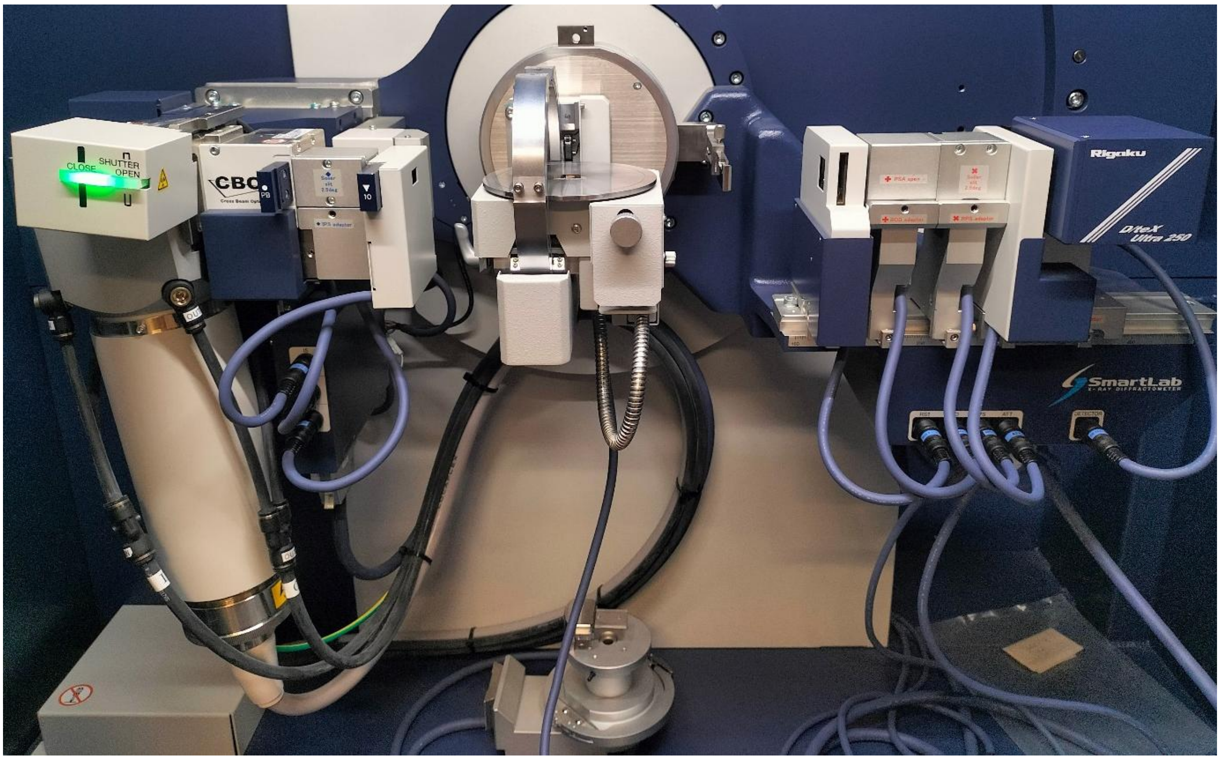
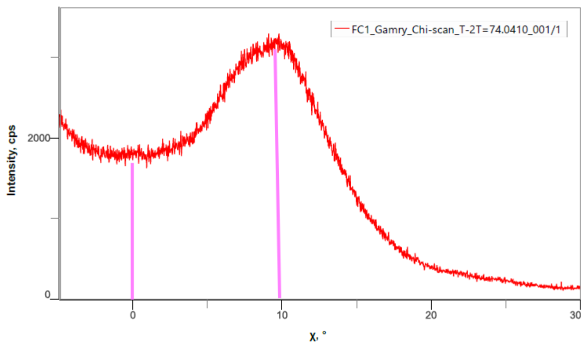
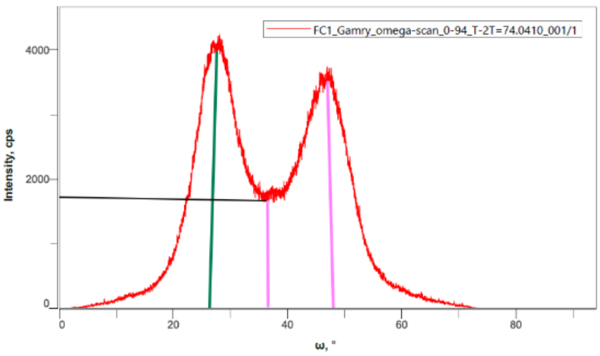
Operational Manual: Overcoming χ Tilt Limitations Using ω Scanning at Fixed 2θ with χ–ϕ Attachment and Optional Ge(220) ×2 Monochromator on the SmartLab SE

