

# Thin Film XRD Training Notebook

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# Before you begin...

- ☐ Complete the required safety training modules on LMS
  - ☐ Laboratory Safety Fundamentals
  - ☐ Hazardous Waste Management
  - ☐ Radiation Safety For Users of Radiation Producing Machines
  - ☐ Compressed Gas Safety
- ☐ Submit a copy of your Training Transcript to Lab Manager
- ☐ Review the MSE Thin Film XRD Policies and Regulations
- ☐ Fill out the Thin Film XRD FAU Authorization Form with PI signature
- ☐ Receive a user name and temporary password for Faces scheduling
- ☐ Arrange a time for Thin Film XRD training with Lab Manager
- ☐ Schedule a 2 hour block on Faces for your training
- ☐ Receive a SmartLab II password

# Thin Film XRD (Rigaku SmartLab II) Operation

- A. XRD Cabinet Overview
- B. Measurement Basics
- C. GUI Basics
  - I. Startup
  - II. XRD Detector
  - III. XRD Optics
  - IV. XRD Sample Attachment
  - V. RS Viewer
  - VI. Utility Activity
  - VII. General (PB) or  $2\theta/\omega$  Scan
  - VIII. Azimuth or Phi ( $\varphi$ ) Scan
  - IX. Reflectivity
  - X. Pole Figure
  - XI. Rocking Curve
  - XII. Reciprocal Space Map (RSM)

- XIII. In-Plane Measurement or  $2\theta_\chi/\varphi$
- XIV. In-Plane Azimuth or Phi ( $\varphi$ ) Scan
- XV. In-Plane Pole Figure
- XVI. In-Plane RSM
- XVII. Monochromator Ge(220)x2
- XVIII. Grazing Incidence XRD or GIXRD
- XIX. Clean-up and Shutdown
- XX. Overnight Scan + Shutdown

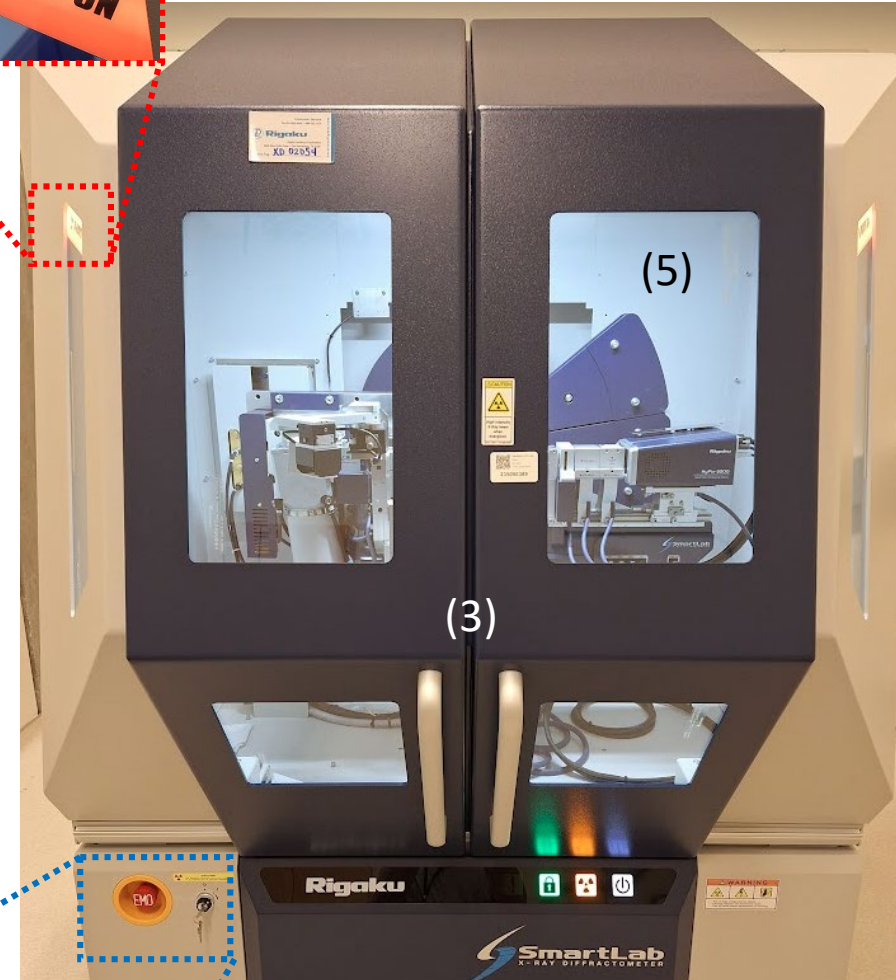
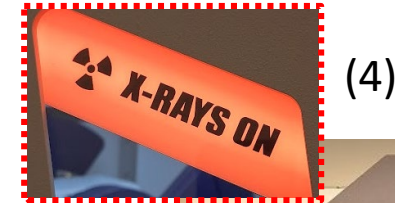
## Troubleshooting

- A. Initial Power Up
- B. Hypix Detector Troubleshooting

# A. XRD Cabinet Overview – 1/2

- This covers the Rigaku SmartLab II XRD Cabinet and its components

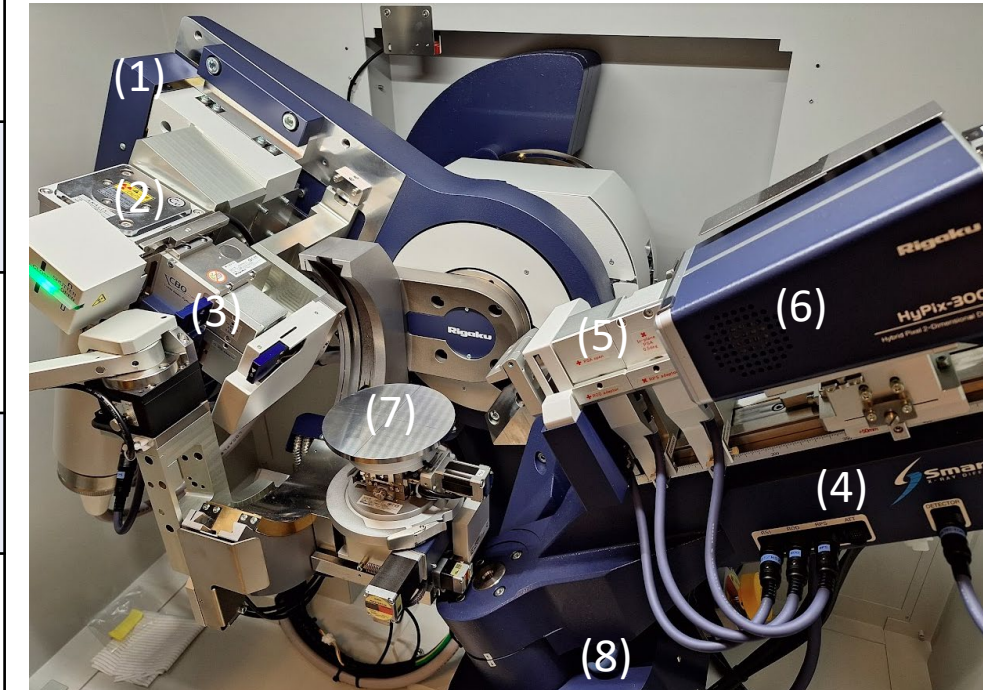
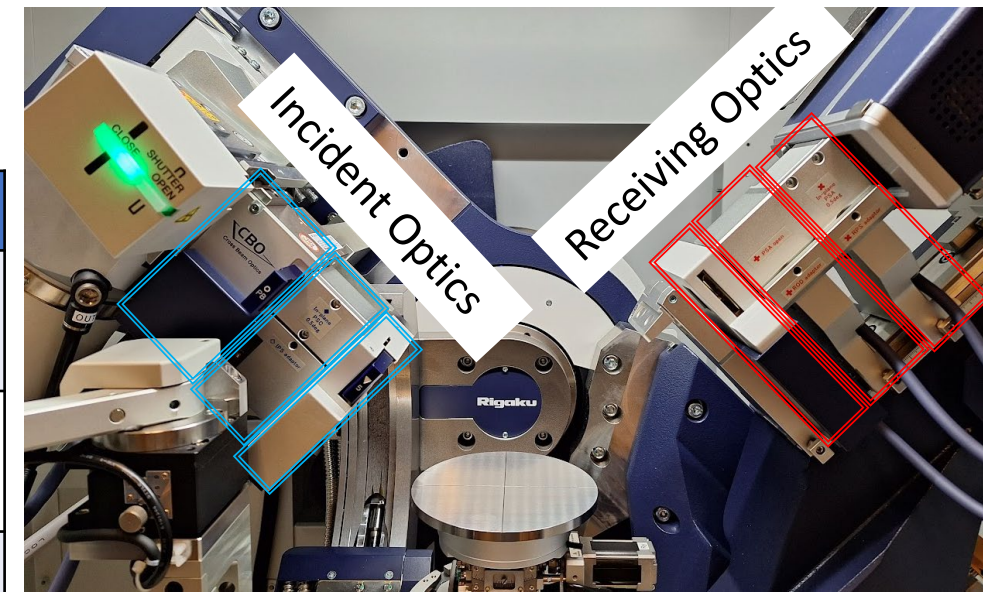
- (1) Power Key: Power key used to start and stop SmartLab
- (2) Emergency OFF Switch: Press this switch in the event of an emergency to cut off the power supply to the main unit
- (3) Door: Opened to change samples and optical devices
- (4) X-Rays ON Lamp: Lights when X-rays are generated
- (5) Observation Window: Window used to observe the inside of the cabinet
- (6) Power-on Indicator: Lights when SmartLab is powered ON
- (7) X-RAYS ON Indicator: Lights when X-rays are generated
- (8) Door-Lock Indicator: Lights when the door is locked
- (9) Alarm Indicator: Flashes when an error occurs





# A. XRD Cabinet Overview – 2/2

(#)	Section	Description
1	Theta_s ( $\theta_s$ ) arm	Arm for controlling X-ray beam incident angle
2	X-ray Generator	X-ray generating device
3	Incident Optics	Optical devices for desired incident X-ray conditions
4	Theta_d ( $\theta_d$ )Arm	Arm for controlling the X-ray detector angle
5	Receiving Optics	Optical devices for desired X-ray receiving conditions
6	Detector	X-ray detector
7	Sample	Adjusts the position and orientation of sample
8	In-Plane Arm ( $\theta_\chi$ )	Theta_d arm used for In-Plane measurements



# B. Measurement Basics – 1/10

- This summarizes the different Scans and Information obtained

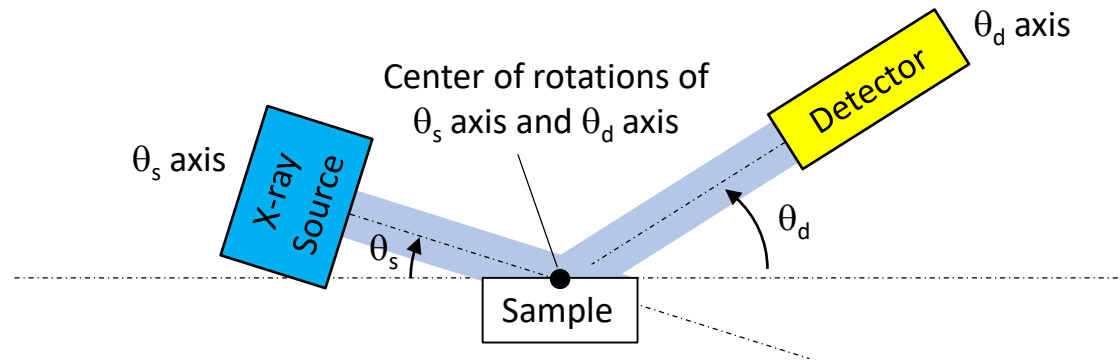
Measurement Technique (Scan)	Information Obtained		Scan Axis
Out-of-Plane (1D)	Information on lattice planes parallel to sample surface → Qualitative analysis and crystal structure		$2\theta/\omega$ (Always $2\theta = 2 \times \omega$ )
Thin Film (1D)	Information near sample surface (applies only to unoriented samples) → Qualitative analysis and crystal structure		$2\theta$ (Incident angle, $\omega$ , is fixed near the critical angle)
In-Plane (1D)	Information on lattice planes near and perpendicular to sample surface → Qualitative analysis and crystal structure		$2\theta_{\chi}/\varphi$ (Incident angle, $\omega$ , is fixed near the critical angle)
Pole Figure (2D)	Information on distribution of specific crystal orientation → Orientation analysis		$\chi(\alpha), \varphi(\beta)$ ( $2\theta$ or sum of $2\theta$ and $2\theta_{\chi}$ is fixed at the diffraction angle)
Preferred orientation and crystallinity measurement (1D)	Information on degree of preferred orientation or crystallinity → Orientation and crystallinity analysis		$\omega, \chi, \text{ or } \varphi$
Rocking Curve (1D)	Information on film structure and crystallinity of epitaxial or single crystal → Crystallinity, film thickness, and composition ratio		$2\theta/\omega$
Reciprocal Space Map or RSM (2D)	Information on d-value of 3-Dimensional components of preferred orientation, crystal orientation, and degree of preferred orientation → Qualitative analysis, orientation analysis, and crystallinity analysis	Information on film structure and crystallinity of epitaxial or single crystal → Crystallinity analysis and epitaxial analysis	$2\theta/\omega, \omega (\chi \text{ or } \varphi)$ $2\theta_{\chi}/\varphi, \varphi (\chi \text{ or } \varphi)$
Reflectivity (1D)	→ Film thickness, density, and surface or interface roughness by fitting		$2\theta/\theta$

# B. Measurement Basics – 2/10

- This covers the Goniometer Optics and Measurement Axes

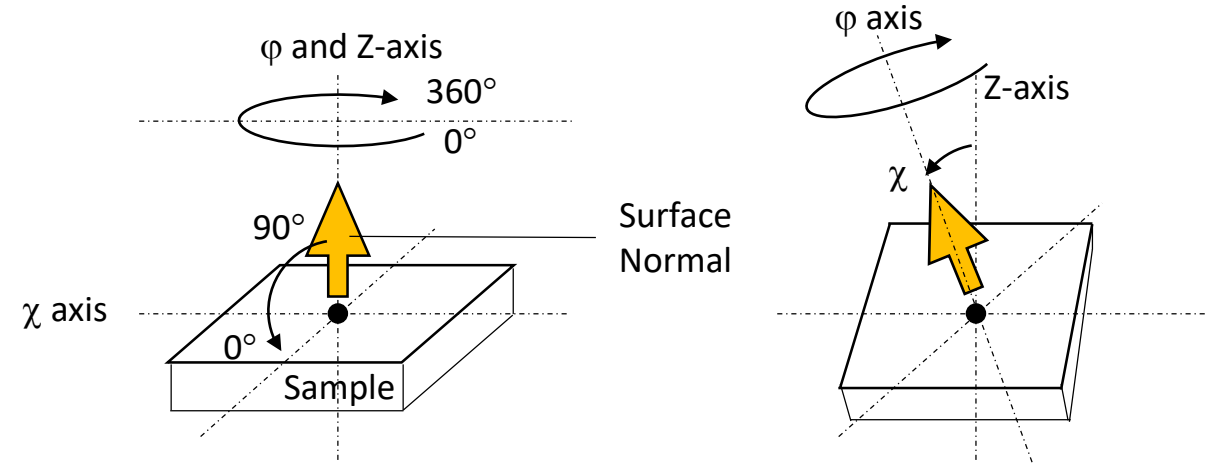
$\theta_s$  axis = axis for setting angle of X-ray Source with respect to the Sample Surface  
(s = "source")

$\theta_d$  axis = axis for setting angle of Detector with respect to the Sample Surface  
(d = "detector")



