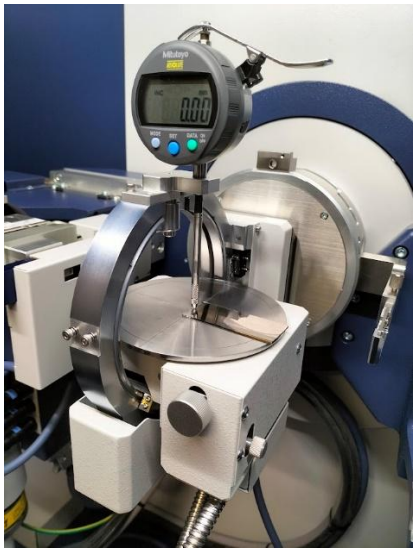
XRR Alignment, Measurements and Analysis with Rigaku SmartLab SE



Dr. Ghulam Sarwar Butt, Dr. Sabieh Anwar, Dr. Sumera Siddique and Hamid Mehmood

https://physlab.org/, Physical Properties Measurements Lab, CentralLab, Department of Physics, LUMS

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XRR Alignment and Measurements and Analysis with Rigaku SmartLab SE

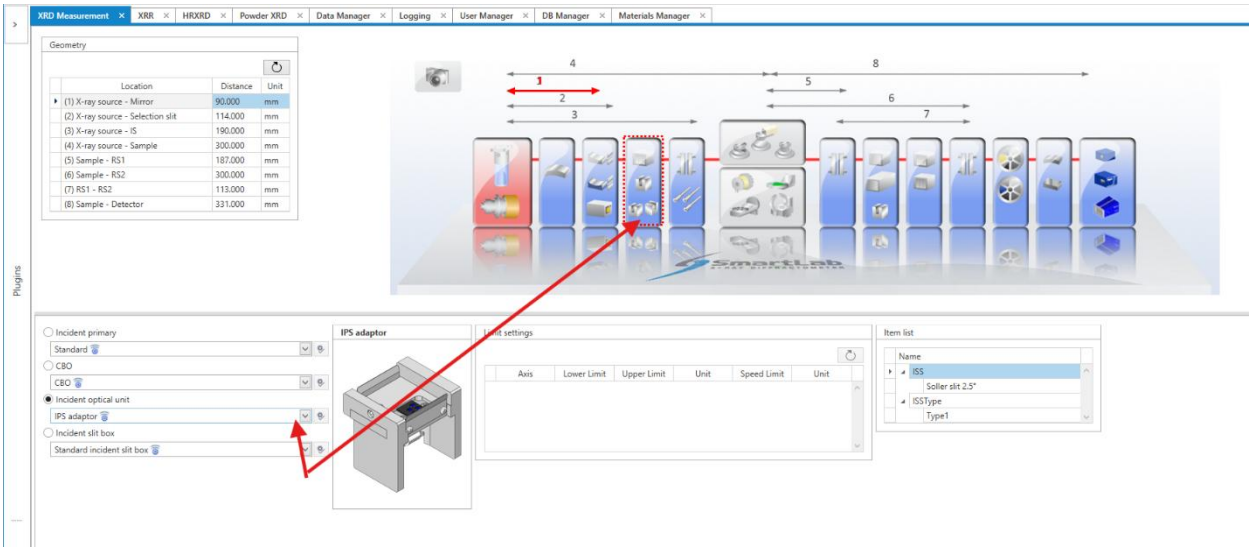
Introduction

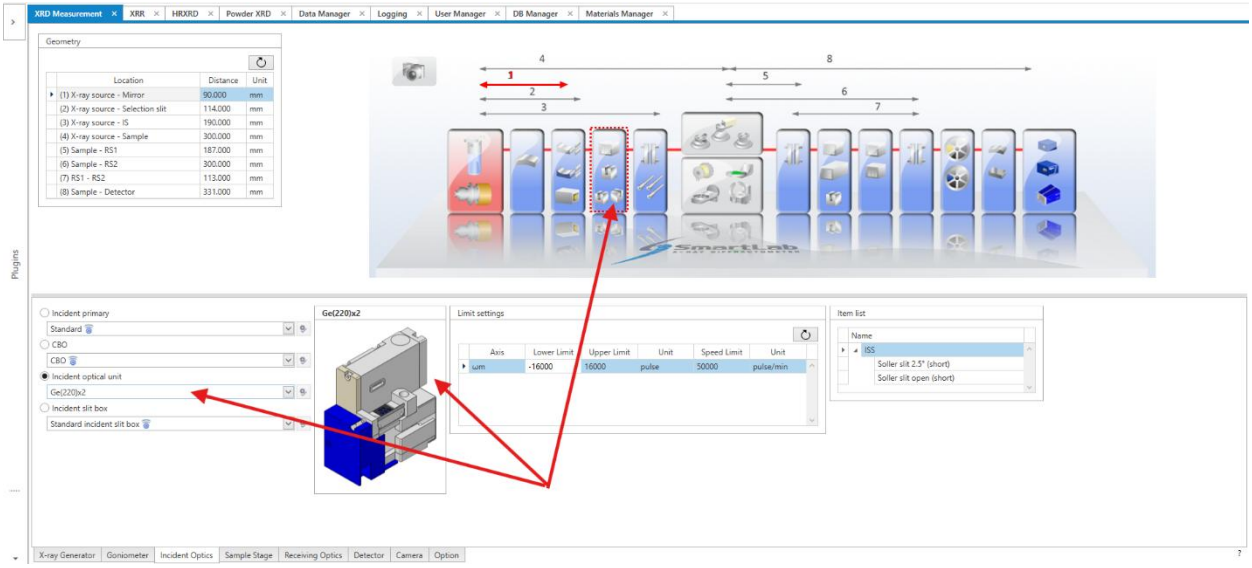
XRD primarily analyses crystal structures, phase identification, and crystallographic properties, XRR is an advanced feature in XRD used to characterize thin film thickness, density, and surface/interface roughness. This makes XRR a valuable complement to XRD for advanced material analysis, particularly for thin films, multilayers, and coatings. X-ray Reflectivity (XRR) measurement on the Rigaku SmartLab SE involves a few key steps: optic alignment, sample alignment, measurement, and data analysis. First, **optic alignment** ensures that the X-ray beam and detector are correctly configured to optimize reflectivity measurements. This includes setting up slits and Soller slits to control beam divergence. **Sample alignment** is critical for accurate XRR, involving adjustments to ensure the sample surface is perfectly parallel to the X-ray beam. The **measurement** process involves scanning over a low-angle 2θ range to capture reflected X-rays and generate a reflectivity curve that contains oscillations (Kiessig fringes) related to film thickness, density, and roughness. Finally, **data analysis** is performed using Rigaku's software, which fits the reflectivity curve to determine the sample's structural properties, such as film thickness, density, and surface/interface roughness, through model-based fitting techniques like Levenberg-Marquardt or genetic algorithms.

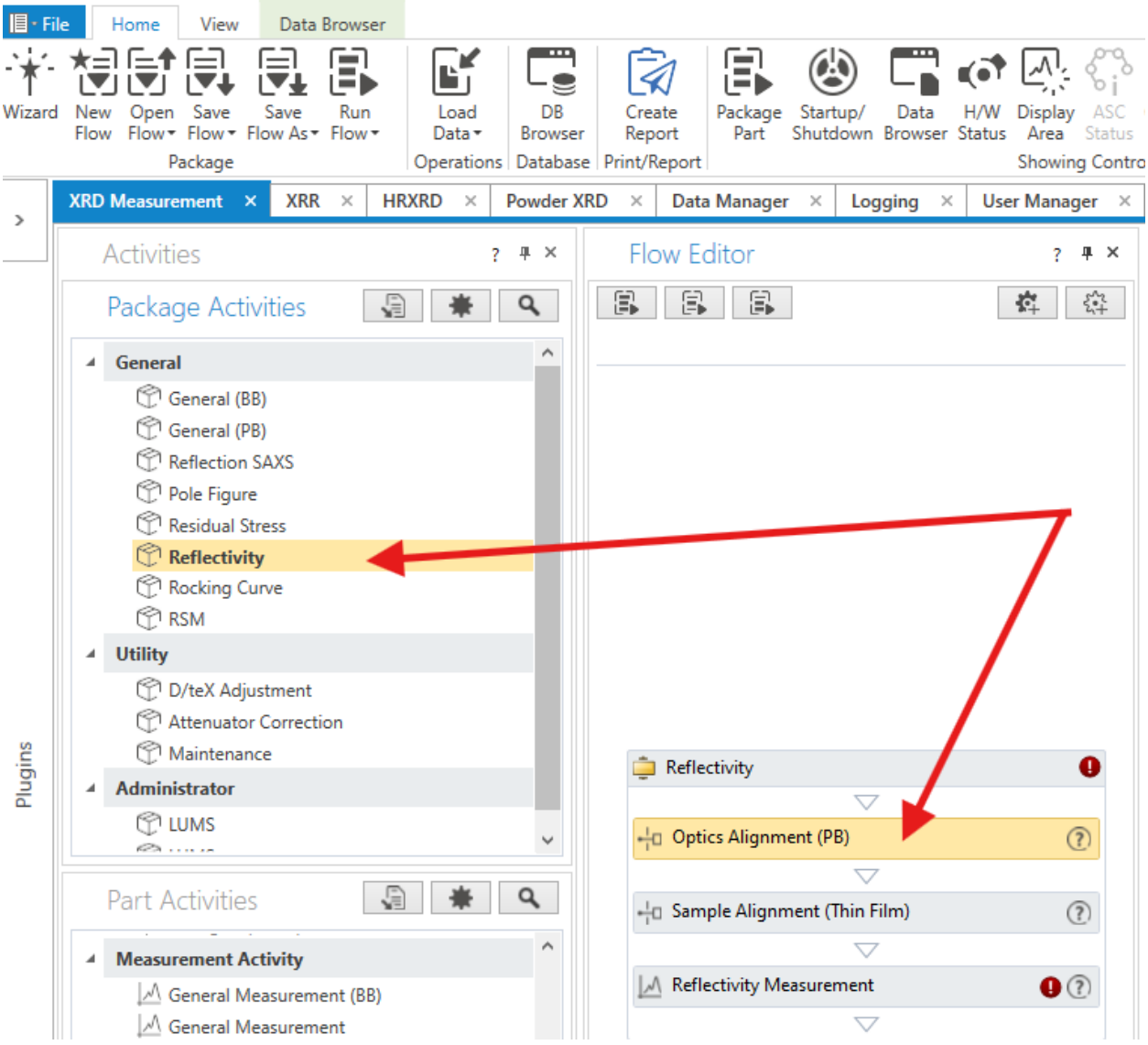
XRR Optic Alignment

XRR Configuration settings

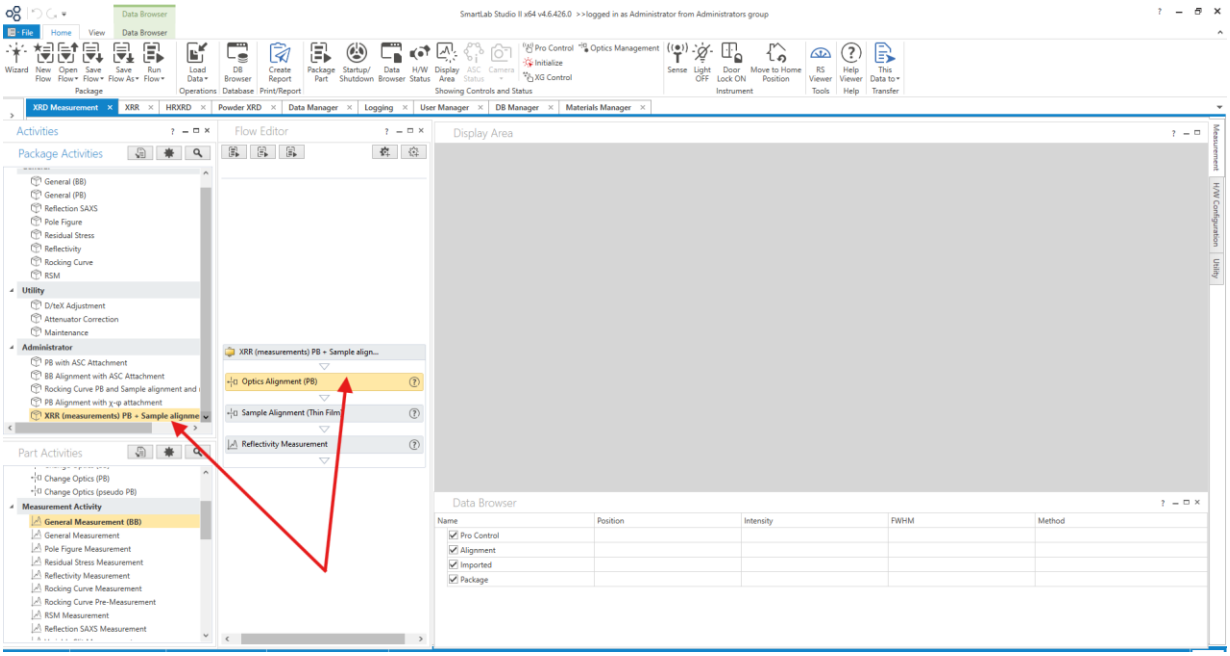
1. Turn OFF XRD unit and Turn OFF SmartLab SE Software
2. Replace Soller Slit 2.5 deg and IPS adapter with Monochromator Ge(220)x2
3. Turn ON XRD unit and Turn ON SmartLab SE Software
    a. Click Hardware Configuration
    b. Select incident optical unit as shown in arrows
    c. Change IPS adapter with Ge(220)x2 and press Apply

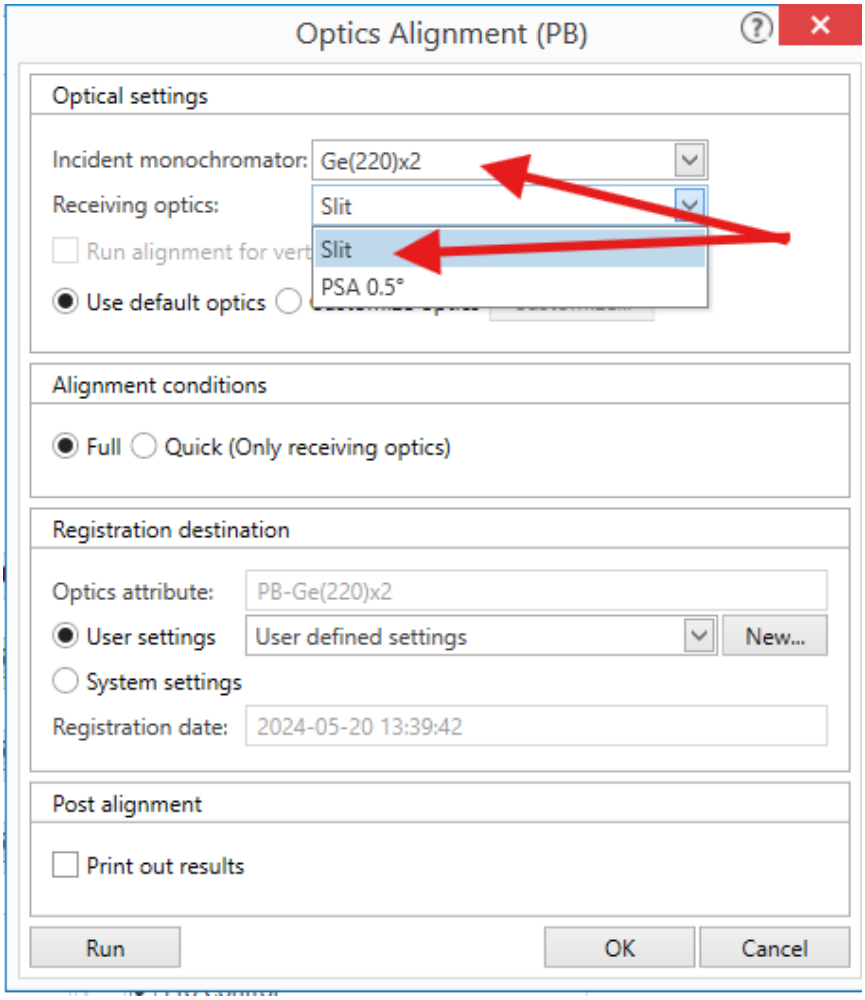


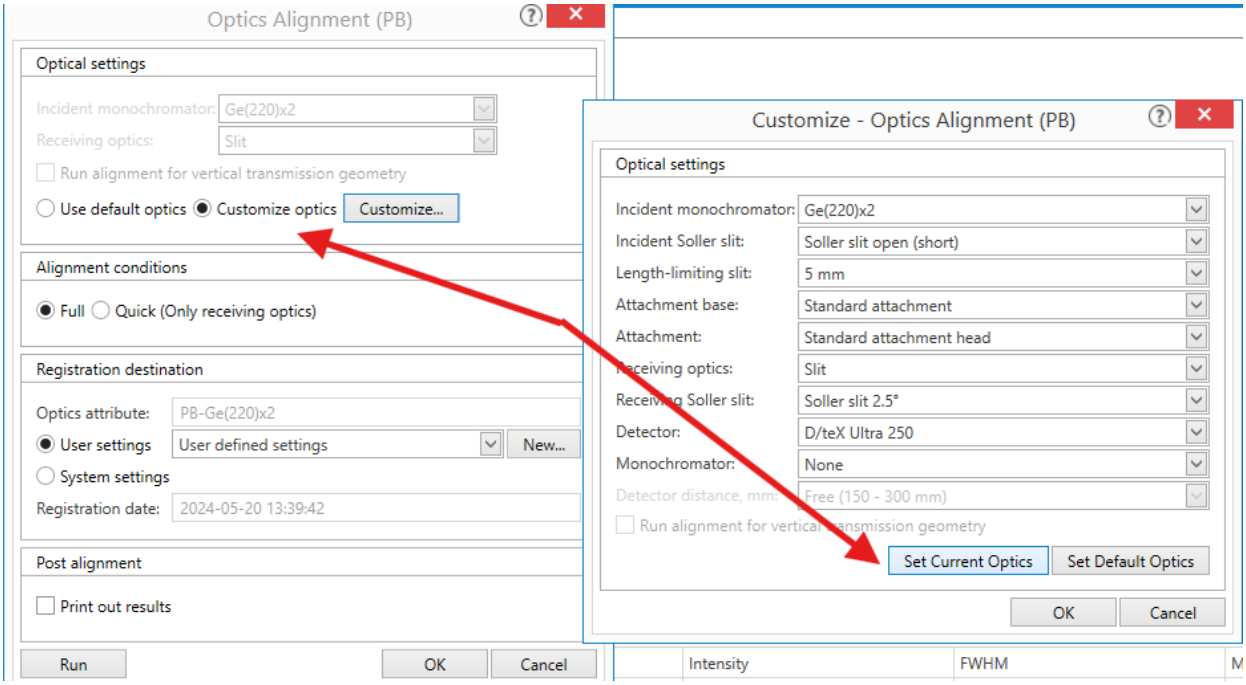


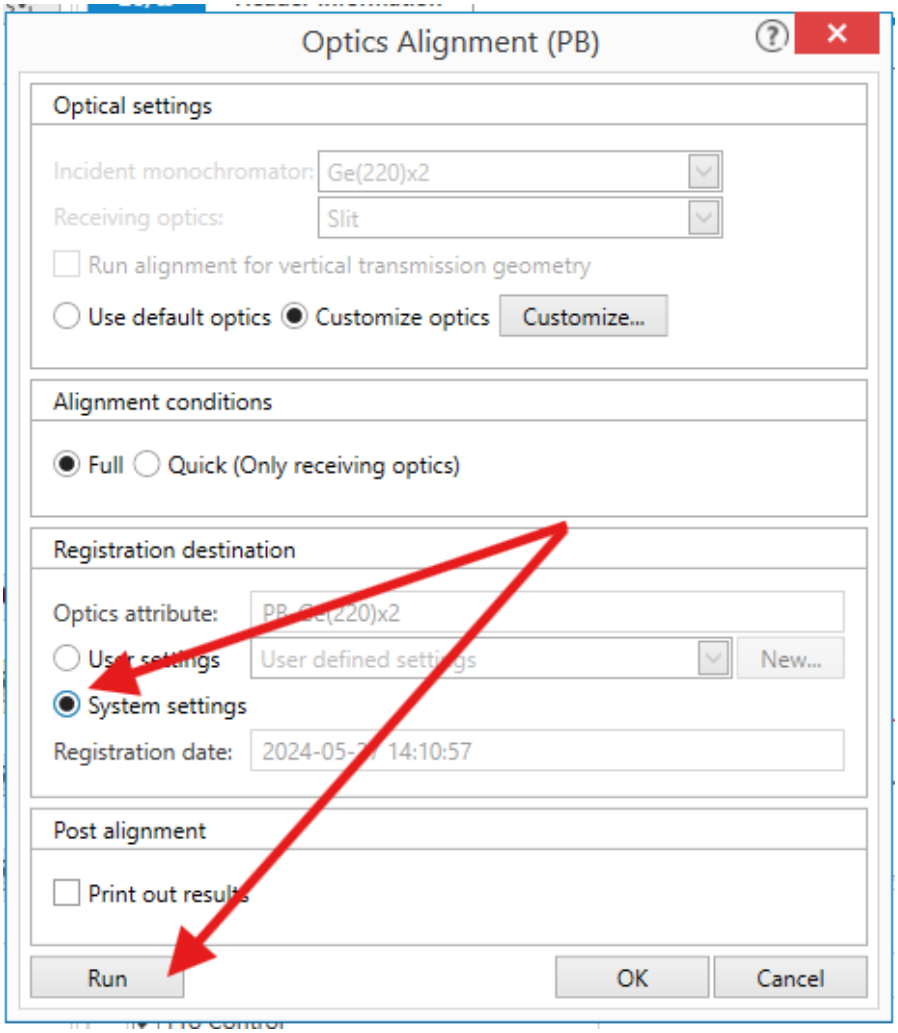

File
Home
View
Data Browser
Wizard
New Flow
Open Flow
Save Flow
Save Flow As
Run Flow
Package
Load Data
Operations
DB Browser
Database
Create Report
Print/Report
Package Part
Startup/ Shutdown
Data Browser
H/W Status
Display Area
ASC Status
Showing Contro
XRD Measurement
XRR
HRXRD
Powder XRD
Data Manager
Logging
User Manager
Activities
Package Activities
General
General (BB)
General (PB)
Reflection SAXS
Pole Figure
Residual Stress
Reflectivity
Rocking Curve
RSM
Utility
D/teX Adjustment
Attenuator Correction
Maintenance
Administrator
LUMS
Part Activities
Measurement Activity
General Measurement (BB)
General Measurement
Flow Editor
Reflectivity
Optics Alignment (PB)
Sample Alignment (Thin Film)
Reflectivity Measurement
Plugins

Or use this


SmartLab Studio II x64 v4.6.426.0 >>logged in as Administrator from Administrators group
XRD Measurement
XRR
HRXRD
Powder XRD
Data Manager
Logging
User Manager
DB Manager
Materials Manager
Activities
Package Activities
Flow Editor
Display Area
Part Activities
Data Browser
Optics Alignment (PB)
Reflectivity Measurement


Optics Alignment (PB)
Optical settings
Incident monochromator:
Ge(220)x2
Receiving optics:
Slit
Slit
PSA 0.5°
Use default optics
Alignment conditions
Full
Quick (Only receiving optics)
Registration destination
Optics attribute:
PB-Ge(220)x2
User settings
User defined settings
New...
System settings
Registration date:
2024-05-20 13:39:42
Post alignment
Print out results
Run
OK
Cancel


Optics Alignment (PB)
Optical settings
Incident monochromator: Ge(220)x2
Receiving optics: Slit
Run alignment for vertical transmission geometry
Use default optics
Customize optics
Customize...
Alignment conditions
Full
Quick (Only receiving optics)
Registration destination
Optics attribute: PB-Ge(220)x2
User settings
User defined settings
New...
System settings
Registration date: 2024-05-20 13:39:42
Post alignment
Print out results
Run
OK
Cancel
Customize - Optics Alignment (PB)
Optical settings
Incident monochromator: Ge(220)x2
Incident Soller slit: Soller slit open (short)
Length-limiting slit: 5 mm
Attachment base: Standard attachment
Attachment: Standard attachment head
Receiving optics: Slit
Receiving Soller slit: Soller slit 2.5°
Detector: D/teX Ultra 250
Monochromator: None
Detector distance, mm: Free (150 - 300 mm)
Run alignment for vertical transmission geometry
Set Current Optics
Set Default Optics
OK
Cancel
Intensity
FWHM


Optics Alignment (PB)
Optical settings
Incident monochromator: Ge(220)x2
Receiving optics: Slit
Run alignment for vertical transmission geometry
Use default optics
Customize optics
Customize...
Alignment conditions
Full
Quick (Only receiving optics)
Registration destination
Optics attribute:
User defined settings
New...
System settings
Registration date:
Post alignment
Print out results
Run
OK
Cancel

Smart Message

Install **standard attachment base** in **diffractometer**.

