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χ and ϕ scan for suppressing peaks of a single crystal substrate in a thin film using a χ–ϕ attachment and Ge(220) ×2 monochromator (optional) with SmartLab SE Rigaku

Version: Chi-Phi Scan-2026-I

Other XRD Operational Manuals

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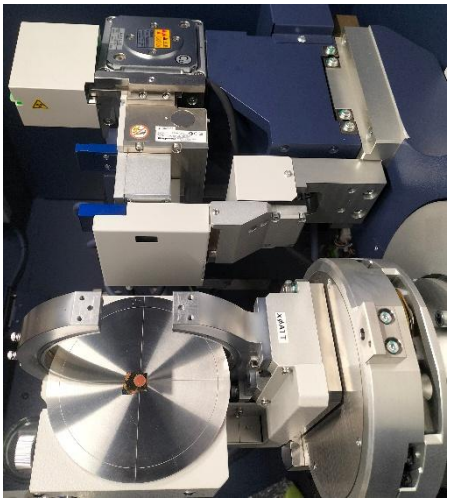
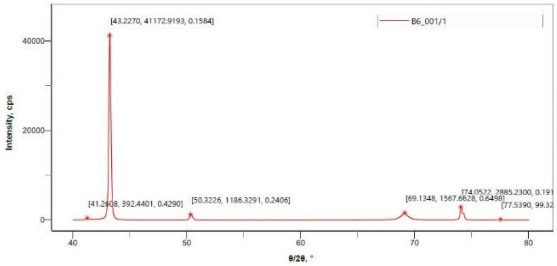
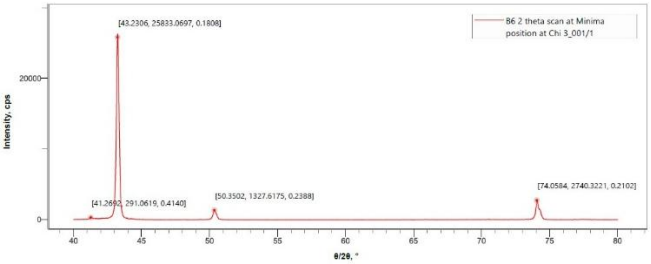






Contents

Abstract

This operation manual describes the use of χ (chi) tilt and ϕ (phi) rotation with the χ–ϕ attachment on the Rigaku SmartLab SE to suppress strong diffraction peaks from a single-crystal substrate during thin-film XRD measurements. By selecting suitable χ and ϕ positions, the substrate can be moved away from its strongest Bragg condition, allowing weak thin-film reflections to be observed more clearly.

Introduction

In thin-film X-ray diffraction analysis, a common challenge is the presence of very intense diffraction peaks from single-crystal substrates. These substrate peaks can dominate the diffraction pattern and may hide the weaker peaks from the thin-film layer. This problem becomes more significant when the thin film is grown on a highly oriented or epitaxial substrate, or when the film peak appears close to a strong substrate reflection.

In this procedure, the Rigaku SmartLab SE equipped with a χ–ϕ attachment is used to reduce the contribution of the single-crystal substrate. First, a normal θ/2θ scan is performed to identify the diffraction peaks from both the substrate and the thin film. Then, a χ scan is carried out by tilting the sample to find angular positions where the intense substrate peak becomes weak or disappears. At these selected χ angles, the substrate planes no longer fully satisfy Bragg's condition, while the broader thin-film diffraction signal may still remain detectable.

A ϕ scan is then used to rotate the sample around its surface normal and identify azimuthal positions where the substrate reflection is either maximum or minimum. The maximum ϕ position represents the strongest substrate diffraction condition, while the minimum ϕ position can be useful for suppressing the substrate signal. By combining χ tilt and ϕ rotation, suitable measurement conditions can be selected to minimize substrate interference and improve the visibility of weak thin-film peaks.

This method is especially useful for polycrystalline or textured thin films deposited on single-crystal substrates, where the film peaks are generally weaker and broader than the sharp substrate peaks. The use of a Ge(220) ×2 monochromator, can further improve angular resolution and peak separation. Overall, this χ–ϕ scan approach provides a practical method for substrate peak suppression and more reliable structural characterization of thin films.

An explanation of the angles theta (θ), 2-theta (2θ), omega (ω), chi (χ), and phi (ϕ).

Symbol (Angle)	**Definition**
θ (theta)	Angle between incident X-ray beam and sample surface.
2θ (2theta)	Total angle between incident and diffracted beams.
ω (omega)	Angle between incident beam and sample surface, often equal to θ.
χ (chi)	Tilt angle of the sample relative to its surface normal.
Φ (phi)	Rotation of the sample about its surface normal.

θ/2θ-Scan (First Step XRD measurements)

The first step in analyzing thin films using the Rigaku SmartLab SE involves performing standard XRD measurements. This process begins with proper optic alignment in Parallel Beam (PB) mode, which ensures the accuracy and precision of the incident and diffracted beams. Following this, sample alignment with the χ–ϕ attachment is carried out to accurately position the sample in all rotational and tilt axes. Once the alignment is complete, a θ/2θ scan is performed to obtain the initial diffraction pattern of the sample. This scan provides an overview of the crystalline phases present and helps in identifying both substrate and thin film peaks.

PB Mode (Parallel Beam)

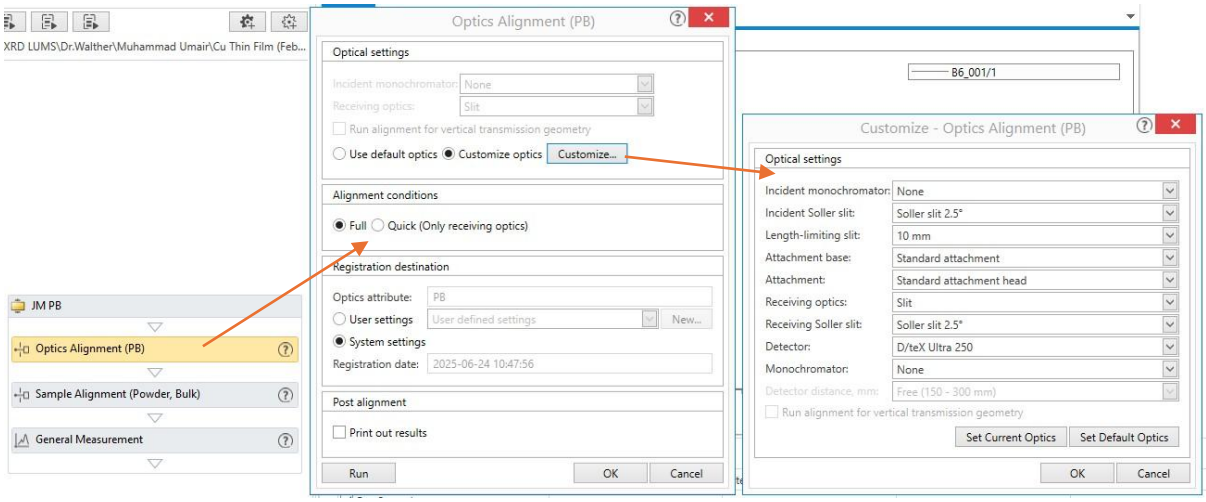
PB mode should be selected for thin films, epitaxial films, multilayers, preferred-orientation samples, grazing-incidence XRD, X-ray reflectivity, rocking curves, reciprocal space mapping, rough/curved surfaces, or samples where exact height alignment is difficult. In this mode, the incident X-ray beam is made nearly parallel using the instrument optics, so the measurement is less affected by sample height error, surface irregularity, and sample shape. PB is therefore more suitable for thin-film and surface-sensitive measurements, where correct geometry and reliable peak position are more important than maximum intensity. However, PB usually gives lower incident beam intensity and lower diffraction peak intensity than BB because part of the X-ray intensity is reduced while making the beam parallel. Therefore, PB should not normally be used as the first choice for routine powder XRD when the powder is flat, thick, well packed, and randomly oriented. If peaks are weak in PB, longer counting time, slower scan speed, or grazing-incidence geometry may be required.