Version: ω-Scan -2026-I

Other XRD Operational Manuals







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Purpose (Why this method is needed)

In thin-film XRD, orientation-dependent measurements are often performed using χ (tilt) and ϕ (rotation) scans at a fixed Bragg reflection. However, χ scanning in the SmartLab SE is limited by instrument geometry, which can lead to incomplete intensity profiles. This method provides a practical solution by using a full-range ω scan at the same fixed Bragg condition to capture the complete intensity variation reliably.

Introduction

X-ray diffraction (XRD) is widely used to investigate the crystallographic orientation and structural properties of thin films. In orientation-dependent analysis, measurements such as χ (tilt) and ϕ (rotation) scans at a fixed Bragg reflection are commonly used to study texture, anisotropy, and to distinguish thin-film signals from substrate contributions

For such measurements, Parallel Beam (PB) mode is preferred, as it provides correct diffraction geometry and reduces sensitivity to sample height errors, making it more suitable for thin films and orientation-dependent studies.

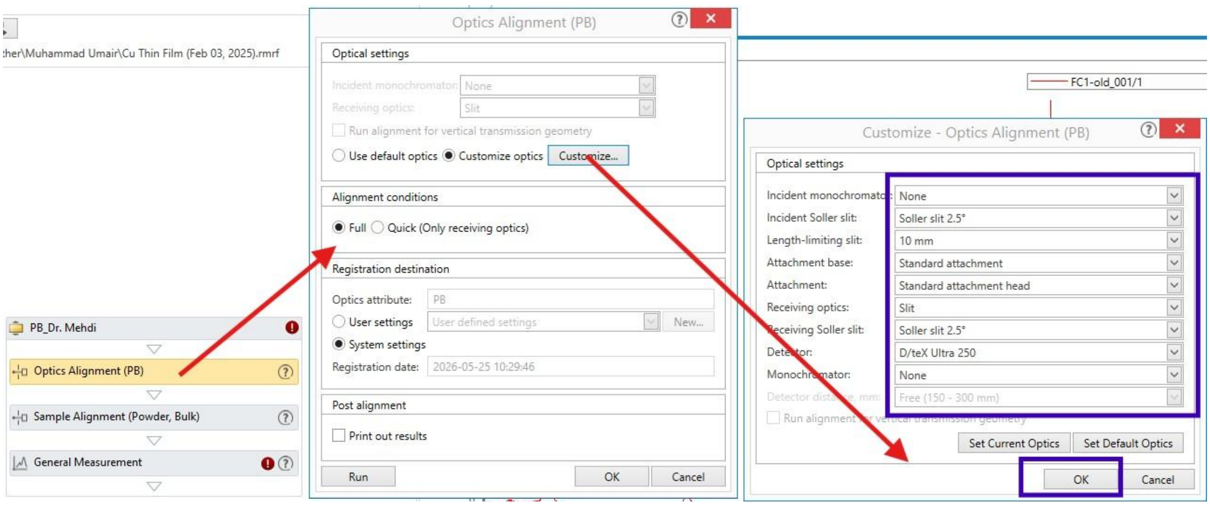
However, in the SmartLab SE system, the accessible χ range is restricted by instrument geometry (typically limited to about −4.9° on the lower side), which prevents the observation of diffraction features lying outside this range. This limitation becomes critical when analyzing intensity variations with tilt, as important features of the diffraction profile may be partially or completely missed.