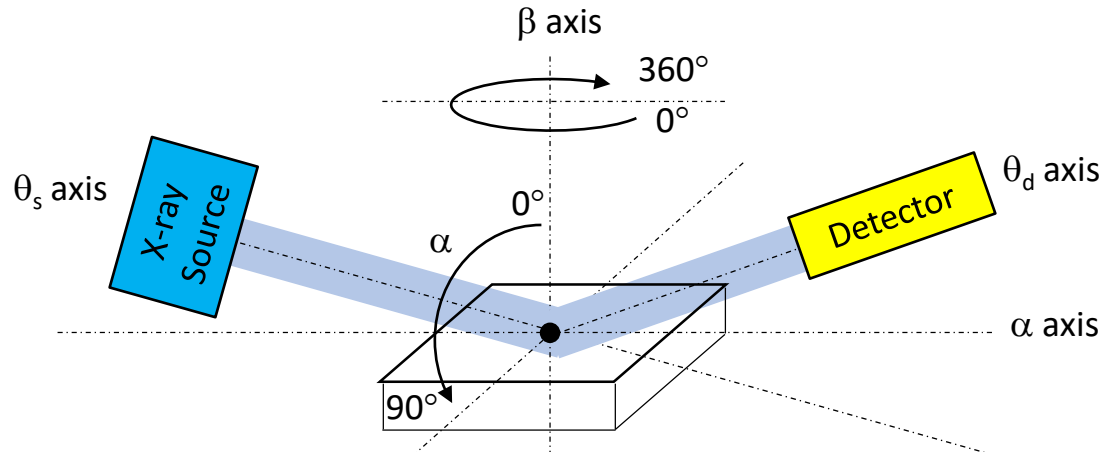
$\phi$  axis = sample rotation axis around Surface Normal axis

$\chi$  axis = axis for setting tilt of Surface Normal with respect to Goniometer Z-axis



$\alpha$  axis = rotation axis for setting tilt angle with respect to Z-Axis

$\beta$  axis = sample rotation axis along Surface Normal (see  $\phi$  axis)



## Out-of-Plane Pole Figure Definitions

$$\alpha \text{ tilt} = 90^\circ - \chi \text{ tilt}$$

$$\beta \text{ rotation angle} = \phi \text{ rotation angle}$$

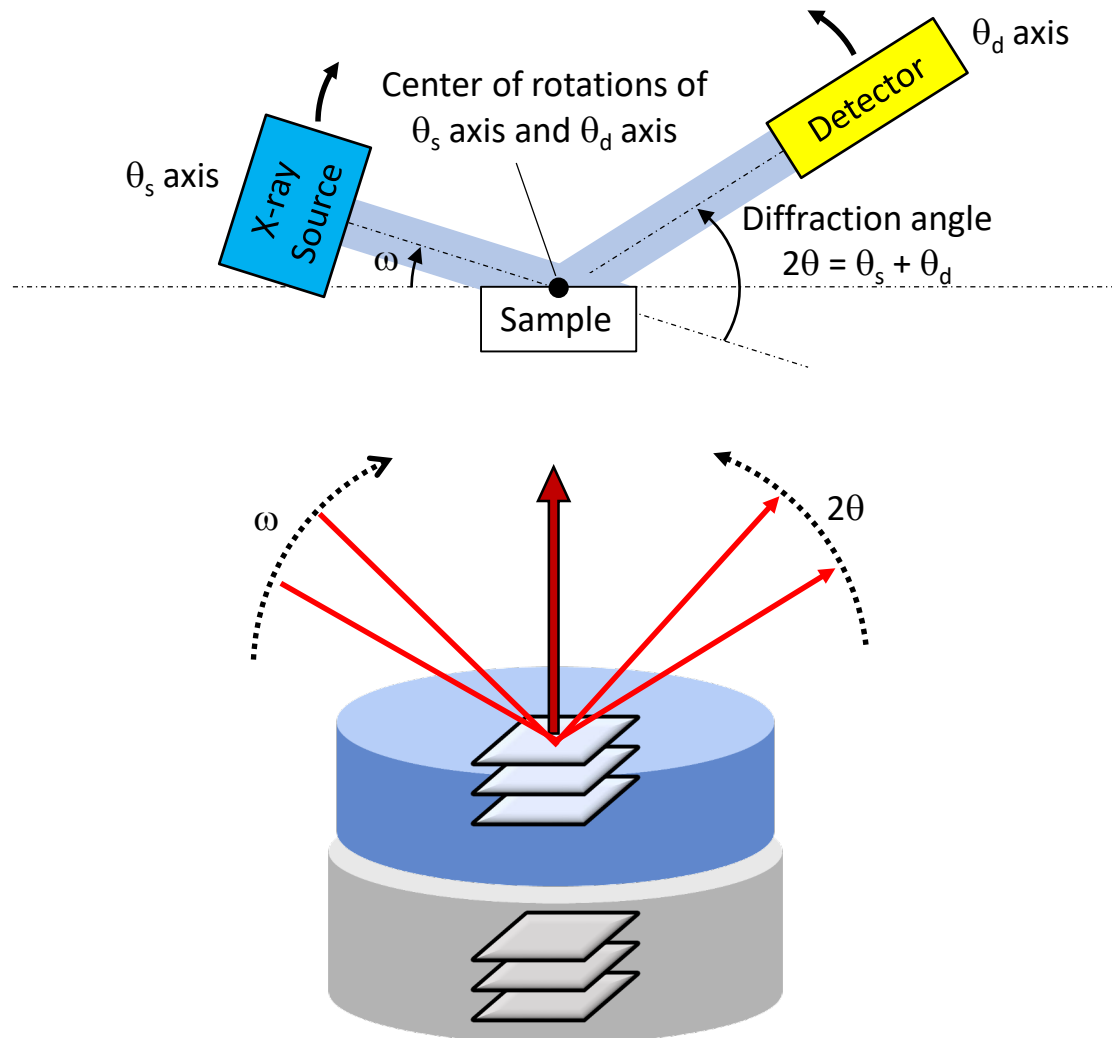
## In-Plane Pole Figure Definitions

$$\alpha \text{ tilt} = \chi \text{ tilt}$$

$$\beta \text{ rotation angle} = \phi \text{ rotation angle}$$

# B. Measurement Basics – 3/10

- This covers the Out-of-Plane (1D) or General (PB) XRD or  $2\theta/\omega$  Measurement



## Movement:

- $2\theta$  is driving arm;  $2\theta$  range =  $-10$  to  $158^\circ$
- $\omega$  is slave arm;  $\omega = \frac{1}{2} (2\theta)$

## Pros:

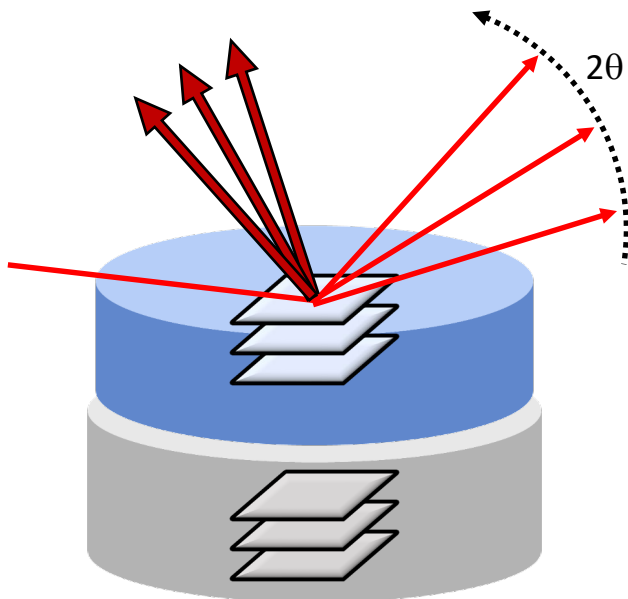
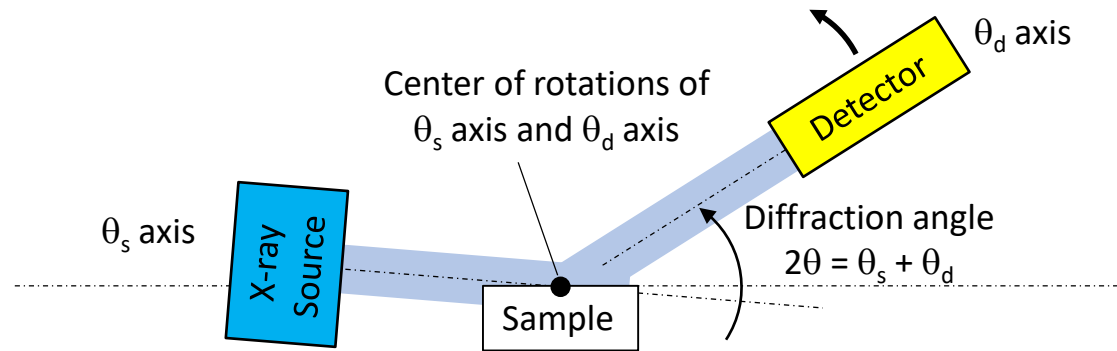
- Used for Qualitative analysis such as:
- Determining presence or absence of a preferred orientation
- Interplanar spacings of lattice planes parallel to surface
- Lattice constants corresponding to these interplanar spacings
- Crystallinity of a crystal lattice parallel to the surface

## Cons:

- Cannot observe lattice planes perpendicular to surface
- Cannot provide information on presence or absence of the in-plane orientation
- Cannot distinguish between a fiber-oriented and a single crystal

# B. Measurement Basics – 4/10

- This covers the Thin Film (1D) or Grazing Incidence XRD or GIXRD Measurement



## Movement:

- $2\theta$  is driving arm;  $2\theta$  range =  $-15$  to  $120^\circ$
- $\omega$  is set near a small critical angle usually between  $0.1$  to  $1^\circ$

## Pros:

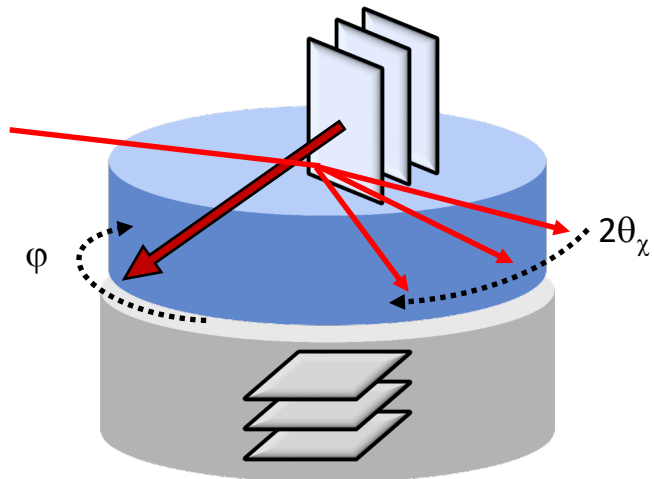
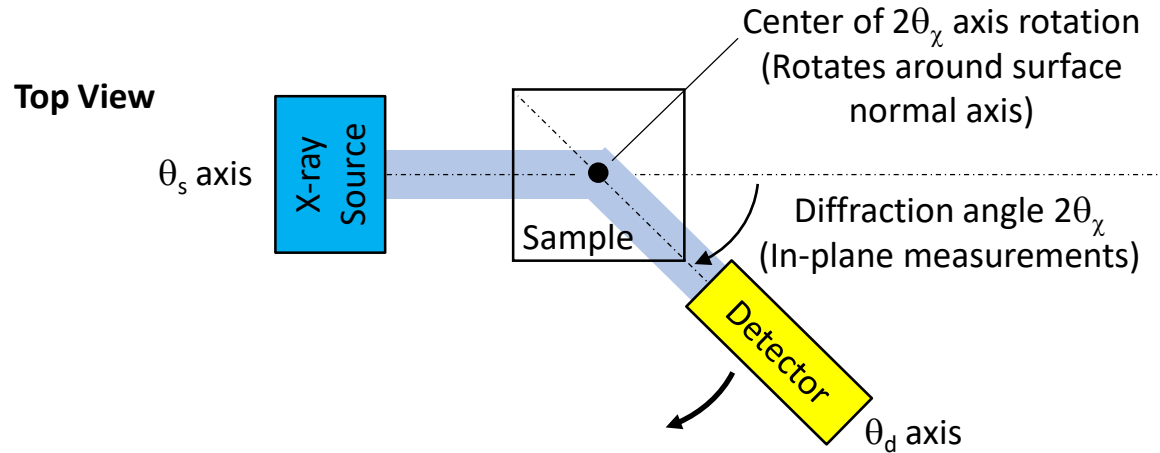
- Avoids scattering from the substrate
- Used for Qualitative analysis such as:
- For unoriented (or weakly oriented) polycrystal samples
- Lattice constants
- Crystallinity of a sample
- Depth dependence of above physical quantities

## Cons:

- Cannot be used to analyze strongly oriented polycrystal sample or single crystal

# B. Measurement Basics – 5/10

- This covers the In-Plane (1D) XRD or  $2\theta_\chi/\phi$  Measurement



## Movement:

- $2\theta_\chi$  is driving arm;  $2\theta_\chi$  range =  $-3$  to  $89^\circ$
- $\omega$  is set near a small critical angle usually between  $0.1$  to  $1^\circ$
- $\phi$  is slave arm;  $\phi = \frac{1}{2} (2\theta_\chi)$

## Pros:

- Similar to GIXRD with respect to depth of analysis
- Used for Qualitative analysis such as:
- Investigating presence or absence of the preferred orientation
- Interplanar spacings of lattice planes perpendicular to the surface
- Lattice constants corresponding to these interplanar spacings
- Crystallinity of the crystal lattice perpendicular to the surface
- Presence or absence of in-plane orientation
- Distinguish between fiber-oriented sample and single crystal or confirm presence or absence of twinning

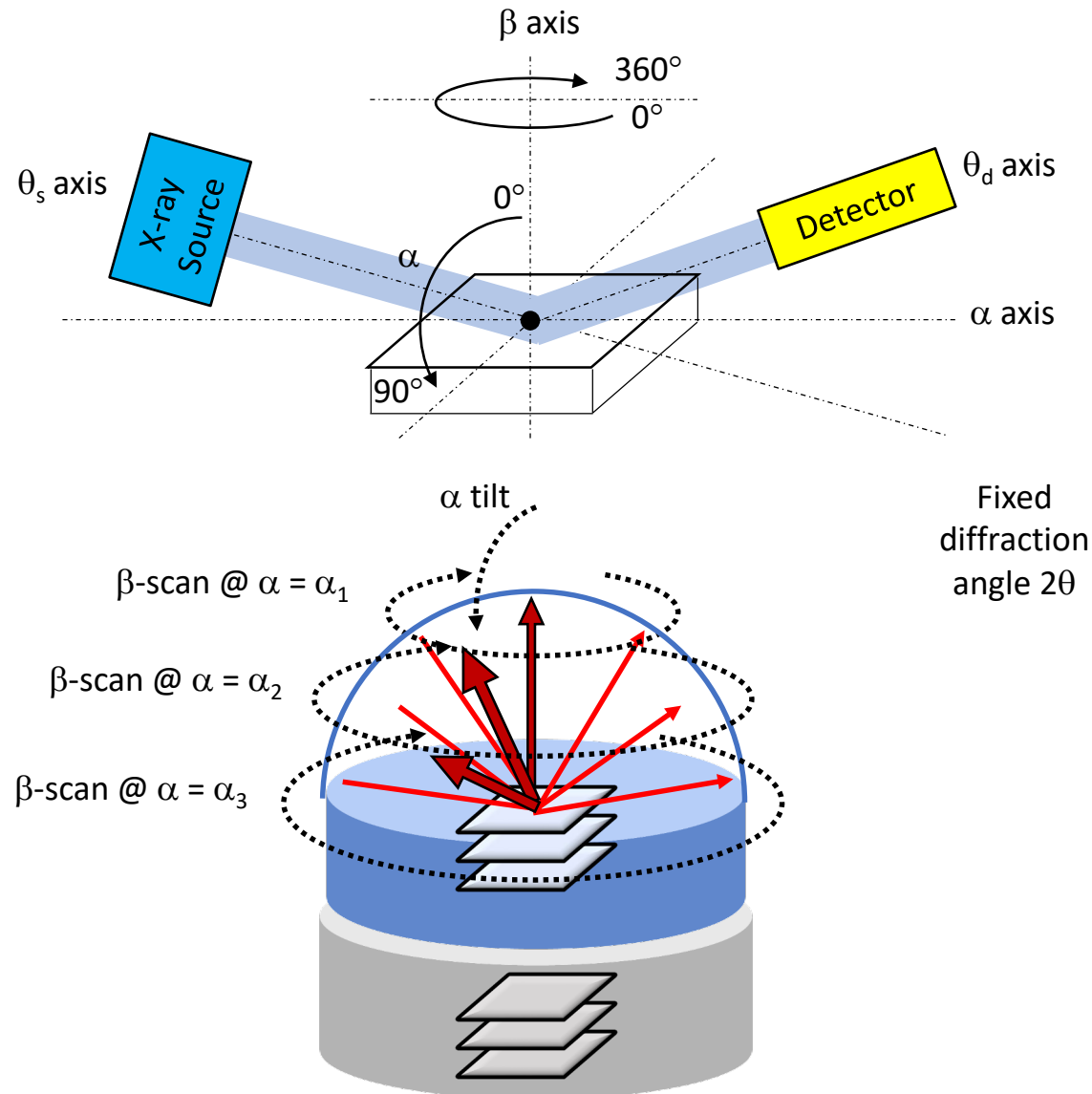
## Cons:

- Cannot observe lattice planes parallel to surface
- Cannot provide information on presence or absence of the out-of-plane orientation



# B. Measurement Basics – 6/10

- This covers the Pole Figure (2D) Measurement



## Movement:

- $2\theta$  is kept constant;  $\omega = \frac{1}{2} (2\theta)$
- $\alpha$  is stepped;  $\alpha$  range =  $-5$  to  $95^\circ$
- $\beta$  is continuously rotated;  $\beta$  range =  $-720$  to  $720^\circ$

## Pros:

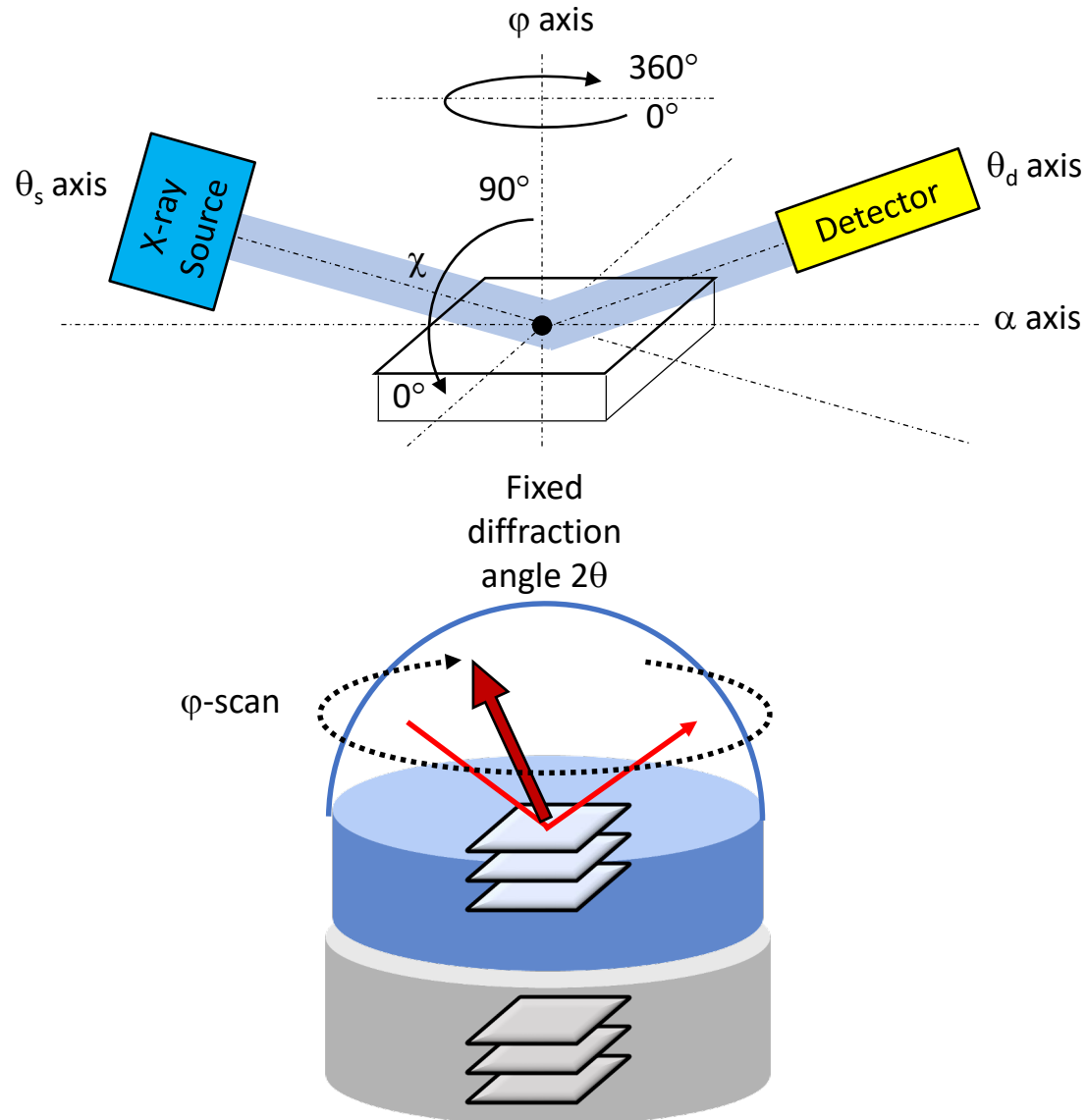
- Measures diffraction intensity distributions by rotating the sample in all directions while keeping the diffraction angle constant
- Direction at high diffraction intensity is observed corresponds to the preferred direction of the pole figure axes – indicating that crystallites with the measurement planes oriented in that direction are dominant

## Notes:

- Remember that  $\alpha = 90^\circ - \chi$  in SmartLab II
- Choose  $\alpha$  step values carefully!
- $\alpha$  step controls the resolution (and max intensity)
- Speed of  $\beta$  scan controls the sign-to-noise ratio of scans

# B. Measurement Basics – 7/10

- This covers the Preferred Orientation (1D) or Azimuth or  $\varphi$  Scan Measurement



## Movement:

- $2\theta$  is kept constant;  $\omega = \frac{1}{2} (2\theta)$
- $\varphi$  is continuously rotated;  $\varphi$  range =  $-720$  to  $720^\circ$

## Pros:

- Same underlying principles for pole figure measurement
- Measures a cross-section of a pole figure measurement
- Measures the spread (width) of diffraction intensity distribution
- Related to degree of preferred orientation (vs randomly orientated sample)
- Related to mosaicity (vs perfect single crystal)
- Quicker to perform than full pole figure measurement

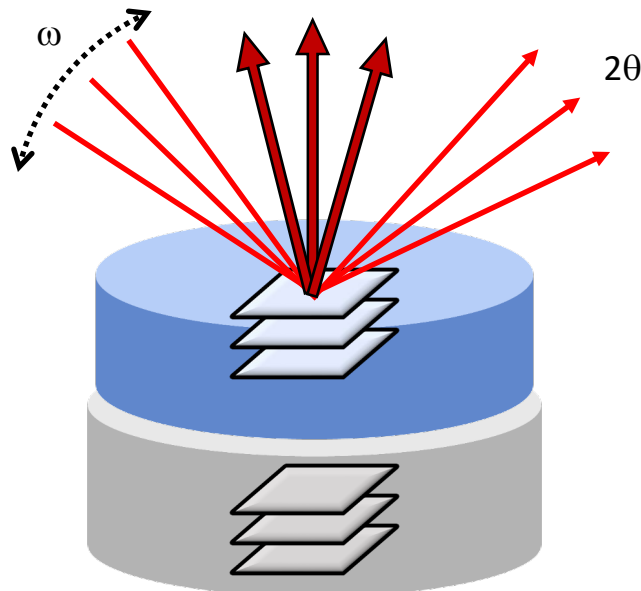
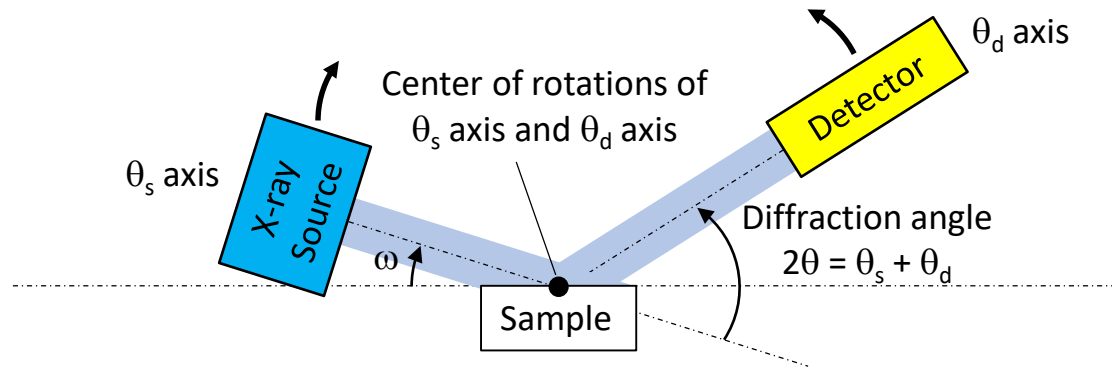
## Notes:

- Remember to optimize  $\chi$  value first!
- Speed of  $\varphi$  scan controls the sign-to-noise ratio of scans



# B. Measurement Basics – 8/10

- This covers the Rocking Curve (1D) Measurement



## Movement:

- $\omega$  is driving arm;  $\omega$  (relative) range =  $-5^\circ$  to  $+5^\circ$
- $2\theta$  is kept constant;  $2\theta = 2\omega$

## Pros:

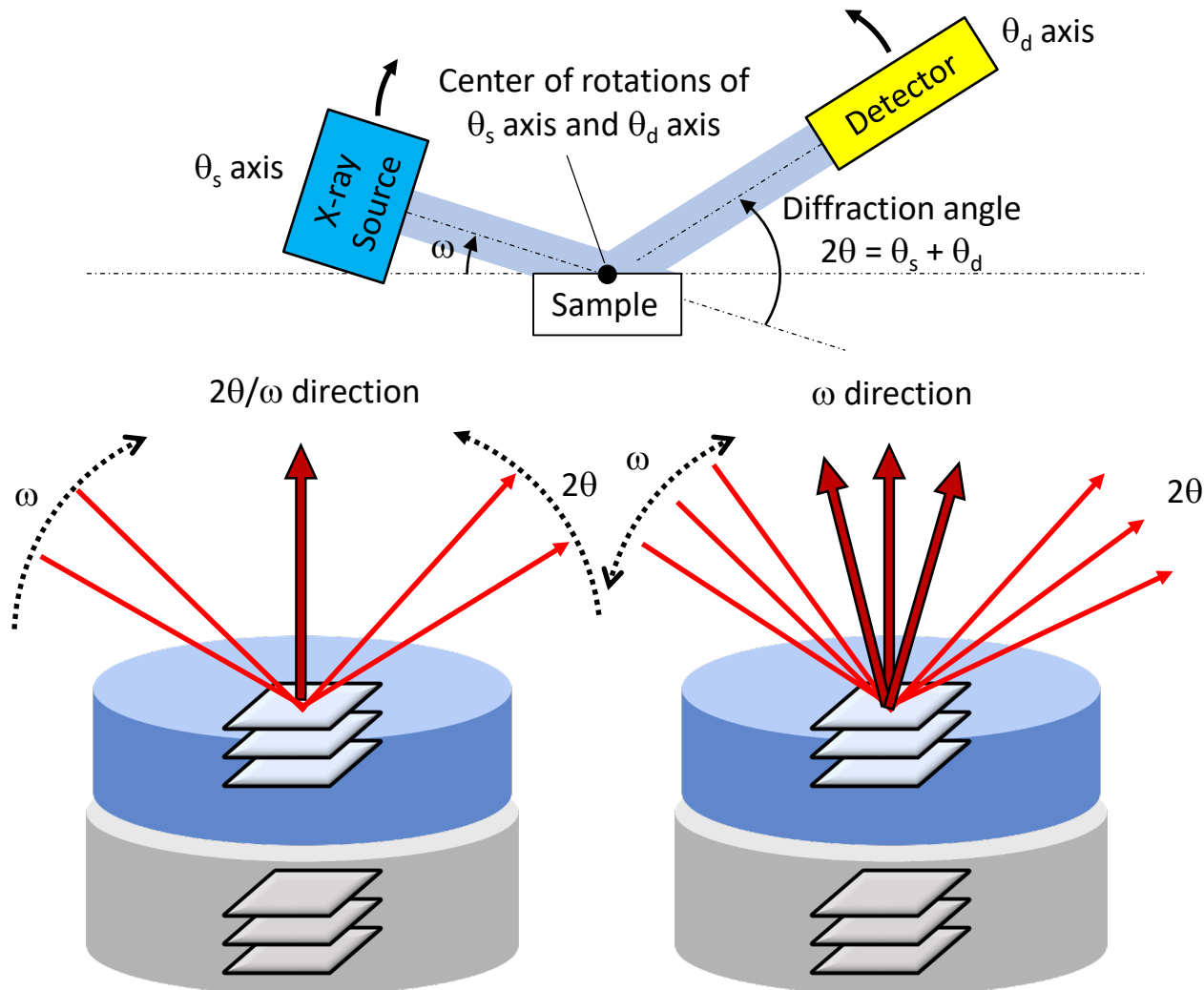
- Measures diffraction intensity distributions along a reciprocal lattice vector
- Planes no longer parallel with sample surface are brought onto the Bragg plane
- Measures changes in interplanar spacing
- Generally used to evaluate the thickness or mixed crystal ratio of an epitaxial film on a sample
- Width of rocking curve depends upon mosaic spread of the grains, density of dislocations, and substrate curvature
- FWHM is recorded and indication of quality of intended epitaxial growth or preferential orientation

## Notes:

- Do not mix up width of rocking curves with the widths on the peaks in the  $2\theta/\omega$  scans

# B. Measurement Basics – 9/10

- This covers the Reciprocal Space Mapping or RSM (2D) Measurement



## Movement:

- $2\theta/\omega$  scan is one mapped direction
- $\omega$  scan is second mapped direction

## Pros:

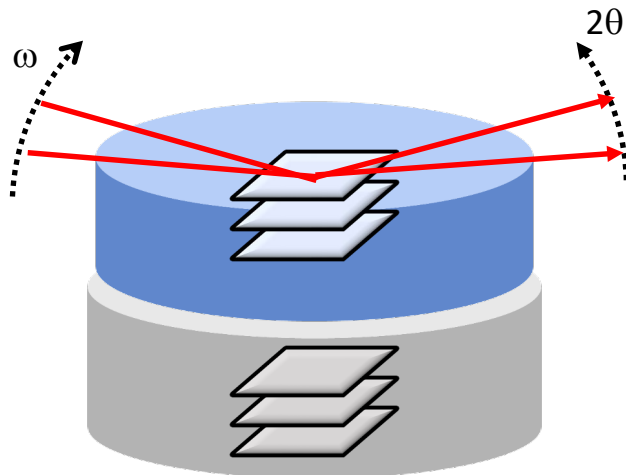
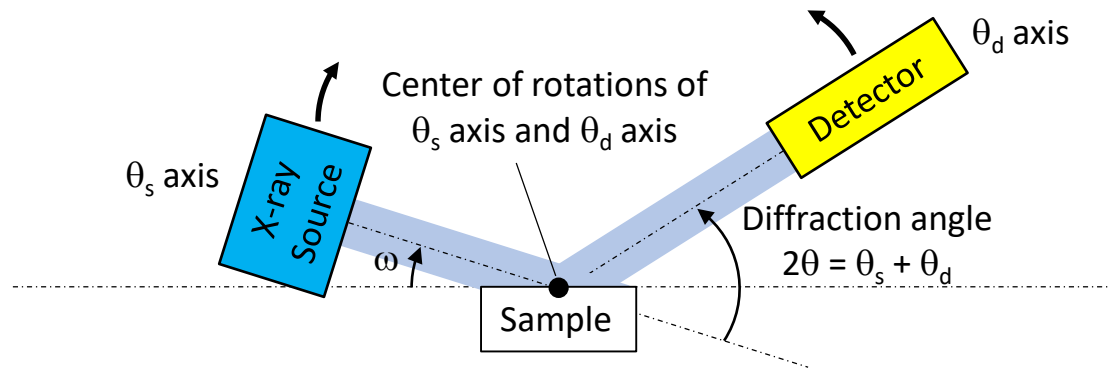
- Measures diffraction intensity distributions and plots result in reciprocal space
- Central coordinates, shapes, and positional relationships of the reciprocal lattice points (film and substrate) appearing in 2D map provides a wide range of info on crystal structure

