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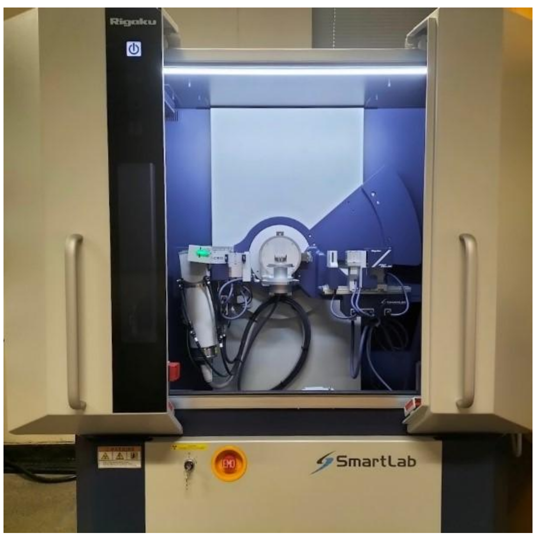
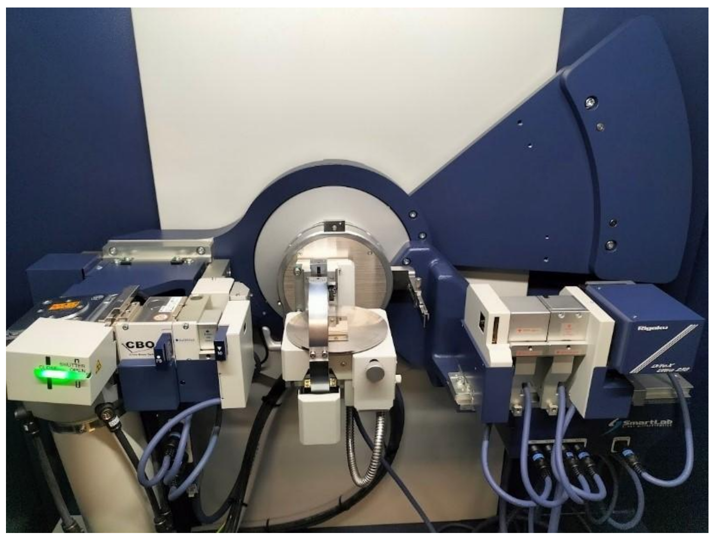
Grazing Incidence X-ray Diffraction (GiXRD): A Practical Guide for Thin Film Characterization Using Rigaku SmartLab SE

Version: GiXRD -2026-I

Other XRD Operational Manuals





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Abstract

This manual describes the practical use of Grazing Incidence X-ray Diffraction (GiXRD) on the Rigaku SmartLab SE for thin-film and surface-sensitive structural analysis. In GiXRD, a low fixed incident angle is used to reduce X-ray penetration into the substrate and enhance diffraction from the film or near-surface region. This approach is useful when conventional θ–2θ XRD shows strong substrate peaks, such as Si(400), that may obscure weak thin-film signals.

Introduction

Thin-film X-ray diffraction often requires a measurement geometry that can reduce the contribution from the underlying substrate. In conventional **θ–2θ XRD**, the X-rays penetrate relatively deep into the sample, so diffraction from a single-crystal or crystalline substrate may dominate the pattern and hide weak peaks from the film. This becomes a major limitation when analyzing thin films, coatings, nanostructures, or near-surface layers. **Grazing Incidence X-ray Diffraction (GiXRD)** is used to overcome this problem by keeping the incident angle **ω** fixed at a very low value, typically in the range of about **0.2° to 1.0°**, while scanning the detector angle **2θ**. Because the X-ray beam strikes the sample at a shallow angle, the penetration depth is reduced and the diffraction signal mainly comes from the thin film or surface region rather than the bulk substrate.