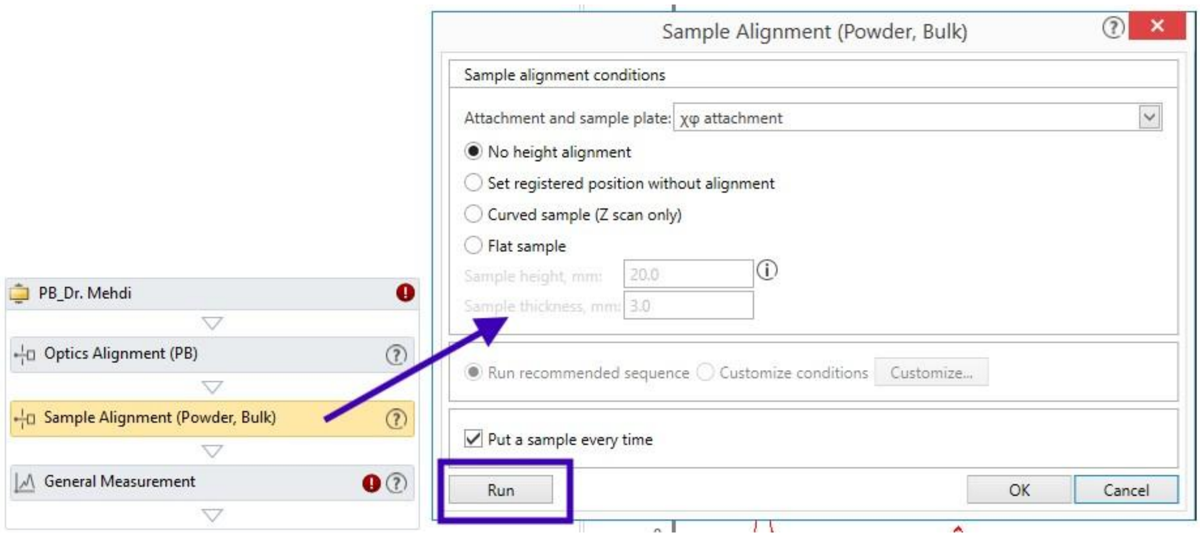
To overcome this limitation, a full-range ω scan can be performed at the same fixed Bragg condition. This approach allows the complete intensity variation to be measured, providing a practical and reliable alternative to χ scanning when the accessible χ range is insufficient for accurate analysis.

Optic and Sample Alignment

Before starting the fixed-2θ χ/ϕ/ω measurements, perform optic alignment in PB mode and then sample alignment. Optic alignment ensures that the incident and receiving optics, slits, detector, and optional monochromator are correctly positioned so the X-ray beam path is stable and well aligned; Rigaku notes that SmartLab SE optics are self-aligning under computer control and support automatic alignment and component recognition.

Sample alignment places the sample surface at the correct measurement position relative to the incident beam and detector, reducing errors caused by sample height or surface-position mismatch. This step is especially important for thin films and orientation-dependent scans, where PB mode is preferred because it provides reliable geometry and reduced sensitivity to sample-height error.





Resolution and Intensity Control

In the SmartLab SE, the incident-side and receiving-side slit combinations, together with optional receiving-side components such as the Kβ filter and monochromator, are selected to balance diffraction intensity and angular resolution. Wider slit settings generally increase X-ray flux and improve peak intensity for weak or poorly crystalline samples, while narrower slits and monochromators improve resolution, peak separation, and background control for highly crystalline samples. Therefore, the optics should be tuned according to the sample quality and measurement objective, using higher-intensity settings for weak signals and higher-resolution settings when precise peak position, shape, or separation is required.

Angle definitions

Use these definitions to avoid confusion when setting scans:

- **θ**: Angle between incident X-ray beam and sample surface.
- **2θ**: Total angle between incident and diffracted beams.
- **ω**: Incident angle (often equals θ in GIXRD measurements).
- **χ**: Tilt angle of the sample relative to its surface normal.
- **ϕ**: Rotation of the sample about its surface normal.

Step 1: Initial θ–2θ Scan

After completing optic alignment in PB mode and sample alignment, an initial θ–2θ scan is performed to identify the diffraction peaks of the sample and select a suitable Bragg reflection for orientation-dependent measurements. In this example, the θ–2θ scan was recorded over 20°–80°, and a clear peak was observed near 2θ ≈ 74°, which was selected for further χ, ϕ, and ω scan studies.