Install **standard attachment head** in **standard attachment base**.

Install **Height reference sample plate** in **standard attachment head**.

Insert **center slit** in **Height reference sample plate**.

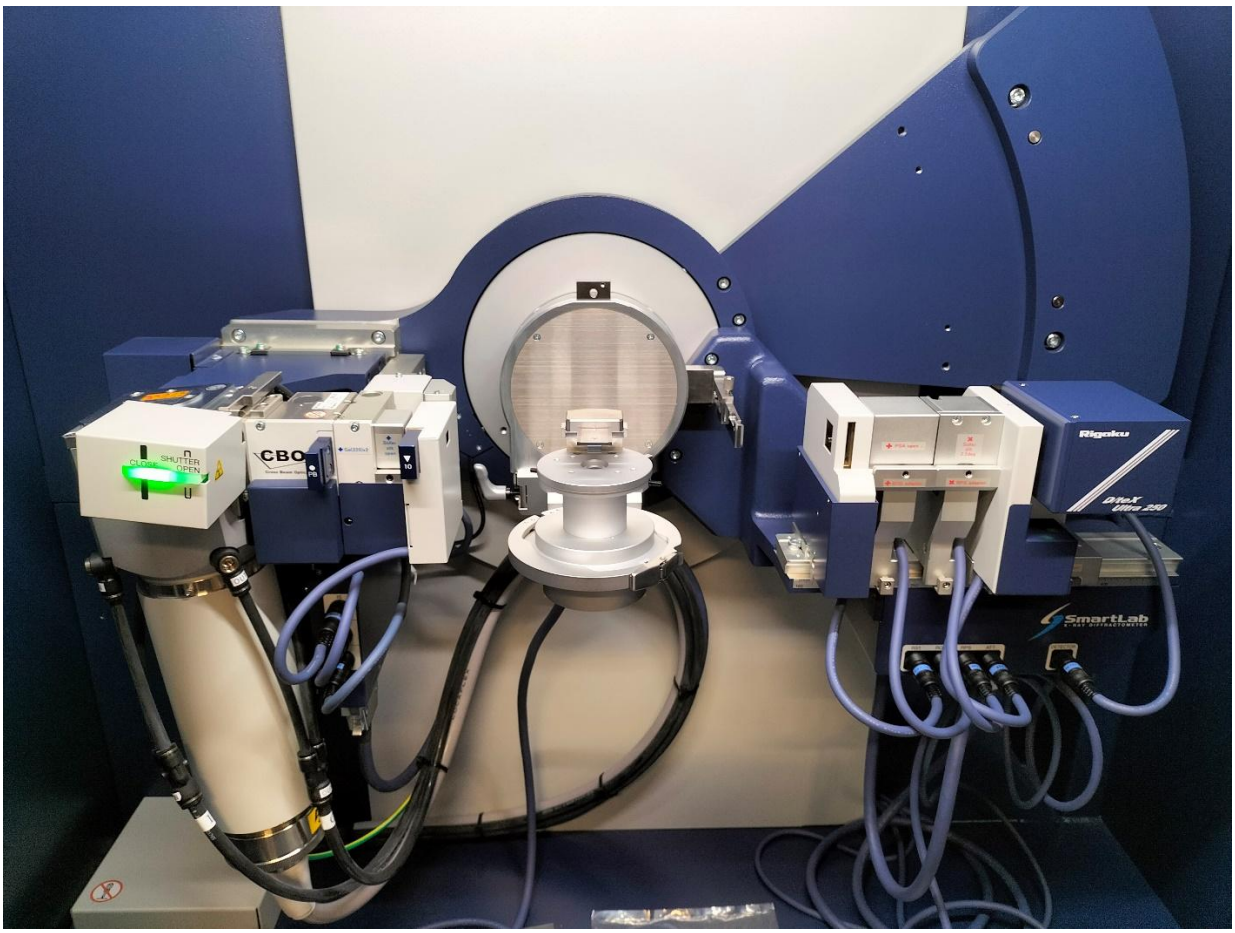
Attach the detector plane of **D/teX Ultra 250** to **311.5** mm.
(Adjust the mark of the **detector adaptor** to **361.5** mm)

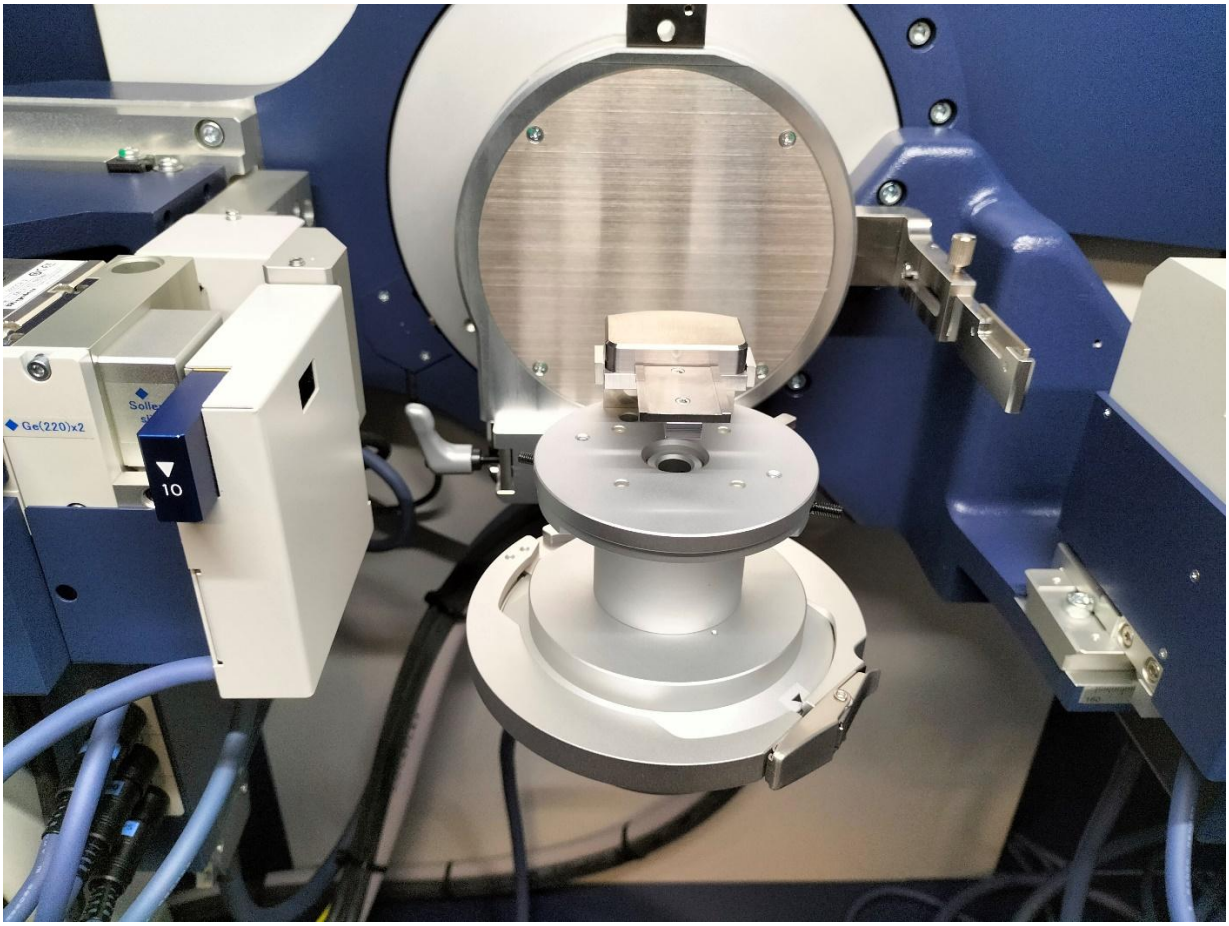
Hide figures

OK

Stop

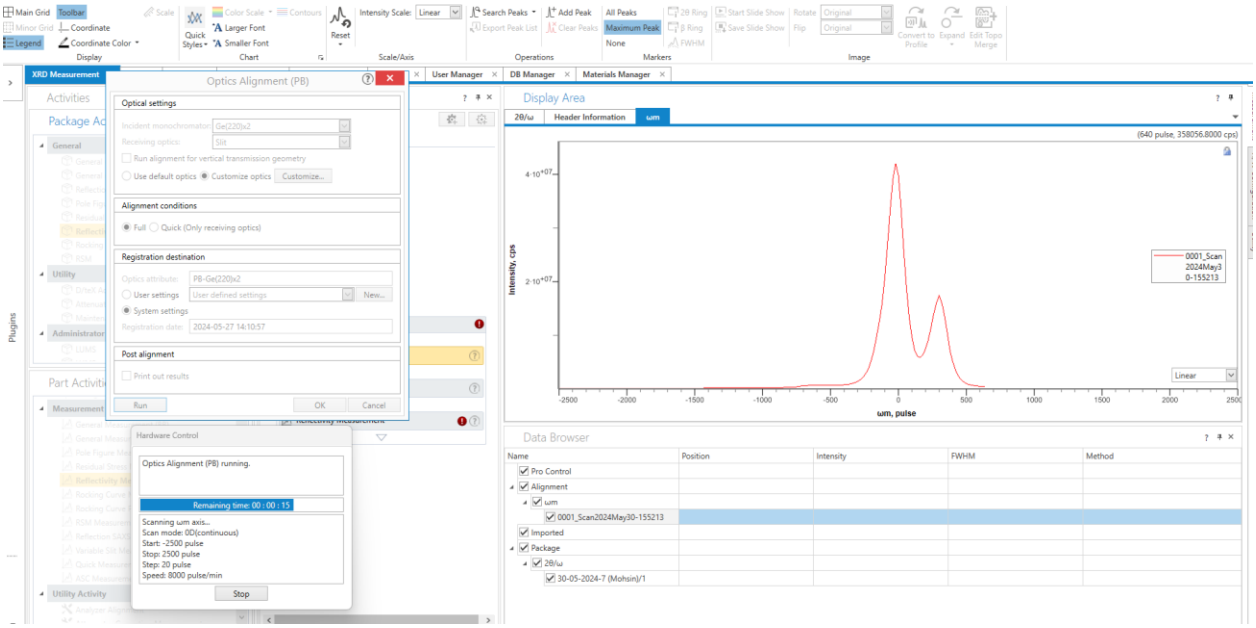
Attachment for general measurements




Ge(220)x2
10

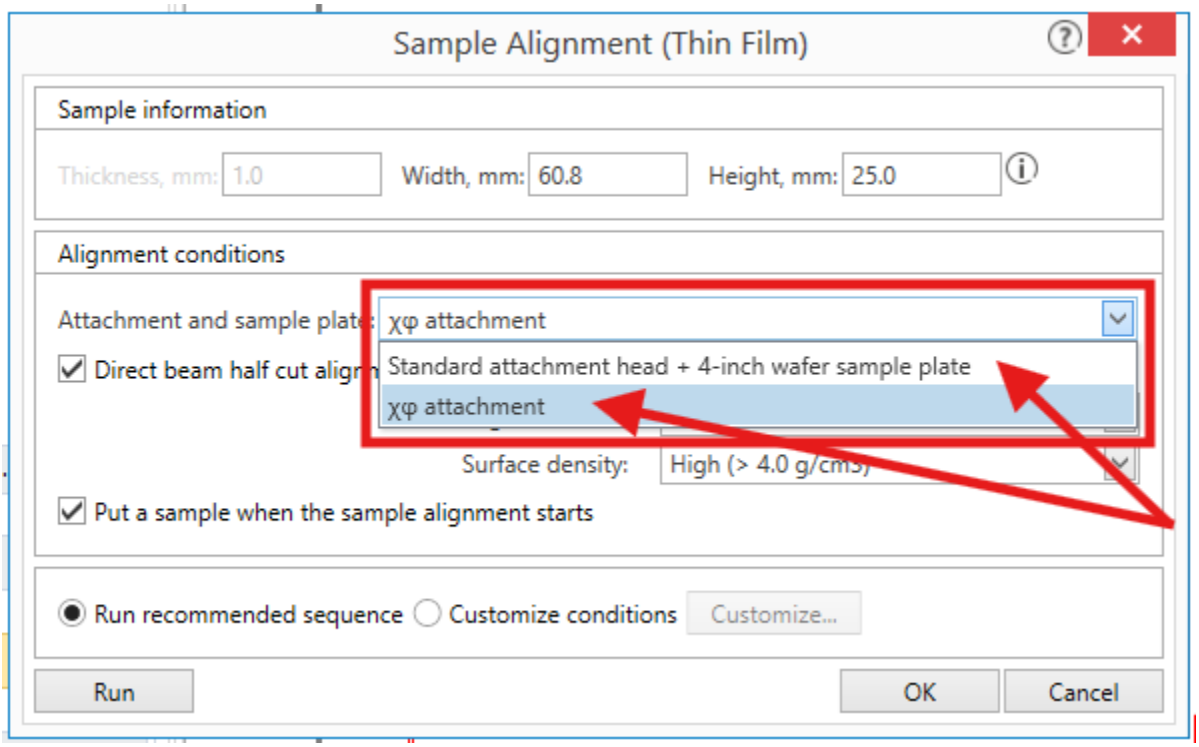
Adjust the mark of the detector adapter to 361.5 mm


tector adaptor
+50mm
300
350
400
ROD
RPS
ATT
DETECTOR

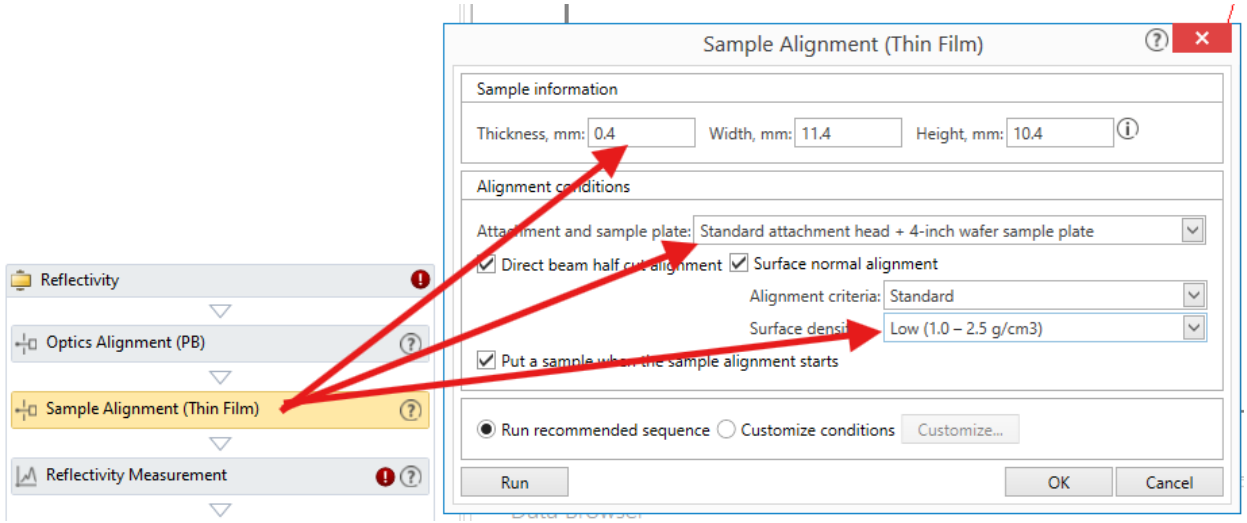


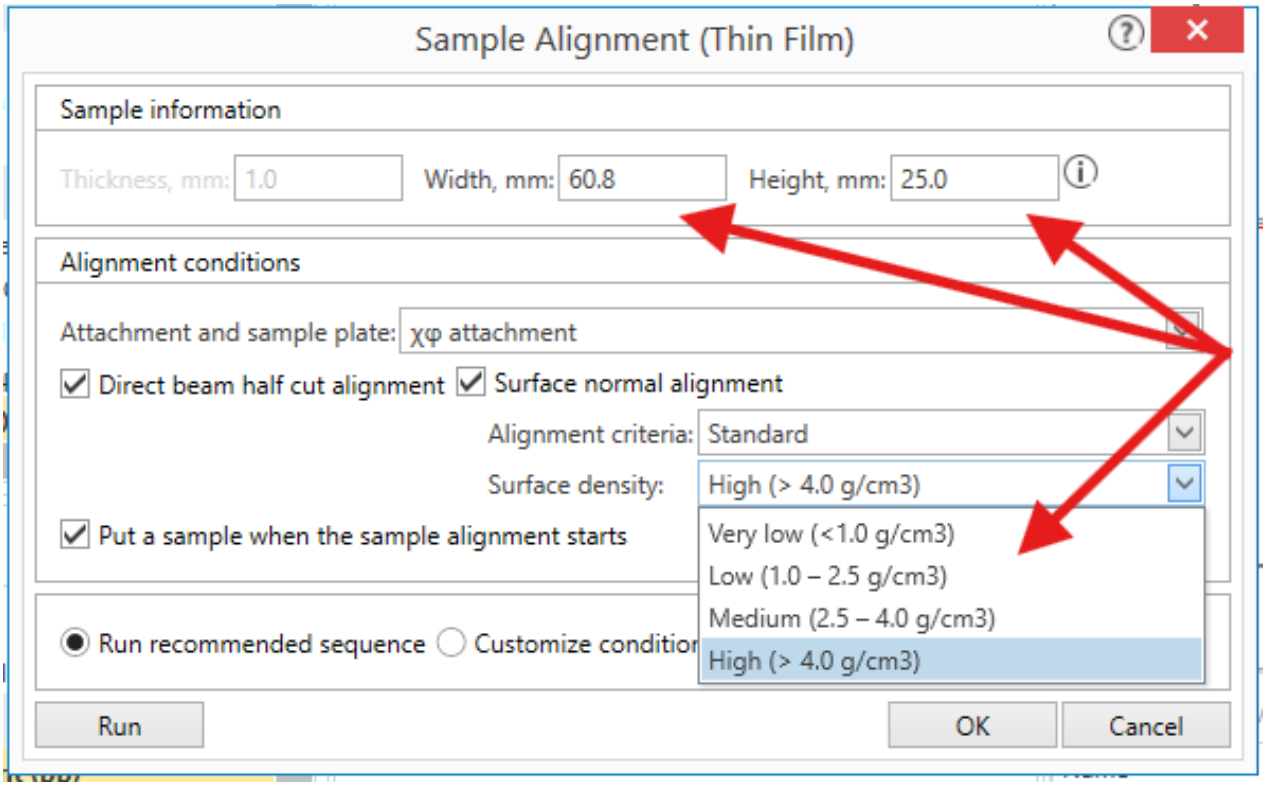
XRR Sample Alignment

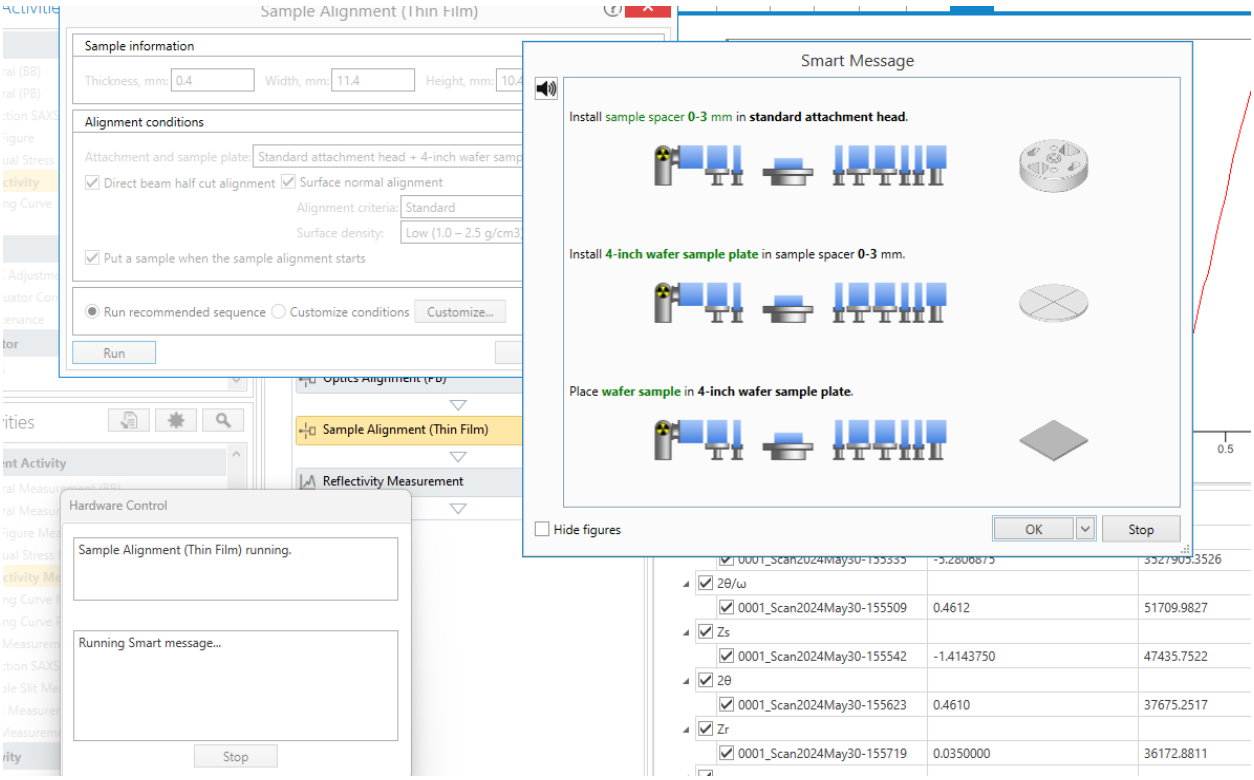
Here we need to select the attachment.



