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

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Physical Properties Measurement Lab.

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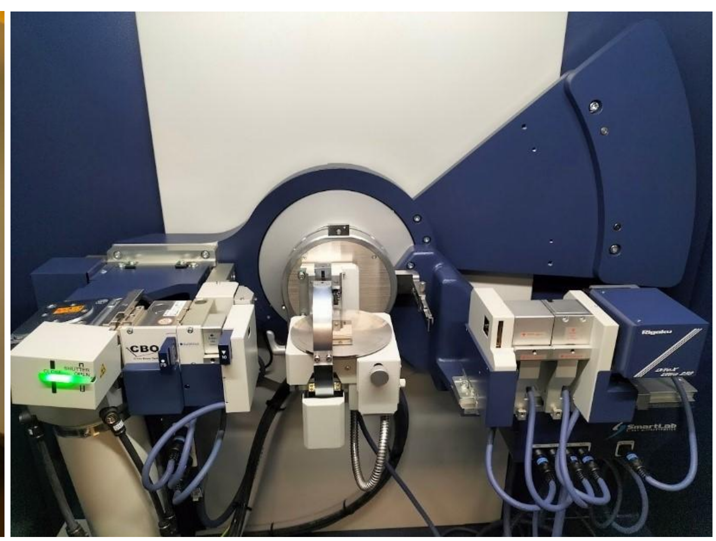

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Introduction

This document provides a practical overview of Grazing Incidence X-ray Diffraction (GiXRD) as implemented on the Rigaku SmartLab SE system. It is intended to guide researchers working with thin films and nanostructures in optimizing experimental conditions and interpreting results, especially in situations where conventional bulk XRD is insufficient.

Note: Basic GiXRD does not require a monochromator; however, a monochromator and Chi-Phi stage are highly recommended for high-resolution measurements, particularly in Parallel Beam (PB) mode.

GiXRD

Grazing Incidence X-ray Diffraction (GiXRD) is a specialized technique used for investigating the structural properties of thin films, surfaces, and nanostructured materials. By using a very low angle of incidence (typically below 1°), GiXRD limits the penetration depth of X-rays, enhancing surface sensitivity and minimizing substrate interference. This makes it highly effective not only for characterizing thin films but also for analyzing nanostructured materials, such as nanoparticles, quantum dots, nanowires, or nanocomposite coatings, especially when they are deposited as surface layers or embedded near the surface. In these systems, GiXRD enables phase identification, crystallite size estimation (via peak broadening), and strain analysis—critical parameters for understanding and optimizing nanoscale functionality. The SmartLab SE system is equipped to perform high-resolution GiXRD, providing valuable insights into these advanced material systems.

Principle of GiXRD

Grazing Incidence X-ray Diffraction (GiXRD) operates by maintaining the incident angle (ω) of the X-ray beam at a fixed, shallow value typically between 0.2° and 1.0°. This small angle restricts the X-ray penetration depth, ensuring that the diffraction signal primarily originates from the surface or near-surface region of the sample. This makes GiXRD highly sensitive to thin films, nanostructured coatings, and surface layers, while effectively suppressing the contribution from the underlying substrate.

Key aspects of the technique:

- Fixed ω (incident angle): Enhances sensitivity to surface layers and thin films
- Scanning 2θ (detector angle): Records diffraction patterns from surface-confined structures

This configuration enables detailed analysis of surface crystallinity, phase composition, strain, and nanocrystalline size without interference from the bulk material.