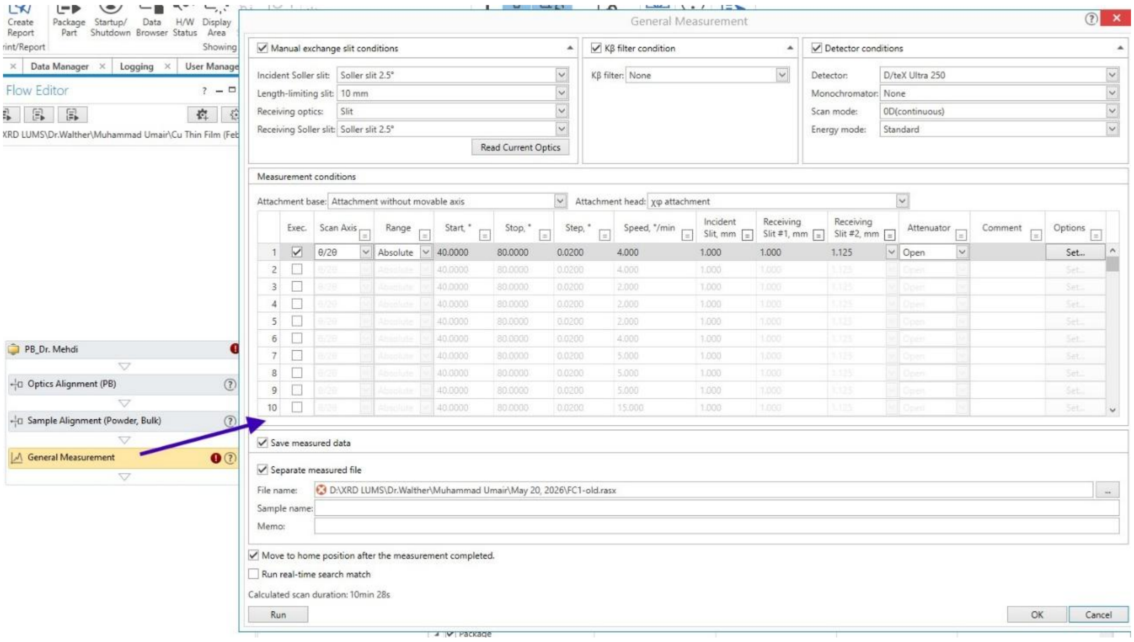


Figure 1: SmartLab SE measurement flow and θ–2θ scan settings in PB mode after optic and sample alignment. The setup is used to identify a suitable Bragg reflection for orientation-dependent ϕ, χ, and ω measurements.