BB Mode (Bragg Brentano)

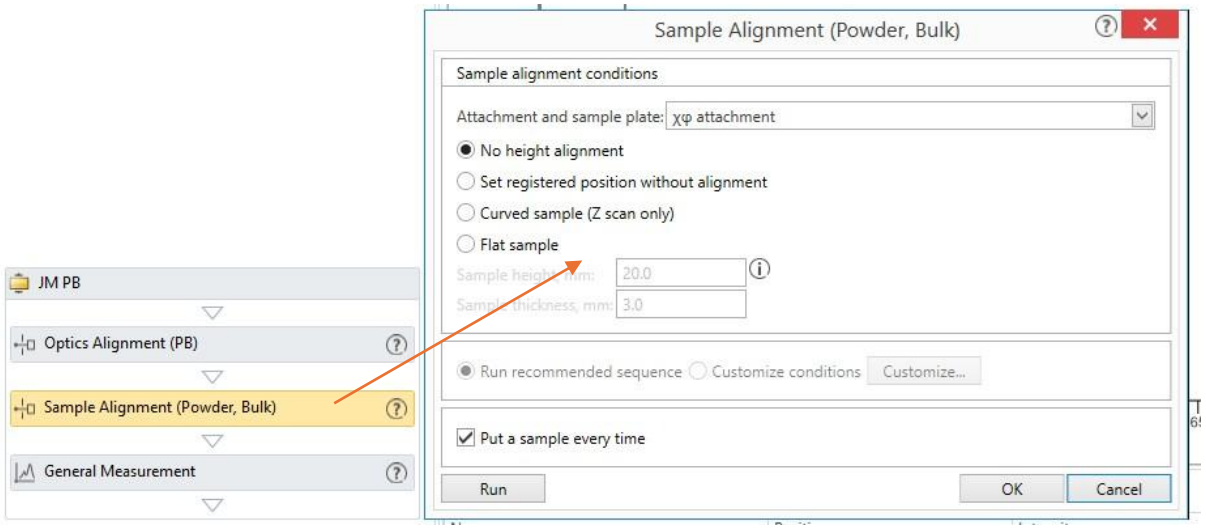
BB mode should be selected for routine powder XRD and bulk polycrystalline samples when the sample surface is flat, smooth, properly packed, correctly positioned, and has enough material to cover the X-ray beam. This is the standard powder-diffraction geometry and is commonly used for phase identification, qualitative and quantitative analysis, and normal powder scans. BB generally provides higher incident beam intensity and stronger diffraction peaks than PB because the beam is divergent/focusing and more X-ray intensity reaches the sample, allowing more sample volume to contribute to diffraction. For this reason, BB is usually the best mode when strong peak intensity is required from well-prepared powder samples. However, BB should not be preferred for very thin films, grazing-incidence measurements, XRR, rocking curves, RSM, rough samples, curved samples, or samples with uncertain height, because BB geometry is more sensitive to sample height and surface-position errors. A simple working rule is: use BB for normal powders when high intensity is needed, and use PB for thin films or

difficult sample geometry when correct geometry and reliable peak position are more important.