This manual provides a practical guide for performing GiXRD measurements on the **Rigaku SmartLab SE**. It explains the basic principle, measurement geometry, parameter selection, comparison with conventional θ–2θ scans, and interpretation of thin-film diffraction data. The method is especially useful when substrate peaks, such as the **Si(400)** reflection, are suppressed in GiXRD but visible in conventional

XRD, confirming the surface-sensitive nature of the technique. Basic GiXRD measurements can be performed without a monochromator; however, for improved resolution and better peak clarity, the use of suitable thin-film optics, a monochromator, and a χ–ϕ stage is recommended, especially when working in **Parallel Beam (PB)** mode or when analyzing weak and broad peaks from nanocrystalline.

GiXRD

Grazing Incidence X-ray Diffraction (GiXRD) is a surface-sensitive XRD technique used to study thin films, coatings, and nanostructured materials. In this method, the incident X-ray beam is fixed at a very small angle, usually below 1°, so that the X-ray interaction is mainly limited to the film or near-surface region. This reduces the contribution from the substrate and improves the visibility of weak diffraction peaks from the deposited layer. GiXRD is useful for phase identification, crystallite size estimation from peak broadening, and strain-related analysis in thin films, nanoparticles, quantum dots, nanowires, and nanocomposite coatings. On the Rigaku SmartLab SE, GiXRD provides a practical method for obtaining structural information from surface and nanoscale material systems.

Principle of GiXRD

Grazing Incidence X-ray Diffraction (GiXRD) is performed by fixing the incident angle ω at a shallow value, commonly between 0.2° and 1.0°, while scanning the detector angle 2θ. At this low angle, the X-ray beam interacts mainly with the film or near-surface region, making the diffraction pattern more representative of the deposited layer rather than the bulk substrate. In this geometry, ω controls the probing depth, while the 2θ scan records the diffraction peaks used for phase identification, crystallinity, peak broadening, and strain-related analysis. This makes GiXRD suitable for studying weak, thin, or nanocrystalline surface layers where conventional θ–2θ XRD may be dominated by substrate signals.

Comparison: GiXRD vs. Conventional XRD

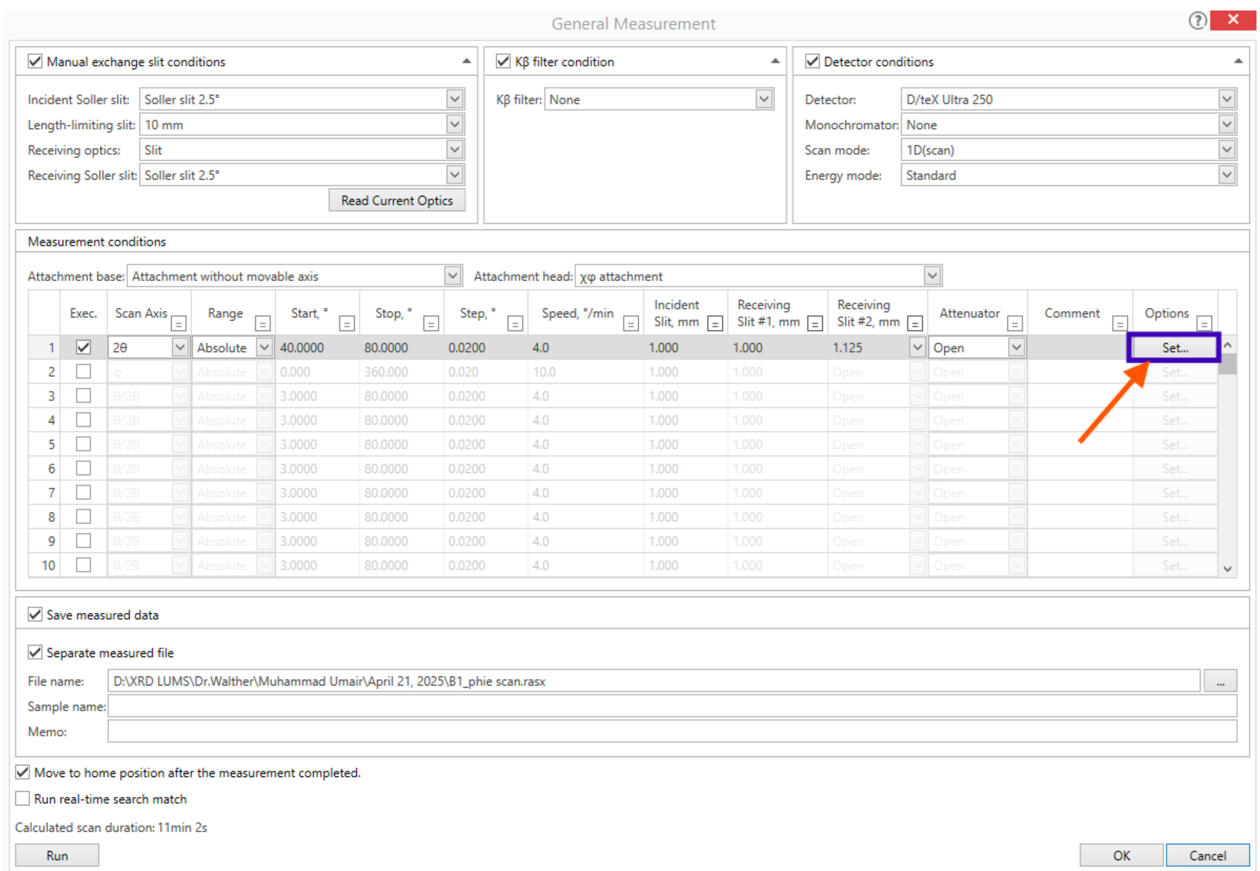
Feature	GiXRD	Conventional XRD / θ–2θ Scan
Measurement Geometry	Incident angle ω is fixed at a very low grazing angle; detector scans 2θ.	Sample angle θ and detector angle 2θ move together in a coupled scan.
Incident Angle	Very small, typically 0.2°–1.0°.	Continuously changes during the scan.
X-ray Penetration	Shallow; mainly probes the film or near-surface region.	Deeper; probes both film and substrate/bulk material.
Substrate Contribution	Strongly reduced, useful for weak thin-film peaks.	Often significant, especially for films on crystalline substrates.
Best Suited For	Thin films, coatings, nanostructures, surface layers.	Bulk materials, powders, and strongly crystalline samples.

