR measurements are used to gauge the structure and perfection of thin films and multilayer interfaces in materials science research and semiconductor fabrication. SAXS analysis provides structural information on macromolecules and nanomaterials, in diverse applications from medicine to rubber manufacturing.

Rietveld refinements allow the determination of a wide range of sample characteristics such as composition, crystallinity, and crystallite size. Fig. 4 shows the X-ray data and refinement results for a sample of hydroxyl apatite. The resultant crystal structure is shown in Fig. 5.

X-ray measurements of advanced materials including semiconductor devices can yield information such as thin film thickness, orientation, perfection, and composition. The high resolution rocking curve measured with Ge 4-bounce monochromator (Fig. 6) and high resolution reciprocal space map measured with triple-axis geometry (Fig. 7) provide structural information from a SiGe hetero epitaxial film on a Si substrate.

SAXS is an important tool in the study of the structure of nanomaterials. Fig. 8 shows the SAXS pattern from a 0.0045% solution of gold nanoparticles. Data analysis of SAXS patterns can yield particle size distributions in both solids and liquids. Fig. 9 shows the particle size distribution calculated from the data collected on the gold nanoparticle solution.

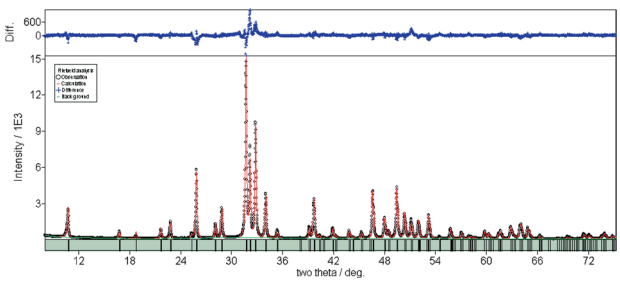

Fig. 4. The Rietveld analysis of hydroxyl apatite.

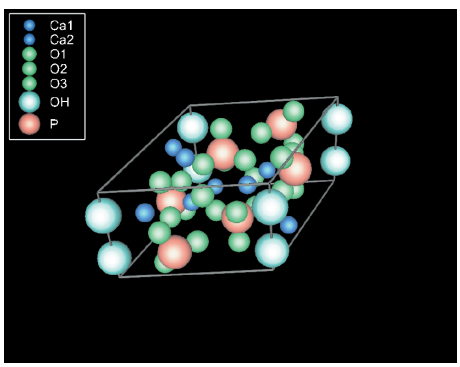

Fig. 5. The refined crystal structure of hydroxyl apatite.

5. Summary

Automatic alignment, CBO, and SmartLab

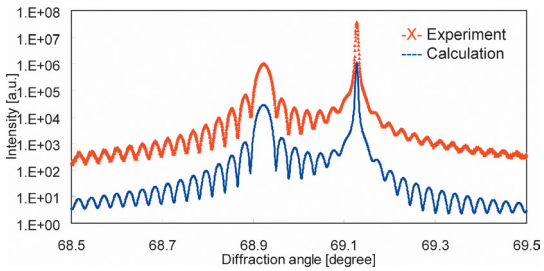

Fig. 6. The experimental and calculated rocking curves of SiGe/Si (004).

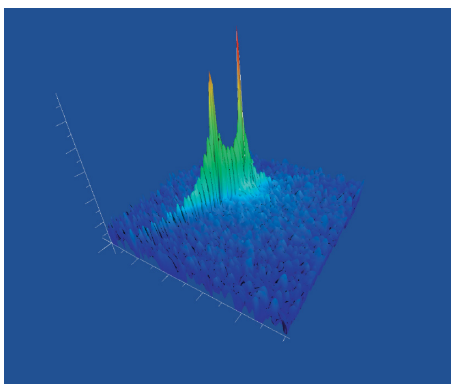

Fig. 7. Reciprocal space map of SiGe/Si (224).

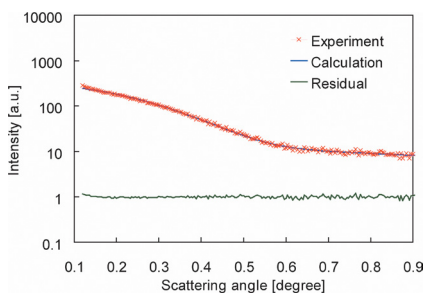

Fig. 8. The small angle scattering pattern from a 0.0045% solution of gold nanoparticles. (Tanaka Kikinzoku Kogyo K. K.)

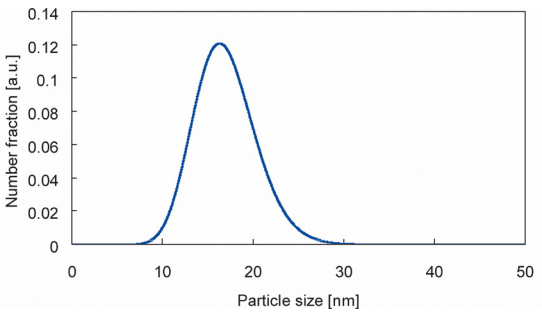

Fig. 9. The calculated particle size distribution.

Guidance software combine to create an extremely flexible, intelligence-based X-ray diffraction instrument. CBO technology allows selection of focusing and parallel beam geometries for the widest possible range of applications. SmartLab Guidance gathers information about samples, suggests measurement configurations, sets up the diffractometer, and executes measurements, with the help of an interactive graphical user interface.

Specifications

X-ray generator		
Maximum rated output	3 kW	Field upgradeable 9 kW
Rated tube voltage-current	20 – 60 kV / 2 – 60 mA	20 – 45 kV / 10 – 250 mA
Target	Cu (standard) (others: optional)	
Goniometer		
Scanning mode	θs/θd coupled or θs, θd independent Optical encoder controlled	
Goniometer radius	300 mm (standard) (others: optional)	
Minimum step	0.0001 deg.	
Chi cradle	χ: -5 – 95 deg. / 0.001 deg. step ϕ: -720 – 720 deg. / 0.002 deg. step Z: -4 – 1 mm / 0.0005 mm	
Attachment (Optional)	Rx, Ry: -5 – 5 deg. X, Y: 20 mm^2, 100 mm ϕ, or 150 mm ϕ	
Sample size	Max. 100 mm ϕ x 3 mm t (standard) (150 mm ϕ, 200 mm ϕ, 3-24mm t optional)	
Optics		
Incidence optics	CBO, Ge 2, 4-bounce monochromators Automatic variable incident slit	
Receiving optics	Automatic variable receiving slit 1 PSA, Ge 2-bounce analyzer Automatic variable receiving slit 2	
Detector		
Scintillation counter	Scintillator NaI, photomultiplier with preamplifier	