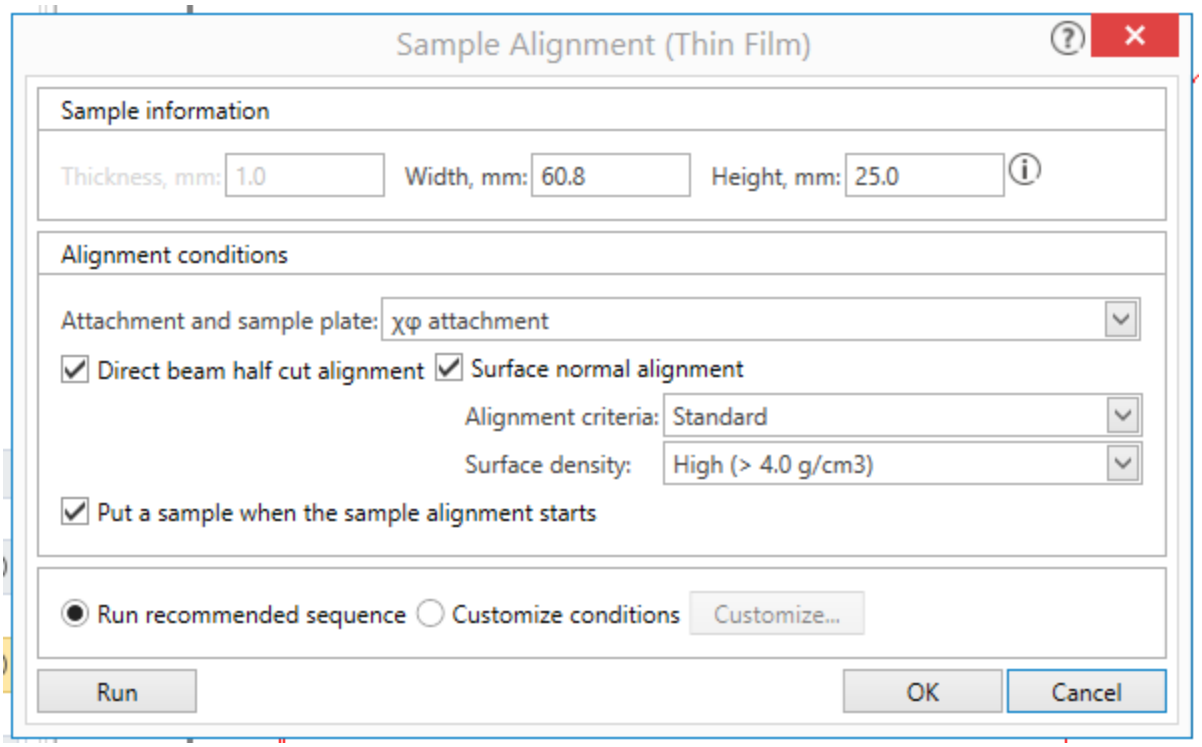
Part1. Attachment 4-inch wafer sample plate. We need to add parameters of thickness, width and height of sample wafer and film density.



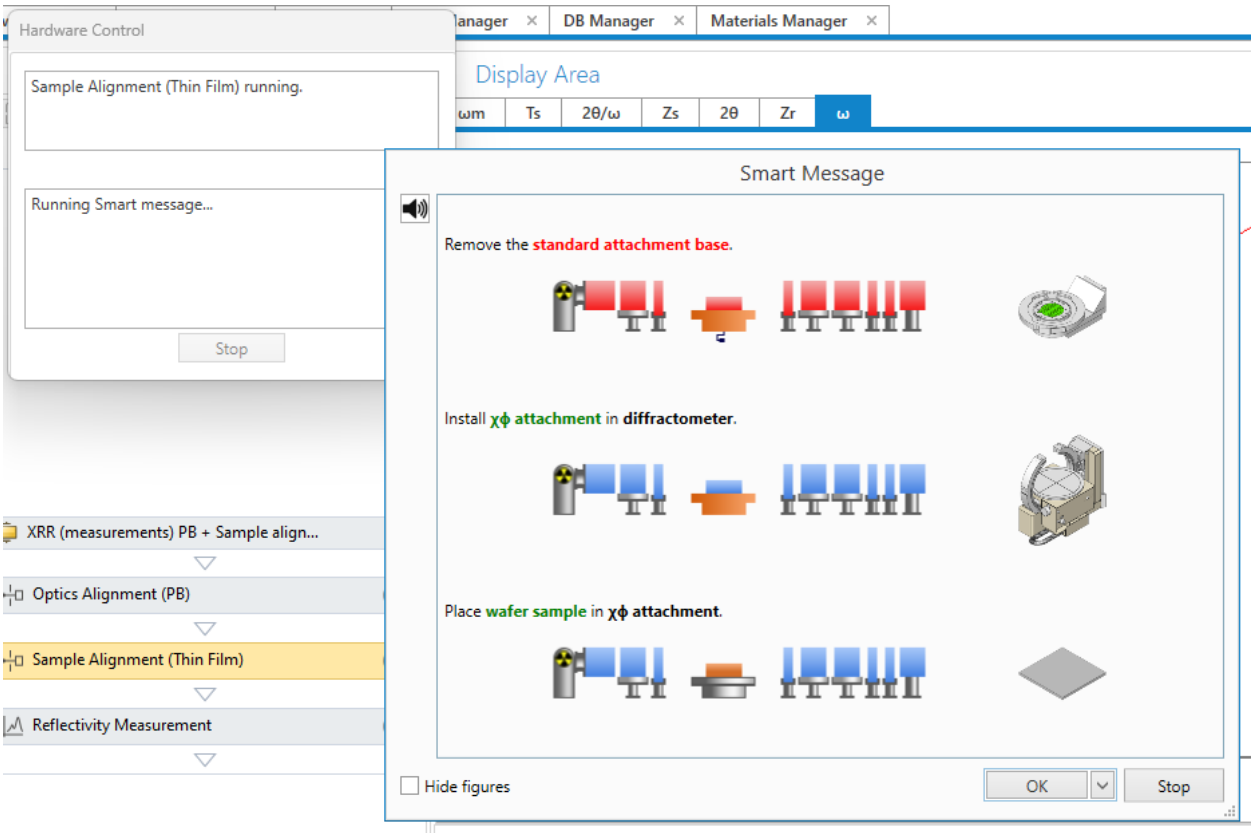


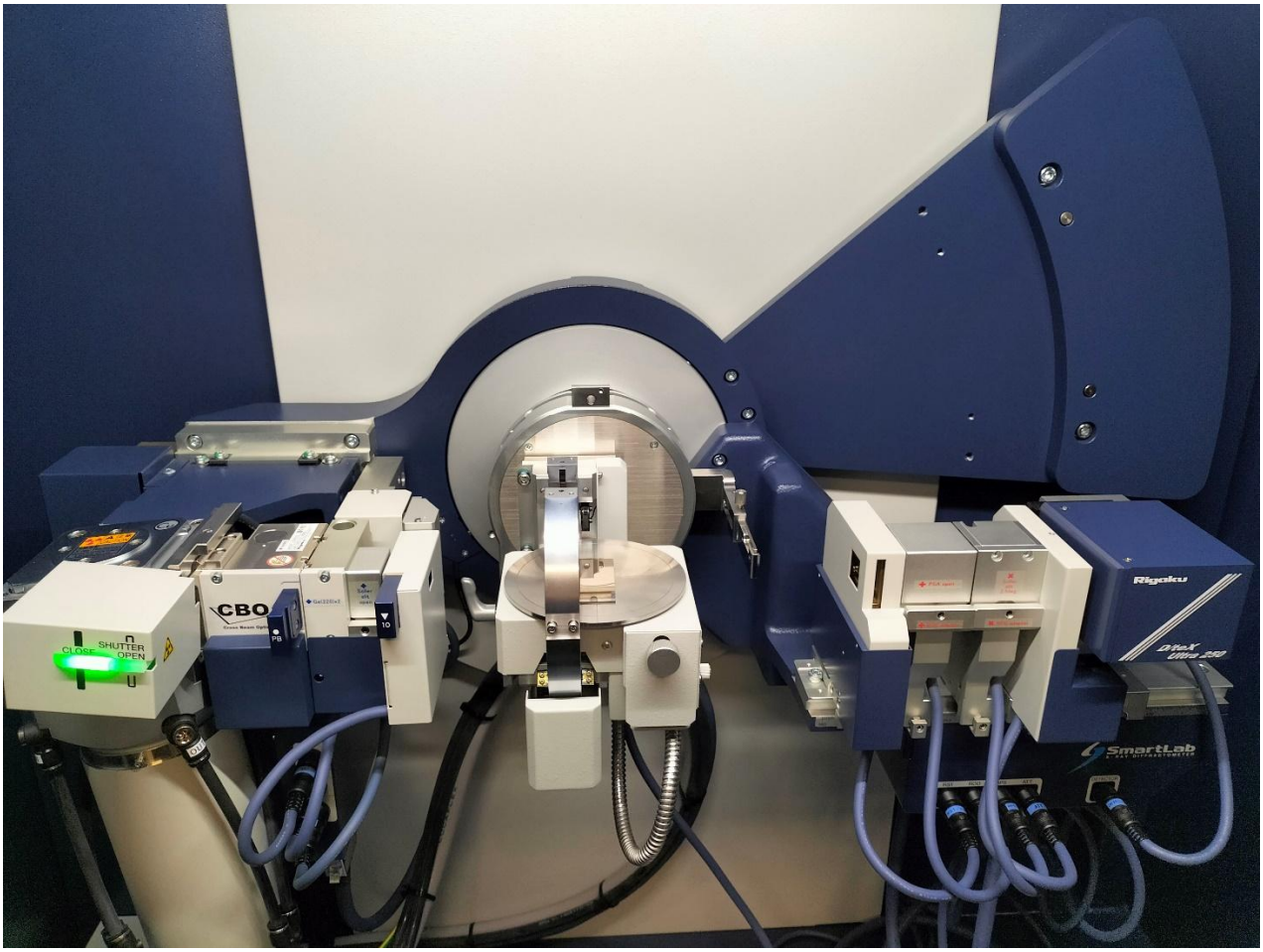


Part2. χϕ attachment. We need to add parameters of width and height of sample wafer.



Replace standard attachment with the χϕ attachment.

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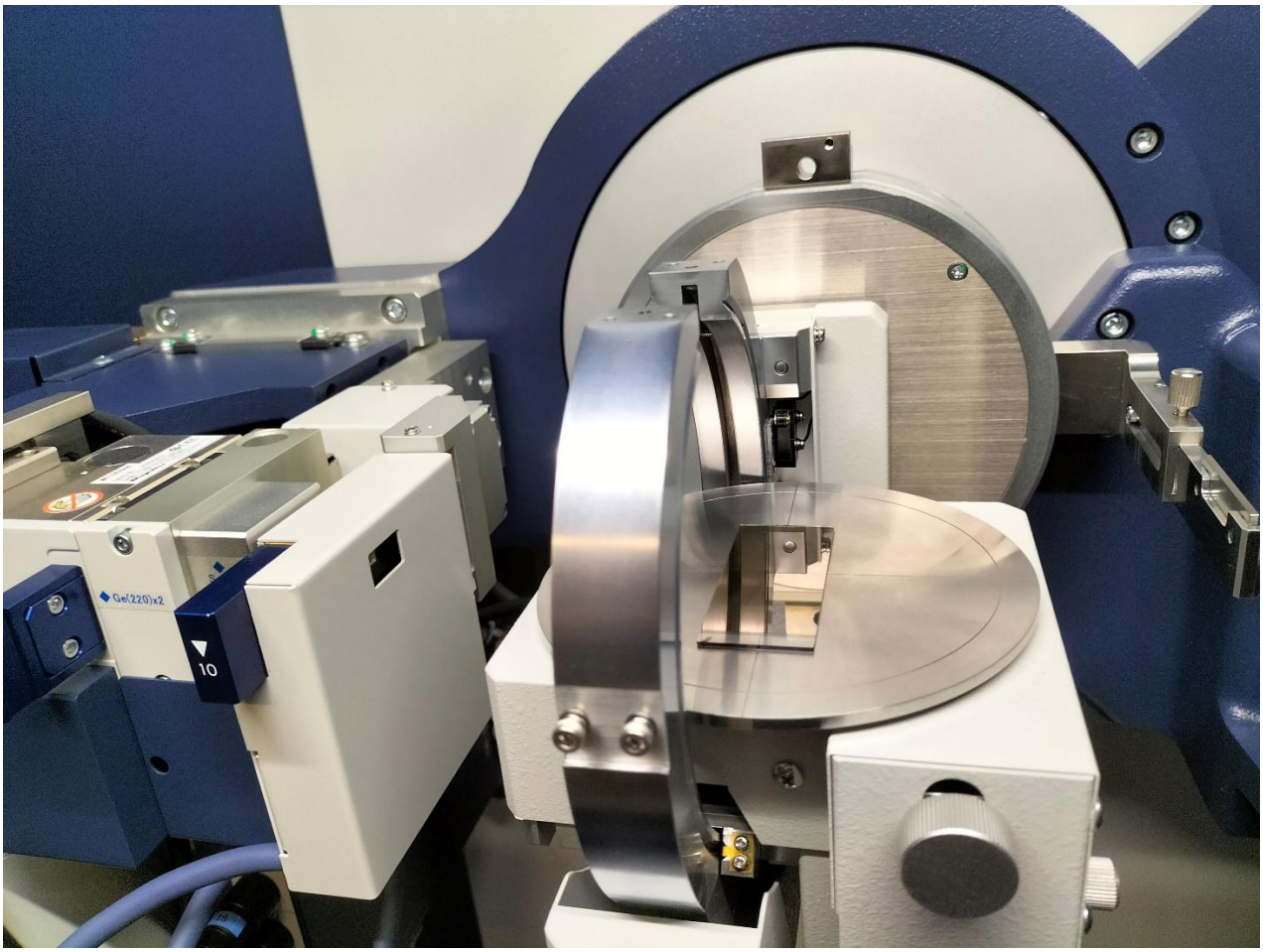
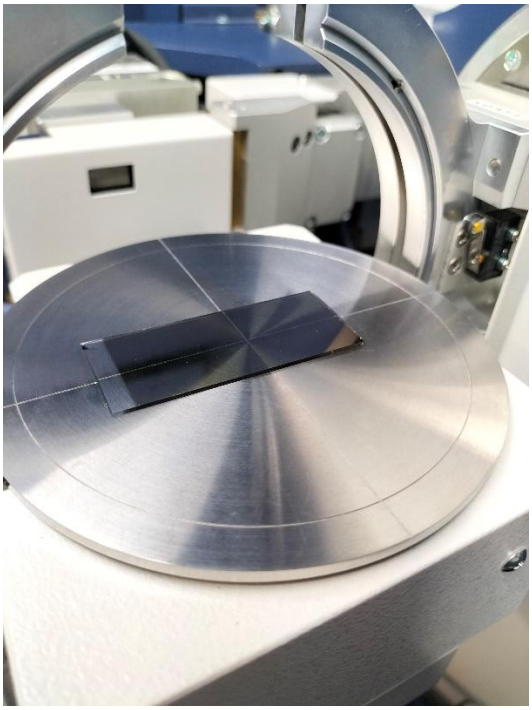
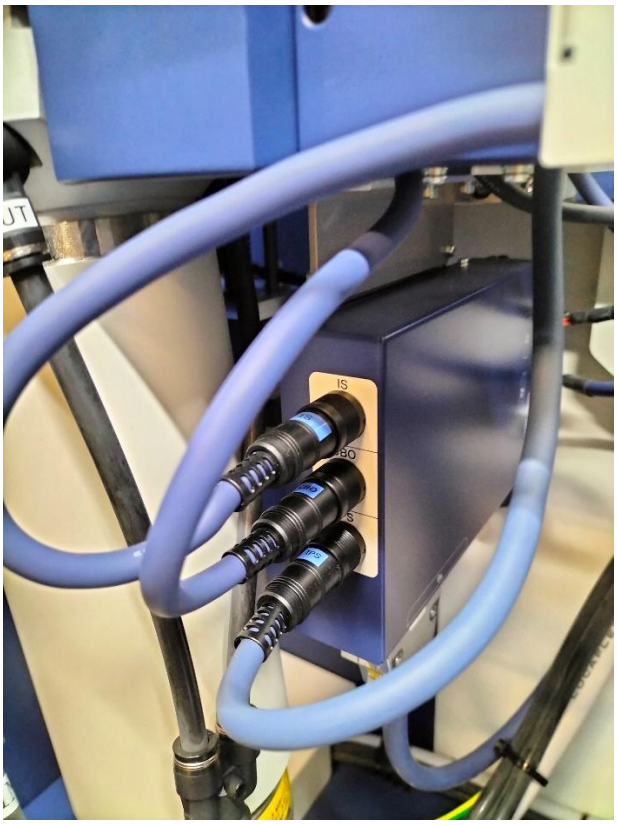
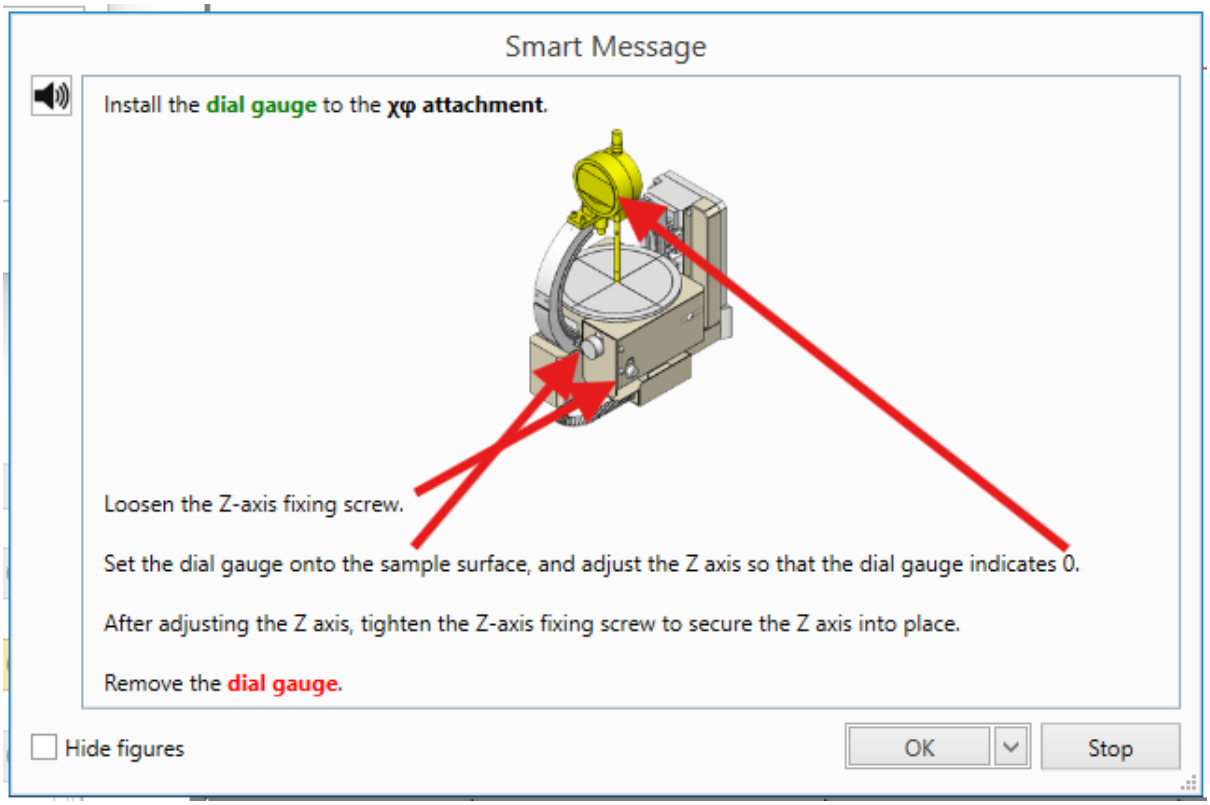
Rigaku
SmartLab