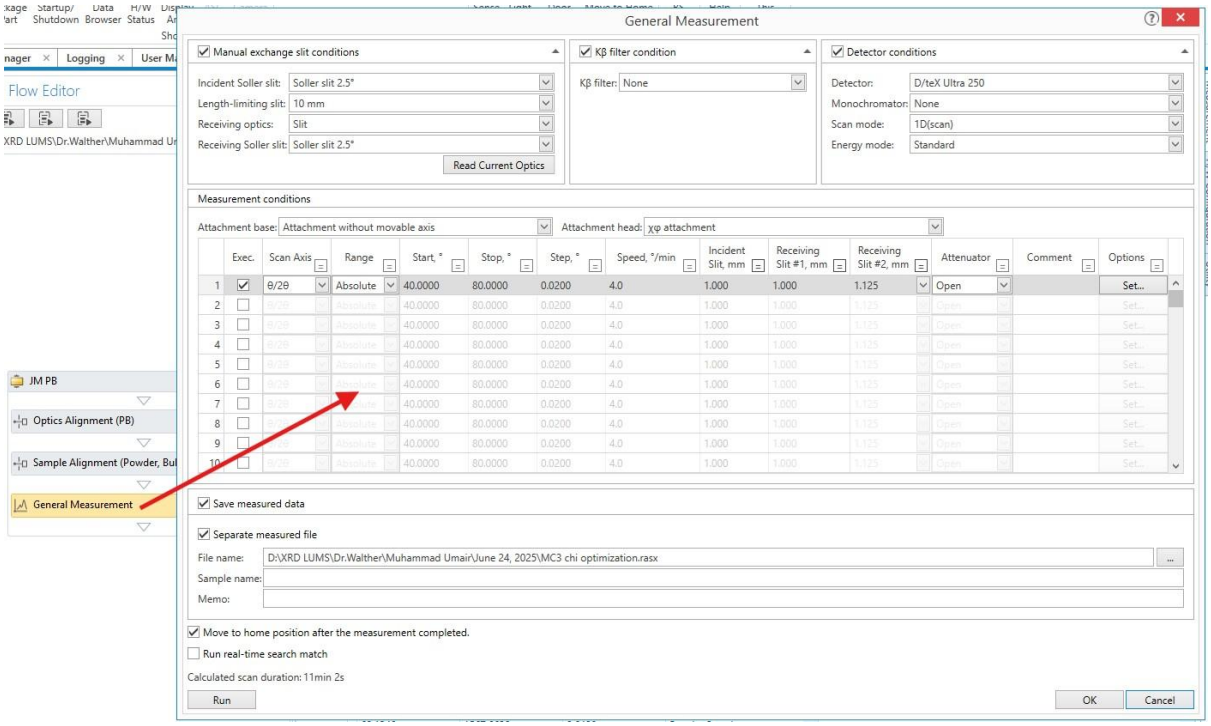
Optic Alignment Settings in PB Mode



Sample Alignment with χ–ϕ attachment



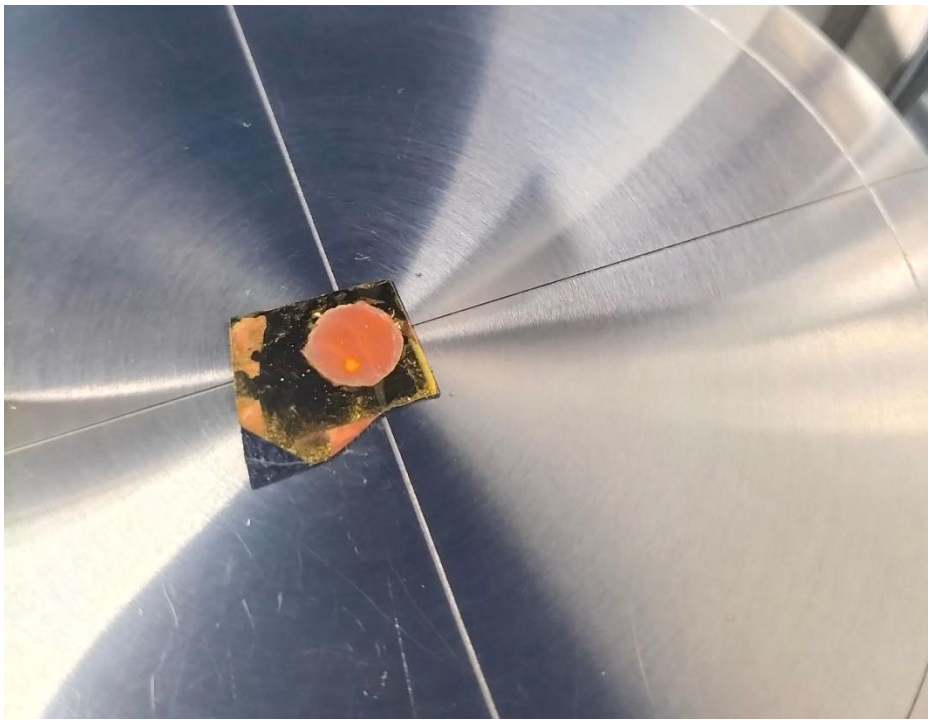
Measurement Settings for θ/2θ scan



XRD θ/2θ-Scan

Sample Name: B6

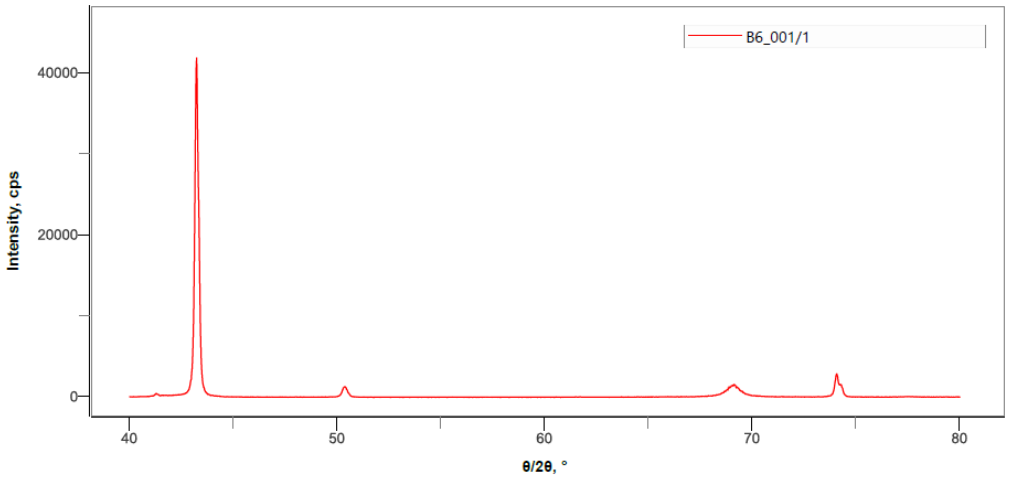
Researcher: Umair (MS- Physics, LUMS)



XRD Measurement Version 4.0

Profile / B6_001/1

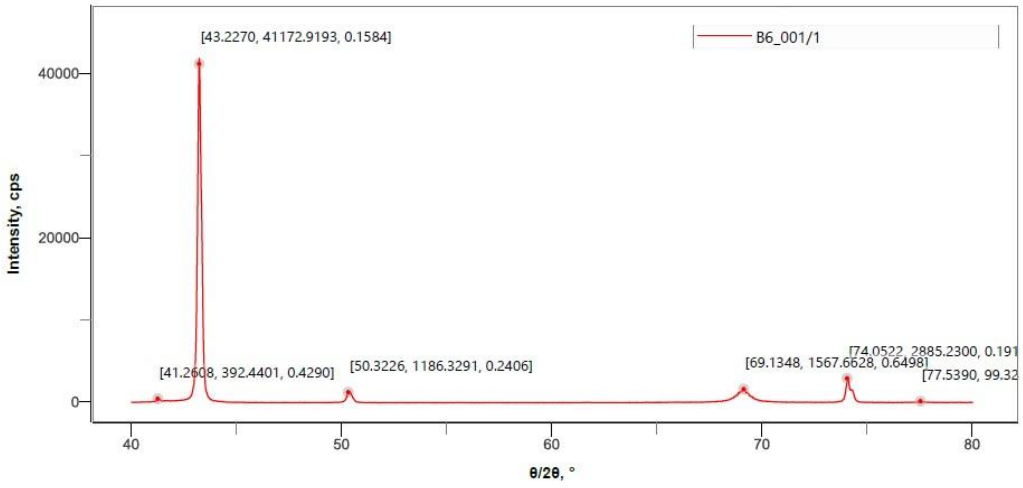
Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-06-26 15:25:06	Scan range	: 40.0000 - 80.0000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.0200 °	Incident slit box	: 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



XRD Measurement Version 4.0

Profile / B6_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-06-26 15:25:06	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



This scan shows the initial diffraction profile of the sample, highlighting both the dominant substrate peak Si(400) and thin film reflections. The intense substrate peak often masks the weaker thin film peaks, necessitating further analysis using a χ-scan

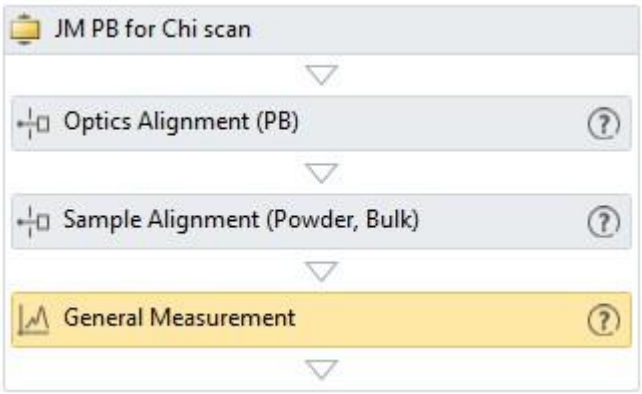
θ/2θ Position	Intensity	FWHM	Peaks
43.227	41172.9193	0.1584	Cu (111)
74.0522	2885.23	0.1916	Cu (220)
69.1348	1567.6628	0.6498	Si (400)
50.3226	1186.3291	0.2406	Cu (200)

Chi χ-Scan (Second Step)

To suppress the dominant Si(400) peak from the single crystal substrate, a χ (Chi) scan is performed. This scan is carried out over a range from –4.9° to 6°, while keeping the 2θ angle fixed at 69.1348°, which corresponds to the Bragg angle of the Si(400) reflection. A separate measurement flow is created specifically for the χ-scan, allowing selective suppression of the substrate peak. It is important to note that during this step, the previously established optic alignment and sample alignment remain unchanged, ensuring consistency and reliability in the scan results.

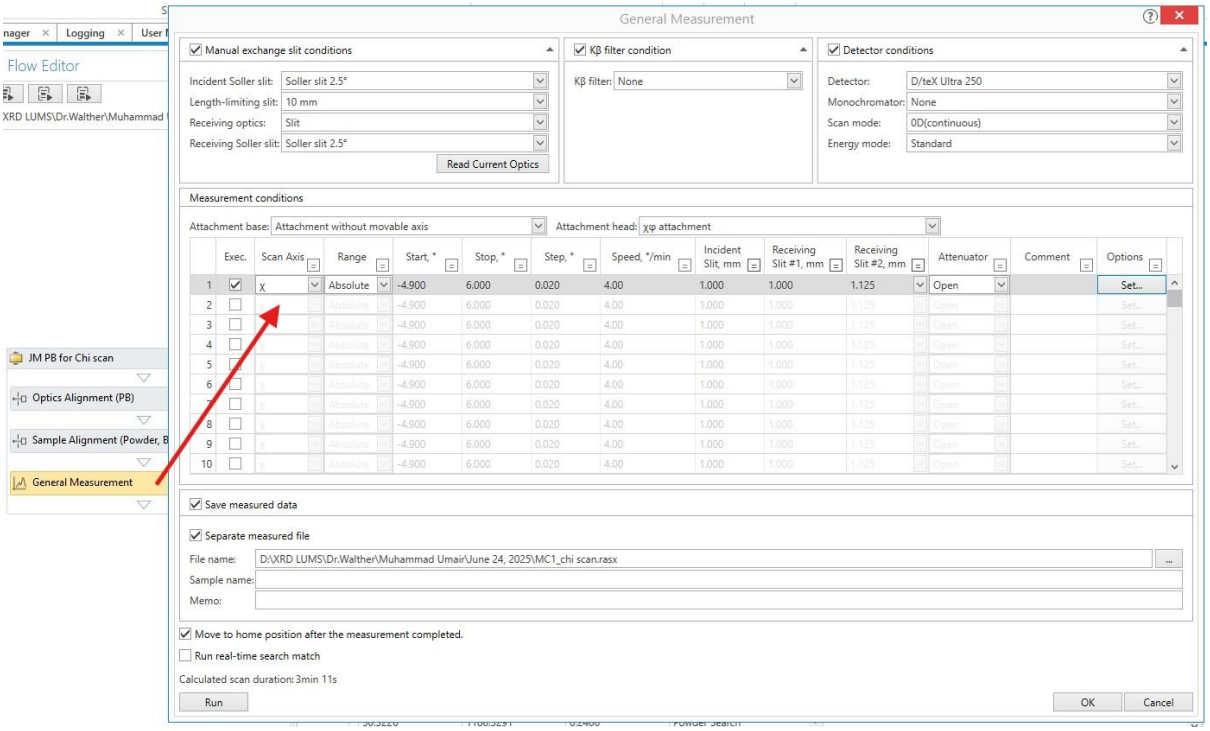
New Flow for χ (Chi) Scan

A dedicated measurement flow was created for the χ-scan, keeping all previous optic and sample alignment settings unchanged to maintain data integrity.