## Notes:

- Each  $\omega$  step  $\rightarrow$   $2\theta/\omega$  scan is performed ( $q_x$ )
- Each  $2\theta/\omega$  step  $\rightarrow$   $\omega$  scan (Rocking Curve) is performed ( $q_y$ )

# B. Measurement Basics – 10/10

- This covers the Reflectivity (1D) Measurement



## Movement:

- $2\theta$  is driving arm;  $2\theta$  range = 0 to  $10^\circ$
- $\omega$  is slave arm;  $\omega = \frac{1}{2} (2\theta)$

## Pros:

- Can be used for crystalline or amorphous samples
- Evaluates thickness, density, and surface or interface roughness of thin-film materials
- Evaluates structure of a multilayer or single layer film
- Measures samples nondestructively

## Cons:

- Requires surface and interfaces are flat
- May require monochromator to yield higher resolution for thicker films

# C. GUI Basics – 1/3

- These slides will teach you GUI Basics

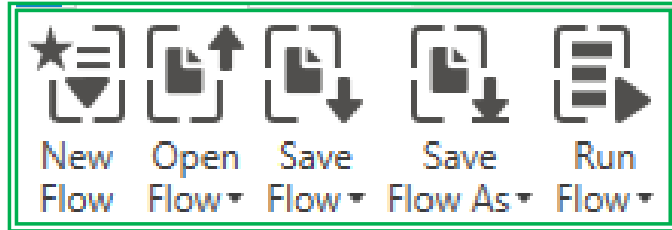
The screenshot displays the XRD Measurement software interface with several key components highlighted and labeled:

- Flow Controls:** A green box highlights the top-left toolbar containing icons for Wizard, Run Flow, and other workflow-related functions.
- Plug-In Tabs:** A yellow box highlights the top toolbar area containing various instrument and data management tabs like Load Data, DB Browser, Create Rep, etc.
- Instrument Controls:** A red box highlights a group of icons on the right side of the top toolbar, including Sense, Light OFF, Door Lock ON, and Move to Home Position.
- Tools:** A blue box highlights another group of icons on the right side of the top toolbar, including RS Viewer, Help Viewer, and This Data to Transfer.
- Activities:** A purple box highlights the left sidebar's 'Package Activities' list, which includes options like Reflection SAXS, Micro Area, Pole Figure, etc.
- H/W Status:** A purple box highlights the 'H/W Status' section in the left sidebar, showing various hardware status indicators.
- Flow Editor:** A yellow box highlights the central 'Flow Editor' workspace, which is currently empty and labeled 'Drop activities here'.
- Display Area for Data:** A green box highlights the 'Display Area' on the right, showing a plot of Intensity (cps) versus 2θ/ω (°) with two prominent peaks.
- Data Browser:** A blue box highlights the 'Data Browser' panel at the bottom, showing a list of data files with checkboxes for selection.
- Startup/Shutdown:** A blue box highlights the 'Startup/Shutdown' panel at the bottom, containing controls for running and stopping the instrument, along with various configuration settings.

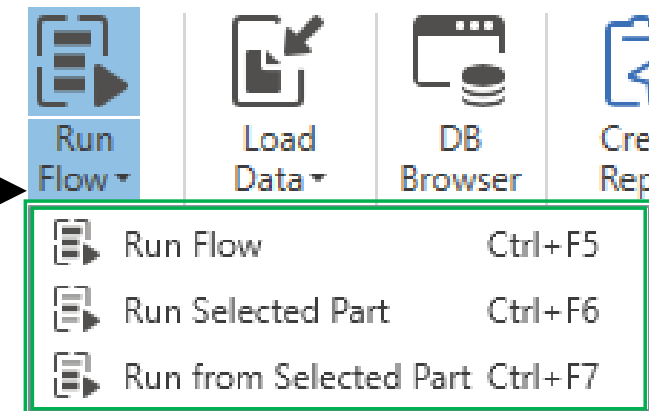
At the bottom of the screen, the status bar shows the current session as 'MSEXRD-PC4 / training' and the time as '3:01 PM'.

# C. GUI Basics – 2/3

- **Flow Controls** – used to control your **Measurement Flow** (or **Measurement Program**)
  - **Wizard** – will help you selected the recommend packages for desired measurement/analysis



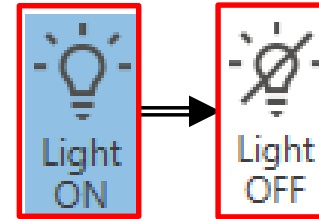
- **New Flow** - remove existing **Flow** and create a **New Flow** package
- **Open Flow** - open existing **Flow** that you have saved in the **File System**
- **Save Flow** - saves your edited **Flow** into the **Files System**
- **Save Flow As** - saves your edited **Flow** as a new file into the **Files System**
- **Run Flow**
  - **Run Flow** – runs your entire **Flow** from top to bottom
  - **Run Selected Part** – only runs the **Selected Part**
  - **Run from Selected Part** will run your entire – runs your flow from top to bottom starting from **Selected Part** (useful for **Shutdown**)



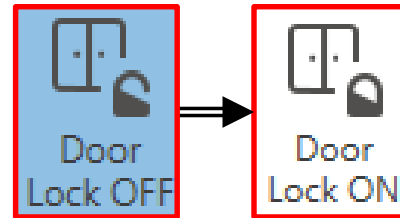
# C. GUI Basics – 3/4

- **Instrument Controls** – useful controls for the **Instrument**

- **Light** – Turns the **Light** ON and OFF inside Cabinet



- **Door Lock** – Toggles the **Door Locks** ON and OFF



- **Move to Home Position** – Moves the **Goniometer** axes to the starting **Home Position** useful for exchanging optics



- **Tools Control** – opens **Reciprocal Space (RS) Viewer** – see **V. RS Viewer**

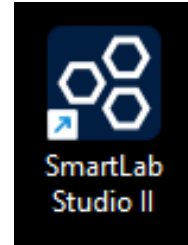


# I. Startup – 1/2

○ This sequence is used for Initial Startup sequence

1. Sign-in on the ***Sign-in Sheet***

2. First ***Double-click*** on ***SmartLab Studio II*** software icon



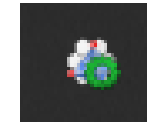
3. Enter your ***Login*** and ***Password***

- Login: *Faces Login*      Password: *Provided by Lab Manager*

4. Confirm that “***XRD Measurement / Ready***” is shown  $\Rightarrow$  **XRD Measurement | Ready** then proceed to ***Step 7***

5. If ***Status*** is “***XRD Measurement / Ready (Not Connected)***”,  $\Rightarrow$  **XRD Measurement | Not ready (Connected)** you will need to follow ***Steps 5-6*** to restart the ***Server***

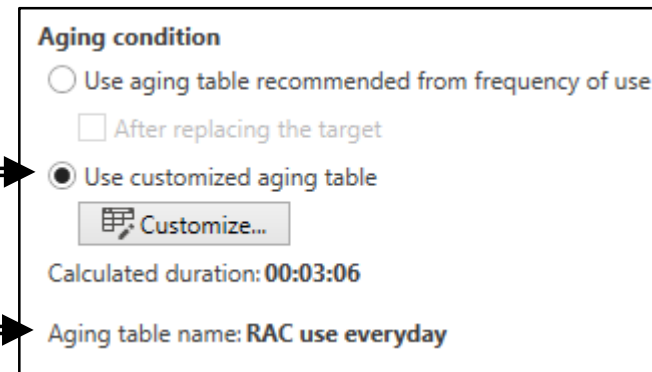
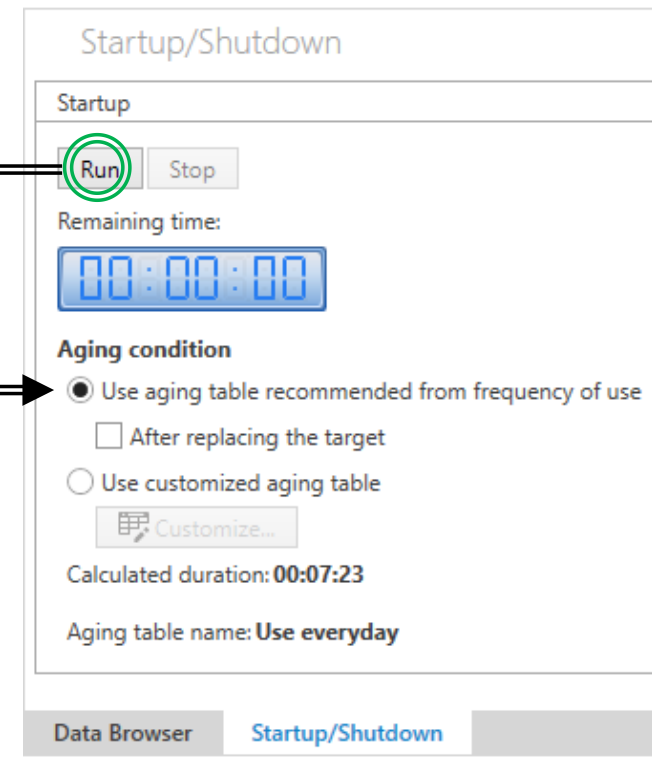
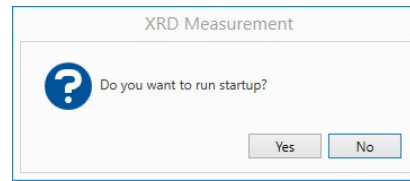
6. Access lower right ***Hidden Icons*** tray,  then find the ***ICServerTaskTray*** icon



7. If icon is not ***Green***, then ***Right-click*** and click on ***Restart*** to enable, then proceed to ***Step 7***

# I. Startup – 2/2

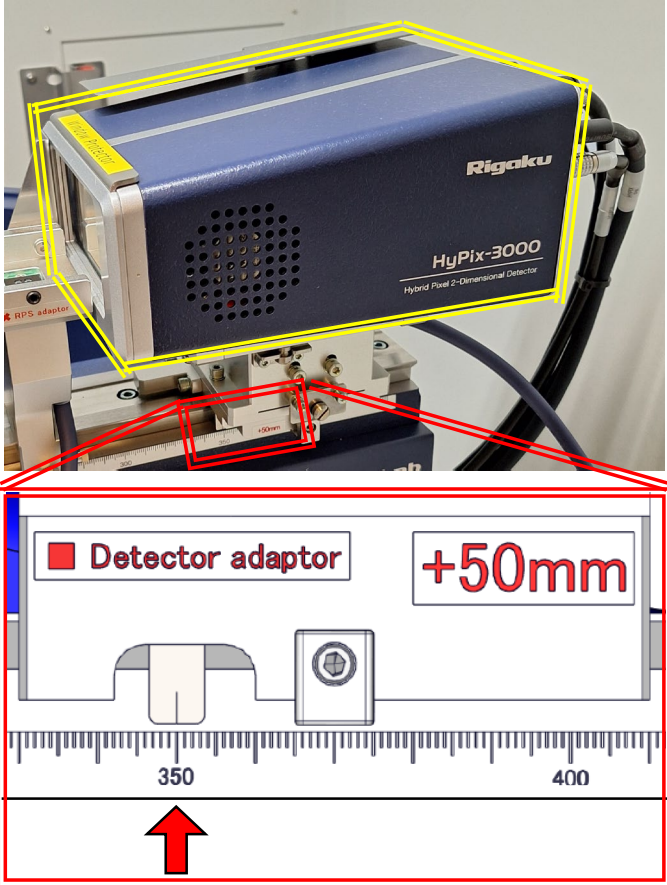
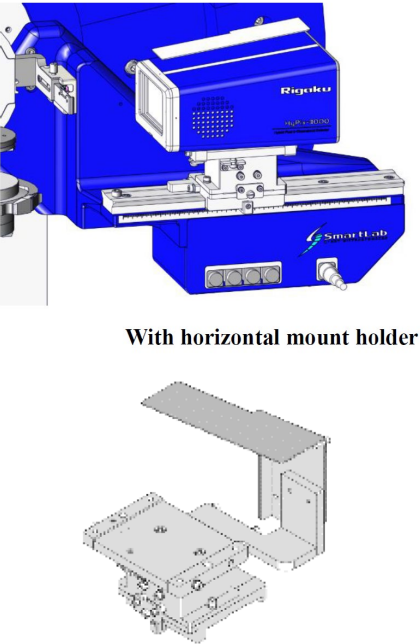
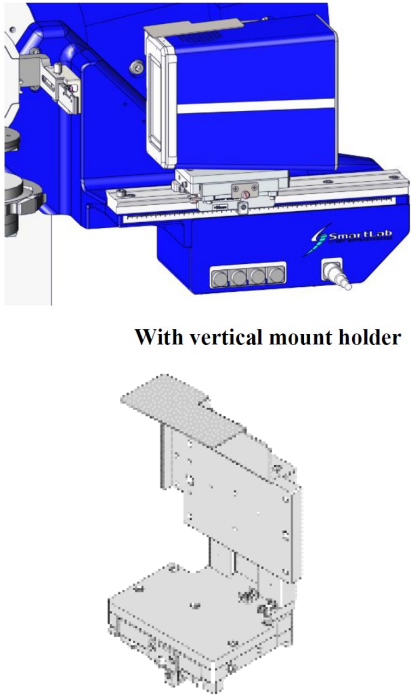

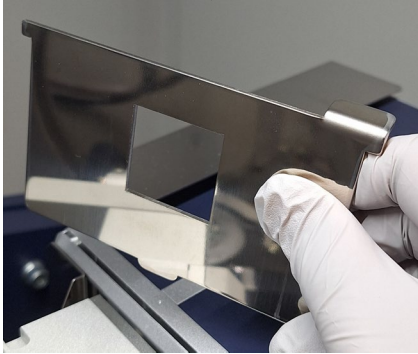
8. Click on the **Startup/Shutdown** tab at the bottom right panel
9. Confirm **Use aging table recommend from frequency of use** is selected
10. The system will automatically choose the appropriate sequence and may take up to 20 minutes to start up depending on frequency of use
11. Click the **Run** button, and confirm with **Yes**
12. Once completed, the **Standby** setting should be at **40 kV** and **30 mA** (1.2 kW)
13. Now let's bring the system up to **Operational Level** by selecting **Use customized aging table** and **RAC use everyday** is selected
14. Click the **Run** button again, and confirm with **Yes**
15. The **Operational Level** should now be set to **40 kV** and **44 mA** (1.8 kW) for normal operation










# II. XRD Detector – 1/1

○ This covers the Detector

Detector	Orientation (Applications)		Window Protector
HyPix-3000 (2D Detector)	Horizontal (Default)	Vertical (Micro Area)	
	 <p>With horizontal mount holder</p>	 <p>With vertical mount holder</p>	<div> ↕ </div> <p><b>Window Protector</b> must be inserted to protect <b>Detector</b> when swapping out <b>Receiving Optics!</b></p> <p>\$\$\$\$\$\$</p>
Mark indicates position of detector adaptor and is +50 mm from the detection plane. i.e. <b>Detector Plane</b> of 300 mm ⇒ <b>Detector Adaptor Position</b> of 350 mm			


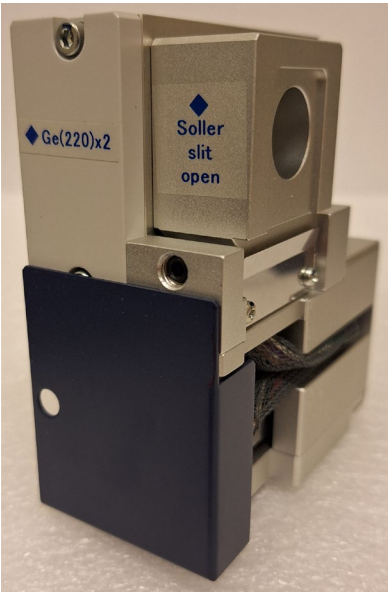
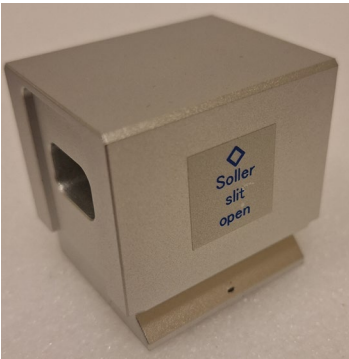