Ge(220)x2
10



Monochromator Ge(220)*2






Mitutoyo
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DATA
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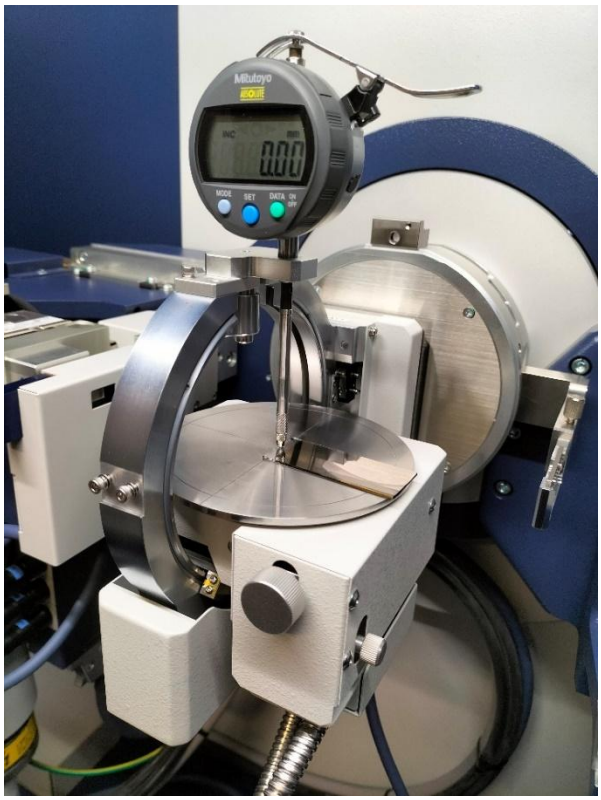
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MODE
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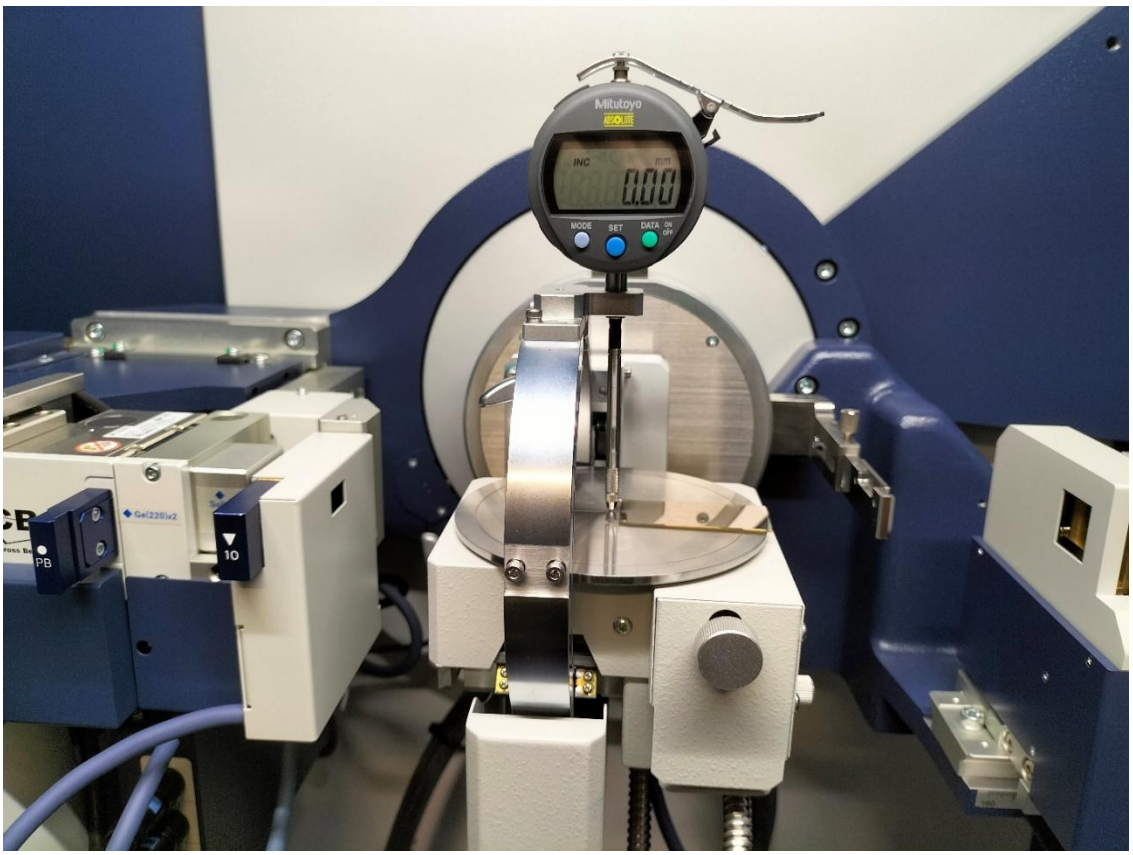
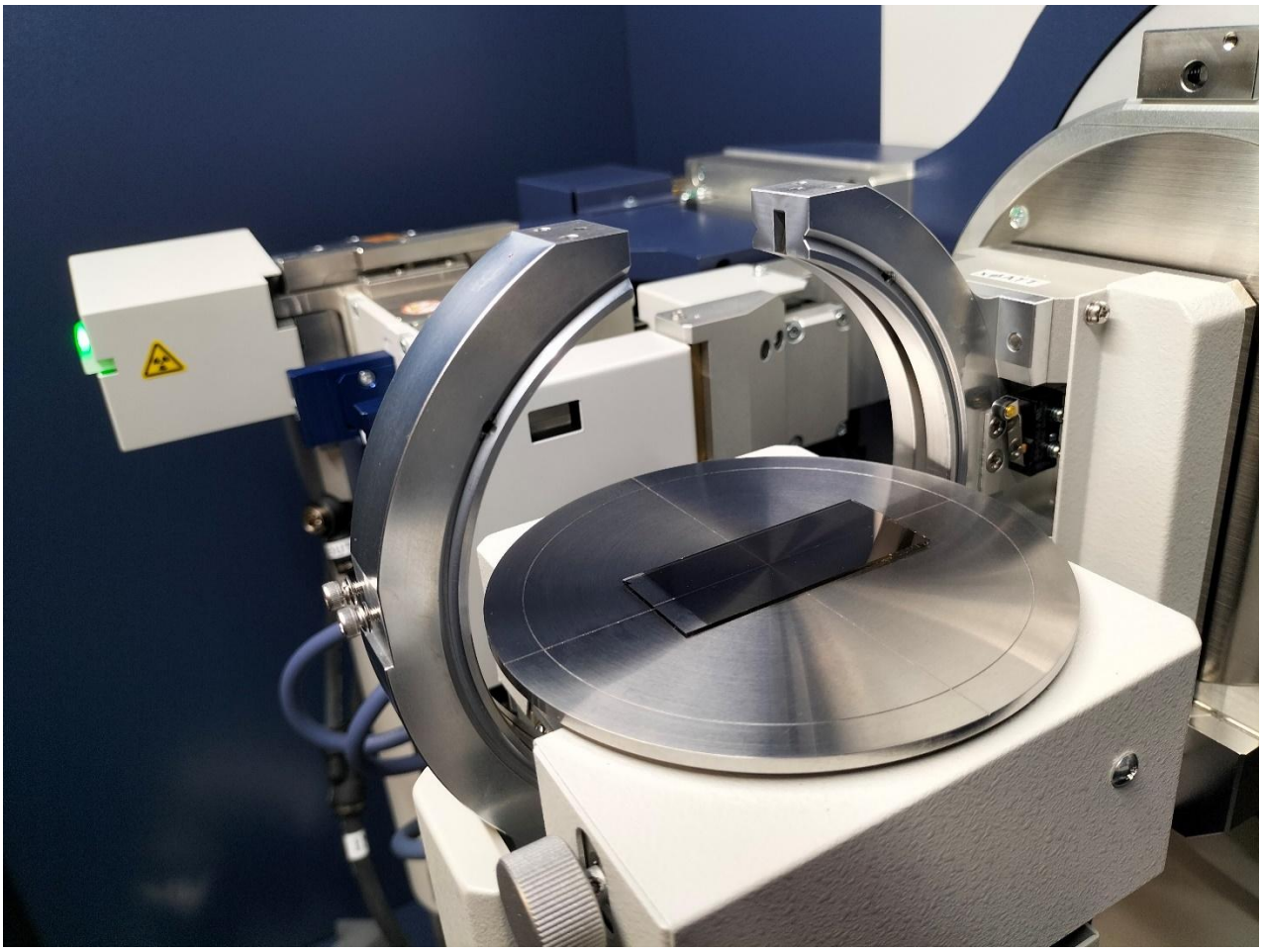
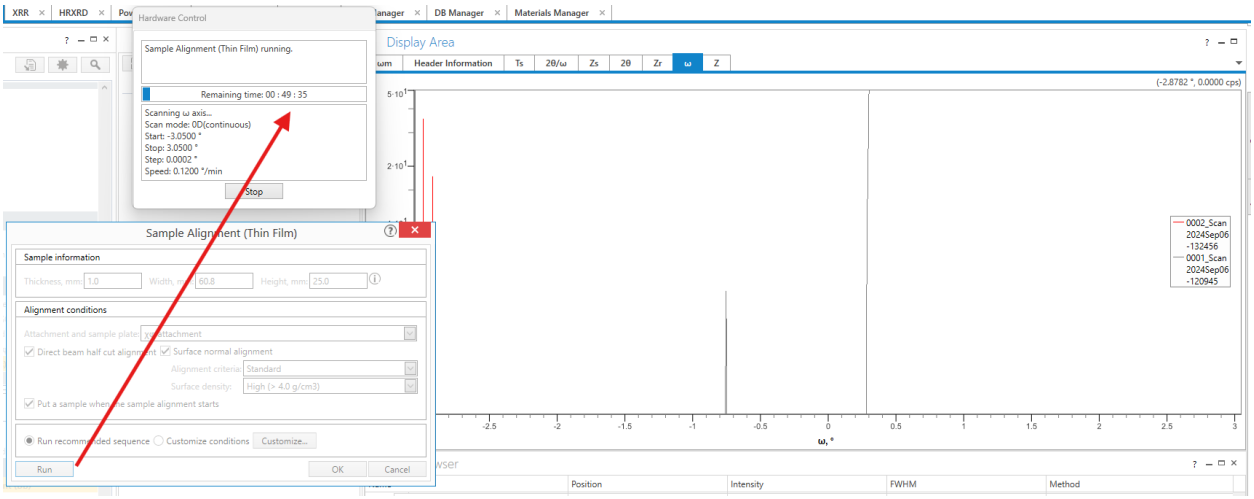
DATA


Mitutoyo
ABSOLUTE
INC
0.00
MODE
SET
DATA
Ge(220)x2
PB
10

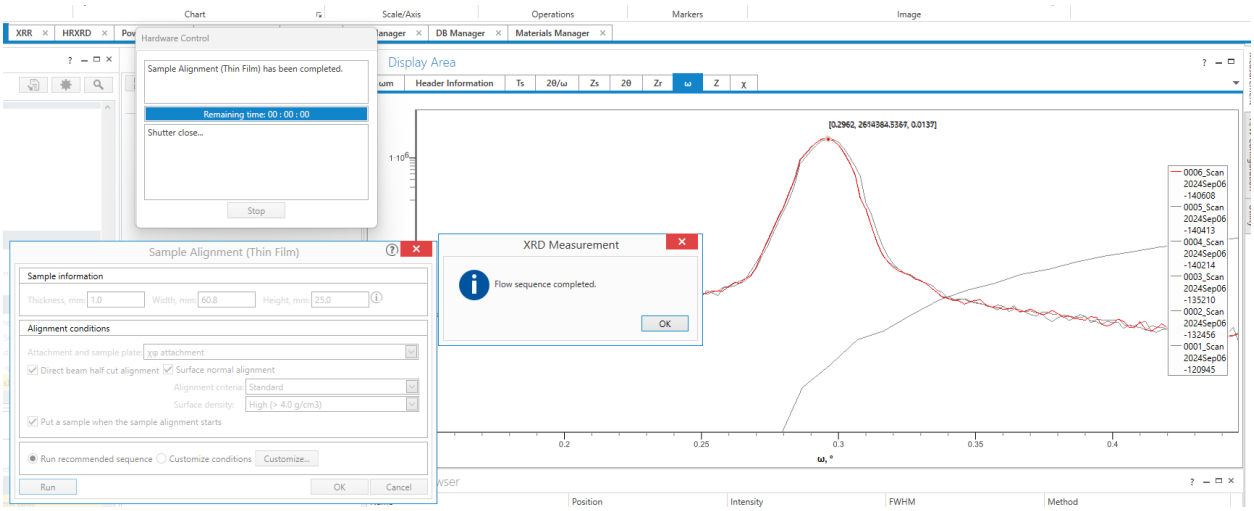
Remove gauge and set sample position as defined in picture.



Sample alignment will take about an hour.

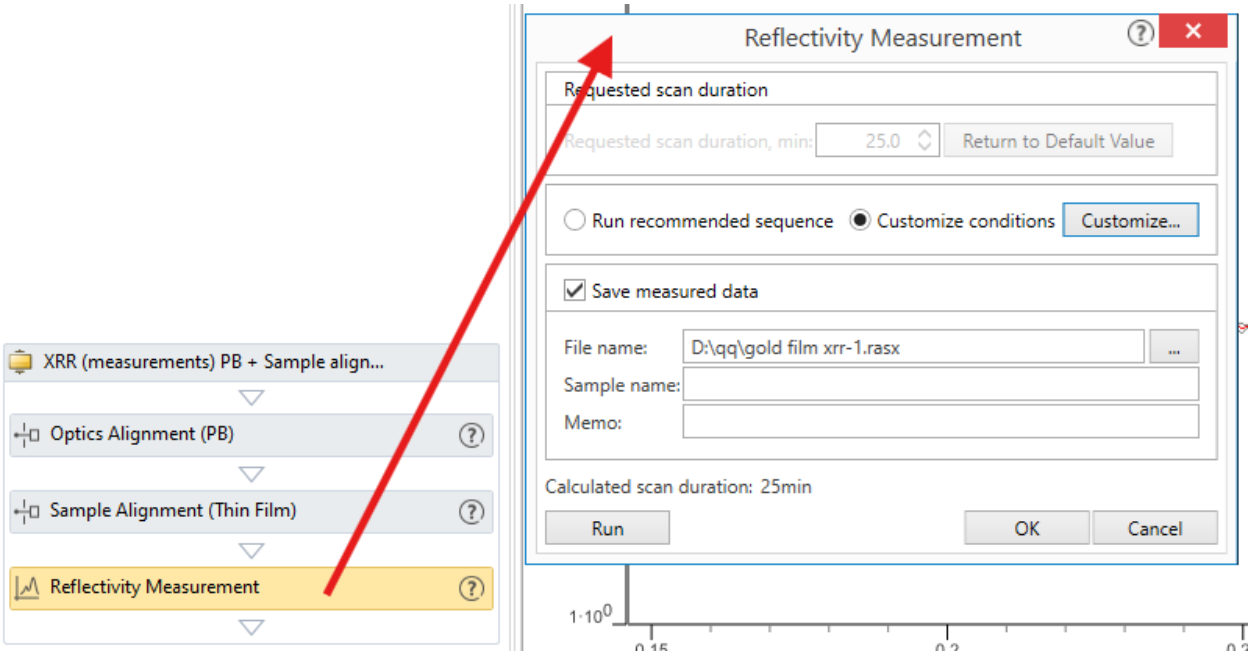


Flow sequence completed.

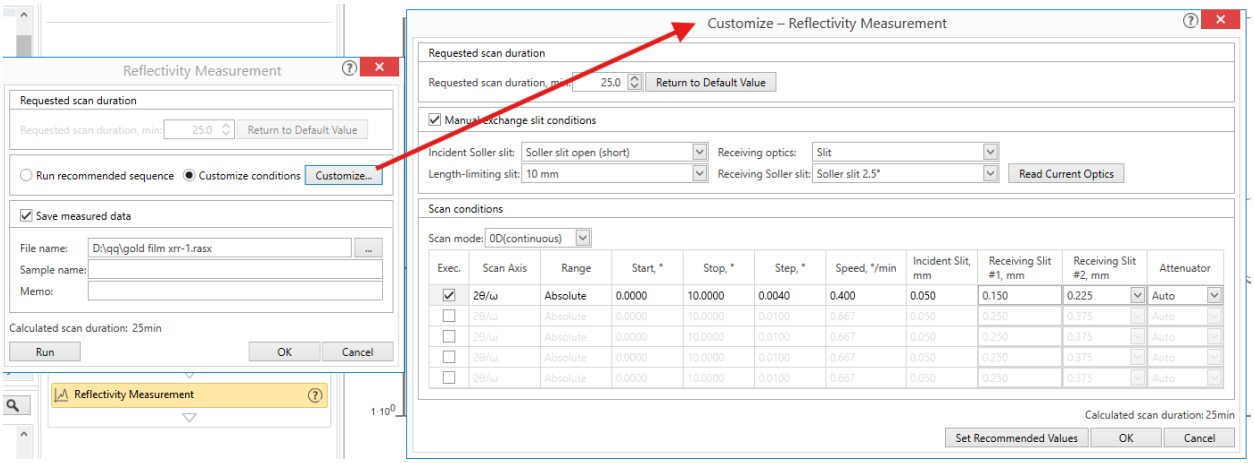


XRR Measurements

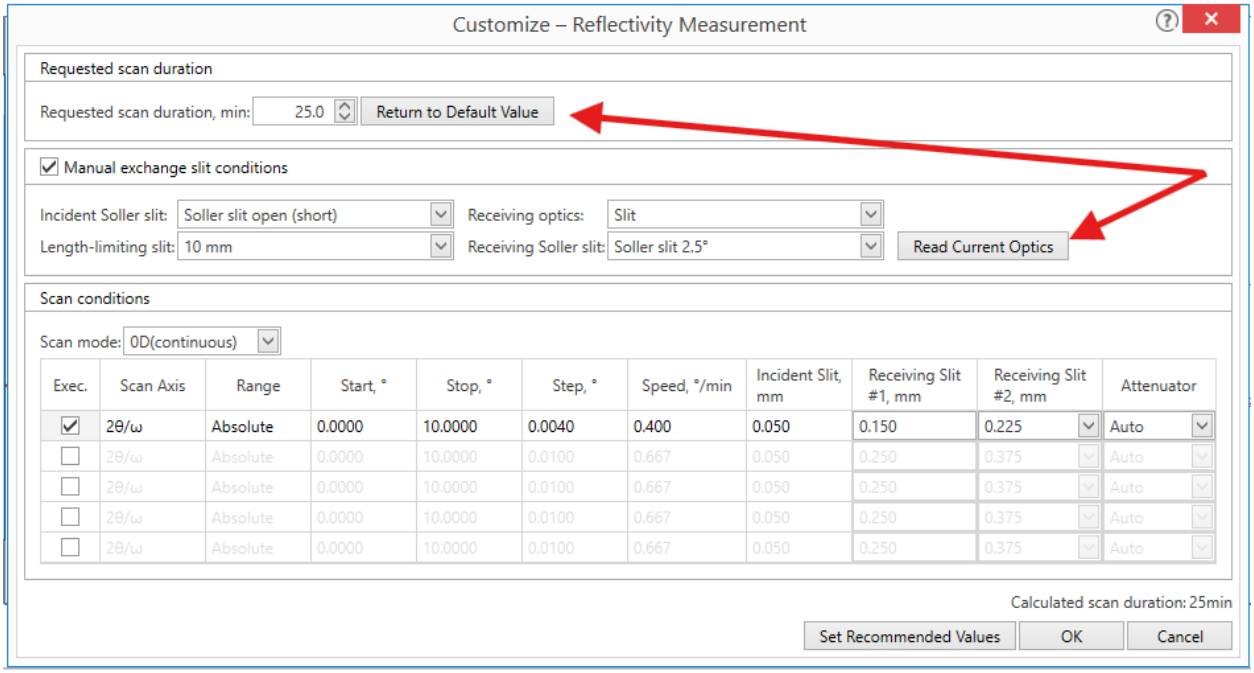
Press Reflectivity measurements



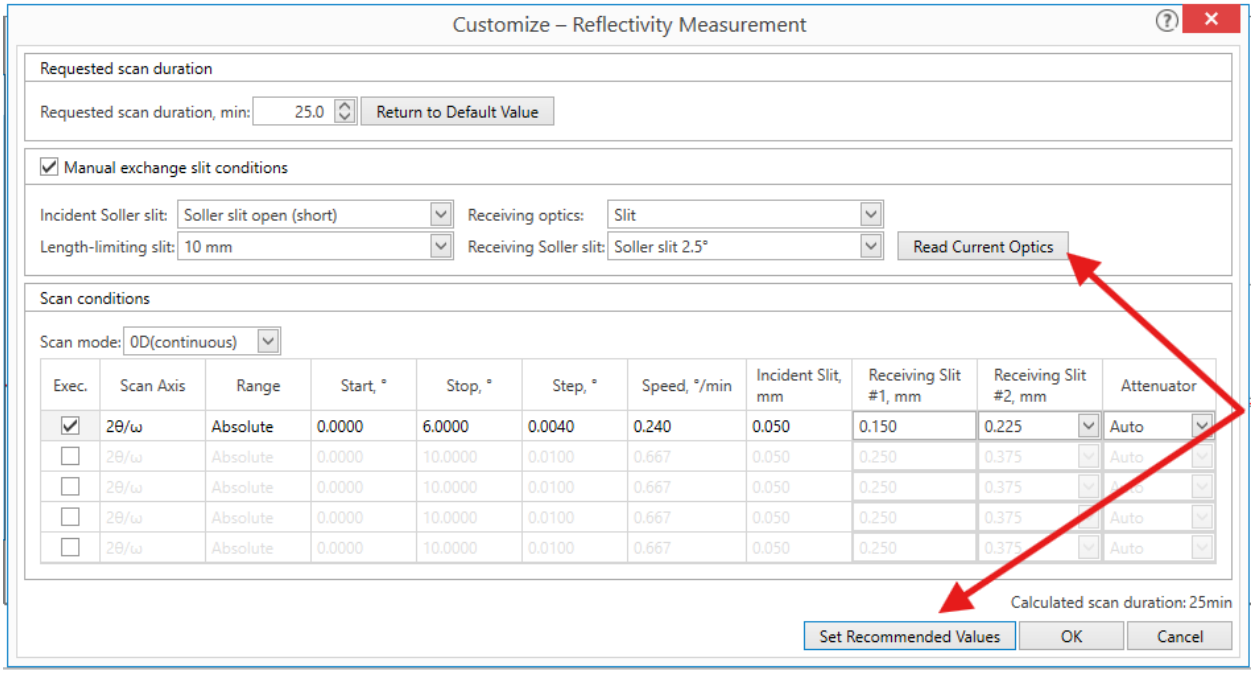
Press Customize for set measurement conditions