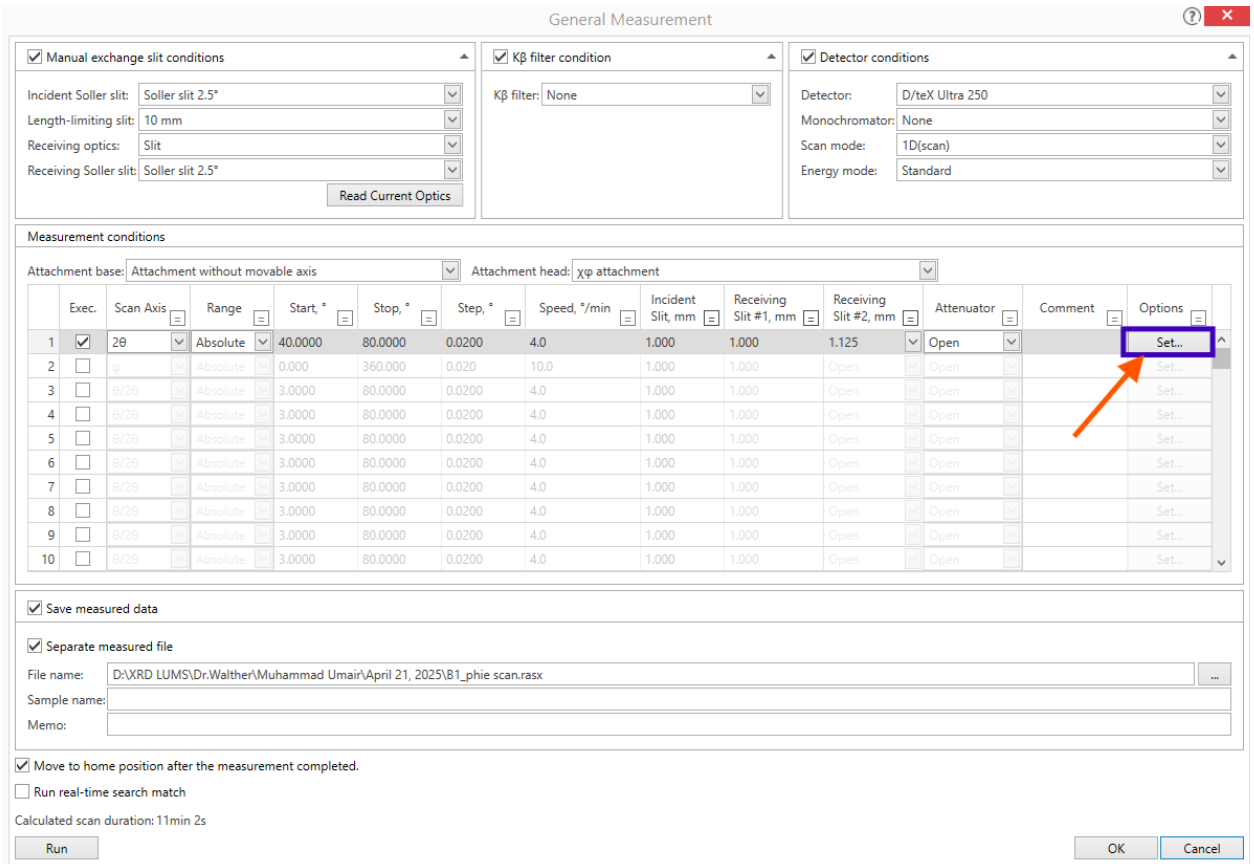
Comparison: GiXRD vs. Conventional XRD

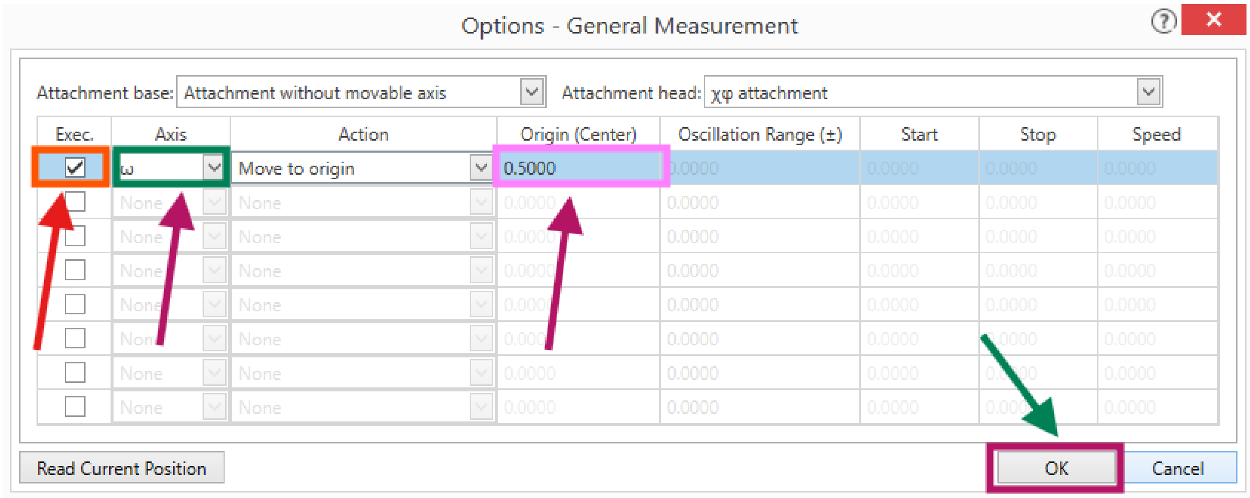
Feature	**GiXRD**	**Conventional XRD (θ–2θ)**
Incident Angle	Fixed (ω) at a very low angle (typically 0.2°–1.0°)	Varies with 2θ (θ = ½ of 2θ scan)
X-ray Scattering Geometry	Grazing incidence: fixed ω, scan 2θ	θ–2θ geometry: coupled scan
Penetration Depth	Shallow: typically 5–100 nm, depending on angle & material	Deep: typically several micrometers (μm)
Ideal for	Thin films, surface layers, nanostructures	Bulk polycrystalline samples
Substrate Interference	Minimized	Significant, especially for thin films
Phase Information	Surface and near-surface phases	Dominantly bulk phase information
Measurement Time	Generally longer due to lower signal intensity	Faster for bulk materials due to stronger signals
Application Fields	Thin film research, coatings, nano-layered structures	Powder diffraction, bulk material characterization