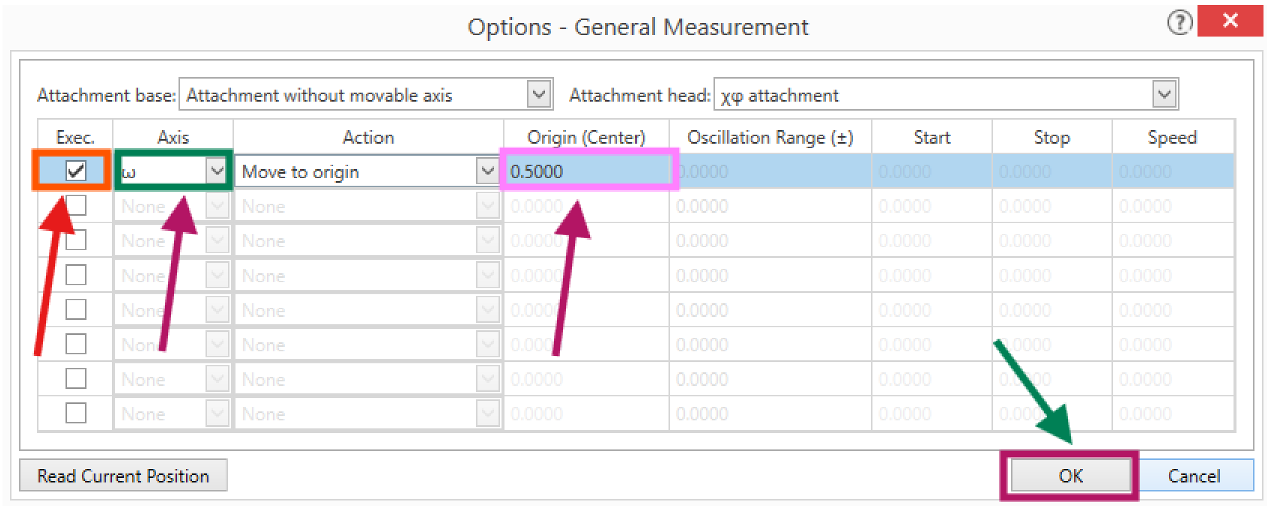
Information Obtained	Surface/film phases, crystallinity, texture, crystallite size, strain.	Bulk phase identification, lattice parameters, crystallinity.
Signal Intensity	Usually weaker; may require longer scan time.	Usually stronger; faster measurement for bulk samples.