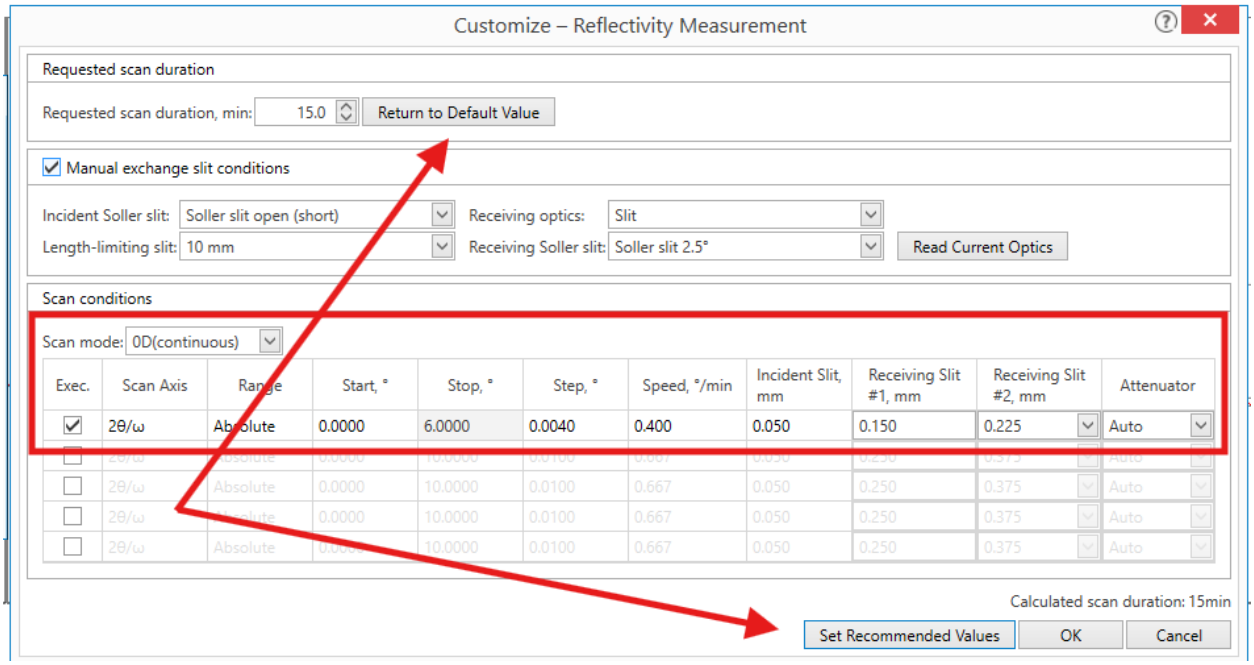
Press read current optics and press default values if default values are suitable for the analysis



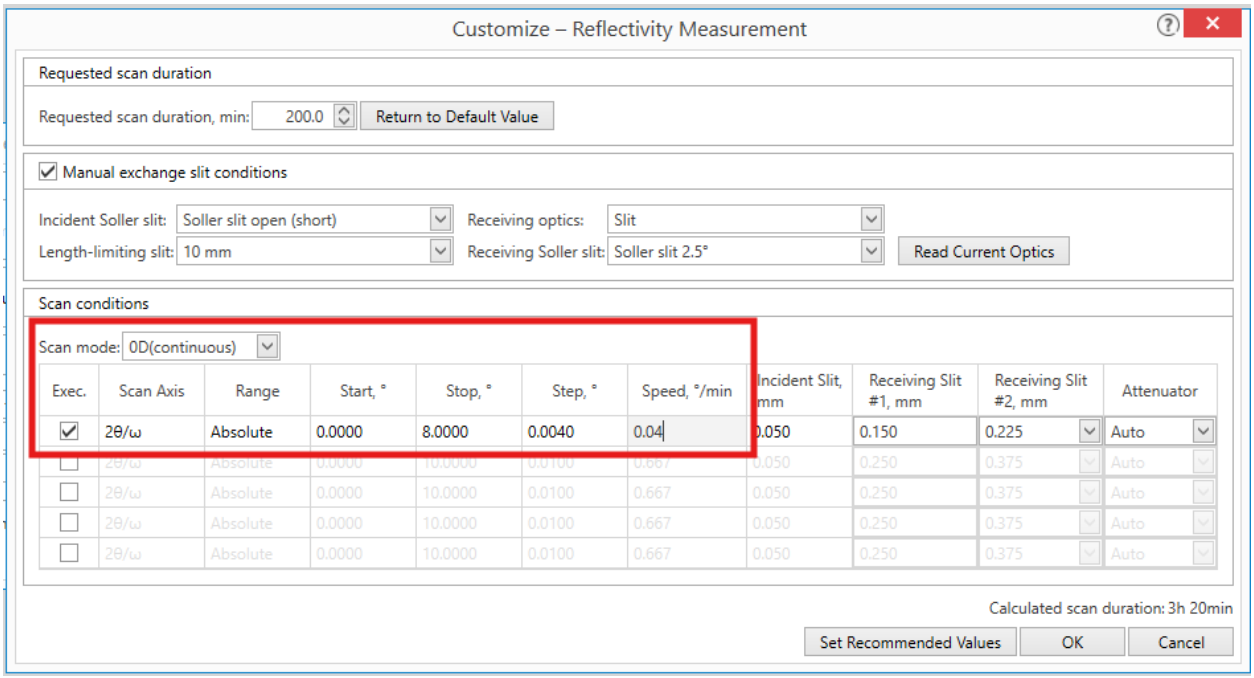
Or press read current optics and press set recommended values



Default values or Recommended values



The better results of the Gold film are achieved with these parameter settings of higher resolution



[More Gold Results]

XRR Data Analysis XRR measurement requirements:

1. The Wafer Size (film + Substrate)
    a. Length (mm) =
    b. Width (mm)=
    c. Thickness optional (mm) =
2. Mark the density of the film (expected)
    a. Very Low (<1.0 g/cm^3
    b. Low (1.0 ~ 2.5 g/cm^3
    c. Medium (<2.5 ~ 4 g/cm^3
    d. High (>4 g/cm^3

XRR analysis requirements:

1. Material of the Film & Substrate e.g. Gold film on Glass.
2. Estimated thickness of the film.
3. If the structure of the film is multilayer, then mention
    a. No. of layers.
    b. Material of each layer.

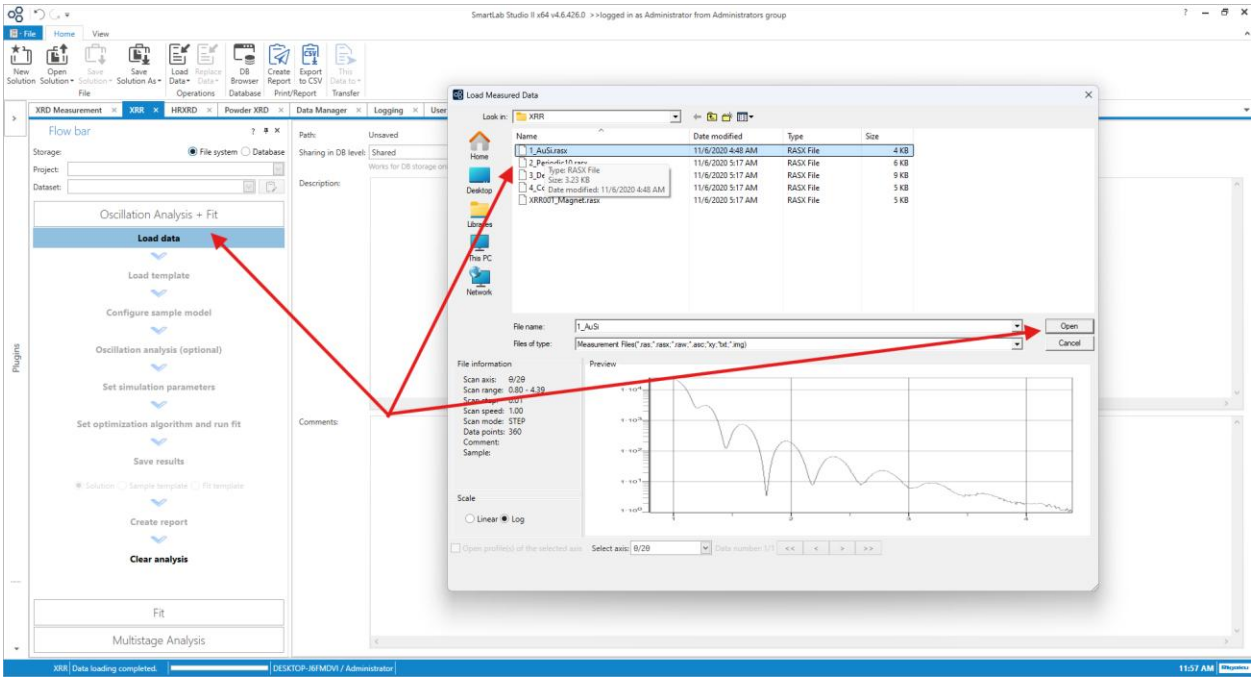
If the material is not available in the SmartLab Studio II database, please provide the necessary information about the film to proceed with the analysis. For details follow the topic (Handling Materials Not in the Database**).**

Summary of XRR measurements and Analysis

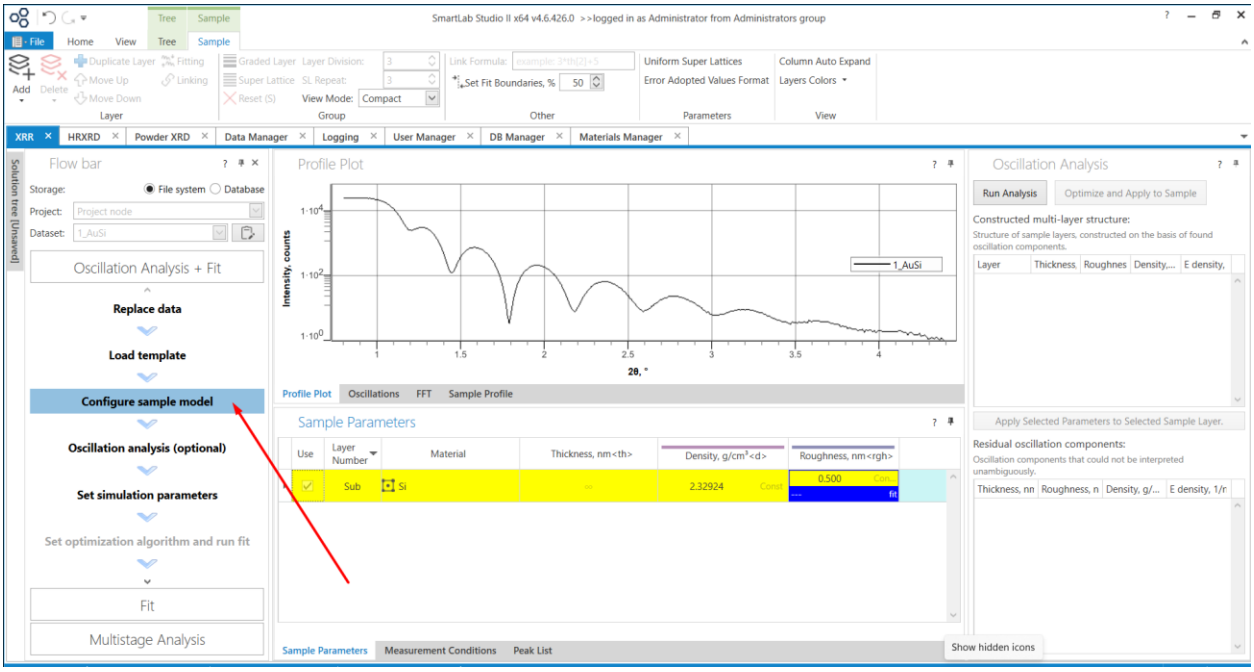
Film Thickness Range	Interpreting the 2θ Range	Fitting Methods
Lower Limit: As low as 1-2 nm, ideal for ultrathin films like monolayers.	Low-Angle Region (0.1° to 1°): Critical for surface roughness and density, with the critical angle visible here.	Levenberg-Marquardt (LM), Genetic Algorithm (GA), Simulated Annealing (SA), Powell's Method,
Upper Limit: Up to a few micrometers, with limitations on thicker films due to reduced oscillation visibility.	Mid-Angle Region (1° to 3°): Contains Kiessig fringes essential for accurate thickness determination.	
	Recommended Range: Typically 0.1° to 4°-5° for optimal results, possibly extending to 8°-10° if needed.	

Analysis Steps

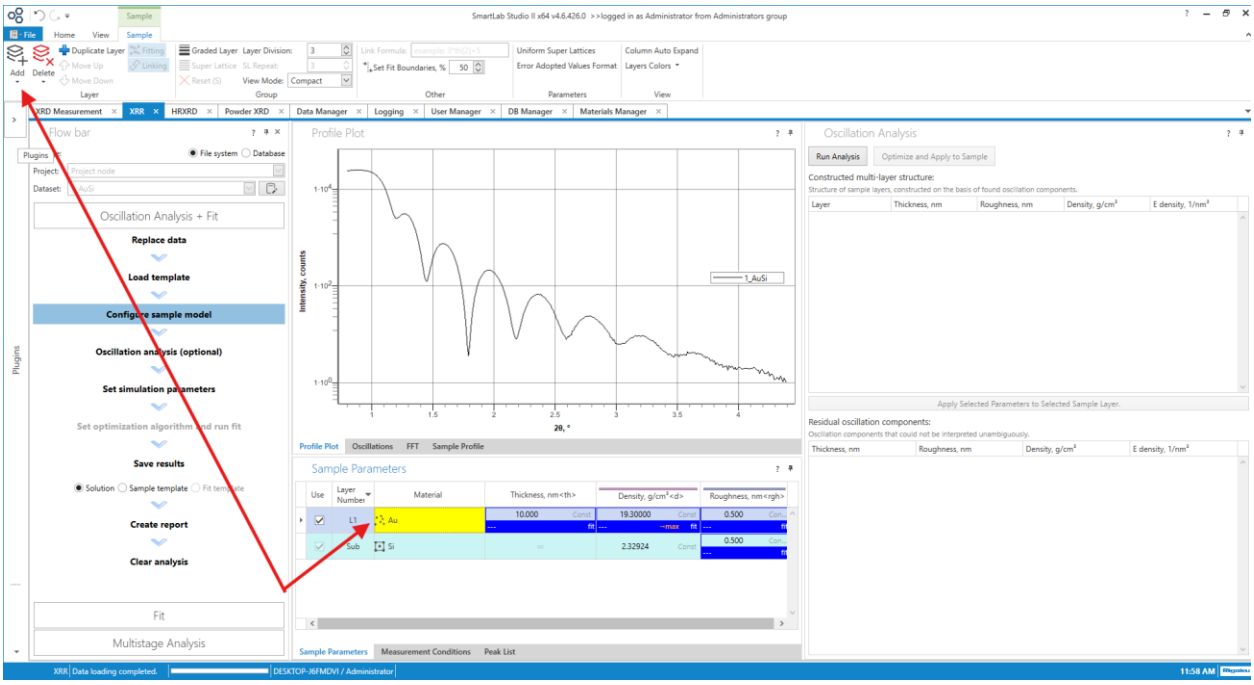
Load XRR Measured Data



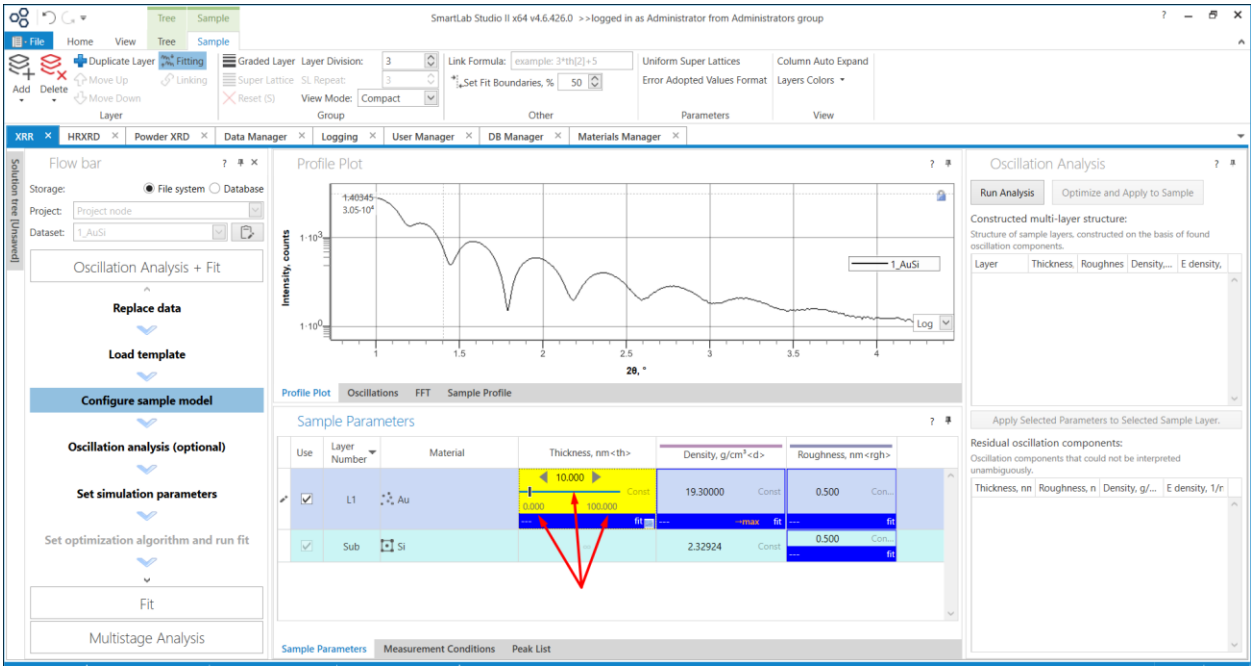
Press Configure sample model



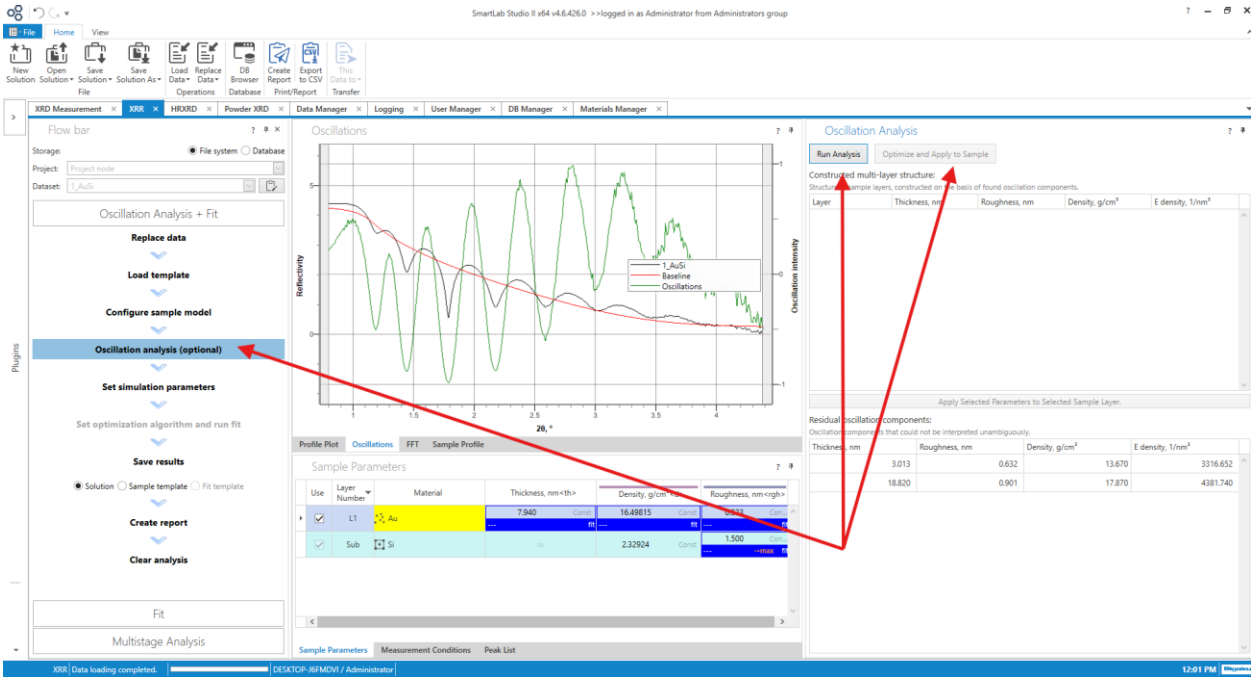
Add Layer and define material (and set it parameters of thickness, density and roughness. This step is optional)