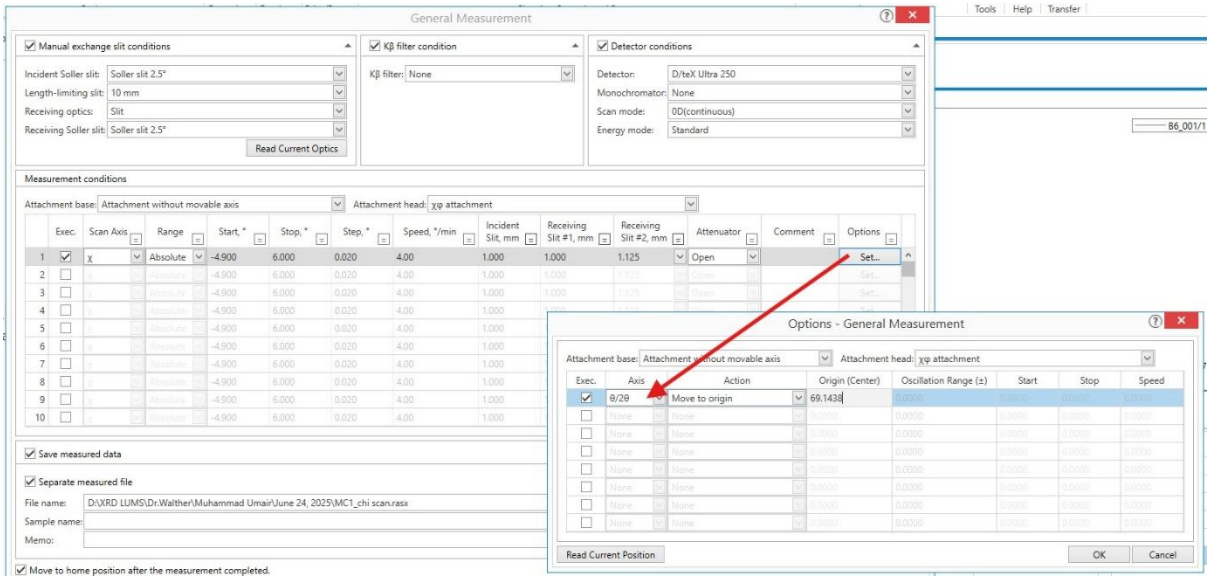


χ (Chi) Scan Settings

χ (Chi) scan settings over a range from –4.9° to 6°.



Keeping the 2θ angle fixed at 69.1348°, which corresponds to the Bragg angle of the Si(400) reflection.



Chi Scan Measurements

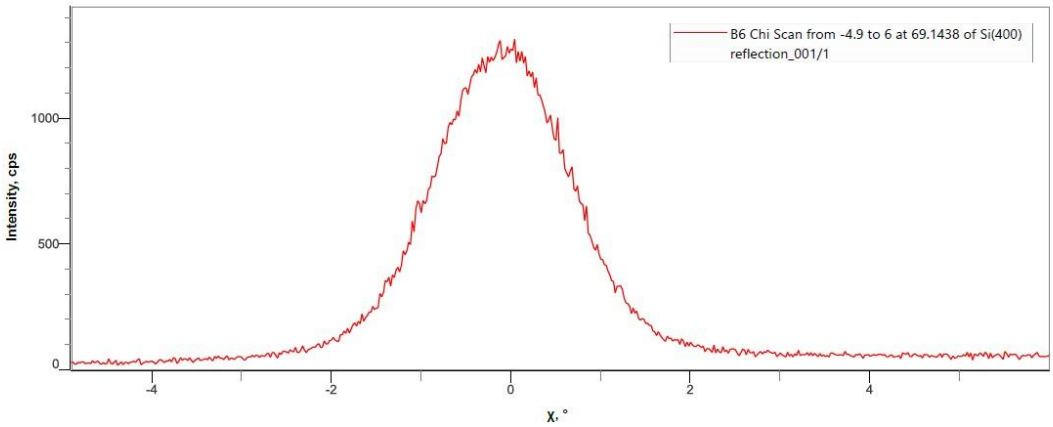
Sample: B6

Range: from -4.9 to 6 (at 69.1438 of Si(400) reflection)

XRD Measurement Version 4.0 2025-07-01 12:03:39

Profile / B6 Chi Scan from -4.9 to 6 at 69.1438 of Si(400) reflection_001/1

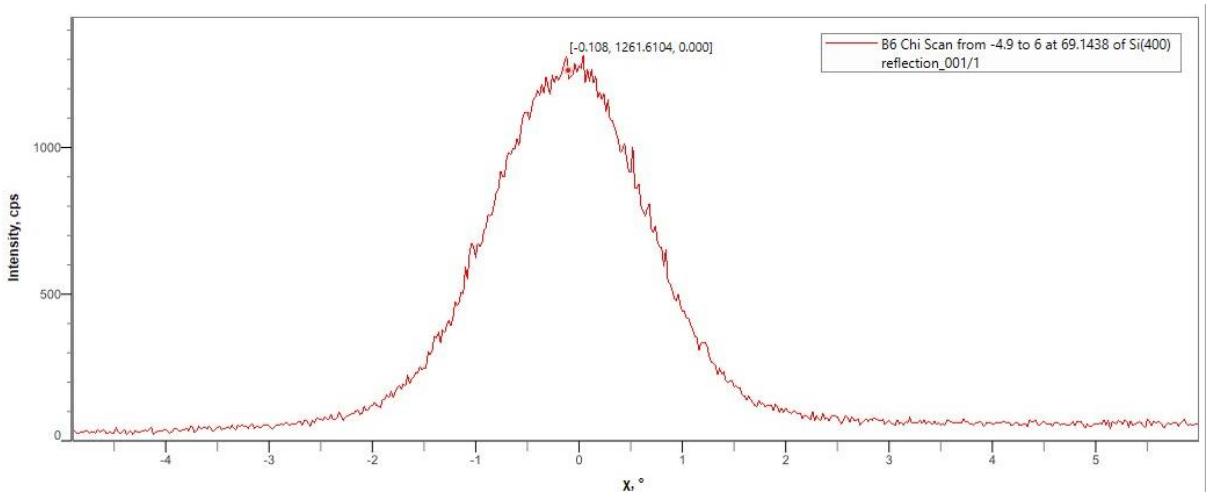
Sample name	:	Scan axis	: χ	Selection slit	: PB
Comment	:	Scan mode	: 0D(continuous)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 11:50:13	Scan range	: -4.900 - 6.000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.020 °	Incident slit box	: 1.000mm
X-ray	: 40 kV, 45 mA	Scan speed	: 1.000 °/min	Length-limiting slit	: 10 mm
Wavelength	: Cu-Kα / 1.54186 Å	χ	: -0.478 °	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



This scan demonstrates how the substrate peak varies with sample tilt. The Bragg condition for the single crystal substrate is met at χ = –0.1°, producing a strong reflection. By tilting the sample the Bragg condition is disrupted, reducing the intensity of the substrate peak at χ = ±3°, and revealing the underlying thin film signal.