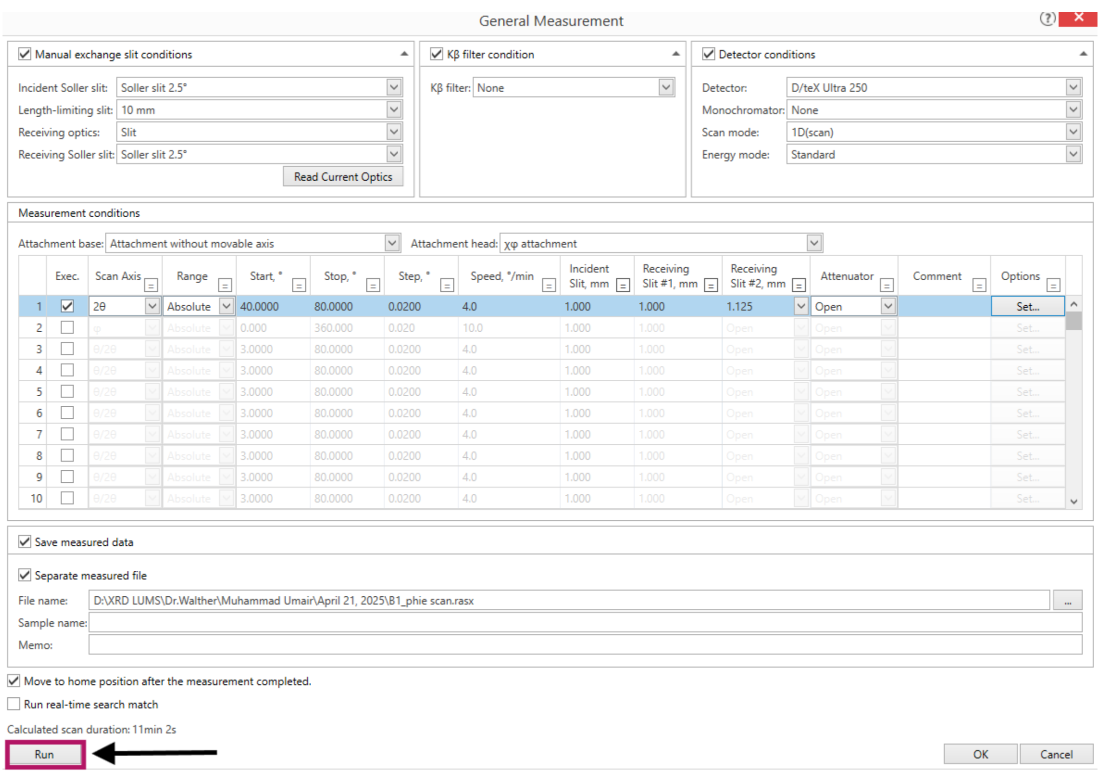
GiXRD measurement settings

The following figures show the main **GiXRD measurement settings** used on the **Rigaku SmartLab SE** system.

- **Incident angle (ω):** Keep ω fixed at a low grazing angle, such as **0.5°**, to measure mainly the thin film or near-surface region.
- **2θ scan range:** Select the 2θ range according to the expected diffraction peaks of the thin-film material. For example, **40°–80°** may be used for targeted analysis, while a wider/full range can be selected when the phase or peak positions are unknown.