# III. XRD Optics – 1/6

- This covers the Incident Optics Unit #1

Incident Optics Unit #1	Incident Optics			
CBO – Cross Beam Optics	Parallel beam method (PB)	Para-focusing method (BB)	Micro Area (MA) 0.5	Micro Area (MA) 0.3
				

# III. XRD Optics – 2/6


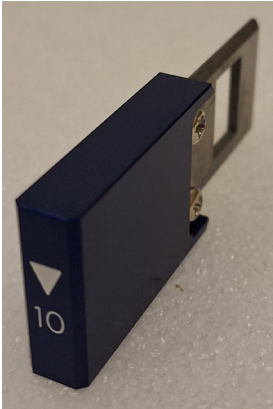




○ This covers the Incident Optics Unit #2

Incident Optics Unit #2		Incident Parallel Slits (Aperture) + IPS Adaptor		
Incident Parallel Slit (IPS) + Adaptor	Ge(220) 2-bounce monochromator	Soller Slit (Open)	Soller Slit (5.0deg)	In-plane PSC (0.5deg)
		 	 	 



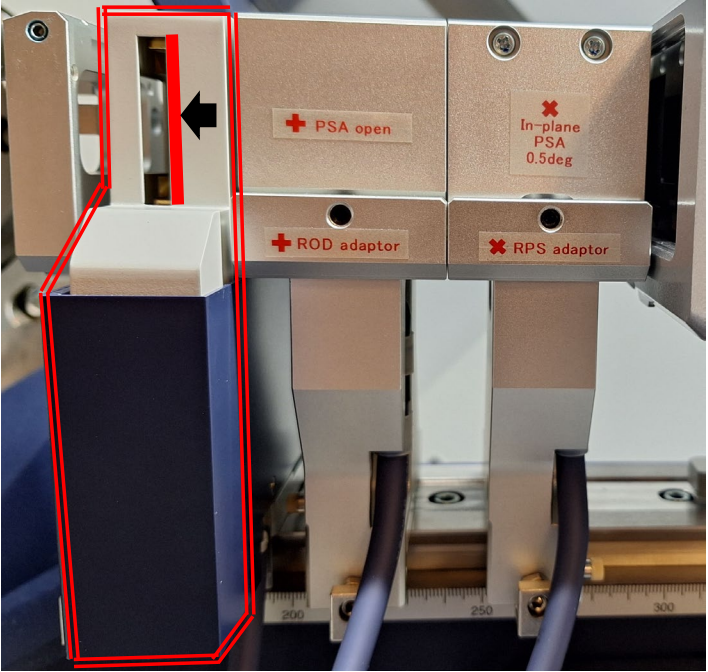
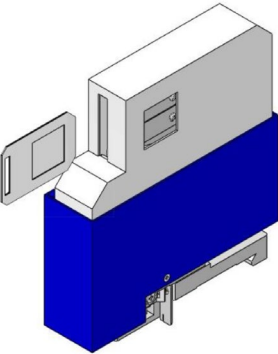



# III. XRD Optics – 3/6

○ This covers the Incident Slit

Incident Slit	Length-Limiting Slit (Aperture)				
	10 mm	5 mm	2 mm	0.5 mm	0.2 mm
 <p>Insert Slit hugging <b>LEFT</b> edge</p>					

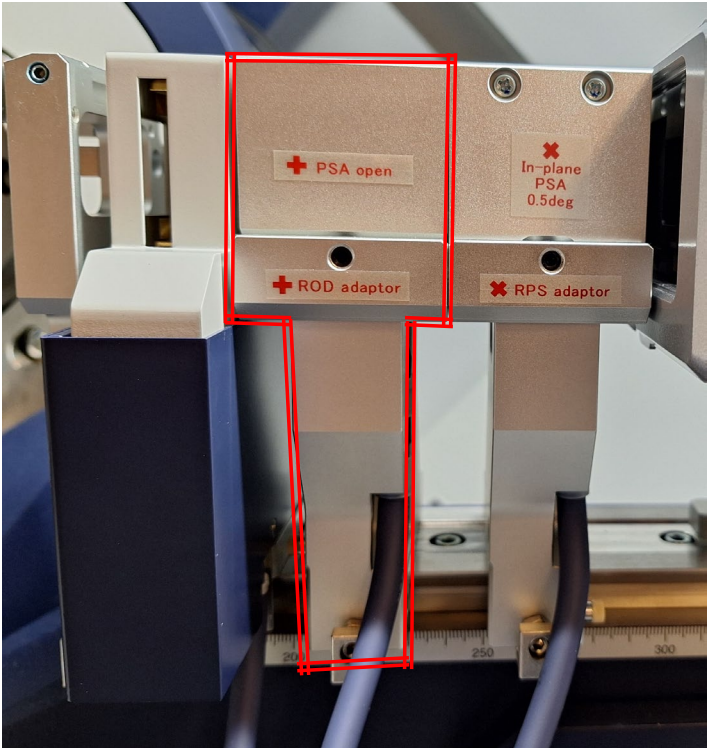




# III. XRD Optics – 4/6

- This covers the Receiving Slit

Receiving Slit	Insertion Diagram	k $\beta$ Filters (Thickness)		
Receiving Slit Box		CuK $\beta$ 1D (23 $\mu$ m)	CuK $\beta$ 15 $\mu$ m (15 $\mu$ m)	9 kW filter
				
Insert Card hugging <b>RIGHT</b> edge				

# III. XRD Optics – 5/6

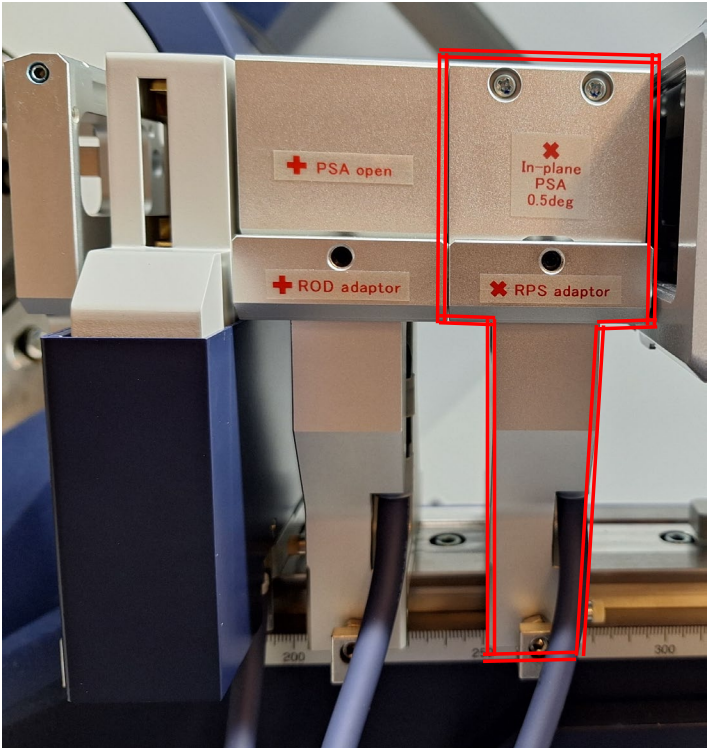
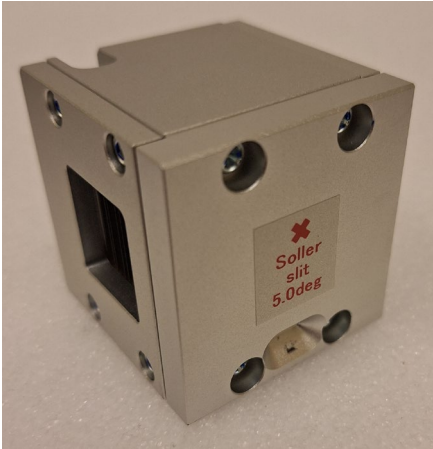



- This covers the Receiving Optics Unit #1

Receiving Optics Unit #1	Parallel Slits Analyzers (Aperture) + ROD Adaptor	
Parallel Slit Analyzer (PSA) + Adaptor	PSA (Open)	PSA (0.5deg)
	 	 



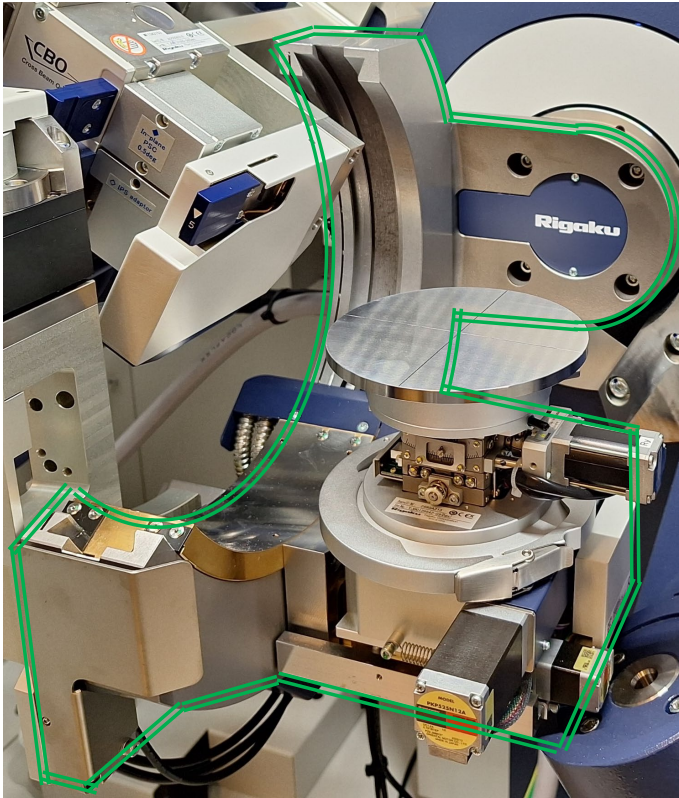
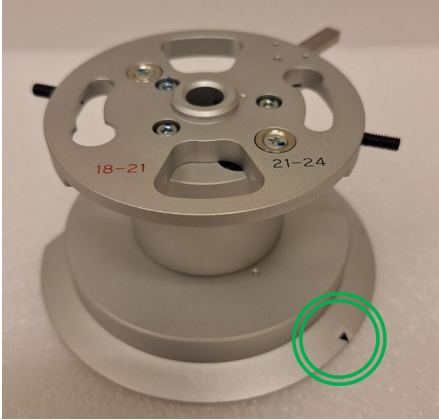
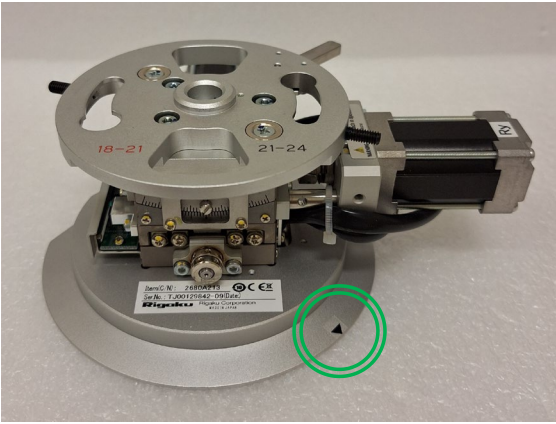
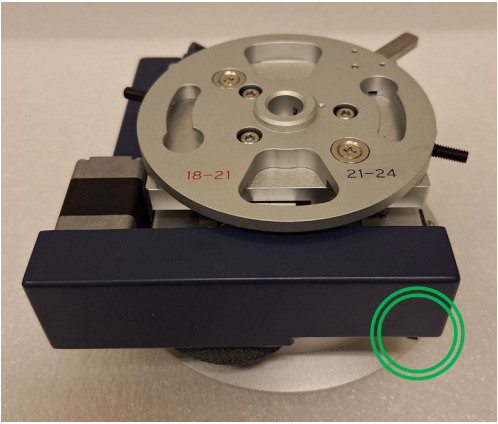
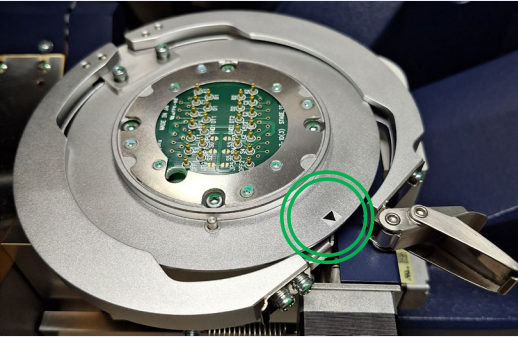
# III. XRD Optics – 6/6

○ This covers the Receiving Optics Unit #2

Receiving Optics Unit #2	Receiving Parallel Slits (Aperture) + RPS Adaptor	
Receiving Parallel Slit (RPS) + Adaptor	Soller Slit (5.0deg)	In-Plane PSA (0.5deg)
	 	 

# IV. XRD Sample Attachment – 1/2

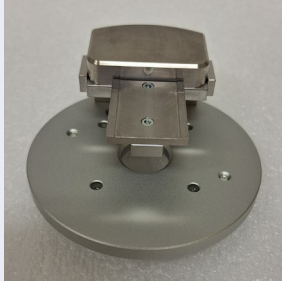
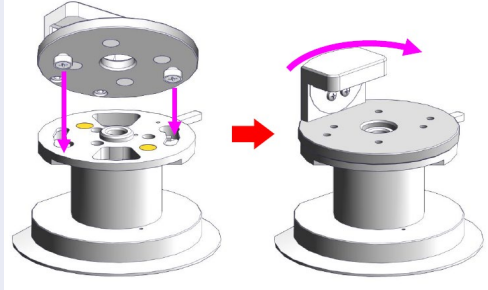
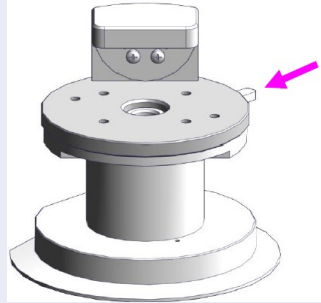

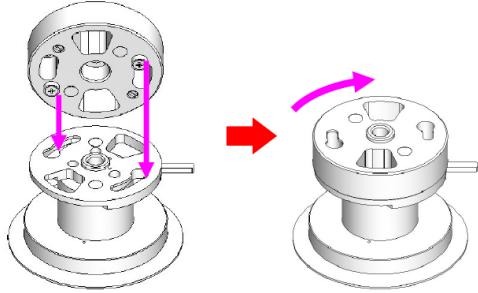
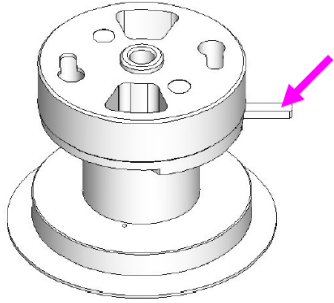
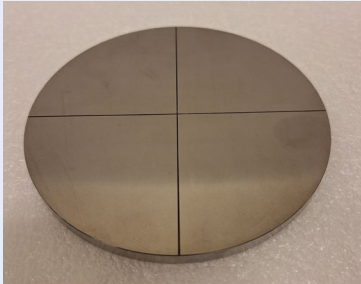
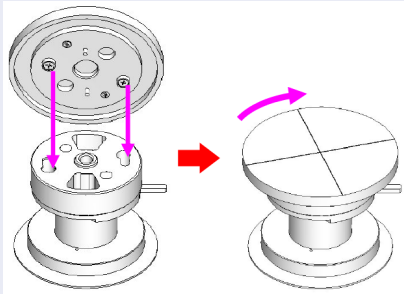
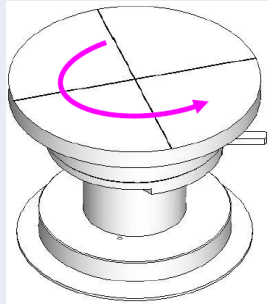
○ This covers the Sample Attachment Heads

Attachment Platform	Attachment Heads (Applications)		
$\chi\phi Z$ Attachment Platform	Standard (Alignment, Bulk Sample)	RxRy (Reflectivity, RSM, In-Plane)	XY-20 mm (Micro-area)
			
		<p>Carefully align <b>Attachment Head</b> to the <b>Attachment Platform</b> via the black triangle ▼ indicator.</p> <p><b>DO NOT BEND OR DAMAGE THE CONNECTOR PINS!</b></p> <p>Secure in place by closing the <b>Clasps</b> and <b>Front Latch</b>.</p>	



# IV. XRD Sample Attachment – 2/2

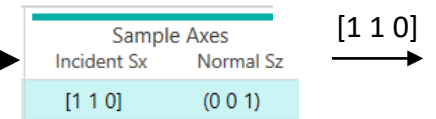
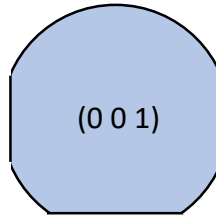
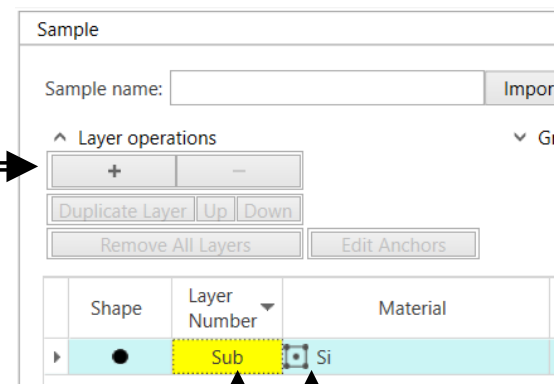
- This covers the Sample Plates (Note: Does not have recognition chips – you must remove even if not instructed!)