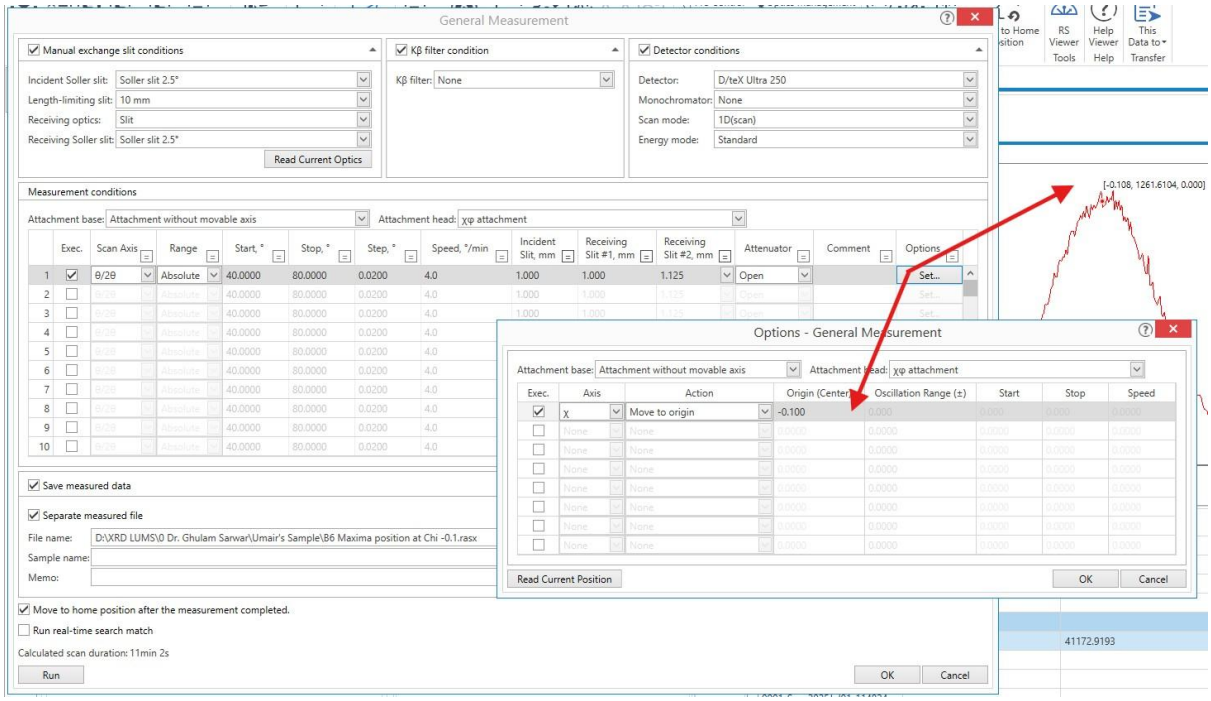
Maxima and Minima Positions for θ/2θ

The χ-scan shows a peak (maximum intensity) at χ = –0.1°, corresponding to strong Si(400) substrate reflection. Suppression of this peak is observed around χ = ±3°, where the substrate signal diminishes significantly.



θ/2θ Scan at Maxima position

Maxima position at Chi -0.1

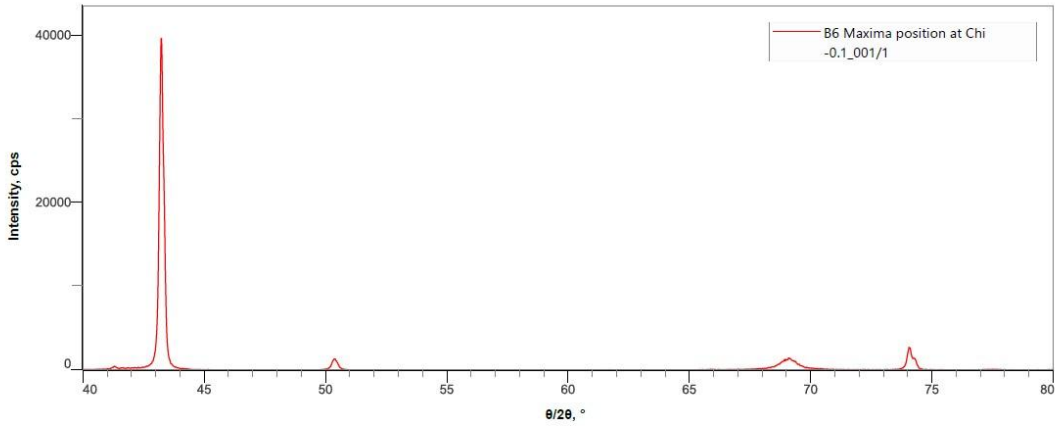


B6 Maxima position at Chi -0.1.

XRD Measurement Version 4.0 2025-07-01 13:22:24

Profile / B6 Maxima position at Chi -0.1_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 12:28:53	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	: -0.1 °	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



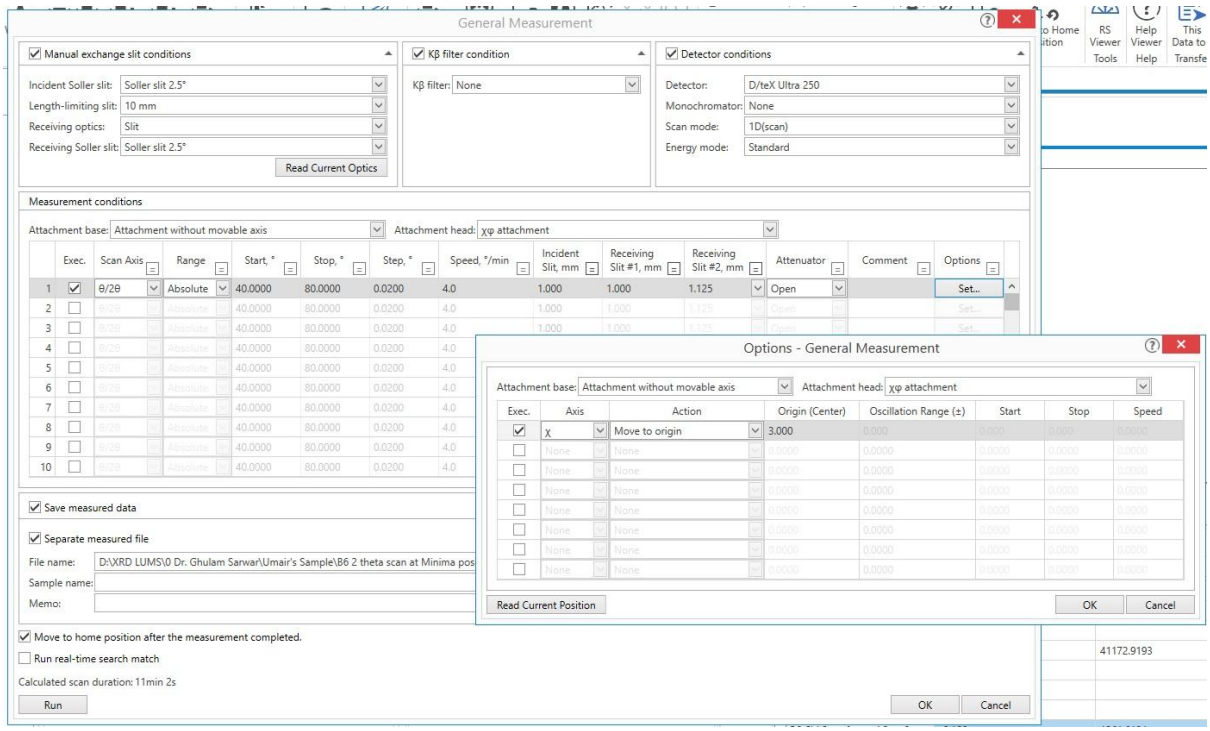
At this tilt angle -0.1° the Si(400) substrate peak is at maximum intensity, which may be a dominating the diffraction pattern and making it difficult to observe any thin film contributions.

θ/2θ Scan at Minima positions

Sample: B6

At this minima position Si(400) reflection will be removed.