Peak Observation in Conventional XRD and GiXRD

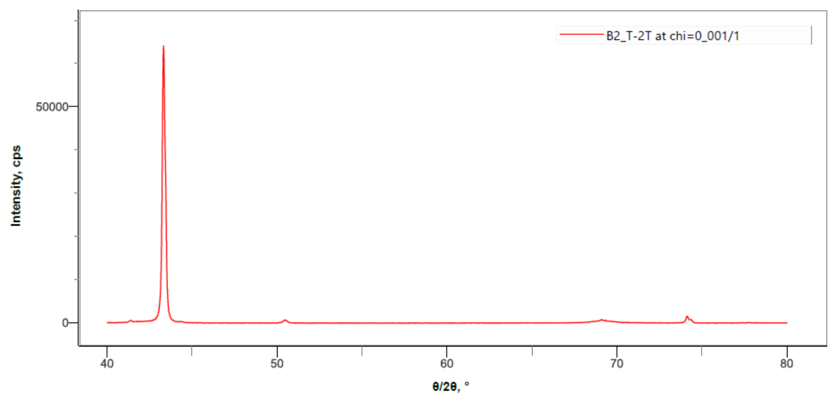
Conventional XRD (θ–2θ) Measurement

In the conventional θ–2θ scan, a Si(400) diffraction peak is observed near $2\theta \approx 69°$, showing that the X-rays penetrate sufficiently to produce diffraction from the underlying silicon substrate.

XRD Measurement Version 4.0

Profile / B2_T-2T at chi=0_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-04-22 15:56:14	Scan range	: 40.0000 - 80.0000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.0200 °	Incident slit box	: 1.000mm
X-ray	: 40 kV, 45 mA	Scan speed	: 4.0000 °/min	Length-limiting slit	: 10 mm
Wavelength	: Cu-Kα / 1.54186 Å	χ	: 0 °	Receiving slit box #1	: 1.000mm
Goniometer	: Standard Goniometer			Filter 1	: None
Attachment base	: Universal Z attachment			Receiving optics unit #1	: PSA open
Attachment head	: χφ attachment			Receiving Soller slit	: Soller slit 2.5°
Detector	: D/teX Ultra 250			Receiving slit box #2	: 1.125mm
Optics attribute	: PB			Receiving Attenuator	: Open
Memo	:			Detector monochromator	: None
				Detector slit	: None



GiXRD Measurements

In the GiXRD scan, the Si(400) substrate peak is not observed, indicating that the low fixed incident angle reduces X-ray penetration into the substrate. This confirms the surface-sensitive nature of GiXRD and its usefulness for enhancing thin-film signals while minimizing substrate contribution.

Reduced peak intensity in GiXRD

In **GiXRD**, the diffraction peak intensity is usually lower than in conventional θ–2θ XRD because the X-ray beam interacts with a smaller near-surface volume of the sample. This reduction in intensity is a normal trade-off for achieving surface sensitivity and minimizing substrate contribution. However, it can also reduce the signal-to-noise ratio, make weak or minor phases harder to detect, and limit the accuracy of quantitative phase analysis unless suitable corrections are applied. Crystallite size and strain analysis can still be performed from peak broadening, but higher noise may reduce the precision of the results. Therefore, GiXRD measurements often require careful optimization, such as longer scan time, smaller step size, or repeated scans, to obtain reliable thin-film diffraction data.

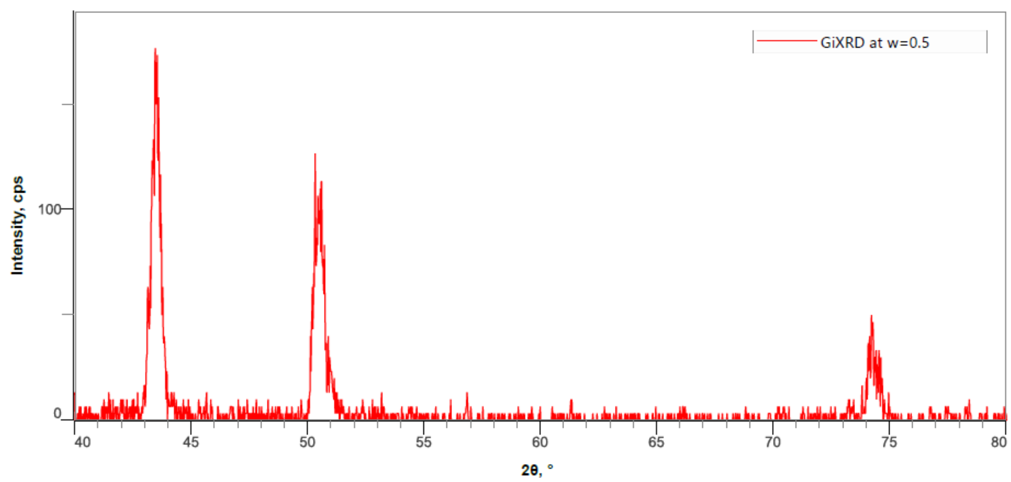
Optimizing GiXRD Settings in SmartLab SE for Improved Peak Clarity