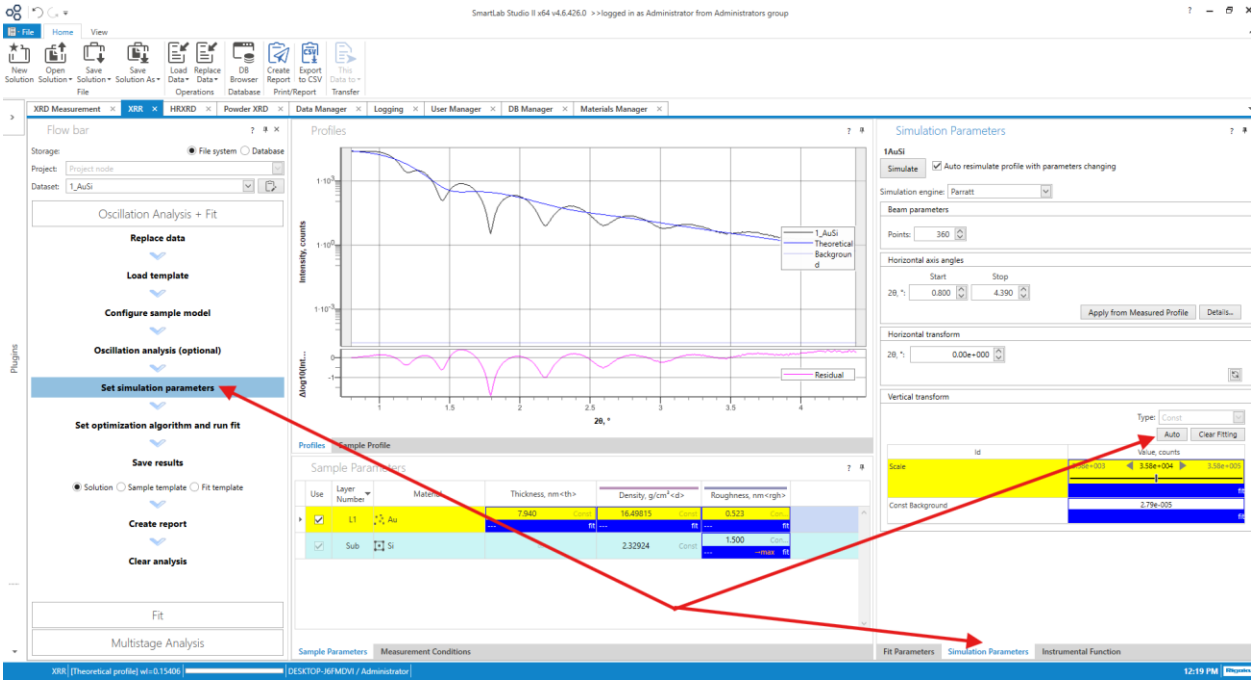
Here we can define thickness and the range of thickness and set this range by defining a minimum and maximum values



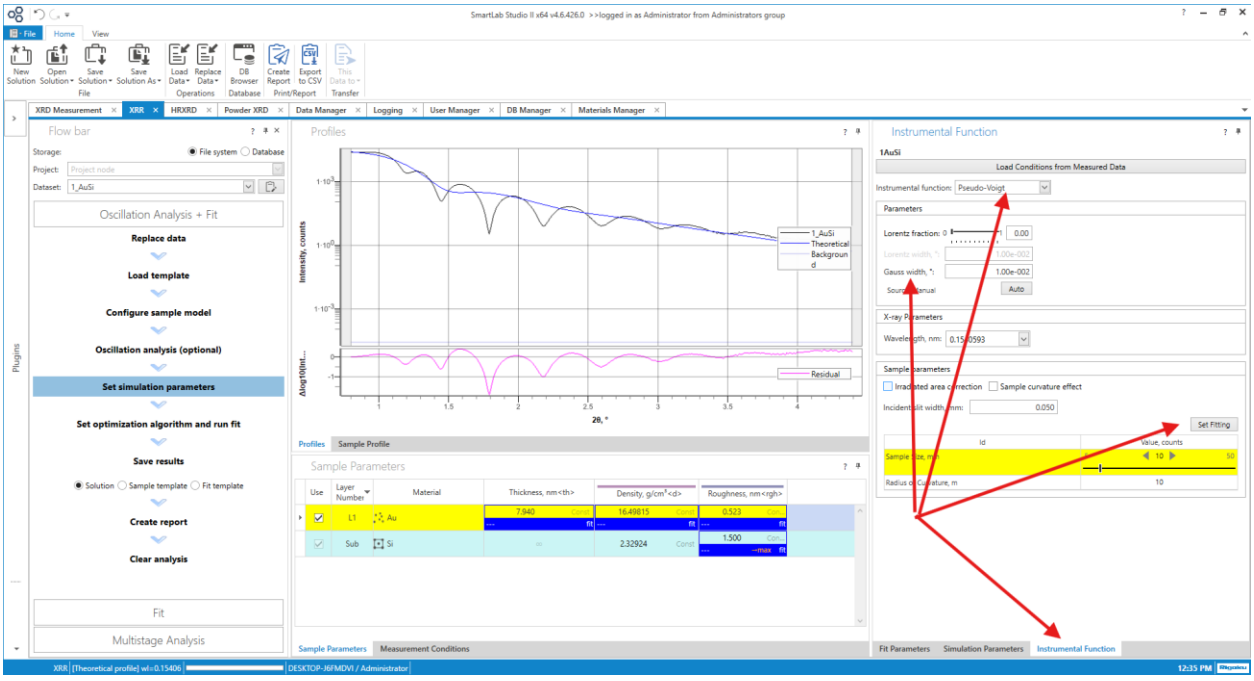
Select Oscillation Analysis and then Run Analysis + Optimize and Apply to Sample



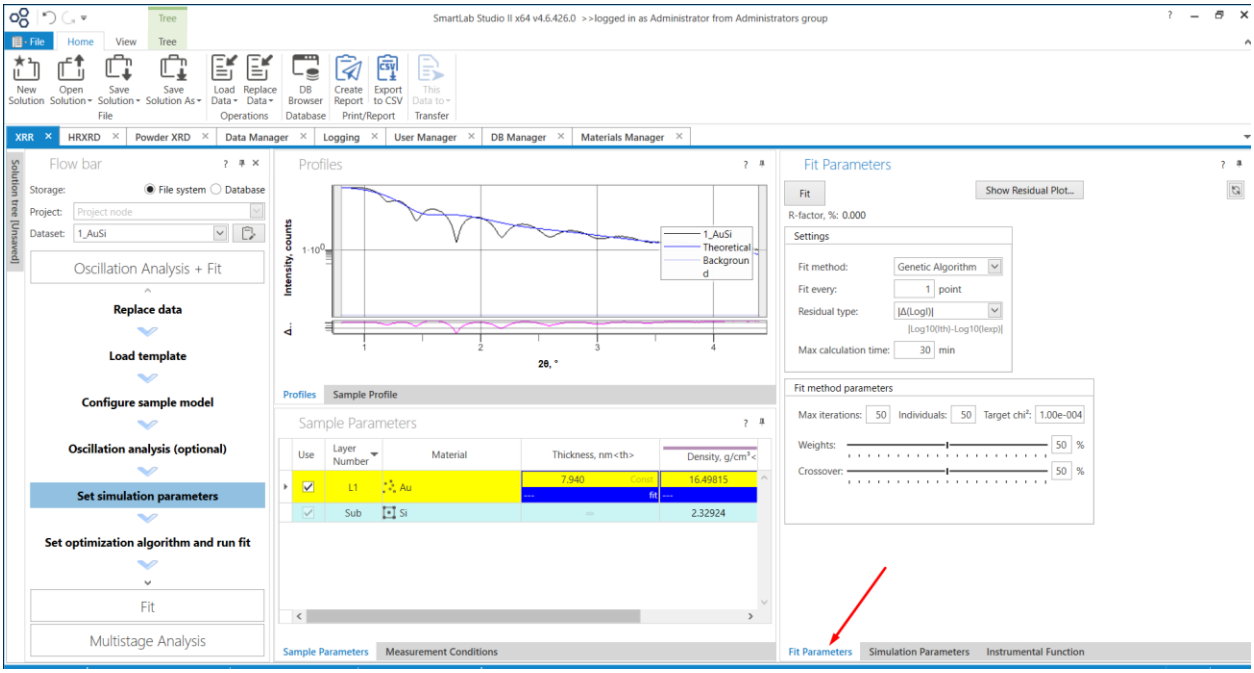
Set Simulation Parameters => Simulation Parameters => Auto



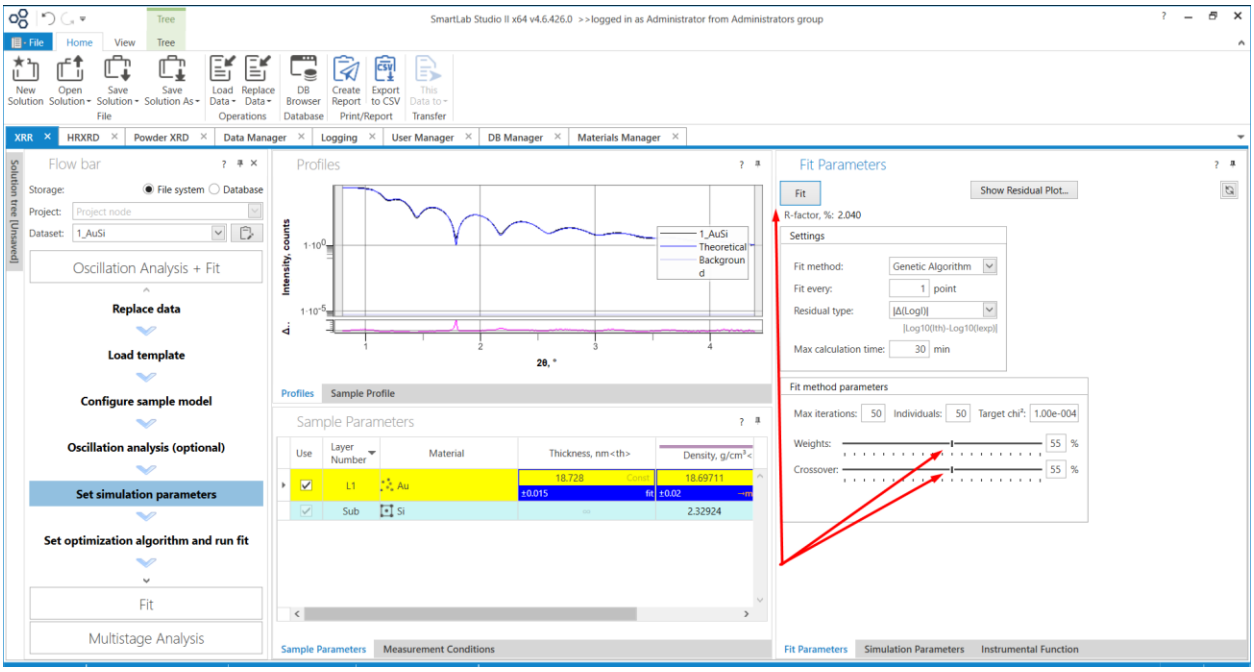
Select Instrumental Function and set Parameters and values (Gauss Width 1.00e-002) as given in arrows and press “Set Fitting”



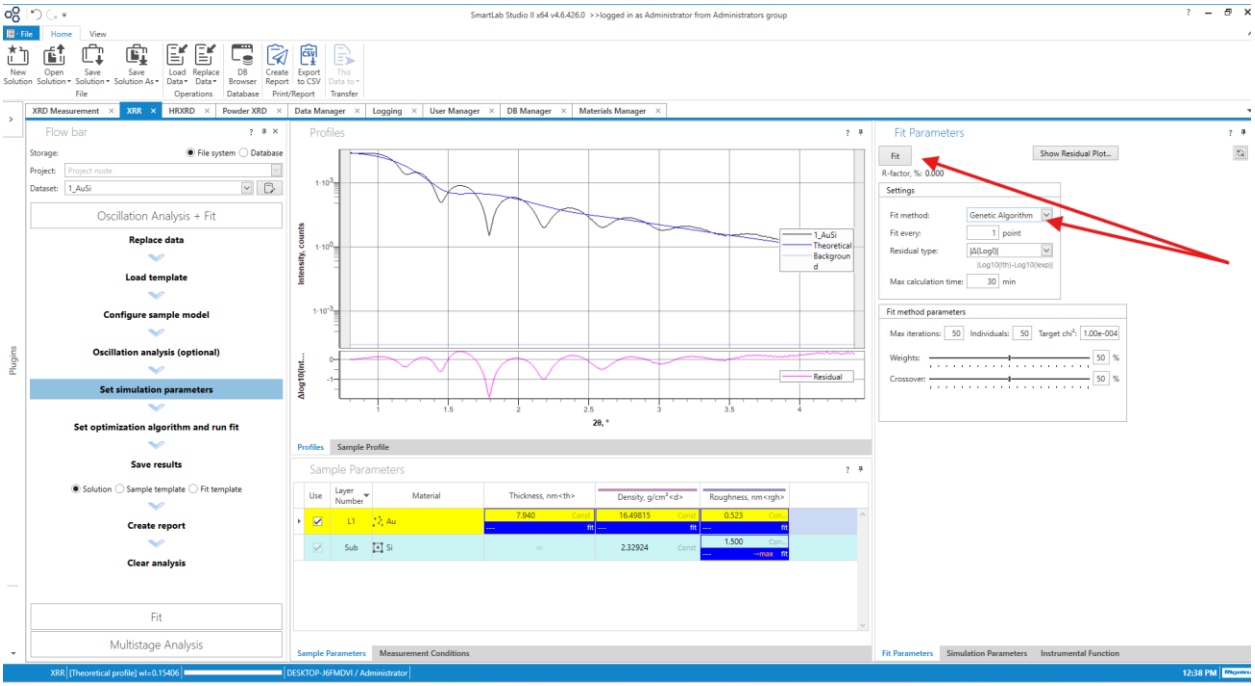
Select Fit Parameters from Genetic Algorithm, Newton Gaussian method, Nelder-Mead method and Parallel Tempring method



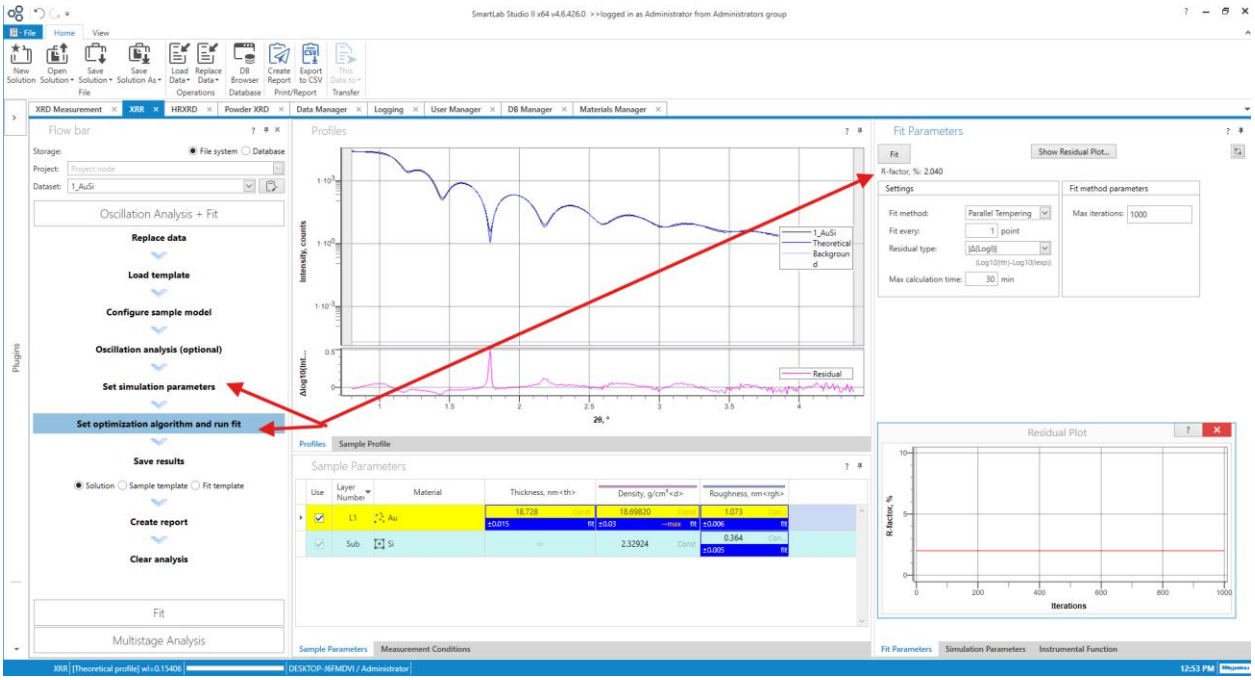
Change “Fit Method Parameters” from 50 to 55 or to 45, and press Fit, it will improve the fitness.



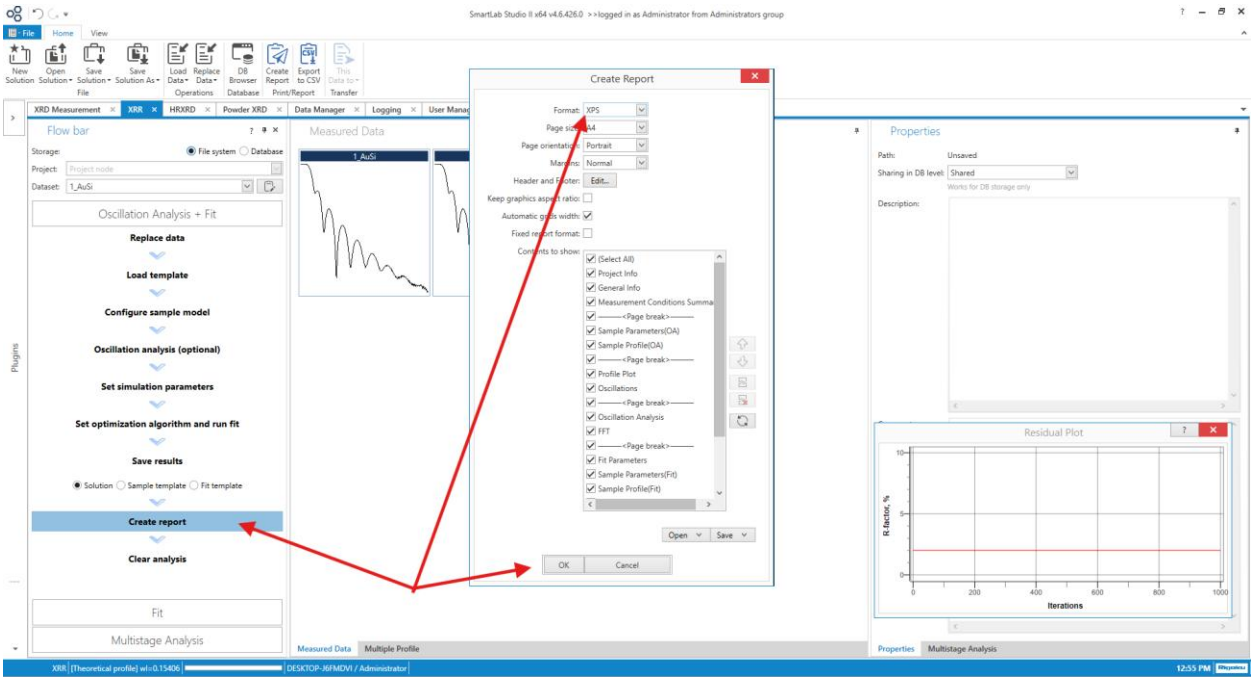
Run Fit with Genetic Algorithm (Select different Fit methods to improve the fitness)



Continue fitting till R-factor is minimized



Create Report



XRR Theory and Concepts

The Rigaku SmartLab SE can accurately measure thin films with thicknesses ranging from a few nanometers (nm) to several micrometers (µm) using X-ray reflectivity (XRR).

Typical Measurement Ranges:

- **Lower Limit**: For ultrathin films, XRR can accurately measure thicknesses as low as 1-2 nm. This is ideal for characterizing monolayers, ultra-thin coatings, and nanostructures.
- **Upper Limit**: XRR can also measure films up to a few micrometers thick, depending on the material and the quality of the measurement setup. For films thicker than this, the reflectivity may become difficult to analyze due to reduced oscillation visibility.

Factors Affecting Accuracy:

- **Surface Roughness**: Higher surface roughness can reduce the accuracy of thickness measurements.
- **Density and Composition**: The film's density and composition also play a role in determining the measurement range and accuracy.
- **Instrument Calibration**: Proper calibration and alignment of the SmartLab SE are crucial for achieving accurate results.

Thickness limit of the film for Accurate XRR measurements

In X-ray Reflectivity (XRR) measurements, the thickness of the thin film plays a significant role in achieving clear and accurate data. For the Rigaku SmartLab SE, the optimal thickness range is typically as follows:

Ideal Thickness Range for XRR on SmartLab SE

- Thin Films up to ~200 nm: XRR is well-suited for films with thicknesses from a few nanometers up to around 200 nm. In this range, the reflectivity oscillations (fringes) are clearly visible, allowing for precise measurement of thickness, density, and roughness.
- Limitations Beyond ~200 nm: As the film thickness increases beyond approximately 200 nm, the reflectivity fringes become closely spaced and harder to distinguish, making it challenging to perform accurate measurements. In some cases, up to 500 nm can be analyzed if the material has very sharp interfaces and low roughness, but accuracy decreases with increasing thickness.

Factors Impacting Thickness Limits

1. Density Contrast: If there's a strong density contrast between the thin film and the substrate, XRR can handle slightly thicker films.
2. Surface and Interface Roughness: Higher roughness blurs the fringes and limits the maximum measurable thickness.
3. Instrument Settings and Resolution: Using fine slits or a monochromator can enhance resolution, potentially allowing measurements for thicker films, though it's still limited.

If your films are significantly thicker and you want structural information, switching to X-ray Diffraction (XRD) might offer better insight into phase and crystallographic structure. For finer control on SmartLab SE, I'd recommend starting with test measurements to see if fringes are visible and adjusting parameters accordingly.

Why Oscillations Appear in XRR

The oscillations you see in X-ray reflectivity (XRR) measurements, known as **Kiessig fringes**, occur due to constructive and destructive interference between X-rays reflected from different interfaces in a thin film structure.

Mechanism of Kiessig Fringes:

1. **Incident X-ray Beam**: When X-rays strike a thin film at a low angle, part of the beam is reflected from the top surface, and part penetrates the film and reflects from the interface between the film and the substrate (or from internal layers in the case of multilayer structures).
2. **Interference**: The two reflected beams (from the top surface and the internal interface) can interfere with each other. Depending on the phase difference between them, this interference can be constructive (resulting in a higher reflected intensity) or destructive (resulting in a lower reflected intensity).

3. **Oscillations**: As the angle of incidence (and hence the path difference between the reflected beams) changes, this interference leads to oscillations in the reflected intensity, which manifest as the Kiessig fringes in the XRR pattern.