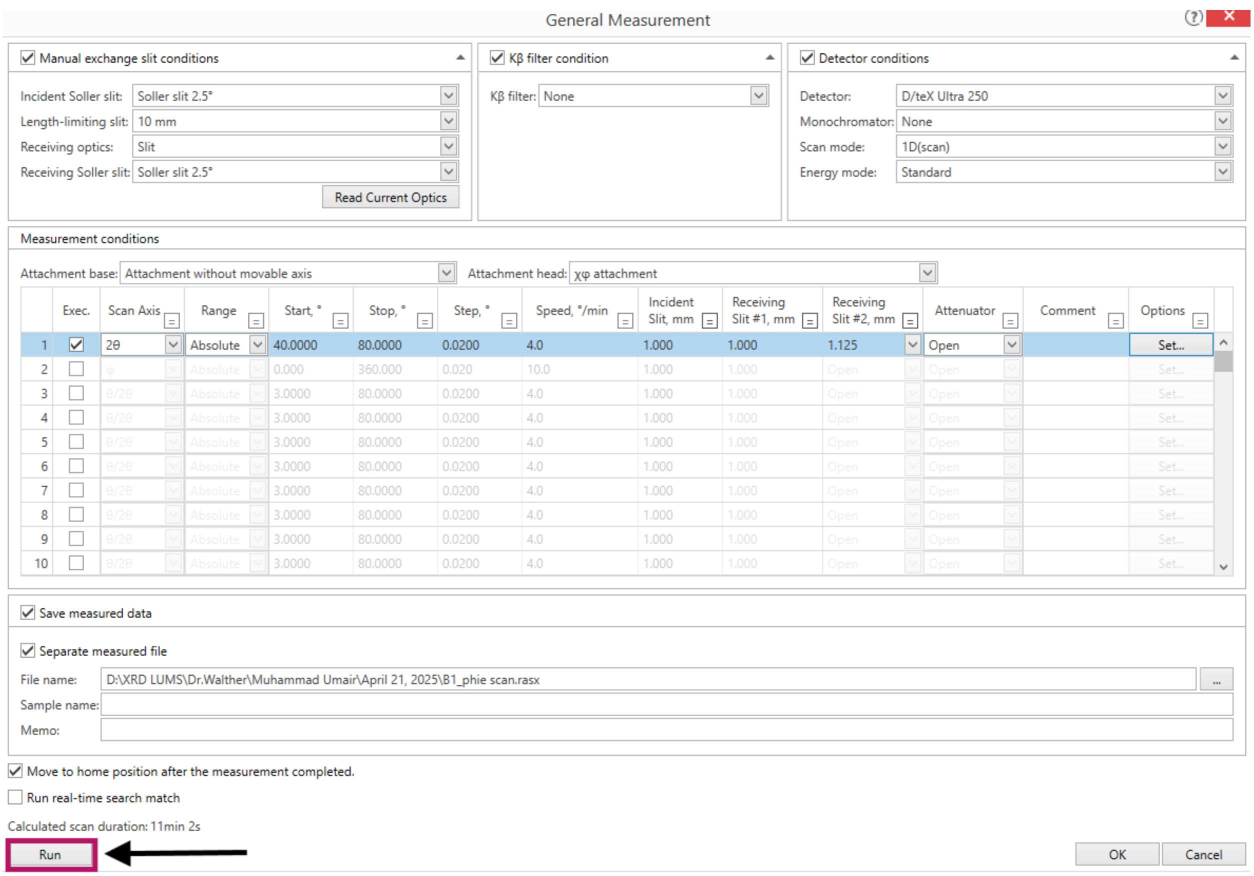
GiXRD measurement settings

The following figures illustrate the **GiXRD measurement setup** on the Rigaku SmartLab SE system:

- **Incident angle (ω):** Set to a small, fixed value (e.g., **0.5°**) to limit X-ray penetration depth and enhance surface sensitivity.
- **2θ scan range:** Typically set from **40° to 80°**, selected based on the expected diffraction peaks of the thin film material under investigation.







Peak Observation in Conventional XRD and GiXRD

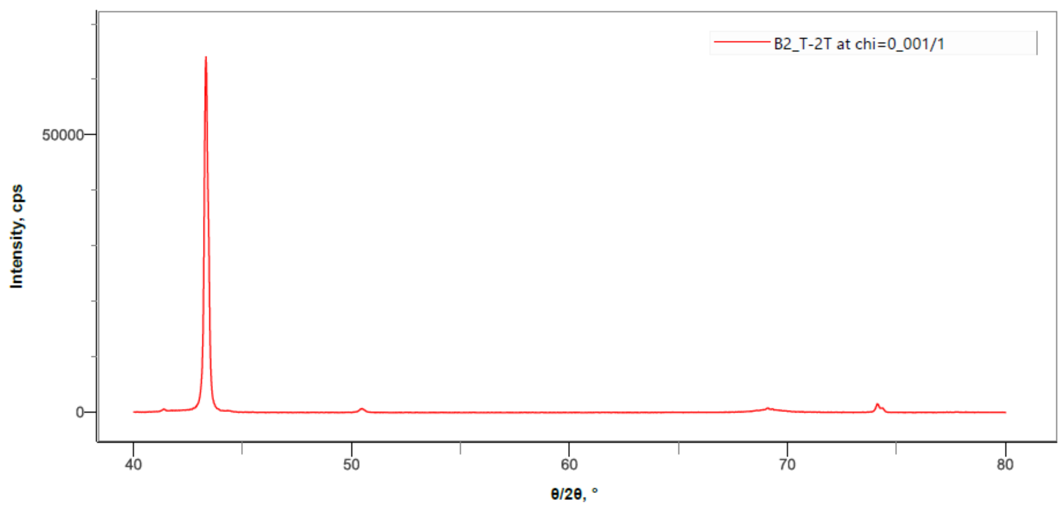
Conventional XRD (θ–2θ) Measurements

In the θ–2θ scan, a weak **Si(400)** diffraction peak appears around **69°**, indicating detectable X-ray 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)** peak is absent, demonstrating that the incident X-rays did not penetrate deep enough to interact with the substrate highlighting the surface sensitivity of the technique.

Reduced peak intensity in GiXRD

The **reduced peak intensity in GiXRD** compared to conventional XRD does have technical implications, but it's generally a **trade-off** that is understood and accounted for in thin film and surface studies.

Technical Effects of Reduced Peak Intensity in GiXRD

Aspect	Effect in GiXRD
Signal-to-Noise Ratio (SNR)	Lower intensity reduces SNR, potentially requiring longer scan times or signal averaging to obtain reliable data.
Peak Detection Sensitivity	Weak or minor phases may be harder to detect due to lower intensity.
Quantitative Analysis	Less accurate for quantitative phase analysis unless corrections are applied.
Crystallite Size/Strain Analysis	Still feasible via peak broadening, but higher noise may affect precision.
Penetration Depth Trade-Off	Lower intensity is the cost of achieving surface sensitivity and minimizing substrate contribution.

Key Point

The **primary goal of GiXRD** is **surface and near-surface structural analysis**, so even though peak intensity is reduced, this is an **acceptable and manageable limitation**. Careful experimental setup (e.g., optimizing detector sensitivity, increasing scan time, or using thin film optics) can mitigate most of these effects.

How to optimize GiXRD settings in the SmartLab SE to improve peak clarity

Below is a concise and practical guide on optimizing **GiXRD settings in Rigaku SmartLab SE** to improve **peak clarity**, especially given the typically lower intensities associated with grazing incidence geometry.