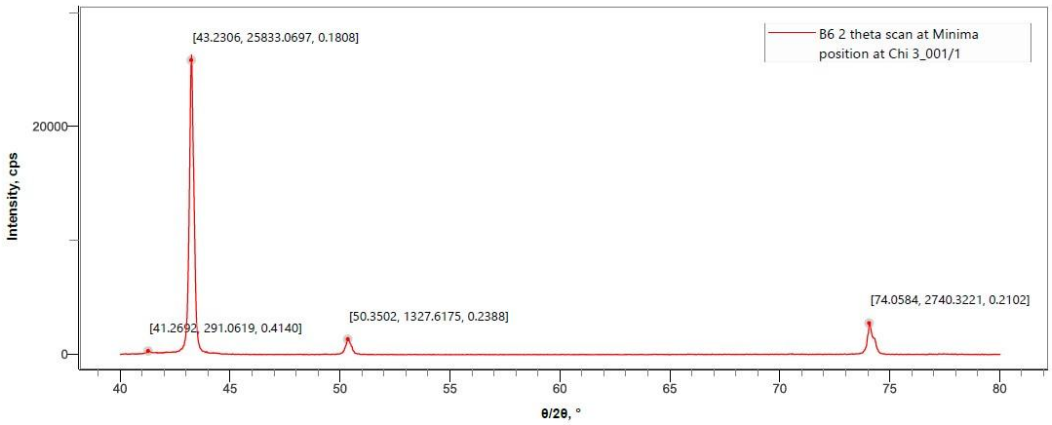
Minima position of χ (Chi) = 3



XRD Measurement Version 4.0 2025-07-01 13:47:55

Profile / B6 2 theta scan at Minima position at Chi 3_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 13:36:53	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	: 3 °	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

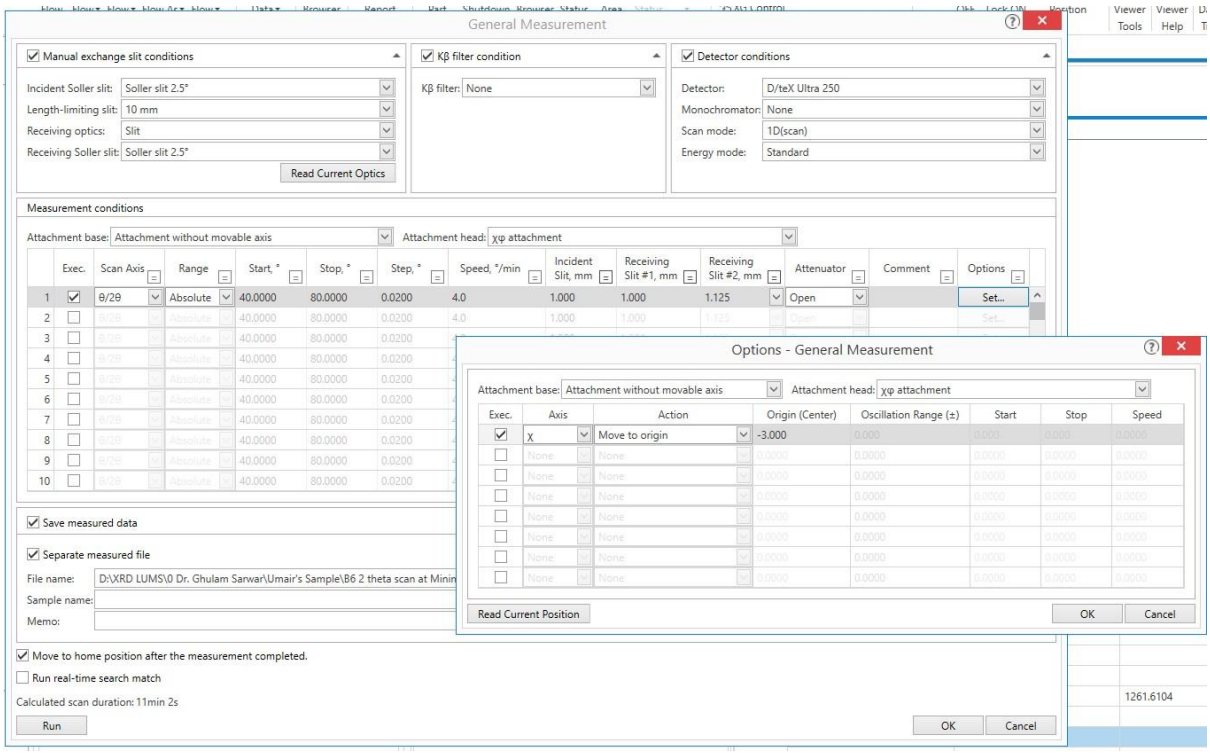


Tilting the sample to χ = +3° significantly reduces the intensity of the Si(400) substrate peak. This suppression enables better visibility of the thin film peaks that may be previously obscured.

Sample: B6

At this minima position Si(400) reflection will be removed.

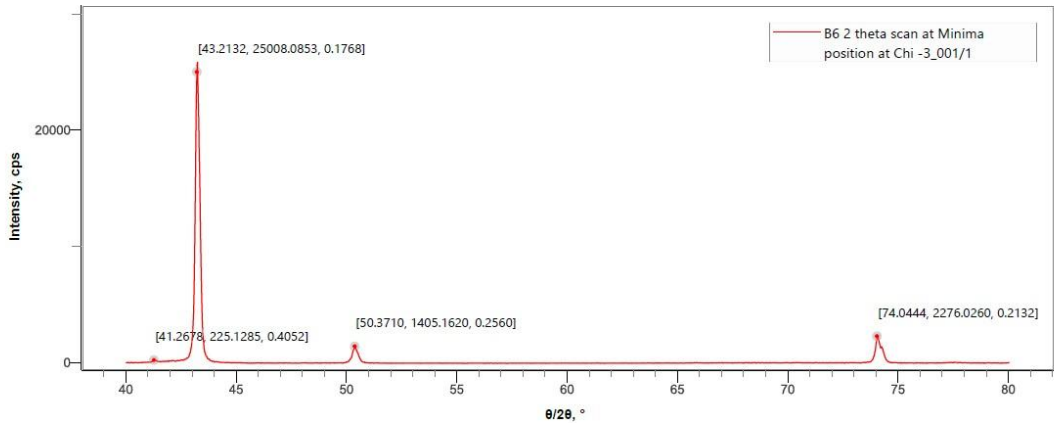
Minima position of χ (Chi) = -3



XRD Measurement Version 4.0 2025-07-01 14:06:52

Profile / B6 2 theta scan at Minima position at Chi -3_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 13:55:56	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	: -3 °	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



Similar to the +3° case, this scan at χ = –3° demonstrates substrate peak suppression. This confirms that substrate suppression is symmetric about the peak and can be achieved by tilting in either direction.

Phi Φ-Scan (Third Step)

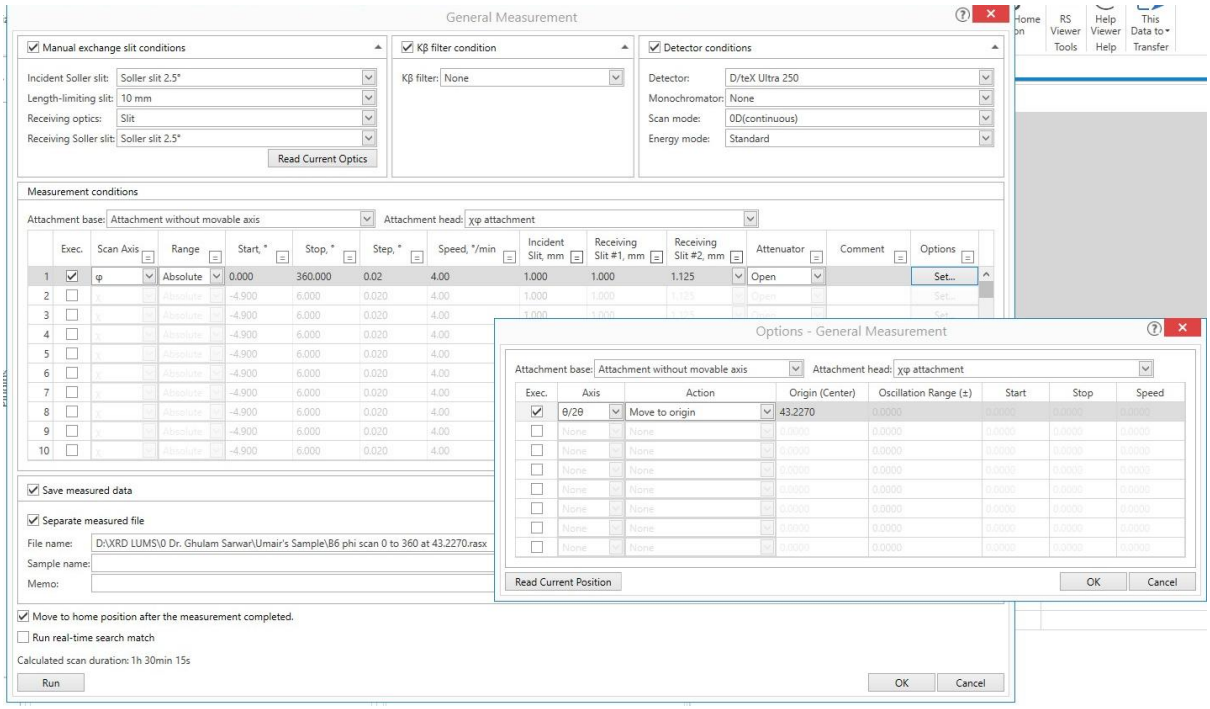
A phi (Φ) scan is typically used to investigate the in-plane crystallographic orientation of a film, particularly when studying epitaxial or textured thin films grown on single-crystal substrates. In this scan, the sample is rotated azimuthally around its surface normal, while keeping the incident angle (θ) and detector angle (2θ) fixed at a specific Bragg reflection. This allows detection of symmetry-related peaks that reveal in-plane rotational alignment between the film and the substrate. Phi scans are especially useful in determining epitaxial relationships, identifying twinning, and assessing in-plane texture or mosaicity in crystalline films.