Information from Oscillations:

- **Film Thickness**: The period of the oscillations is directly related to the thickness of the film. Thicker films produce more closely spaced oscillations.
- **Density and Roughness**: The amplitude and decay of the oscillations can provide information about the film's density, surface roughness, and interface quality.

Why Oscillations Become Noise at Higher 2θ Angles

As you move to higher 2θ angles in XRR measurements, several factors contribute to the oscillations becoming less distinct and eventually appearing as noise:

1. **Decreasing Intensity**:
    - As the angle increases, the intensity of the reflected X-rays decreases exponentially due to the reduced reflectivity at higher angles.
    - With lower signal intensity, the fringe visibility diminishes, making the oscillations harder to distinguish from noise.
2. **Instrumental Limitations**:
    - The signal-to-noise ratio decreases at higher angles due to limitations in the detector sensitivity and the X-ray source's intensity.
    - Background noise and scattered radiation become more prominent, masking the weaker oscillations.
3. **Surface and Interface Roughness**:
    - Roughness at the film's surface or interfaces causes scattering, which can blur the fringes at higher angles.
    - If the roughness is significant, it can cause the oscillations to decay more rapidly and merge into the noise.
4. **Multiple Reflections and Complexity**:
    - At higher angles, multiple reflections and refractions within the film can lead to complex interference patterns that are difficult to resolve, further contributing to the noise.

For optimal data analysis, it's essential to focus on the range where the oscillations are clear and distinct, typically in the lower to mid-angle regions of the XRR spectrum.

It is generally advisable to focus on the area where the Kiessig fringes are clear and exclude the noisy region for XRR calculations when using the SmartLab SE. Here's why and how to approach this:

Why Focus on the Clear Kiessig Fringes?

1. **Accuracy of Data**:
    - The clear Kiessig fringes represent the reliable interference pattern that corresponds directly to the film's thickness, density, and roughness.
    - Using this well-defined region ensures that the fitting models accurately represent the physical characteristics of the film.
2. **Signal-to-Noise Ratio**:
    - Including the noisy regions in the calculation can degrade the signal-to-noise ratio, leading to less accurate parameter determination.
    - Noise can introduce artifacts in the data analysis, leading to incorrect conclusions about the film properties.
3. **Model Fitting**:
    - The fitting algorithms used in XRR analysis, such as those in the SmartLab SE software, work best with clear, well-defined oscillations.
    - Excluding the noisy data allows the algorithm to focus on the meaningful data, improving the fit quality and the accuracy of the extracted parameters.

How to Focus on the Kiessig Fringes in SmartLab SE:

1. **Data Trimming**:
    - **Select Range**: When setting up your analysis, select the 2θ range that includes the clear Kiessig fringes. This typically involves starting from the critical angle and extending through the region where the fringes are distinct.
    - **Exclude Noise**: Cut off the data at the point where the oscillations become indistinguishable from noise.
2. **Fitting the Data**:
    - Use the SmartLab SE's software to fit the XRR data within the selected range.
    - The software may allow you to manually adjust the fitting range, ensuring that the focus remains on the usable data.
3. **Cross-Validation**:
    - After fitting, cross-validate the results by considering physical parameters such as known film density and expected thickness. This helps confirm that the fitting model accurately reflects the sample's characteristics.

The Rigaku SmartLab SE offers four fitting methods for XRR analysis, which typically include different optimization algorithms and approaches to model the reflectivity data. While it may be tempting to use all four methods to cover all bases, it's essential to understand their strengths and how to choose the most appropriate one for your specific analysis. Here's a guideline on how to approach this:

Understanding the Fitting Methods

- **Levenberg-Marquardt (LM) Algorithm**:
    - **Strength**: This method is widely used for non-linear least squares optimization and is effective for converging quickly when the initial model is close to the actual data.
    - **Use When**: Your initial parameters are well-known, and you need a fast, reliable fit.
- **Genetic Algorithm (GA)**:
    - **Strength**: GA is a global optimization method that is particularly useful when you have little information about the initial parameters or when the parameter space is large and complex.
    - **Use When**: The model fitting may involve multiple local minima, and a broader search of the parameter space is required.
- **Simulated Annealing (SA)**:
    - **Strength**: This method is another global optimization technique that is less likely to get stuck in local minima, providing robust solutions over complex parameter spaces.
    - **Use When**: The data may involve a complex landscape of solutions, and you need to ensure that the global minimum is found.
- **Powell's Method**:
    - **Strength**: This method is a direction-set optimization technique, which can be faster but might not always find the global minimum.
    - **Use When**: The problem is well-behaved and doesn't require a global search.

Should You Use All Four Methods?

Advantages of Using Multiple Methods:

- **Cross-Verification**: Running multiple fitting methods can provide a way to cross-verify results. If different methods converge on similar parameters, this increases confidence in the fit.
- **Robustness**: By trying different methods, you can ensure that the solution isn't biased by the choice of the fitting algorithm, especially in complex or poorly understood systems.

Potential Downsides:

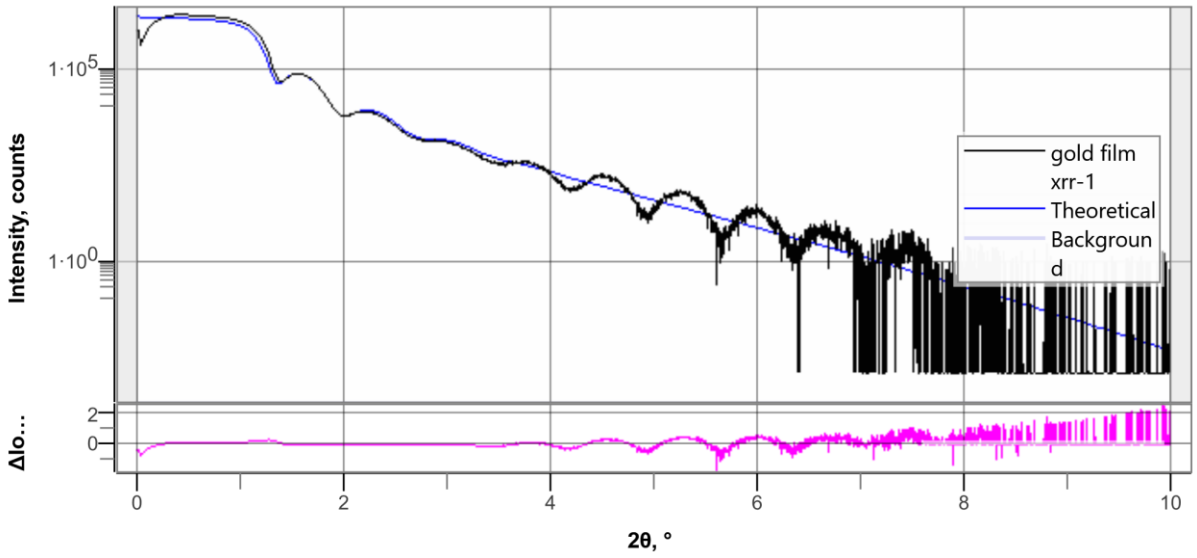
- **Time-Consuming**: Running all four methods can be time-consuming, especially if you have a large number of samples or if some of the methods (like GA or SA) take longer to converge.
- **Overfitting**: There's a risk of overfitting if you choose the results that simply give the best numerical fit without considering the physical meaning of the parameters.

Recommended Approach

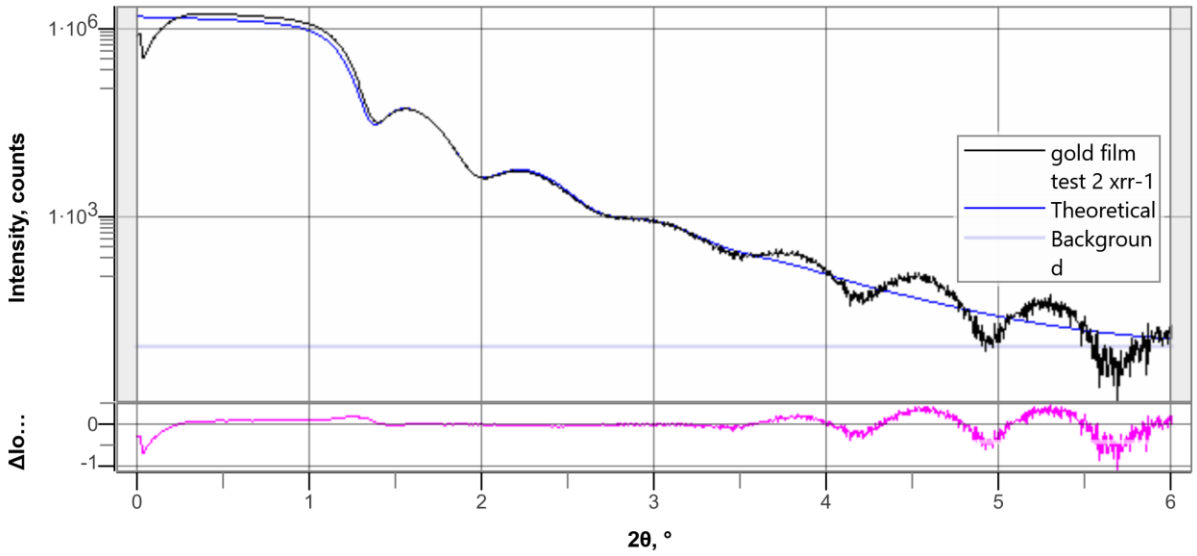
- **Start with a Robust Method**: Begin with a method like the Genetic Algorithm (GA) or Simulated Annealing (SA) if you suspect the model is complex or if you're unsure about initial parameters. These methods are more likely to find a global minimum.
- **Fine-Tuning with LM**: Once you have a general fit from a global method, use the Levenberg-Marquardt (LM) method to fine-tune the fit, as it is faster and often more precise when close to the solution.
- **Cross-Verification**: If the fitting is critical, or if you're dealing with an unfamiliar material, run another method like Powell's for cross-verification.
- **Check Consistency**: Compare the results across methods. Consistent results reinforce the reliability of the parameters. Significant discrepancies may indicate that the model needs adjustment or that certain parameters are not well-constrained.

Using all four methods can be beneficial for cross-validation, but it may not always be necessary. A practical approach is to use a global method like GA or SA for initial fitting, followed by LM for refinement. If the results are critical, consider using an additional method for verification. This strategy balances thoroughness with efficiency.

The 2θ range Selection for the best XRR results



Layer Numb er	Material	Thickness, nm<th>		Density, g/cm³<d>		Roughness, nm<rgh>	
L1	Au	10.579	±0.06	17.64665	±0.19	0.405	±0.003
Sub	Si			2.32924		1.072	±0.017



Layer Numb er	Material	Thickness, nm<th>		Density, g/cm³<d>		Roughness, nm<rgh>	
L1	Au	10.609	±0.03	18.13896	±0.09	0.454	±0.005
Sub	Si			2.32924		1.110	±0.01

The 2θ range is crucial in XRR measurements, as it determines the quality of the reflectivity data and the ability to accurately model the thin film's properties.

Interpreting the Graph:

1. **Low-Angle Region (0.1° to 1°)**:
    - This region is where total external reflection occurs, and it provides information about the surface roughness and density of the film.
    - The critical angle (around 0.2° to 0.4° in many materials) can be seen in this region.
2. **Mid-Angle Region (1° to 3°)**:
    - This is where the oscillations (known as Kiessig fringes) appear. These fringes are related to the thickness of the film and are essential for determining the film's thickness accurately.
    - For thin films, capturing several oscillations is crucial to obtain a precise fit during analysis.
3. **High-Angle Region (3° to 6° and beyond)**:
    - As the angle increases, the intensity of the reflected X-rays decreases, and the fringes may become less pronounced or disappear.
    - This region may provide information about the interface roughness and additional layers if present.

Recommended 2θ Range:

- **Starting Angle**: Around 0.1° to 0.2°, to capture the critical angle and total reflection.
- **Upper Limit**: Typically, a range up to 4° to 6° is sufficient for most thin films. However, if you see that the oscillations continue beyond 6°, you might want to extend the range to 8° or 10°.

For Your Sample:

Based on the graph for 2θ range from 0 to about 10°:

- **Ideal Range**: A 2θ range from **0.1° to about 4°-5°** seems appropriate for this measurement.
- **Adjustment**: If you notice more oscillations or features at higher angles, consider extending the range slightly beyond 5° to capture more data.

This range should provide a good balance between capturing the critical features and maintaining measurement efficiency. Adjustments can be made depending on the specific requirements of your analysis.

Significance of X-ray reflectivity (XRR) Kiessig fringes

X-ray reflectivity (XRR) fringes, also known as Kiessig fringes, arise due to interference between X-rays reflected from different interfaces within a thin film sample, such as between the film surface and the film-substrate interface. These fringes are highly informative for thin film analysis, and their characteristics—such as amplitude, spacing, and shape—can reveal important details about the sample.

Significance of XRR Fringes:

1. **Thickness of the Film:**
    - The spacing between fringes is inversely proportional to the film thickness. Closer fringes indicate a thicker film, while wider fringes represent a thinner film. This is one of the primary pieces of information extracted from XRR measurements.
2. **Density of the Film:**
    - The amplitude of the fringes is related to the electron density contrast between the layers (film and substrate). Higher density contrast between the layers will produce higher fringe amplitudes, while lower contrast will lead to smaller amplitudes. For instance, if a film is denser than the substrate, the fringes will have a higher amplitude.
3. **Surface and Interface Roughness:**
    - The sharpness or decay of the XRR fringe amplitude also depends on the roughness of the film surfaces and interfaces. Higher roughness tends to dampen the fringe amplitude more quickly as the rough surface causes scattering, reducing the coherent interference necessary for strong fringes. Smooth films will maintain higher amplitudes over a longer range.

Why Some Samples Have Higher Amplitude Fringes:

- **Higher Density Contrast:** A greater difference in density between the film and substrate results in larger amplitude fringes.
- **Lower Surface/Interface Roughness:** If the film has a smooth surface and a well-defined interface with the substrate, the fringes will have a higher amplitude.

Why Some Samples Have Smaller Amplitude Fringes:

- **Lower Density Contrast:** A smaller difference in density between the film and substrate will reduce the fringe amplitude.
- **Higher Surface/Interface Roughness:** Increased roughness scatters X-rays and diminishes the coherence of reflected beams, leading to reduced fringe amplitudes and faster damping.

In summary, the amplitude of XRR fringes provides insight into the material's density contrast and the roughness of its surfaces and interfaces.

Gold Film Results

Path: Unsaved
Sharing in DB level: Shared
Content:
Oscillation analysis: 1

Fits: 1
Measured data: 2

General Info

Analysis date	05/09/2024 4:50:42 pm	Measured data name	gold film xrr-3.rasx
Analyst	Administrator	Measurement start time	2024-09-04 10:43:15
Sample name	Sample	Comment	
Sample name	Sample		

Measurement Conditions Summary

gold film xrr-3

X-ray generator	40 kV, 50 mA	Scan step	0.002 °
Scan mode	0D(continuous)	Scan axis	2θ/ω
Scan speed	0.04 °/min		

gold film xrr-3

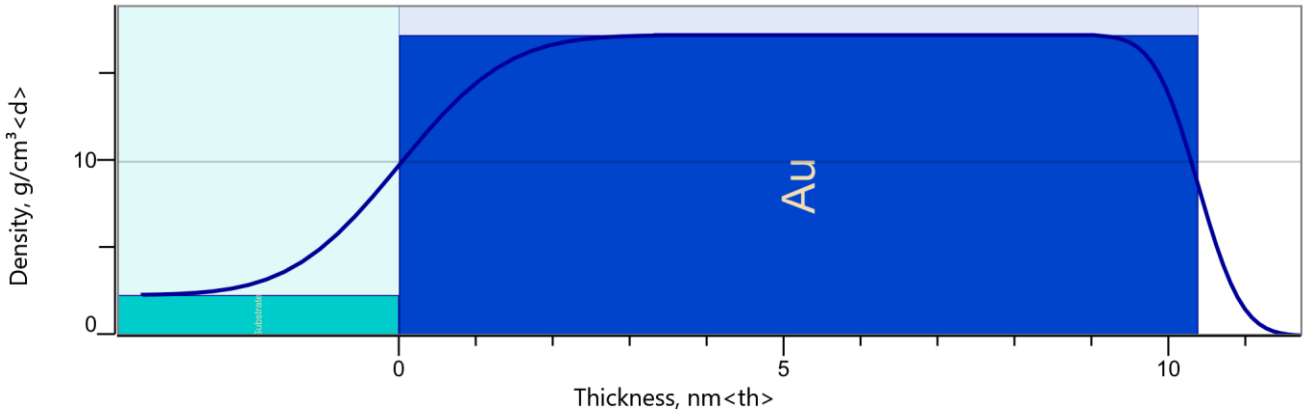
X-ray generator	40 kV, 50 mA	Scan step	0.002 °
Scan mode	0D(continuous)	Scan axis	2θ/ω
Scan speed	0.04 °/min		

Oscillation Analysis

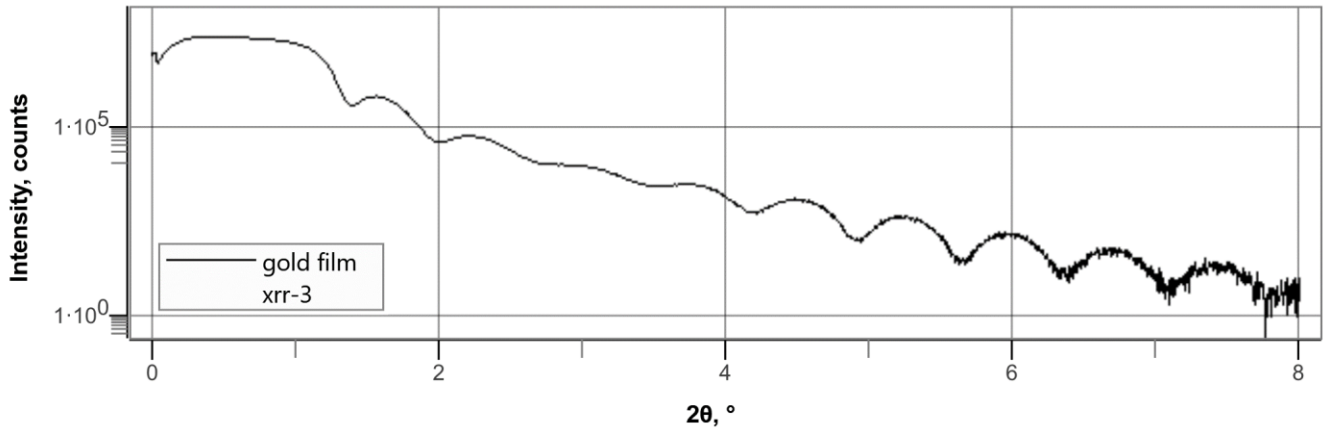
Sample Parameters

Use	Layer Number	Material	Thickness, nm<th>		Density, g/cm³<d>		Roughness, nm<rgh>	
v	L1	Au	10.371	---	17.18074	---	0.451	---
v	Sub	Si			2.32924		1.109	---

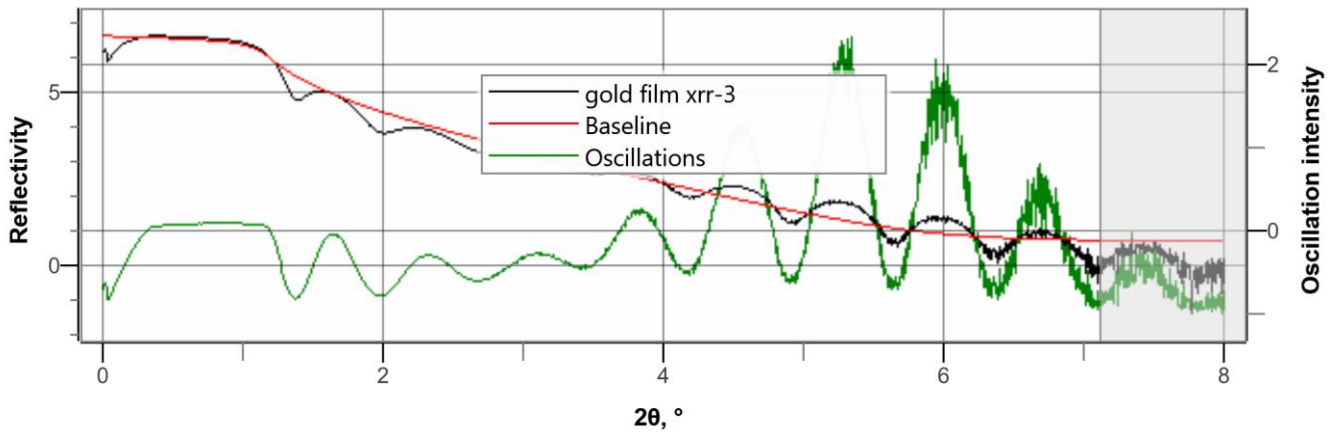
Sample Profile



Profile Plot



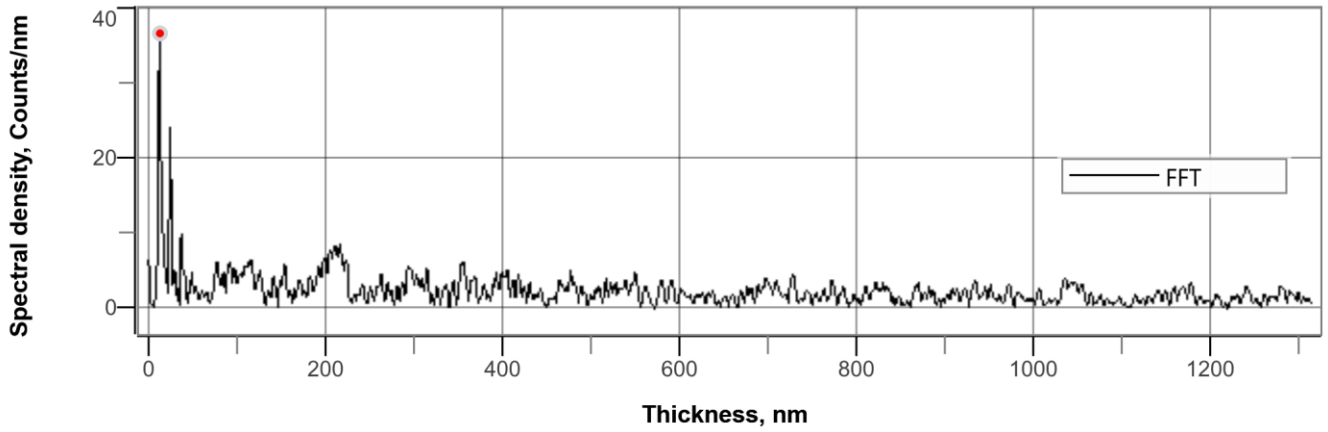
Oscillations



Residual oscillation components:

Thickness, nm	Roughness, nm	Density, g/cm³	E density, 1/nm³
1.696	0.494	2.788	814.563
11.908	0.678	12.149	2972.283

FFT



Fit container

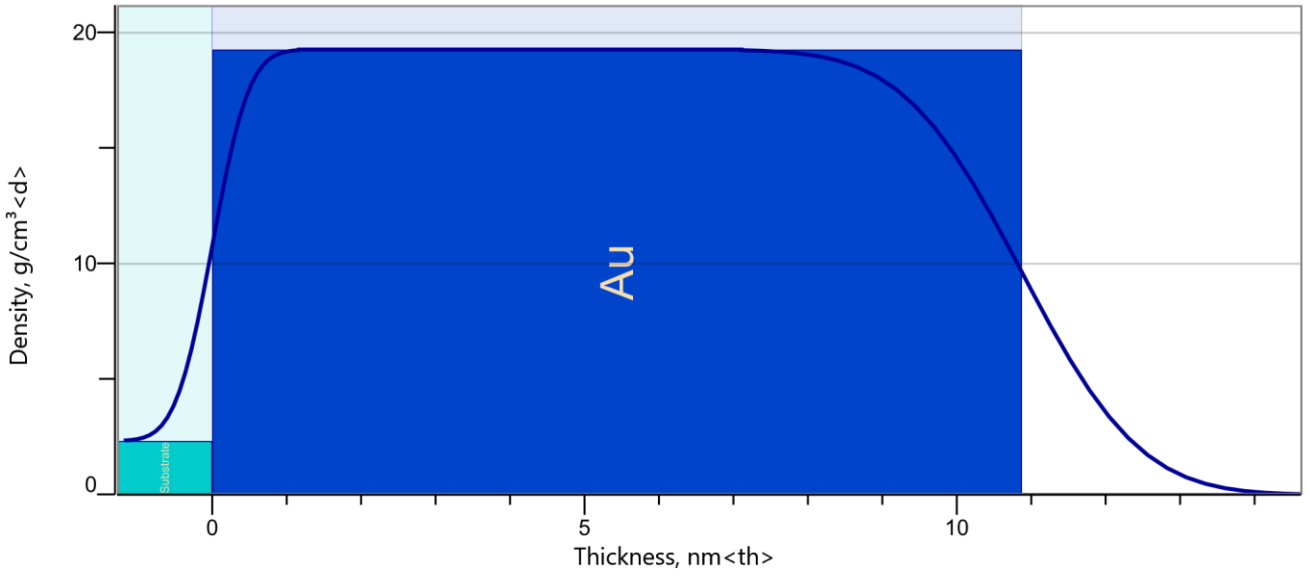
Fit Parameters

R-factor, %: 3.920
Fit every: 1 point
Residual type: |Δ(LogI)|
Max iterations: 500
Tolerance: 1.00e-010

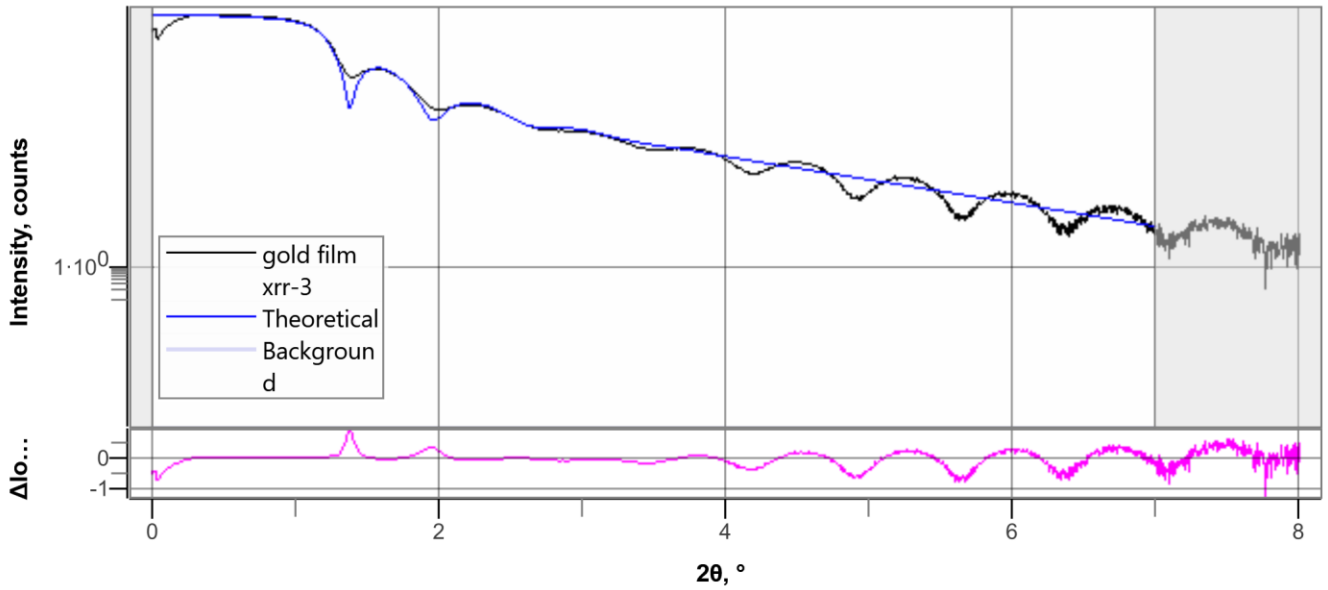
Sample Parameters

Use	Layer Numbe r	Material	Thickness, nm<th>		Density, g/cm³<d>		Roughness, nm<rgh>	
v	L1	Au	10.860	±0.06	19.25634	±0.2	1.254	±0.03
v	Sub	Si			2.32924		0.388	±0.004

Sample Profile



Profiles



Fit ranges: 0° - 7°

Simulation Parameters

Points: 2001
2θ,°: 0.000 - 8.000
step=0.004
offset=0.000e+000

Instrumental Function

Instrumental function: Pseudo-Voigt
Lorentz fraction: 0.00
Lorentz width: 1.00e-002
Gauss width: 1.00e-002
Irradiated area correction: Off
Sample curvature effect: Off
Radius of curvature, m: 10
Sample size, mm: 10
Wavelength, nm: 0.1540593

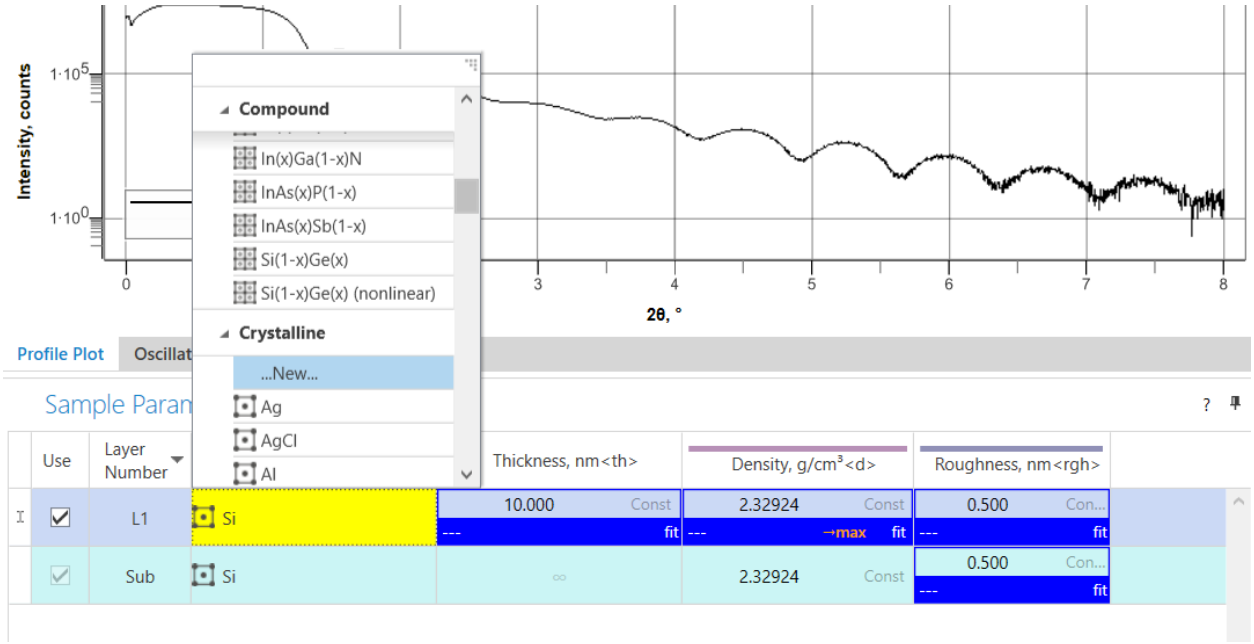
Handling Materials Not in the Database

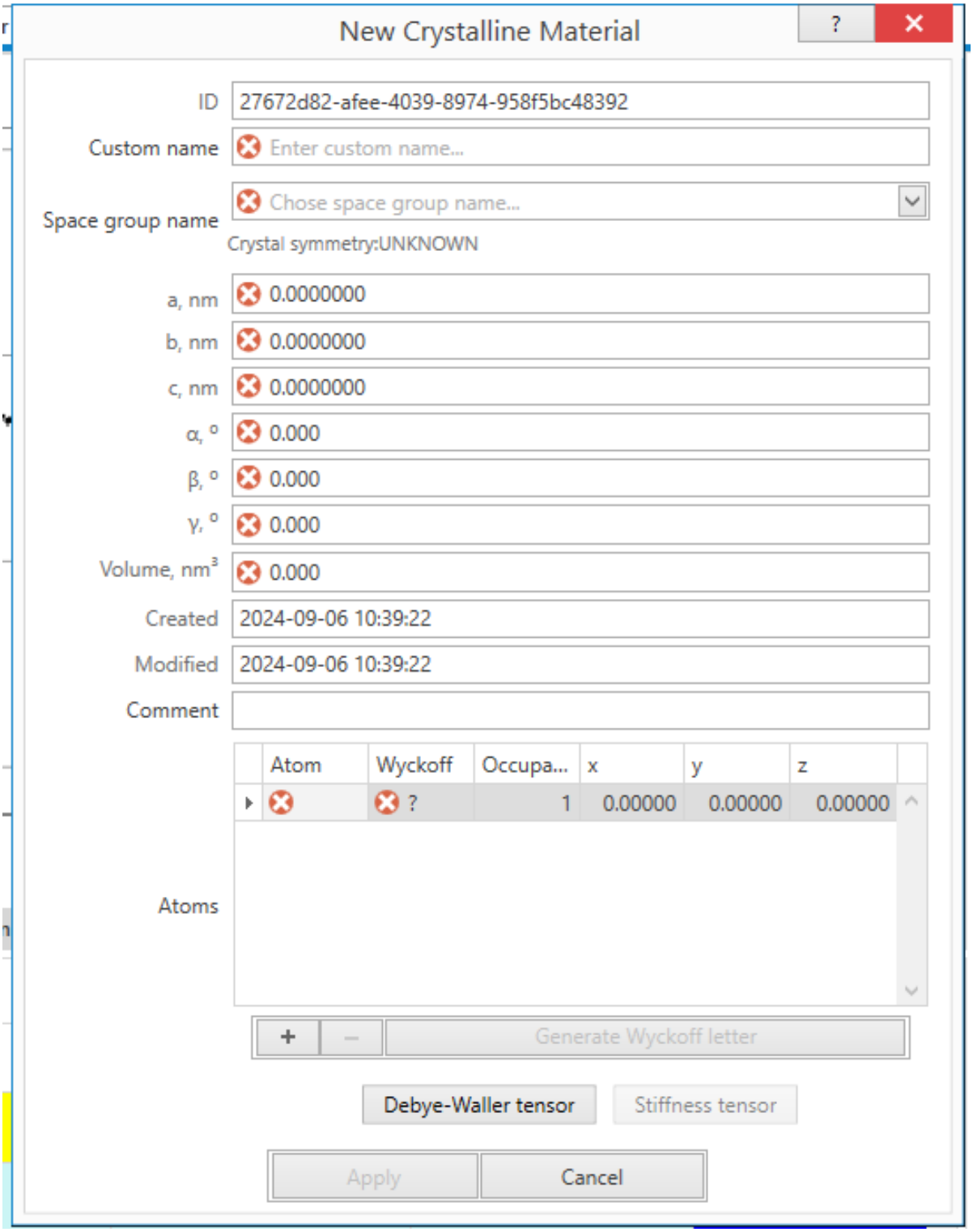
If the material is not available in the database for XRR measurements, you can still perform analysis by manually defining key parameters of the material in the XRR software. Here's what you can do:

1. **Input Known Material Properties**: Enter the known properties such as density, refractive index (δ and β values), and atomic composition. These values can usually be found in scientific literature or through separate material analysis.
2. **Estimate Unknown Properties**: If certain properties like density or thickness are unknown, you can still perform the measurement and use the software to fit these parameters based on the measured reflectivity curve.
3. **Use Similar Materials**: If the exact material isn't available, you can select a similar material from the database and modify its properties according to your needs. This works well if you're working with a material of similar density or atomic composition.
4. **Custom Database Entry**: Some advanced XRR software allows you to create custom entries in the database by defining the material's characteristics, allowing you to reuse it for future measurements.
5. **Fitting Models**: The software often provides fitting algorithms (e.g., Levenberg-Marquardt, Genetic Algorithm, Simulated Annealing) that can help optimize the unknown parameters based on the reflectivity data.

By manually adjusting these parameters, you can still achieve accurate XRR results even when the material is not pre-defined in the system's database.

For new crystalline material




New Crystalline Material
ID
27672d82-afee-4039-8974-958f5bc48392
Custom name
Enter custom name...
Space group name
Chose space group name...
Crystal symmetry:UNKNOWN
a, nm
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b, nm
0.0000000
c, nm
0.0000000
α, °
0.000
β, °
0.000
γ, °
0.000
Volume, nm³
0.000
Created
2024-09-06 10:39:22
Modified
2024-09-06 10:39:22
Comment
Atom
Wyckoff
Occupa...
x
y
z
?
1
0.00000
0.00000
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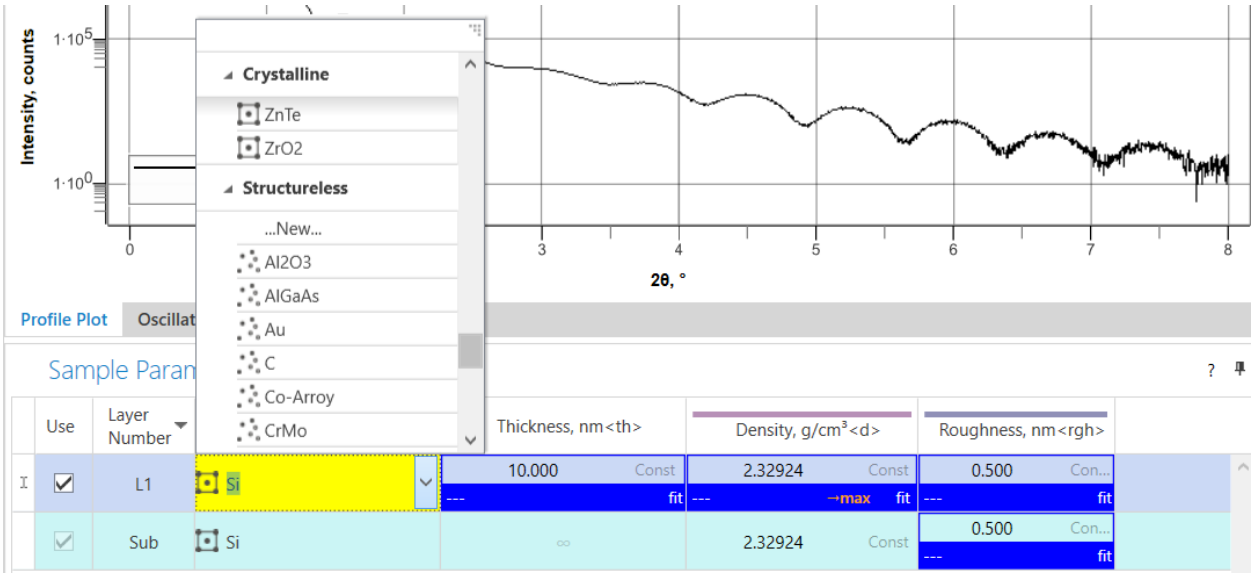
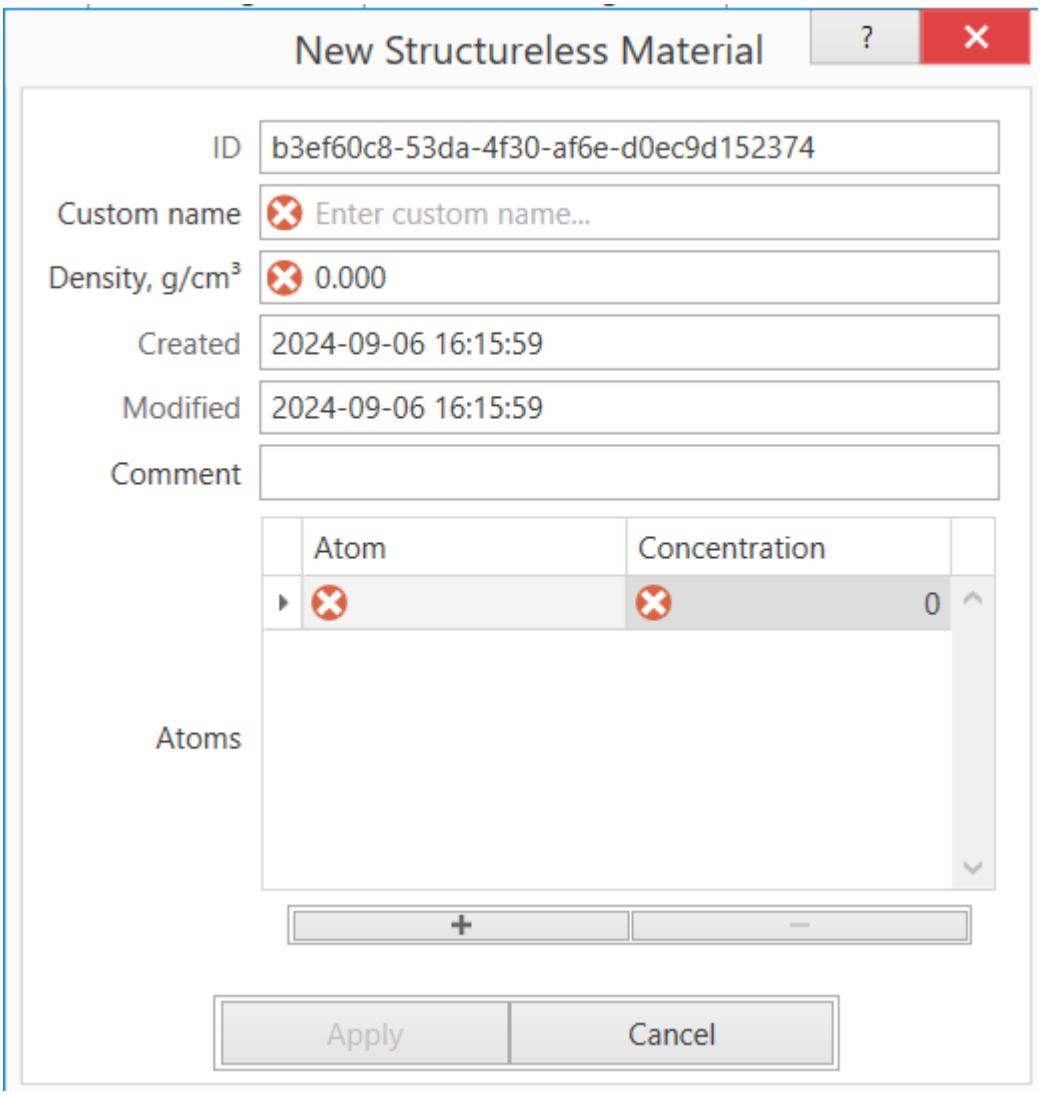
Atoms
Generate Wyckoff letter
Debye-Waller tensor
Stiffness tensor
Apply
Cancel

For new structureless material





To perform XRR analysis for a new crystalline material in the Rigaku SmartLab SE software, you need to input accurate crystallographic parameters. Here's how you can source the correct values:

1. **Lattice Constants (a, b, c, α, β, γ):** Lattice constants refer to the set of six parameters that define the geometry of a unit cell in a crystal lattice
2. **Space Group:** It specifies the symmetry group of the crystal lattice, which defines how the unit cell repeats in space.
3. **Wyckoff Positions:** It represents the symmetry-equivalent positions of atoms in the unit cell based on the space group.
4. **Atom Types and Coordinates (x, y, z):** Atomic coordinates are usually expressed in terms of fractional coordinates, (x, y, z). This coordinate system coincides with the cell axes (a, b, c) and relates to the position of the atom in terms of the fraction along each axis.
5. **Volume:** The calculated volume of the unit cell based on the lattice parameters and angles

Recommended Approach:

1. **Search in Databases:** Use crystallographic databases like ICSD (Inorganic Crystal Structure Database), COD (Crystallography Open Database) or Pearson's Crystal Data to search for your material or closely related ones.
2. **Experimental Methods:** If your material is new or unavailable in databases, perform XRD or similar characterization techniques to gather the necessary lattice parameters and atomic coordinates.
3. **Literature Review:** You can source this data from specialized literature or material property databases such as Materials Project or MatWeb. If the material is not in the database, starting with a closely related material for initial analysis and refining the parameters experimentally would be a good approach.