Optimizing GiXRD Settings in SmartLab SE for Improved Peak Clarity

1. Select Optimal Incident Angle (ω)

- Choose a **fixed grazing angle** (typically between **0.3° and 1.0°**) based on your film's thickness:
    - For ultrathin films (< 10 nm): use 0.2°–0.3°
    - For thicker films (~100 nm): 0.5°–1.0°
- A **very low angle** reduces substrate contribution but may also reduce intensity; balance is key.

2. Increase Counting Time

- Use **longer dwell times per step** (e.g., 1–5 seconds or more).
- This improves **signal-to-noise ratio** and helps distinguish weak peaks.

3. Use Narrow Step Size

- Use a **step size of 0.01° or smaller** in 2θ.
- Helps in resolving closely spaced or broad peaks from nanocrystalline films.

4. Employ Thin Film Optics

- Ensure the **incident beam path** uses **parallel beam or monochromator optics**.
- Avoid divergence slits that allow deeper penetration.
- **Length-limiting slits** and **Soller slits** can improve resolution by reducing beam spread.

5. Use High-Sensitivity Detector Mode

- Set the **D/teX Ultra or 1D detector** to high-sensitivity or integrated mode to collect more counts.
- If available, use **multi-channel detection** to speed up acquisition while maintaining quality.

6. Background Reduction

- Apply **background correction** in SmartLab Studio II to eliminate diffuse scattering.
- Minimize air scatter by using **beam path enclosures** or **anti-scatter slits**.

7. Sample Preparation

- Ensure the **sample surface is smooth, flat, and uniformly coated** to avoid artifacts.
- Mount the sample to maintain a **perfectly level geometry** with the beam.

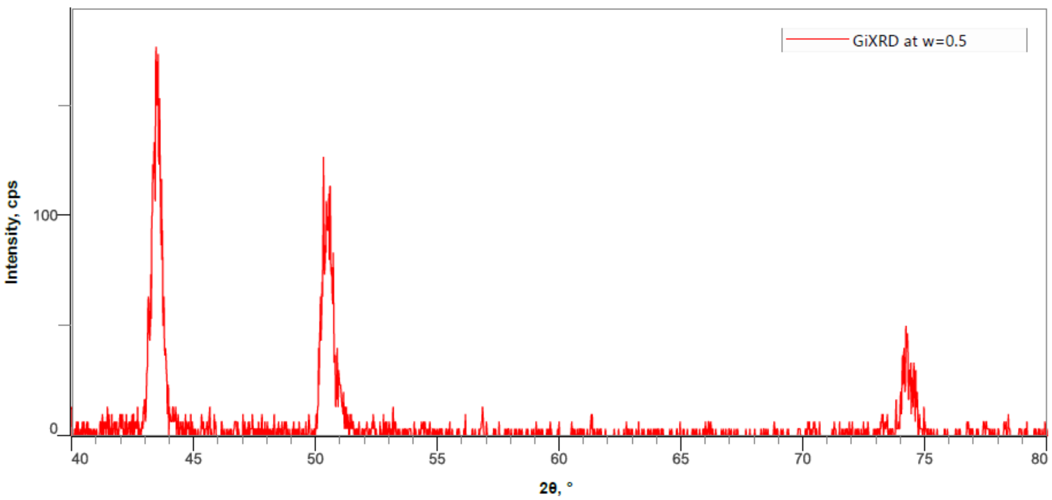
8. Temperature Stability

- Use **low-temperature drift** stages or ensure **room temperature stability**, as thermal expansion can shift peak positions or broaden them.

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



Interpretation

These observations confirm that:

- **Conventional XRD** captures diffraction signals from both the **thin film and the underlying substrate**.
- **GiXRD** selectively probes the **surface and near-surface regions**, effectively **suppressing substrate peaks**, making it highly suitable for **thin film characterization**.

Conclusion

Grazing Incidence X-ray Diffraction (GiXRD) is a highly effective technique for the structural analysis of thin films. Unlike conventional XRD, GiXRD enhances surface sensitivity by employing a fixed, shallow incident angle, which limits X-ray penetration into the substrate. The comparison between θ~2θ and GiXRD scans in this study clearly highlights the advantages of GiXRD, most notably, the suppression of substrate reflections such as the Si(400) peak. This enables a more accurate and focused investigation of the film's surface structure and composition.

Rigaku Website

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