Φ (phi) Settings

Φ scan: 0° to 360° at 2θ = 43.2270° for Cu(111) thin-film reflection

This Φ scan was performed to observe the azimuthal intensity variation of the **Cu(111)** thin-film reflection. The objective was to check whether the Cu film shows any preferred in-plane rotational alignment, texture, or possible epitaxial relationship with the substrate.

sample: B6

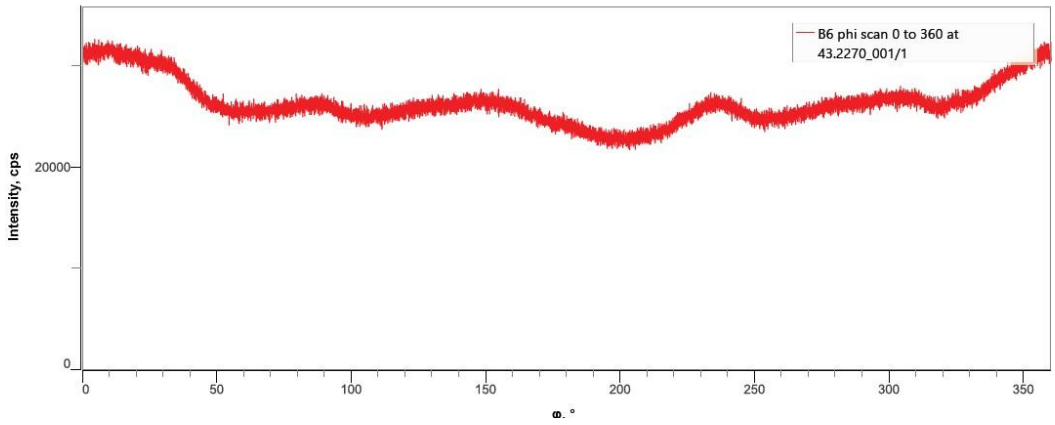


Φ (phi) Measurements

XRD Measurement Version 4.0 2025-07-01 15:45:21

Profile / B6 phi scan 0 to 360 at 43.2270_001/1

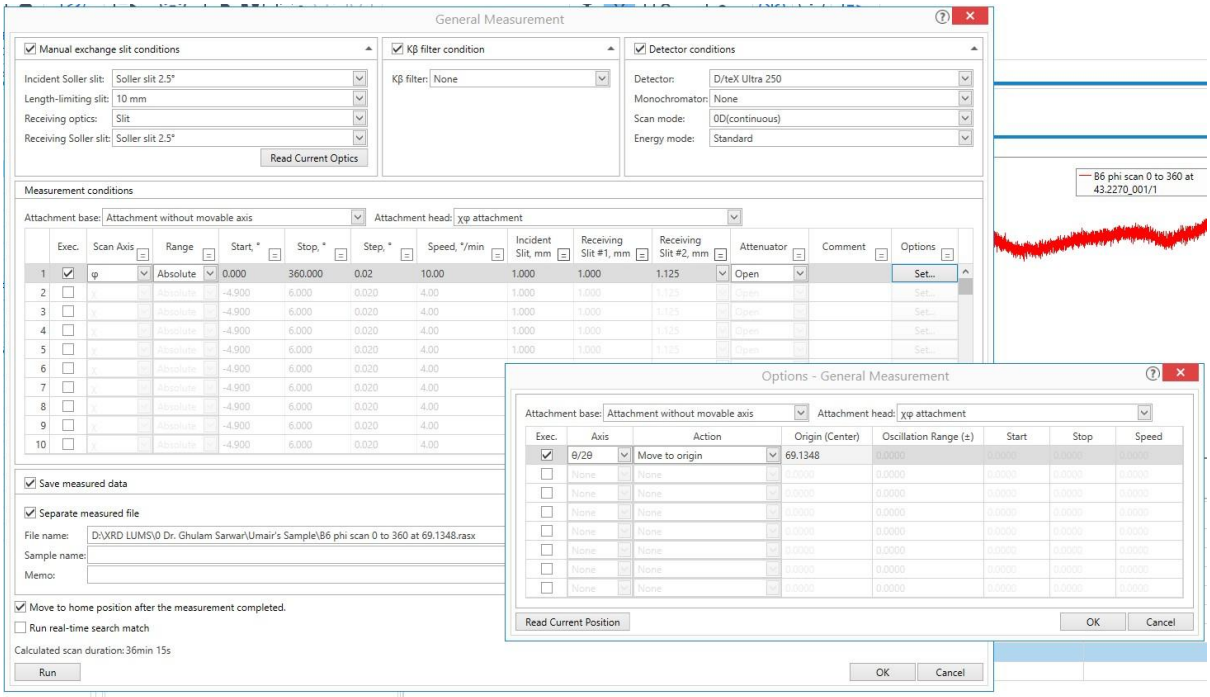
Sample name	:	Scan axis	: φ	Selection slit	: PB
Comment	:	Scan mode	: 0D(continuous)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 14:19:30	Scan range	: 0.000 - 360.000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.020 °	Incident slit box	: 1.000mm
X-ray	: 40 kV, 45 mA	Scan speed	: 10.000 °/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



The Φ scan from 0° to 360° was performed at 2θ = 43.2270°, corresponding to the Cu(111) thin-film reflection, to examine the azimuthal intensity variation of the film. The graph shows a broad and nearly continuous intensity variation without sharp periodic peaks, indicating that the Cu film does not show clear in-plane rotational symmetry or well-defined epitaxial alignment under this measurement condition. This suggests that the Cu(111) film peak is mainly related to out-of-plane texture, while the in-plane orientation may be weak, random, or not clearly resolved using this reflection.

Φ scan: 0° to 360° at 2θ = 69.1348° for Si(400) substrate reflection

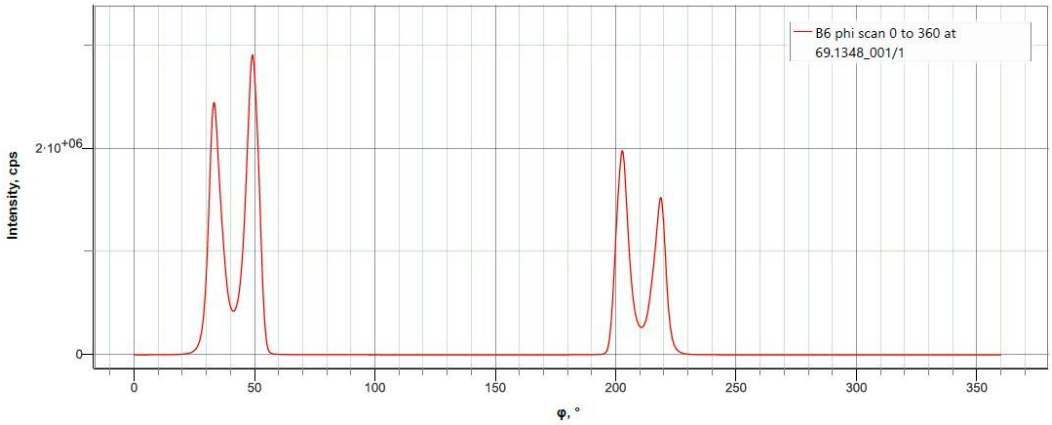
This Φ scan was performed to study the azimuthal response of the **Si(400)** single-crystal substrate reflection. The objective was to identify Φ positions where the substrate peak becomes strong or weak, which helps in selecting suitable angles to reduce substrate contribution during thin-film measurements.