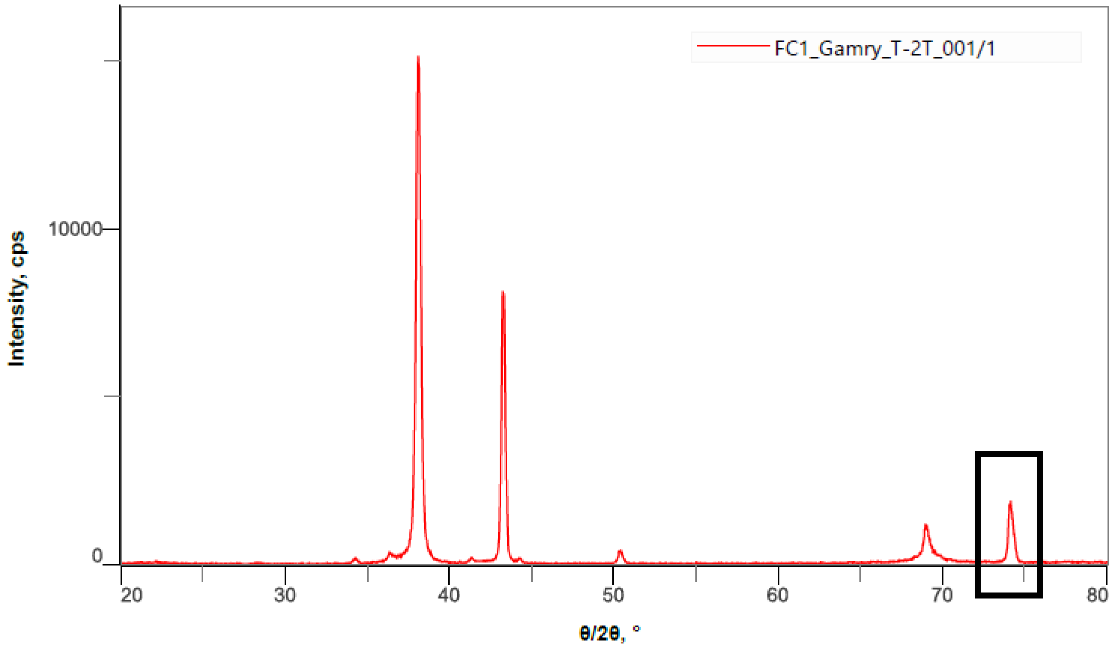


Figure 2: Initial θ–2θ scan recorded over 20°–80°, showing a selected Bragg reflection near 2θ ≈ 74°. This peak was used as the fixed Bragg position for subsequent ϕ, χ, and ω scans.

Step 2: ϕ Scan at Fixed Bragg Position

For the ϕ scan, the scan axis was set to ϕ and the sample was rotated from 0° to 360° while keeping the selected Bragg condition fixed at 2θ ≈ 74°. This configuration allows the intensity of the selected reflection to be monitored as a function of in-plane sample rotation, helping to identify any preferred azimuthal orientation or in-plane texture before proceeding to χ and ω scan measurements.

A ϕ scan was then performed at this fixed Bragg position to examine whether the diffraction intensity changes with in-plane rotation of the sample. The ϕ-scan shows an almost uniform intensity distribution, with an average intensity of about 3200 cps, indicating that the selected reflection does not show strong in-plane anisotropy under this measurement condition. This confirms that the selected peak is suitable for further tilt-dependent analysis using χ and ω scans.

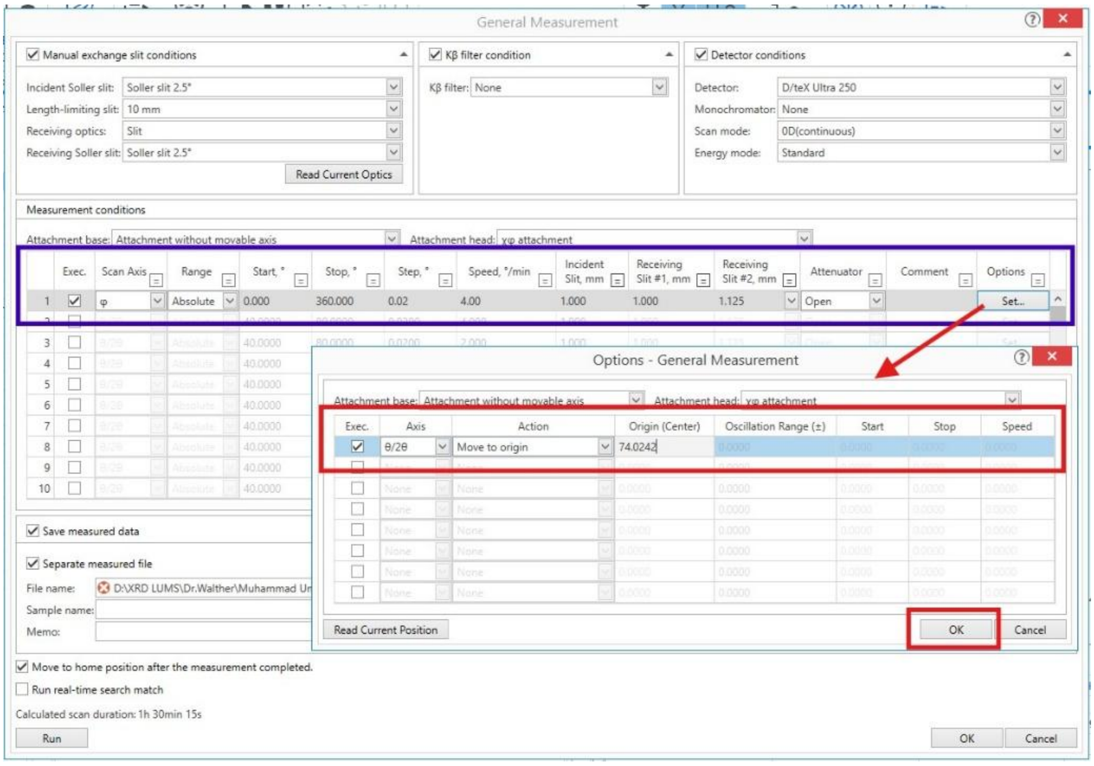


Figure X: SmartLab SE ϕ-scan settings at fixed 2θ ≈ 74°, with ϕ rotation from 0° to 360° for checking in-plane intensity variation of the selected Bragg reflection.