Sample Plate	Picture	Installation	Removal
Height Reference Sample			
Sample Spacer			
Wafer Sample Plate			

# V. RS Viewer – 1/2

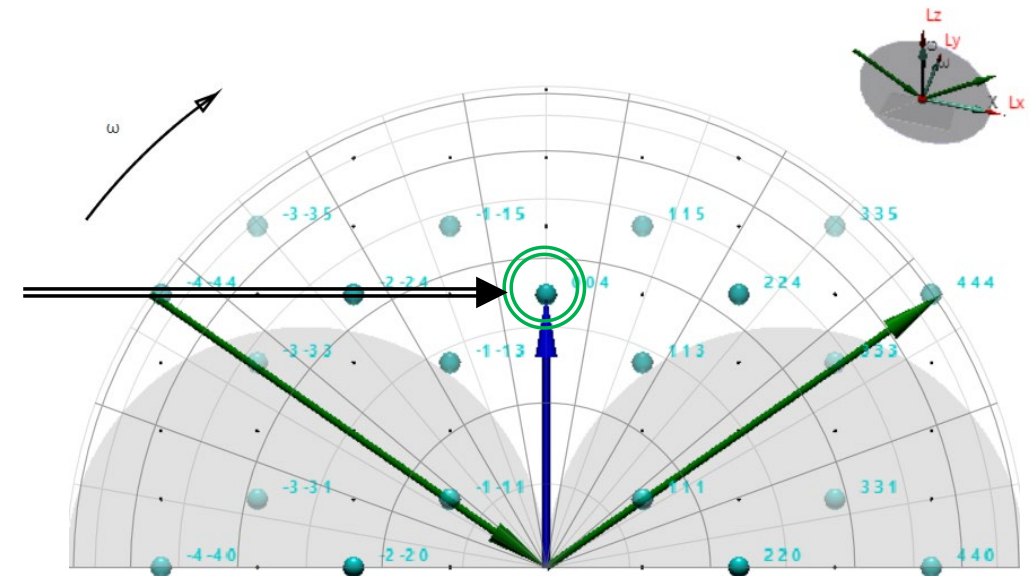
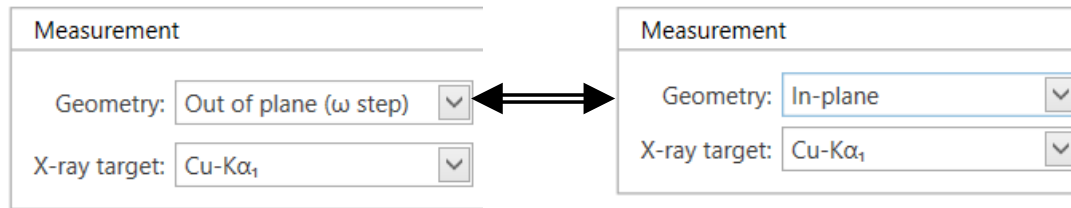
○ These slides will teach you about the Reciprocal Space (RS) Viewer

1. Click on the **RS Viewer** icon at the top of the **XRD Measurement** plugin
2. Click on the ? icon to open up the **RSViewer\_UserManual\_en.pdf** to review the tutorial
3. Add and edit layers to build up your substrate + films
4. Edit the **Samples Axes** (if known) to correlate actual sample to reciprocal lattice (e.g. Si wafer)
5. Set **Geometry** for your scans: **Out of plane** or **In-Plane**



[1 1 0]

6. Enter in desired reflection plane in **Origin** or select the **Shape Icon**



## V. RS Viewer – 2/2

7. The goniometer settings to achieve desired diffraction will be displayed:


- Out-of-Plane – No Tilt Scans:  $\chi$  and  $\phi$  are both zero
- Out-of-Plane – Tilted Scans:  $\chi$  and/or  $\phi$  are non-zero values
- In-Plane Scans:  $2\theta_\chi$  is diffraction angle

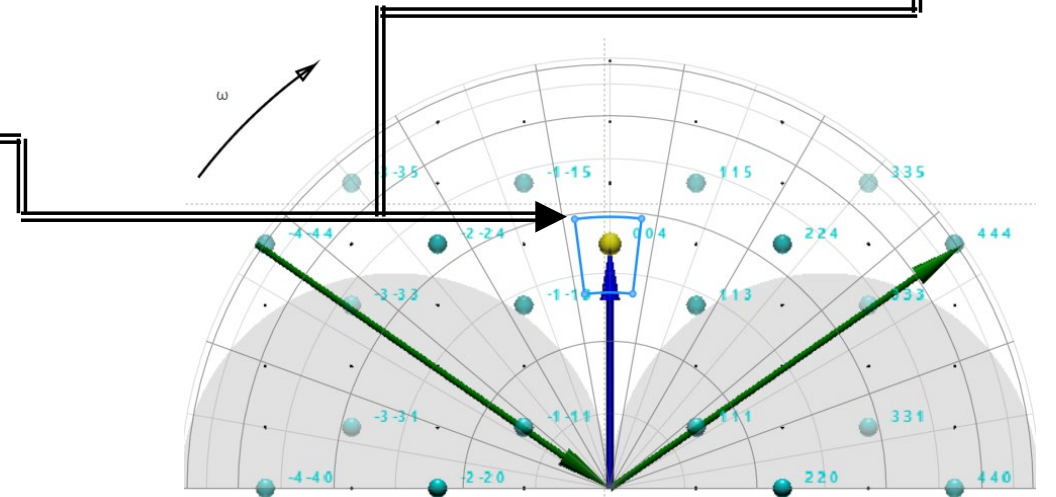
Out of Plane No Tilt	Out of Plane Tilted	In-Plane
$\omega_i$ : 34.5652	$\omega_i$ : 14.2213	$\omega_i$ : 0.0000
$2\theta/\omega_i$ : 69.1304	$2\theta/\omega_i$ : 28.4425	$2\theta/\omega_i$ : 0.0000
$\chi_i$ : 0.0000	$\chi_i$ : 54.7356	$\chi_i$ : 0.0000
$\varphi_i$ : 0.0000	$\varphi_i$ : -90.0000	$\varphi_i$ : 113.6515
$2\theta\chi_i$ : 0.0000	$2\theta\chi_i$ : 0.0000	$2\theta\chi_i$ : 47.3030

8. In order to achieve desired diffraction orientation, you will need to click on ***Move Axes***

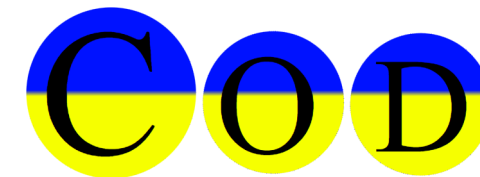
☐ Show area

9. Click on **Show area** checkbox will show you the corresponding scanned area for performing **Reciprocal Space Mapping (RSM)**

10. The area used for **RSM** can be manually adjusted visually  and then applied to **RSM Measurement** step via **Send Area**
  - Only works if **RS Viewer** is launched from **RSM Measurement**



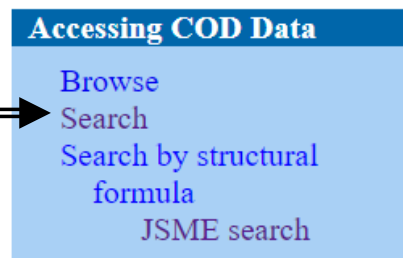
# V. RS Viewer – 3/4



- These slides will teach you how to load your sample information if not in the database already

11. Go to the following Crystallography Open Database (COD) website: <https://www.crystallography.net/cod/>

12. Click on the **Search**



13. Search for your desired sample using your desired method (e.g. Text, Journal, Chemical Formula, Elements, etc...)

14. Identify the desired sample information you want to import

COD ID ▲	Links	Formula ▲	Space group ▲	Cell parameters	Cell volume ▲	Bibliography
<a href="#">1001452</a>	<a href="#">CIF</a>	Ba <sub>2</sub> Cu <sub>3</sub> O <sub>7</sub> Y	<a href="#">P m m m</a>	3.8128; 3.8806; 11.6303 90; 90; 90	172.1	Capponi, J J; Chaillout, C; Hewat, A W; Lejay, P; Marezio, M; Nguyen, N; Raveau, B; Soubeyroux, J L; Tholence, J L; Tournier, R Structure of the 100 K Superconductor Ba~2~ Y Cu~3~ O~7~ between (5- 300)K by Neutron Powder Diffraction <a href="#">Europhysics Letters</a> , <b>1987</b> , <i>3</i> , 1301-1307

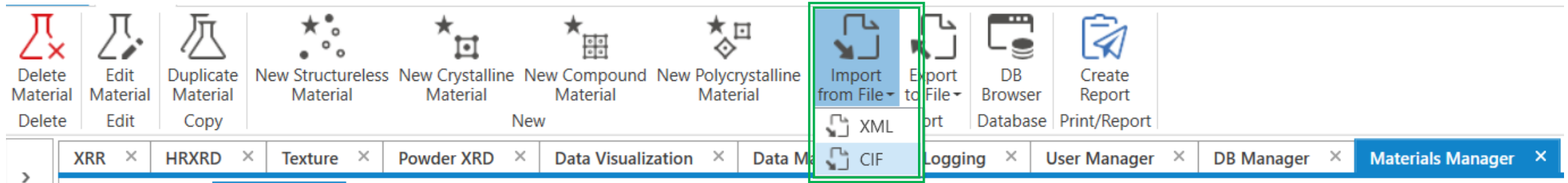
15. Click on the **CIF** link and download the file into your **CIF Folder**

16. Click on the **Materials Manager** in **SmartLab II**

Materials Manager X

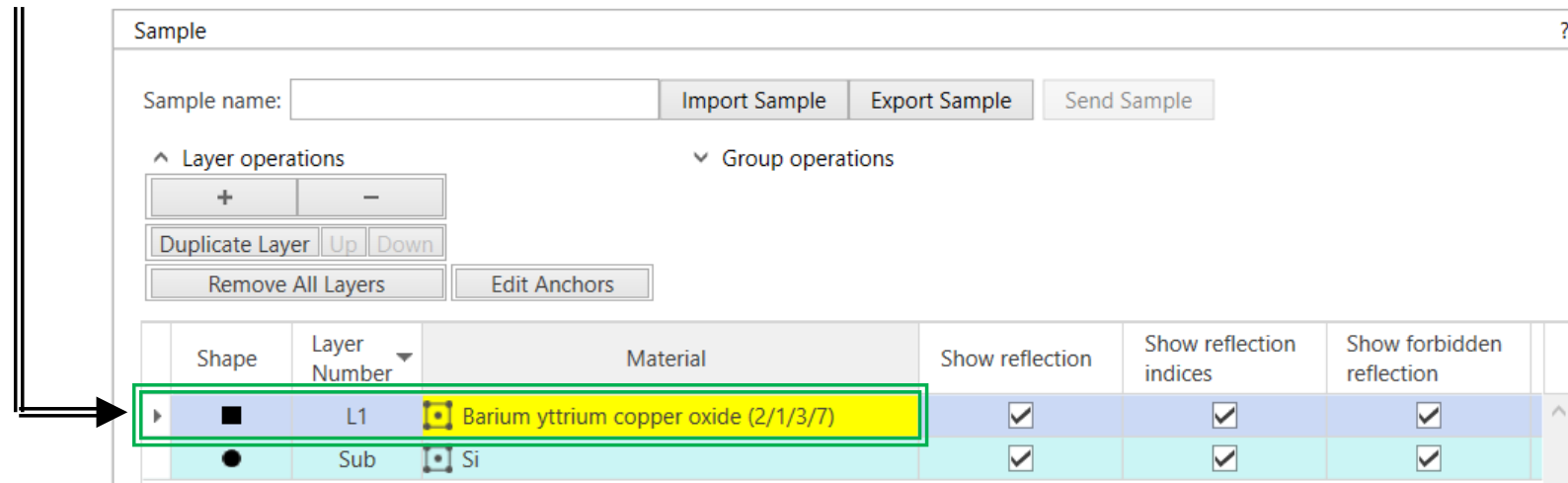
# V. RS Viewer – 4/4

17. Click on the **Import from File** tab and select **CIF**



18. Select the **CIF** file that you had downloaded

19. The sample information should now be available for you in the **Material** selection in **RS Viewer**



# VI. Utility Activity – 1/2

- This sequence will perform a Mirror Alignment and HyPix Adjustment after using the Monochromator Ge(220)x2

1. Select the **Mirror Alignment** activity under **XRD Measurement > Part Activities**

2. Drag the **Mirror Alignment** activity into the **Flow Editor** in **Sequence**

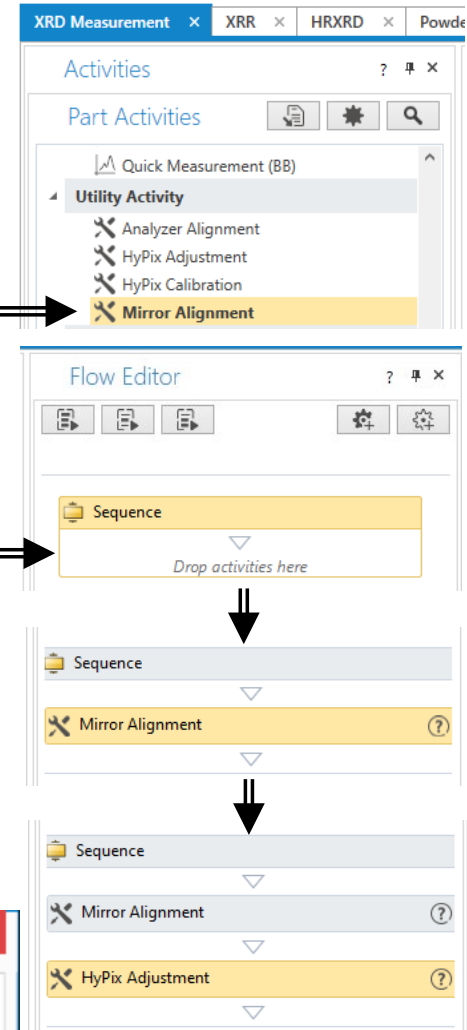
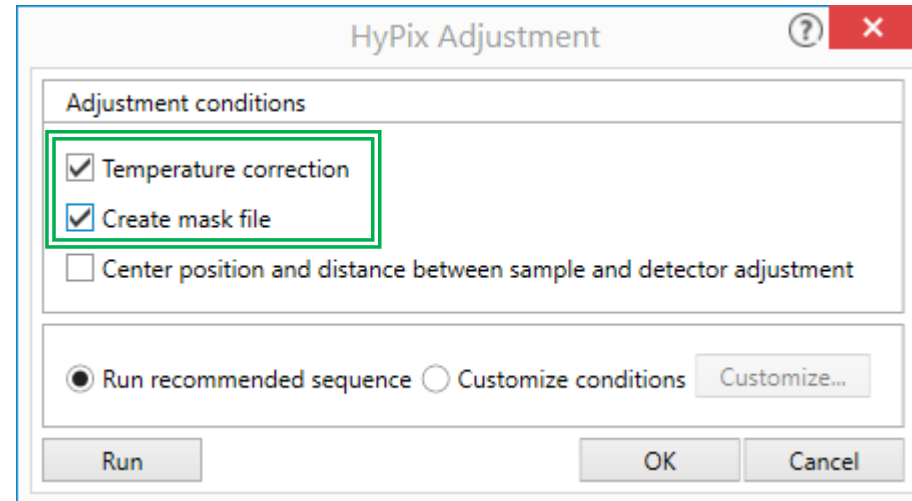
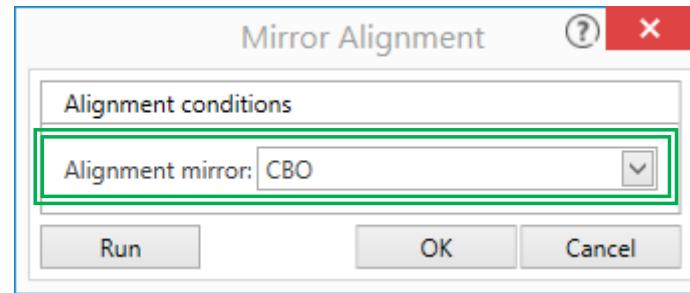
3. Drag the **HyPix Adjustment** activity in **Sequence**