XRD Measurement Version 4.0 2025-07-01 16:32:20

Profile / B6 phi scan 0 to 360 at 69.1348_001/1

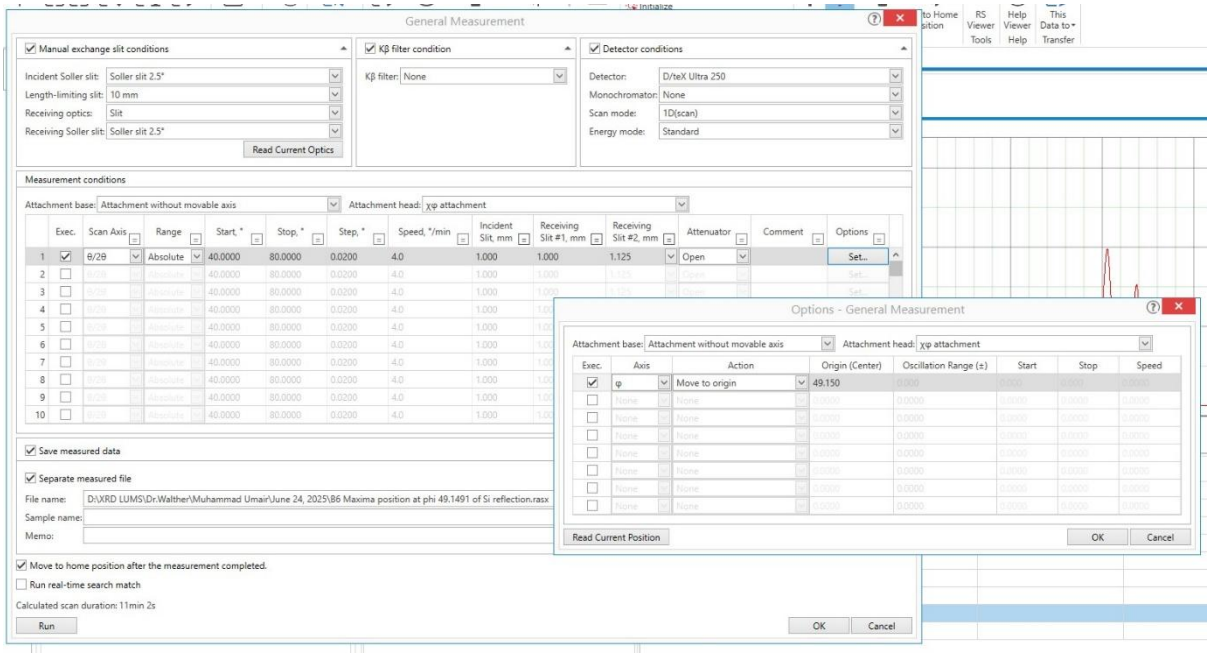
Sample name	:	Scan axis	: φ	Selection slit	: PB
Comment	:	Scan mode	: 0D(continuous)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 15:54:35	Scan range	: 0.000 - 360.000 °	Incident Soller slit	: Soller slit 2.5°
Operator name	: Administrator	Scan step	: 0.020 °	Incident slit box	: 1.000mm
X-ray	: 40 kV, 45 mA	Scan speed	: 10.000 °/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



The Φ scan from **0° to 360°** was performed at **2θ = 69.1348°**, corresponding to the **Si(400)** single-crystal substrate reflection, to examine the azimuthal response of the substrate under the selected tilted condition. The graph shows very intense and sharp substrate peaks at specific Φ positions because, at these angles, the Si(400) planes are correctly aligned with the X-ray beam and detector, so Bragg's condition is satisfied. At other Φ angles, the planes rotate away from the diffraction condition, causing the intensity to become very low or nearly zero. This confirms the highly directional

diffraction behavior of the single-crystal Si substrate, and the low-intensity Φ regions can be selected to reduce substrate contribution during thin-film measurements.

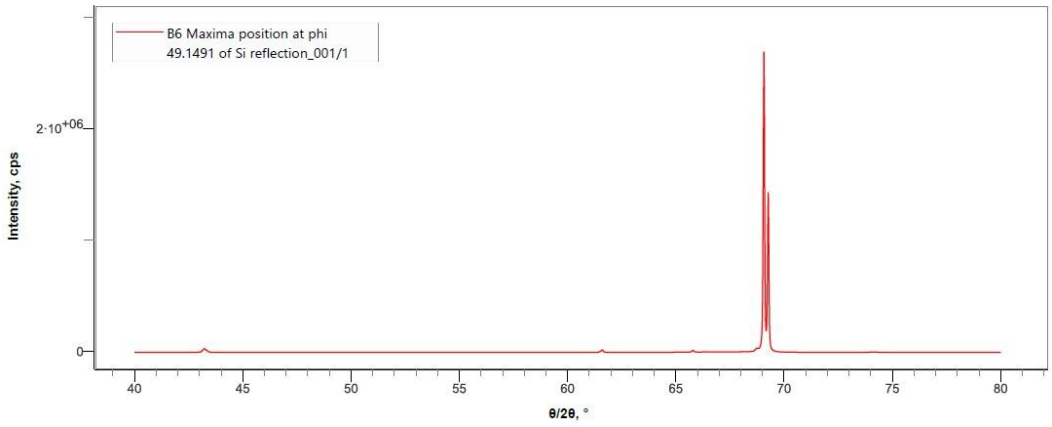
Set the θ/2θ scan at Φ = 49.1491°, where the Si reflection shows maximum intensity



XRD Measurement Version 4.0 2025-07-01 17:05:54

Profile / B6 Maxima position at phi 49.1491 of Si reflection_001/1

Sample name	:	Scan axis	: θ/2θ	Selection slit	: PB
Comment	:	Scan mode	: 1D(scan)	Incident optics unit	: IPS adaptor
Date of measurement	: 2025-07-01 16:40:50	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



A θ/2θ scan was performed at the Φ position of maximum Si substrate intensity, Φ = 49.1491°, as identified from the previous Φ scan of the Si(400) reflection. In this setting, the sample was fixed at the selected Φ angle and scanned over 2θ = 40°–80° to observe the diffraction response when the substrate is strongly aligned with the X-ray beam. The

measurement shows a very intense and sharp peak near 2θ ≈ 69.13°, confirming that this Φ position corresponds to the maximum diffraction condition of the Si(400) single-crystal substrate reflection.

Description on ϕ-scan

Φ scans were performed from 0° to 360° at two fixed diffraction positions: 2θ = 43.2270° for the Cu(111) thin-film reflection and 2θ = 69.1348° for the Si(400) substrate reflection. The Cu(111) ϕ-scan was used to examine the azimuthal response of the thin film and to check for any preferred in-plane rotational alignment or texture. The Si(400) ϕ-scan was used to identify the ϕ positions where the single-crystal substrate reflection becomes maximum or minimum. Sharp maxima indicate that the substrate planes satisfy Bragg's condition at specific ϕ angles, while low-intensity regions indicate substrate suppression and can be selected to improve the visibility of weak thin-film peaks.

Conclusion

The combined χ-scan and ϕ-scan procedure using the χ–ϕ attachment on the Rigaku SmartLab SE is an effective approach for reducing strong diffraction signals from a single-crystal substrate and improving the visibility of weak thin-film peaks. The θ/2θ scan first identifies the film and substrate reflections, while the χ scan helps find tilt positions where the substrate no longer satisfies the Bragg condition and its peak is strongly suppressed. The ϕ scan further identifies azimuthal positions where the substrate intensity is maximum or minimum, allowing suitable measurement positions to be selected. In this way, dominant substrate peaks such as Si(400) can be minimized, making it easier to detect and analyze thin-film reflections such as Cu(111). This method is especially useful for thin films grown on single-crystal substrates, and the use of a Ge(220) ×2 monochromator can further improve peak resolution and measurement quality. Overall, χ–ϕ scanning provides a practical and reliable method for substrate peak suppression and thin-film structural characterization.