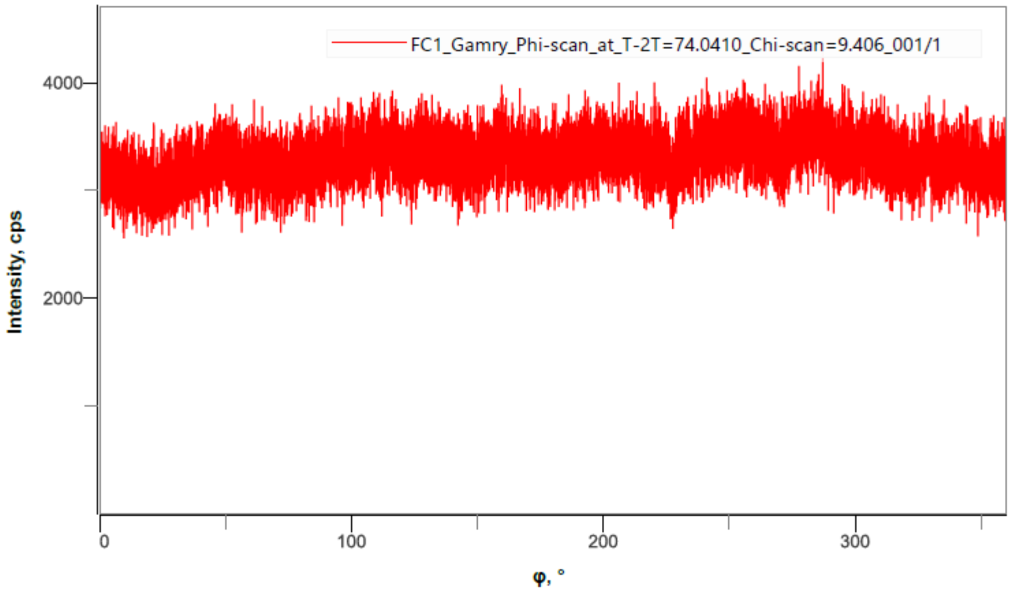


Figure 3: ϕ scan at the fixed Bragg position near 2θ ≈ 74°, showing nearly uniform intensity around 3200 cps. The weak variation with ϕ indicates no strong in-plane anisotropy under this measurement condition.

Step 3: χ Scan at Fixed Bragg Position

After confirming the ϕ-scan response, a χ scan was performed at the same fixed Bragg position near 2θ ≈ 74° to study how the diffraction intensity changes with sample tilt. The χ scan was carried out within the accessible range of the SmartLab SE, from approximately −4.9° to 30°. The intensity profile shows a minimum near χ ≈ 0° and increases toward higher χ values, reaching a strong maximum around χ ≈ 9–10°, followed by a gradual decrease at larger χ angles. However, the continuation of the intensity profile toward χ < −4.9° could not be recorded due to the χ-tilt limitation of the instrument. This incomplete profile indicates the need for an alternative scan method, such as a full-range ω scan at fixed 2θ, to capture the missing tilt-dependent intensity variation