4. Click on **Mirror Alignment** activity and select **CBO**, and click **OK**

5. Click on **HyPix Adjustment** activity and confirm only the first 2 options are checked, and click **OK**

a) **Temperature correction**

b) **Create mask file**





# VI. Utility Activity – 2/2

6. Click on **Run Flow** to perform Mirror Alignment and HyPix Adjustment automatically

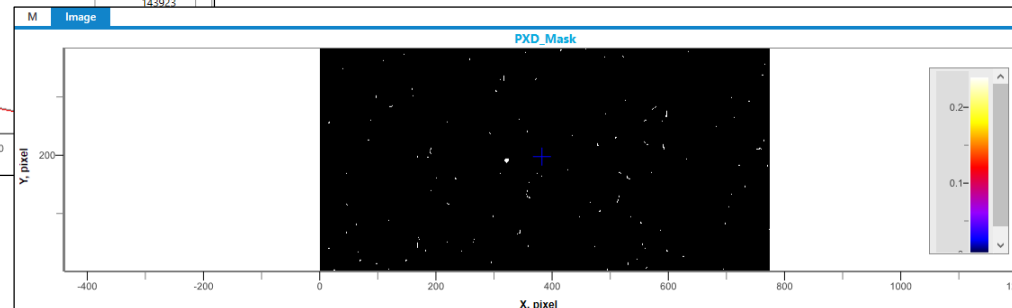
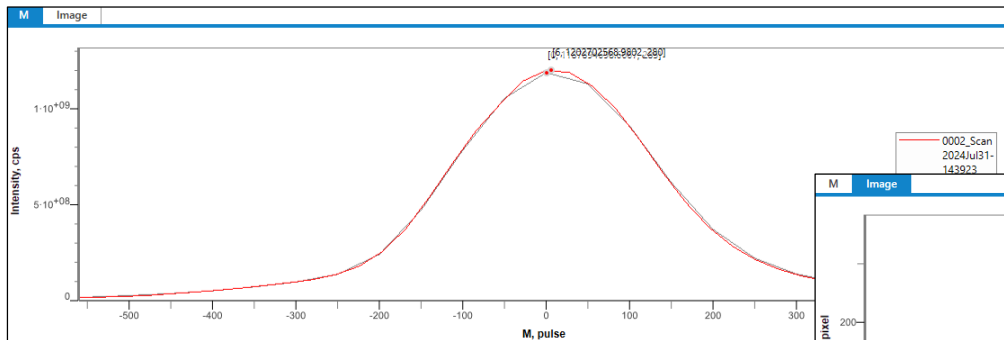
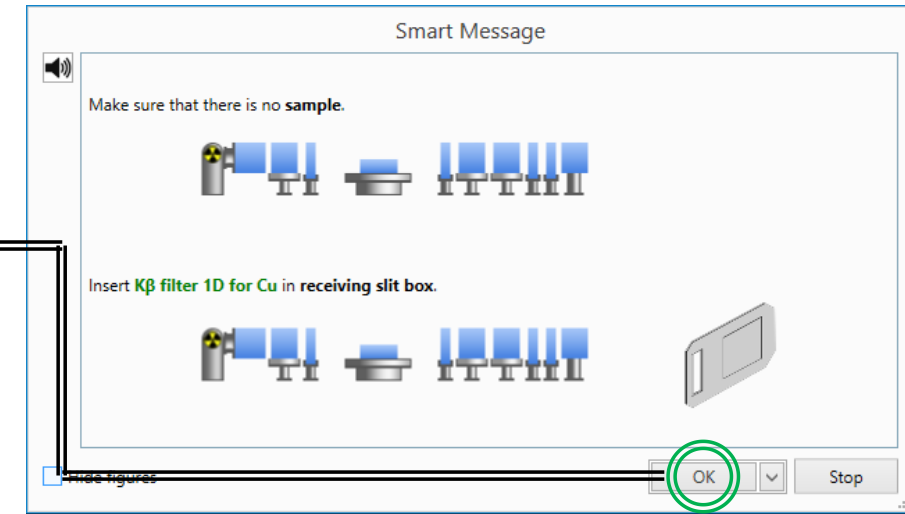


7. A **Smart Message** will appear indicating all the optics and attachments that need to be **removed** (indicated in **RED**) and those that need to be **installed** (indicated in **GREEN**)

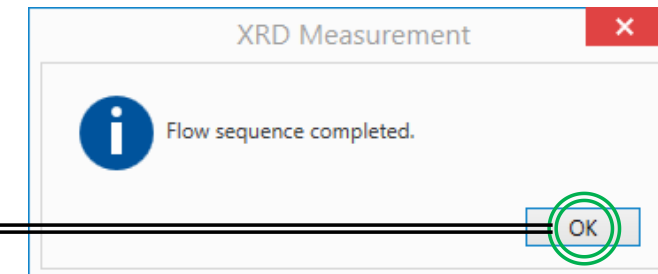
8. Proceed to perform each step in sequence and click **OK** when completed

Note: Your **Smart Message** may differ from example shown

9. **Mirror Alignment** will be performed first, followed by **HyPix Adjustment**



10. Wait for the **Flow sequence completed** prompt to continue by clicking **OK**



# VII. General (PB) or $2\theta/\omega$ Scan – 1/7

○ This sequence will perform a General  $2\theta/\omega$  scan using Parallel Beam optics

1. Select the **General (PB)** package under **XRD Measurement > Package Activities**

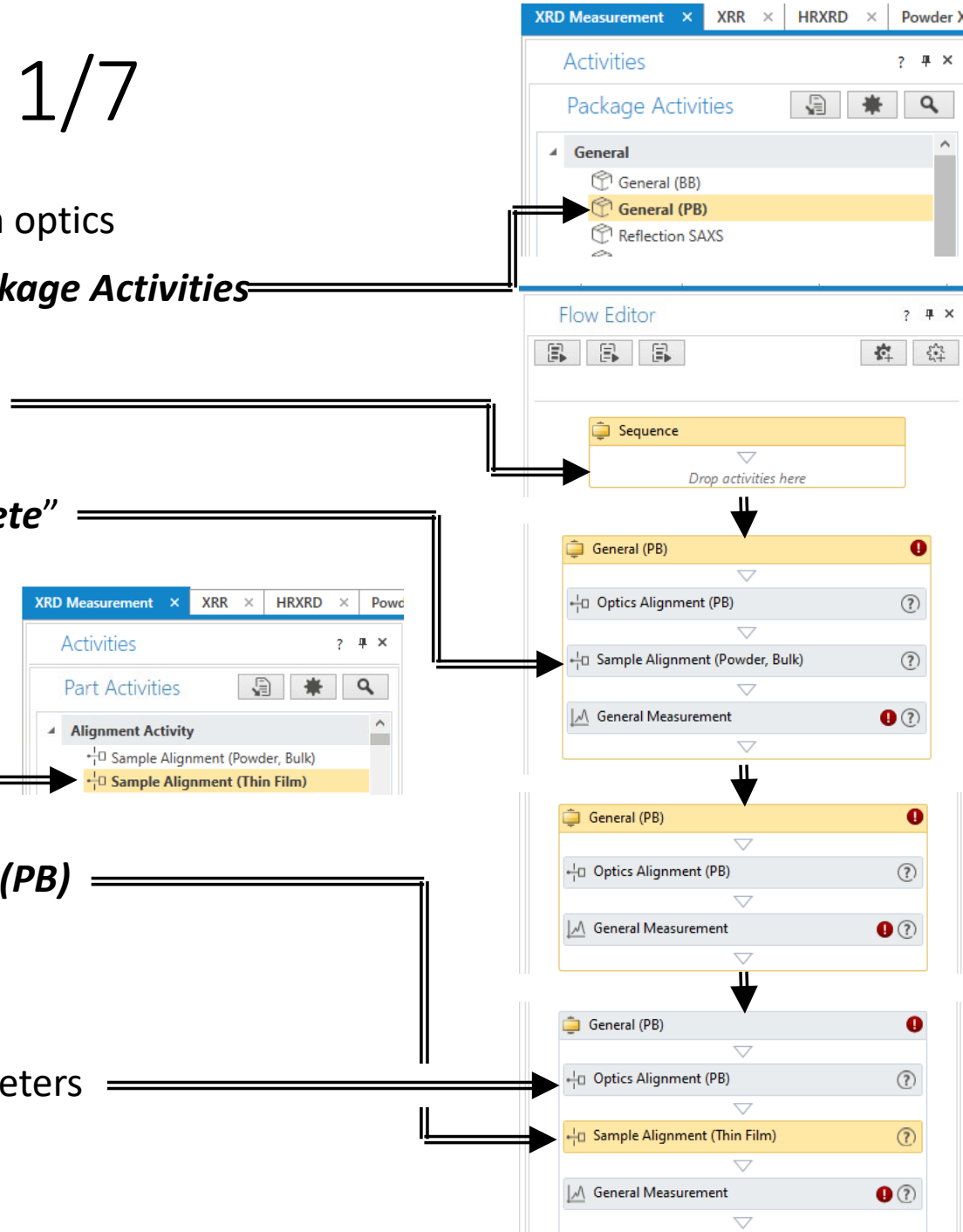
2. Drag the **General (PB)** package into the **Flow Editor** in **Sequence**

3. Right-click on **Sample Alignment (Powder, Bulk)** and select “Delete”

4. Find **Sample Alignment (Thin Film)** under **Part Activities**

5. Drag **Sample Alignment (Thin Film)** under the **Optics Alignment (PB)**

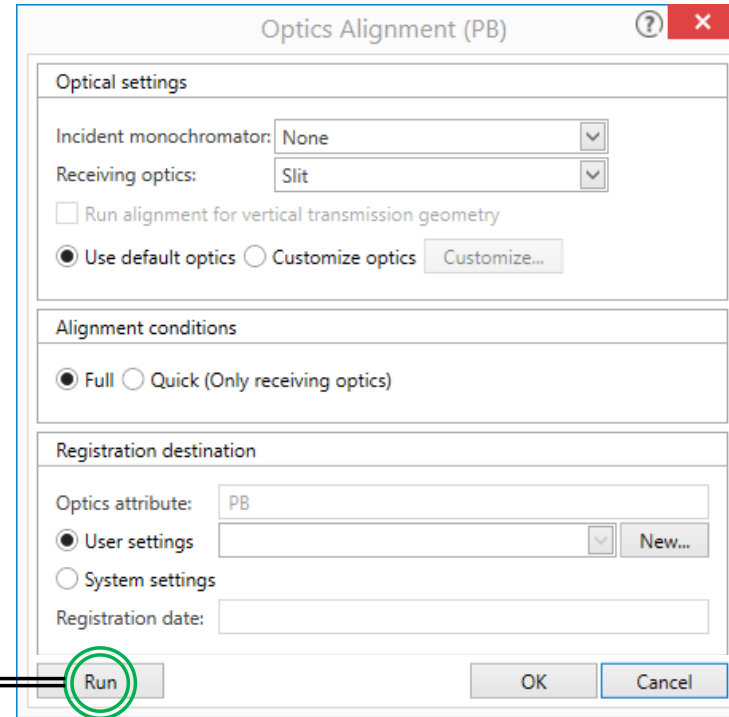
6. Left-click on **Optics Alignment (PB)** tab to confirm default parameters



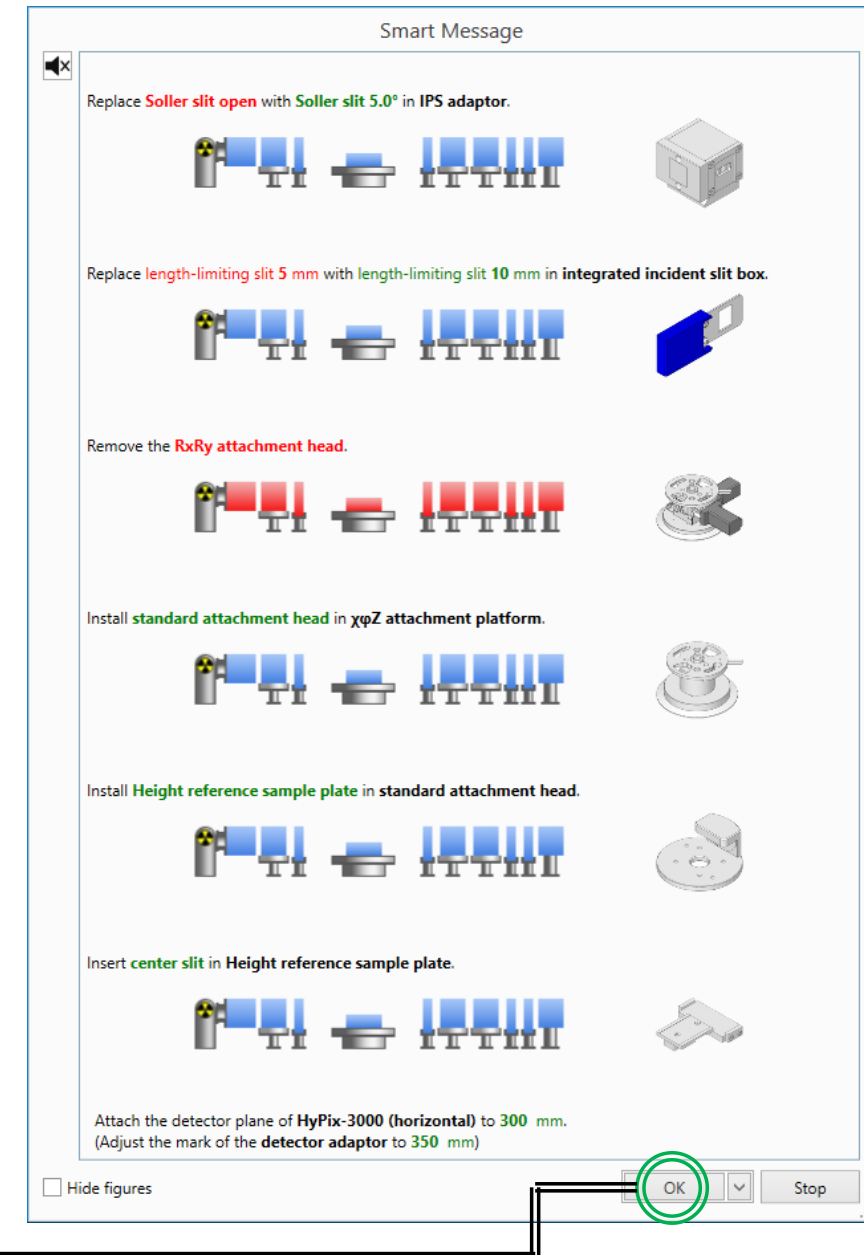


# VII. General (PB) or $2\theta/\omega$ Scan – 2/7

7. Confirm **Use default optics** is selected under **Optical settings**
8. Confirm **Full** is selected under **Alignment conditions**
9. Confirm **User settings** is selected, then click **Run**