The main purpose of GiXRD is surface and near-surface structural analysis, so reduced peak intensity is a normal and acceptable trade-off for minimizing substrate contribution. To improve peak clarity on the Rigaku SmartLab SE, the incident angle ω should be carefully selected according to film thickness, typically using a lower angle for very thin films and a slightly higher angle when more film volume is needed. Longer counting time, smaller 2θ step size, and repeated scans can improve the signal-to-noise ratio and help detect weak or broad peaks. The use of suitable thin-film optics, parallel-beam configuration, proper slits, and high-sensitivity detector settings can further enhance peak quality. Good sample preparation is also important; the surface should be flat, smooth, and well aligned to avoid artificial broadening or intensity loss.

XRD Measurement Version 4.0

Profile / GiXRD at w=0.5

Sample name	:	Scan axis	: 2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-04-21 15:21:33	Scan range	: 40.0000 - 80.0000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.0200 °	Incident slit box	: 1.000mm
X-ray	: 40 kV, 45 mA	Scan speed	: 4.0000 °/min	Length-limiting slit	: 10 mm
Wavelength	: Cu-Kα / 1.54186 Å	ω	: 0.5 °	Receiving slit box #1	: 1.000mm
Goniometer	: Standard Goniometer	χ	: 0 °	Filter 1	: None
Attachment base	: Universal Z attachment			Receiving optics unit #1	: PSA open
Attachment head	: χφ attachment			Receiving Soller slit	: Soller slit 2.5°
Detector	: D/teX Ultra 250			Receiving slit box #2	: 1.125mm
Optics attribute	: PB			Receiving Attenuator	: Open
Memo	:			Detector monochromator	: None
				Detector slit	: None



Discussion of Measurement Results

The measurement settings show that the GiXRD scan was performed using Cu-Kα radiation (λ = 1.54186 Å) with a fixed low incident angle ω = 0.5°, χ = 0°, parallel beam (PB) optics, and a D/teX Ultra 250 detector. Under this grazing-incidence condition, the X-ray beam mainly probes the thin film or near-surface region rather than the bulk silicon substrate. Therefore, the absence or strong reduction of the Si(400) peak near 2θ ≈ 69° confirms that GiXRD effectively suppresses substrate diffraction. In contrast, the conventional θ–2θ scan allows deeper X-ray penetration and can detect diffraction from both the thin film and the underlying Si substrate.