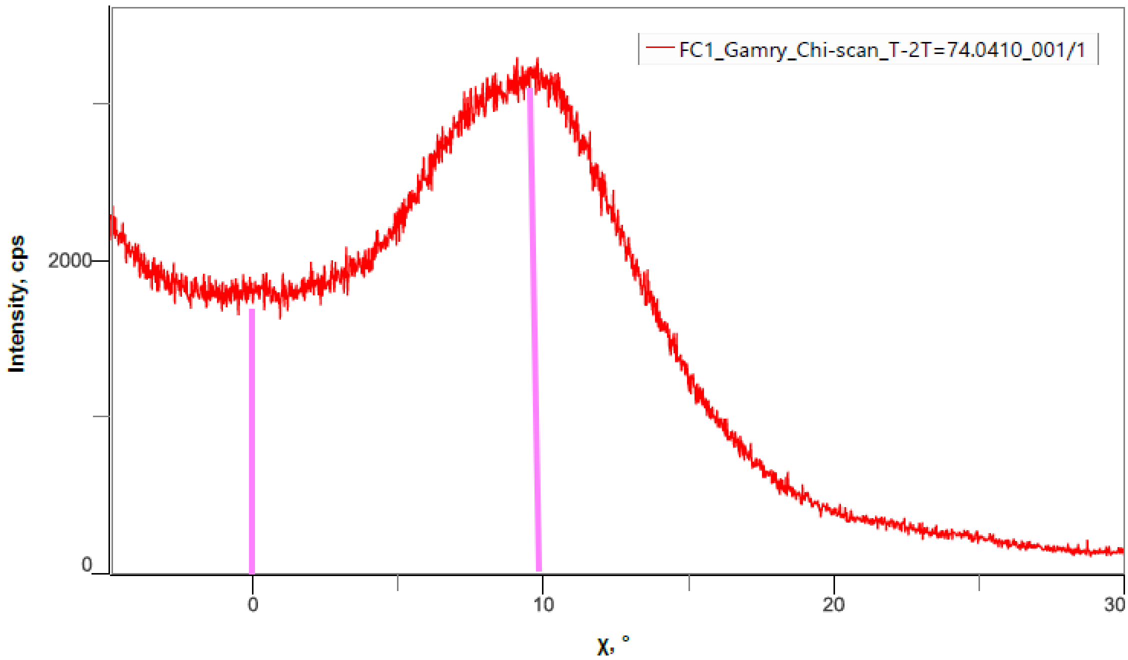


Figure X: χ scan at fixed **2θ ≈ 74°** over **χ = −4.9° to 30°**, showing the accessible tilt-dependent intensity profile before ω-scan correction.

Step 4: Full-Range ω Scan at Fixed Bragg Position

To overcome the limited χ range, a full-range ω scan was performed at the same fixed Bragg position near 2θ ≈ 74°. In this scan, the ω axis was scanned from 0° to 94°, while the detector position was kept fixed at the selected Bragg angle. This configuration allowed the missing tilt-dependent intensity variation, which could not be fully observed in the χ scan, to be captured more completely. The ω-scan profile revealed a clear double-peak structure, with a central minimum of about 1900 cps and a higher-intensity peak of about 3700 cps, consistent with the trend observed in the χ scan. This confirms that ω scanning at fixed 2θ is an effective practical method for extending the orientation-dependent analysis when χ scanning is restricted by instrument geometry.