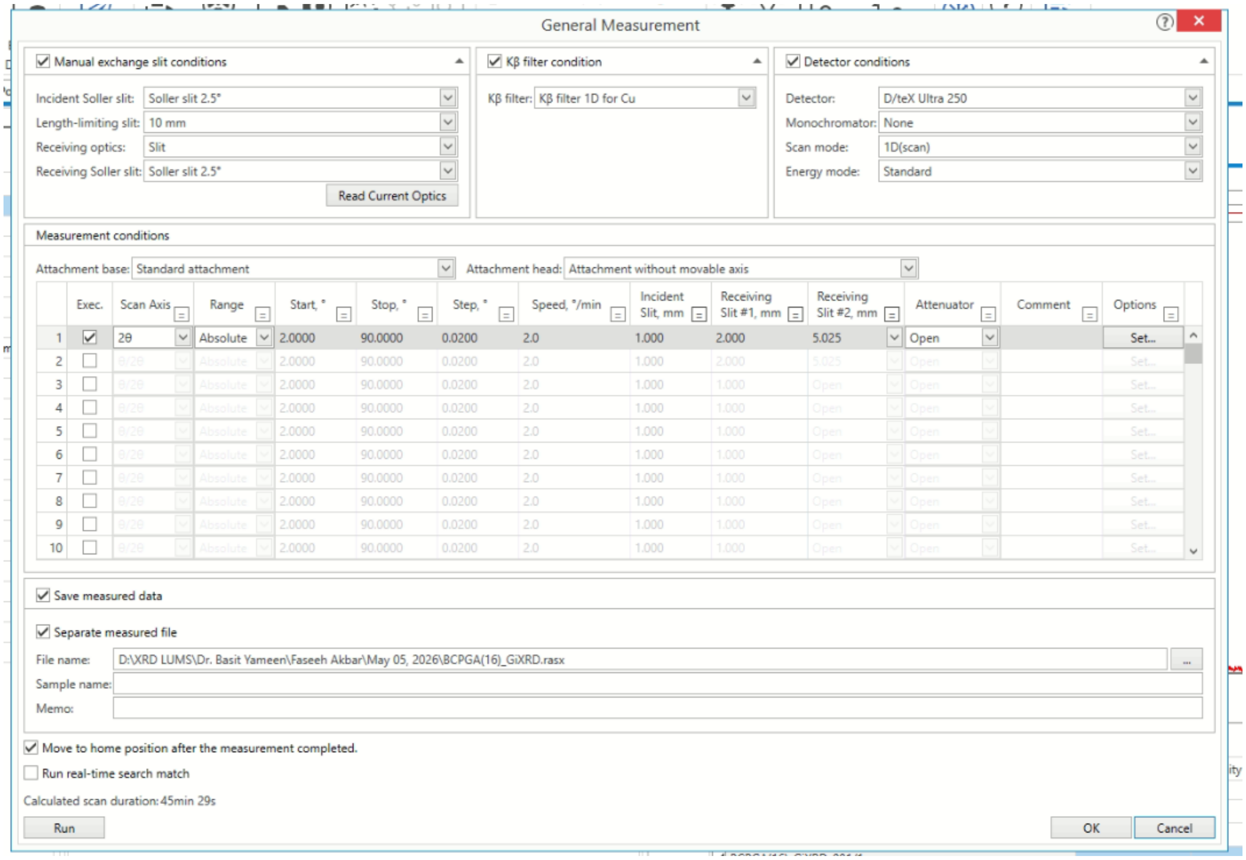
Conclusion

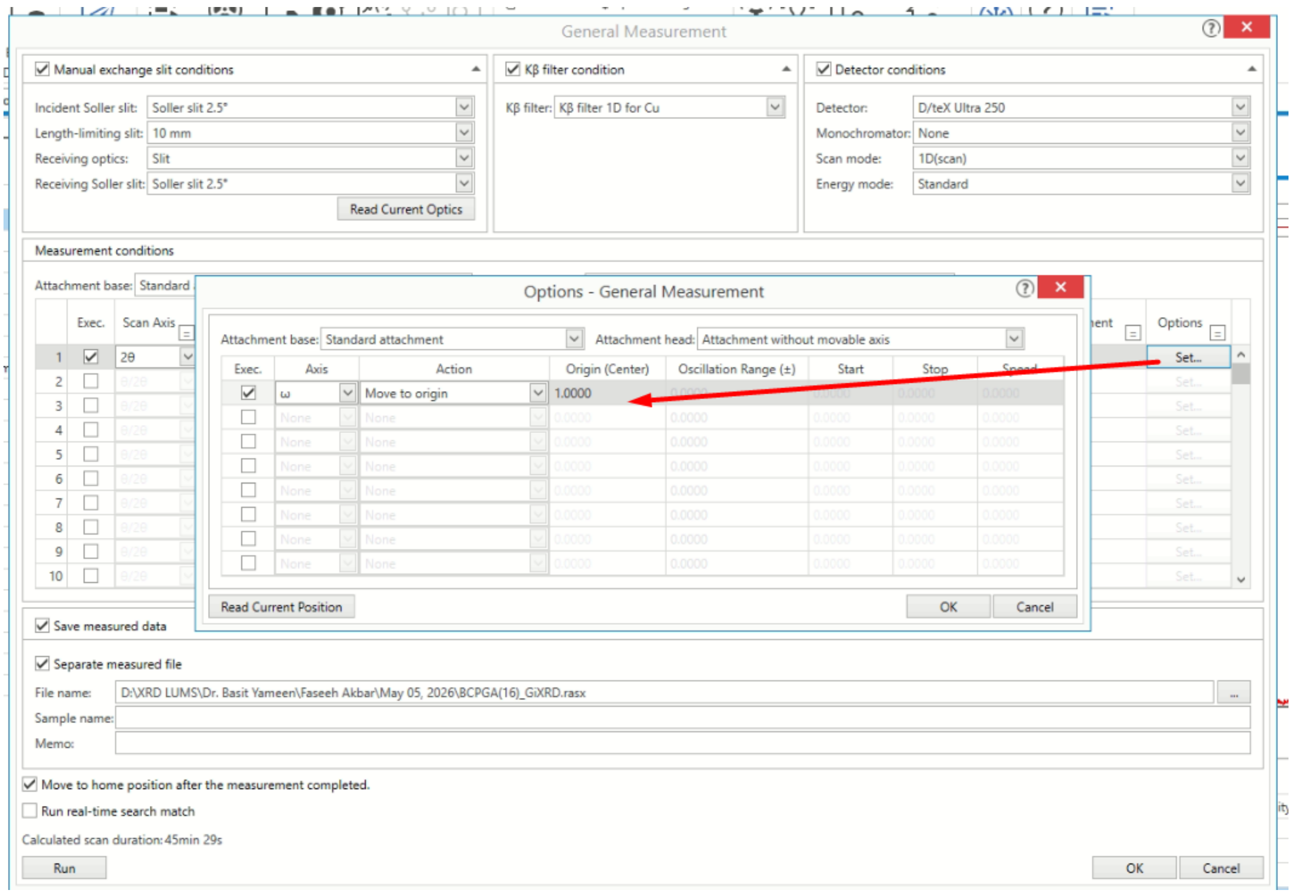
The comparison between conventional **θ–2θ XRD** and **GiXRD** confirms the advantage of grazing-incidence geometry for thin-film analysis. By using a fixed shallow incident angle, such as **ω = 0.5°**, GiXRD reduces the penetration depth of X-rays and minimizes the contribution from the silicon substrate. As a result, substrate reflections such as **Si(400)** are suppressed, allowing the diffraction response from the thin film or near-surface region to be observed more clearly. This makes GiXRD a practical and effective method for structural characterization of thin films, coatings, and nanostructured surface layers on crystalline substrates.

Rigaku Website

https://rigaku.com/products/x-ray-diffraction-and-scattering/xrd/smartlab-se

Improved Settings




General Measurement
Manual exchange slit conditions
Incident Soller slit: Soller slit 2.5°
Length-limiting slit: 10 mm
Receiving optics: Slit
Receiving Soller slit: Soller slit 2.5°
Read Current Optics
Kβ filter condition
Kβ filter: Kβ filter 1D for Cu
Detector conditions
Detector: D/teX Ultra 250
Monochromator: None
Scan mode: 1D(scan)
Energy mode: Standard
Measurement conditions
Attachment base: Standard
Exec.
Scan Axis
2θ
Options
Set...
Options - General Measurement
Attachment base: Standard attachment
Attachment head: Attachment without movable axis
Exec.
Axis
Action
Origin (Center)
Oscillation Range (±)
Start
Stop
Speed
ω
Move to origin
1.0000
0.0000
None
Read Current Position
OK
Cancel
Save measured data
Separate measured file
File name: D:\XRD LUMS\Dr. Basit Yameen\Faseeh Akbar\May 05, 2026\BCPGA(16)_GiXRD.rasx
Sample name:
Memo:
Move to home position after the measurement completed.
Run real-time search match
Calculated scan duration: 45min 29s
Run
OK
Cancel