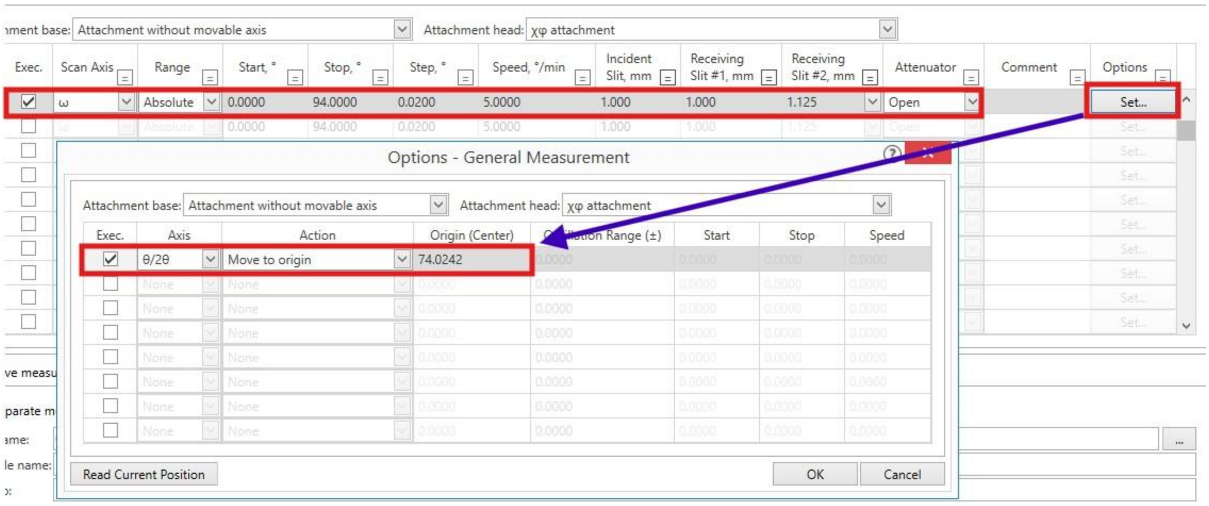


Figure X: SmartLab SE settings for the full-range **ω scan** at fixed **2θ ≈ 74°**, with ω scanned from **0° to 94°**.

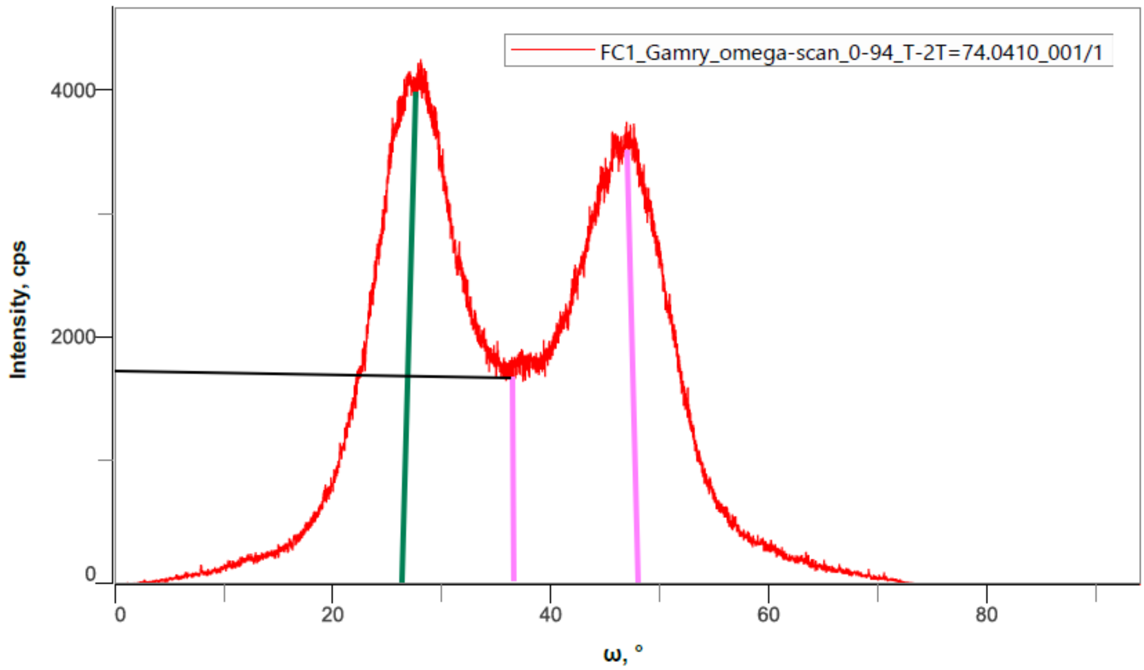


Figure X: Full-range **ω scan** at fixed **2θ ≈ 74°**, showing a complete double-peak intensity profile beyond the accessible χ range.

In this scan, the ω axis was scanned from **0° to 94°**, which was within the valid range displayed by the SmartLab SE software for the selected χ–ϕ attachment configuration, while the detector position was kept fixed at the selected Bragg angle.

Conclusion

The χ scan at the fixed Bragg position provided useful information about the tilt-dependent intensity variation of the selected reflection; however, the lower χ side could not be fully measured due to the angular limitation of the SmartLab SE geometry. To overcome this restriction, a full-range **ω scan at fixed 2θ** was performed under the same Bragg condition. This approach successfully revealed the complete double-peak intensity profile and confirmed the trend observed in the χ scan. Therefore, ω scanning at fixed 2θ is a practical and reliable alternative when χ scanning cannot capture the full orientation-dependent diffraction behavior. This method enables more complete analysis of thin-film texture and diffraction intensity variation while maintaining safe and controlled instrument operation.