10. A **Smart Message** may appear indicating all the optics and attachments that need to be **removed** (indicated in **RED**) and those that need to be **installed** (indicated in **GREEN**)

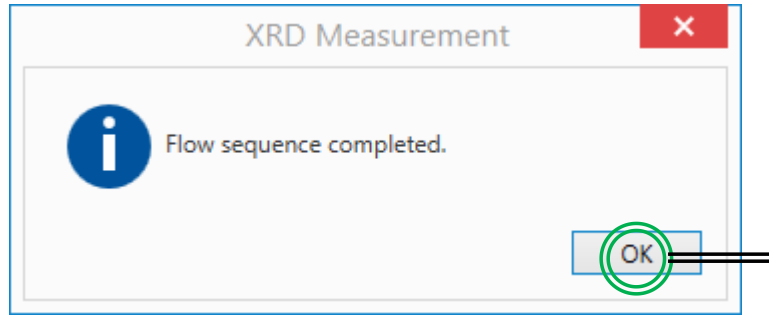


11. Click **OK** when completed

# VII. General (PB) or $2\theta/\omega$ Scan – 3/7

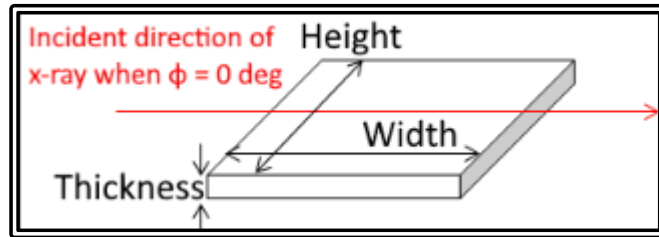
12. System will perform **Optics Alignment** on various axes  
(Average time  $\approx$  4 minutes)

13. Wait for the **Flow sequence completed** prompt to continue by clicking **OK**

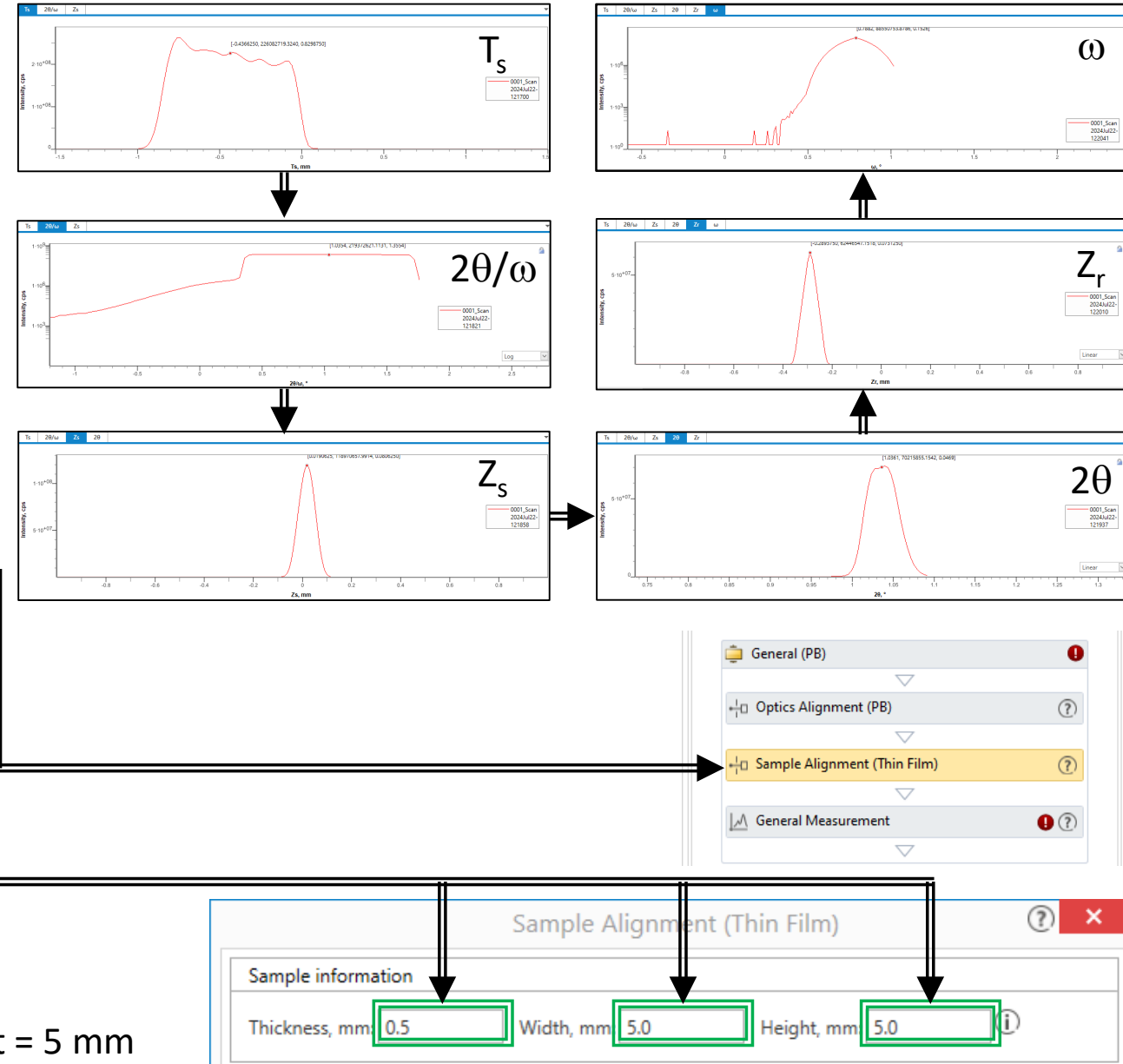


14. Left-click on **Sample Alignment (Thin Film)** to set **Sample Info**

15. Input your **Sample Info** per the dimensions



• For training with Silicon: Thickness = 0.5 mm; Width & Height = 5 mm

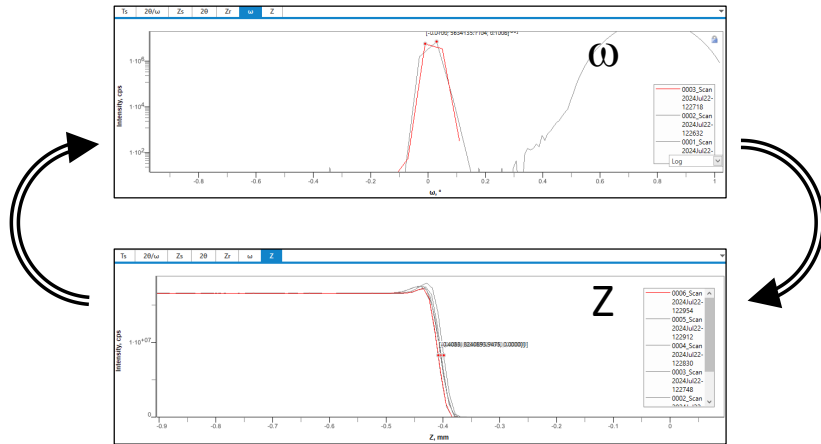


# VII. General (PB) or $2\theta/\omega$ Scan – 4/7

16. Confirm the following are checked:

- Direct beam half cut alignment
- Surface normal alignment
- Put a sample when the sample alignment starts

17. Click **Run** (Average time  $\approx$  6 minutes)



The **Sample Alignment (Thin Film)** dialog box contains the following settings:

- Sample information:** Thickness, mm: 0.5; Width, mm: 5.0; Height, mm: 5.0.
- Alignment conditions:**
  - Attachment and sample plate: RxRy attachment head + 4-inch wafer sample plate
  - ☒ Direct beam half cut alignment
  - ☒ Surface normal alignment
  - Alignment criteria: Standard
  - Surface density: High (> 4.0 g/cm<sup>3</sup>)
  - ☒ Put a sample when the sample alignment starts
- Run recommended sequence** (selected) or **Customize conditions** (with **Customize...** button).
- Run** button (circled in green).
- OK** and **Cancel** buttons.

18. If **Sample Alignment (Thin Film)** fails, try changing surface density

- For training with Silicon: Surface density should be set to High

19. Wait for the **Flow sequence completed** prompt to continue by clicking **OK**

The **XRD Measurement** dialog box displays the message **Flow sequence completed.** and an **OK** button (circled in green).

# VII. General (PB) or $2\theta/\omega$ Scan – 5/7

20. Left-click on **General Measurement** to set scan conditions

	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator	Comment	Options
1	<input checked="" type="checkbox"/>	2θ/ω	Absolute	68.0000	71.0000	0.0100	4.000	1.000	1.000	1.100	Open		Set...
2	<input type="checkbox"/>	θ/2θ	Absolute	3.0000	80.0000	0.0100	4.000	1.000	1.000	1.100	Open		Set...

21. Select  $2\theta/\omega$  for the **Scan Axis**

22. Adjust the following parameters based on your desired scan conditions

- **Start, °:** Enter starting scan position for  $2\theta$  angle (e.g.  $68^\circ$ )
- **Stop, °:** Enter ending scan position for  $2\theta$  angle (e.g.  $71^\circ$ )
- **Step, °:** Enter scan step size for  $2\theta$  angle (e.g.  $0.01^\circ$ ) – controls resolution or spacing of data points
- **Speed, °/min:** Enter the scan speed (e.g.  $4^\circ/\text{min}$ ) – controls the signal/noise (S/N) ratio

23. The following can be increased if you wish to increase the x-ray exposure to your sample in the width dimension

- Incident Slit, mm
  - Receiving Slit #1, mm
  - Receiving Slit #2, mm
- } Default values are automatically chosen based on sample dimensions

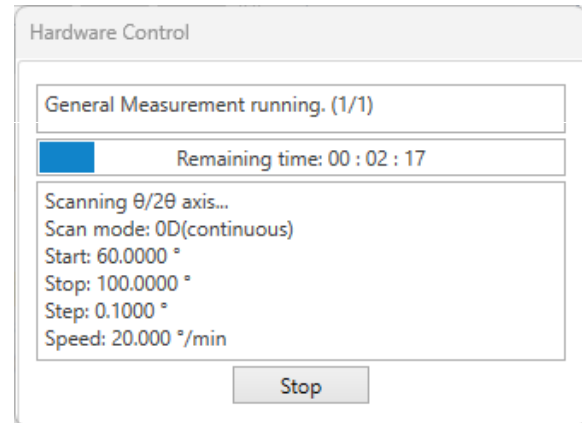
# VII. General (PB) or $2\theta/\omega$ Scan – 6/7

24. If you wish to save your data, click on the **Save measured data** box  
(Note: You can always save your data after collection!)

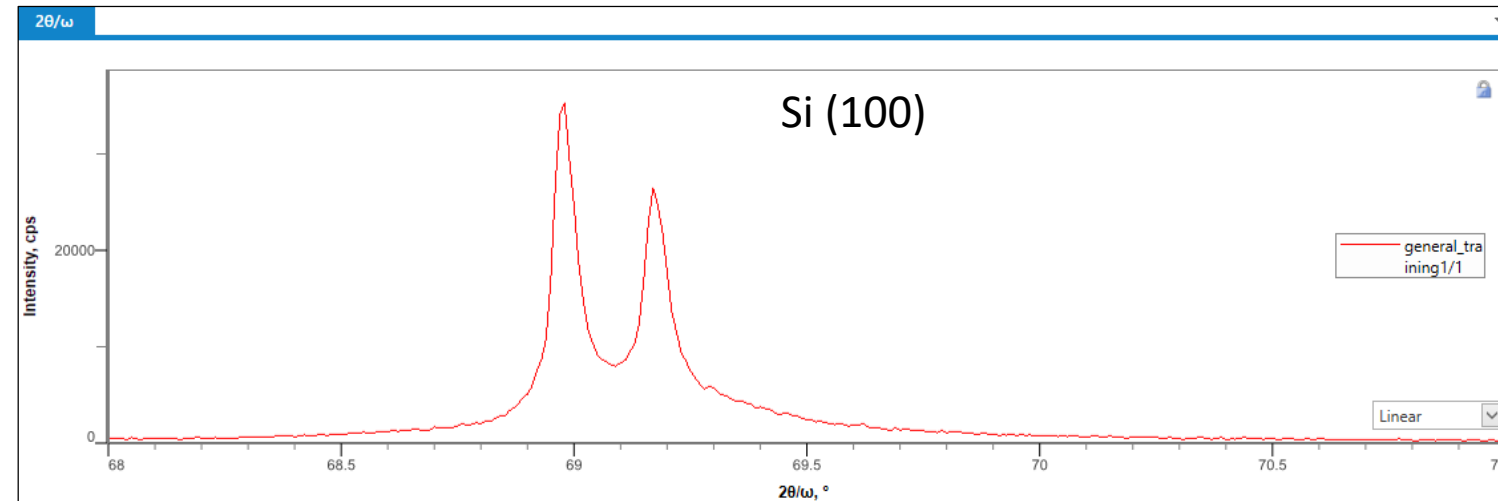
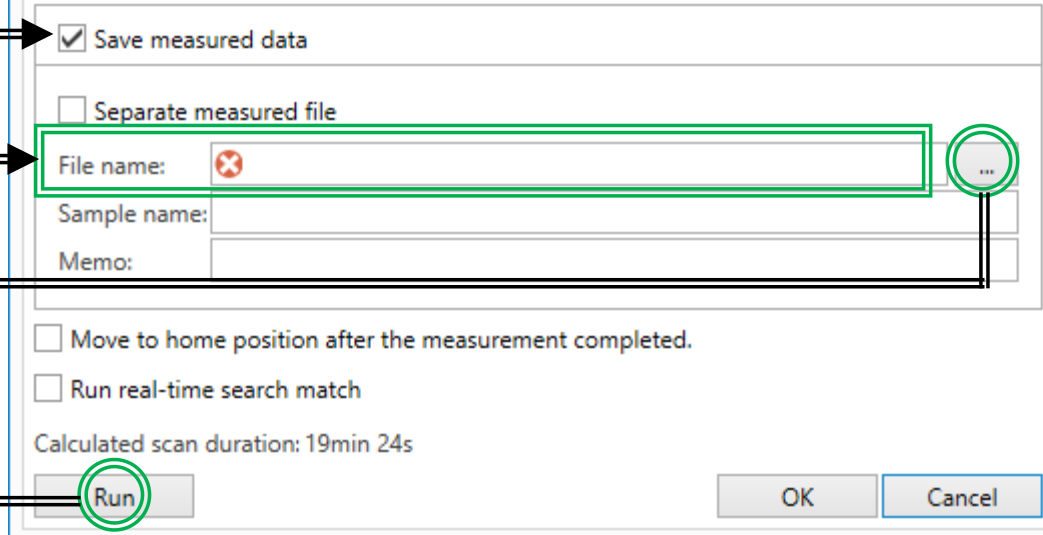
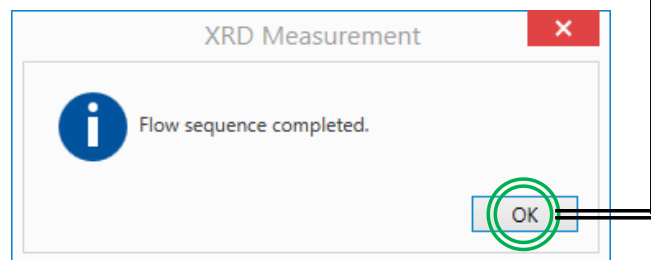
25. Input your desired **File name** and **File location** here

26. Click **Run**

27. The **Remaining time** left in the scan will be displayed



28. Click **OK** when complete



# VII. General (PB) or $2\theta/\omega$ Scan – 7/7

○ Note: The following is for training purposes to learn how to move goniometer axes

24. Open the **RS Viewer** and find the Si (111) plane for diffraction

25. Make sure that you have **SECURED YOUR SAMPLE** before continuing!

26. Click on **Move Axes**

27. The **Goniometer** will now reposition the axes per the values described here

28. Input new **Start** and **Stop** values

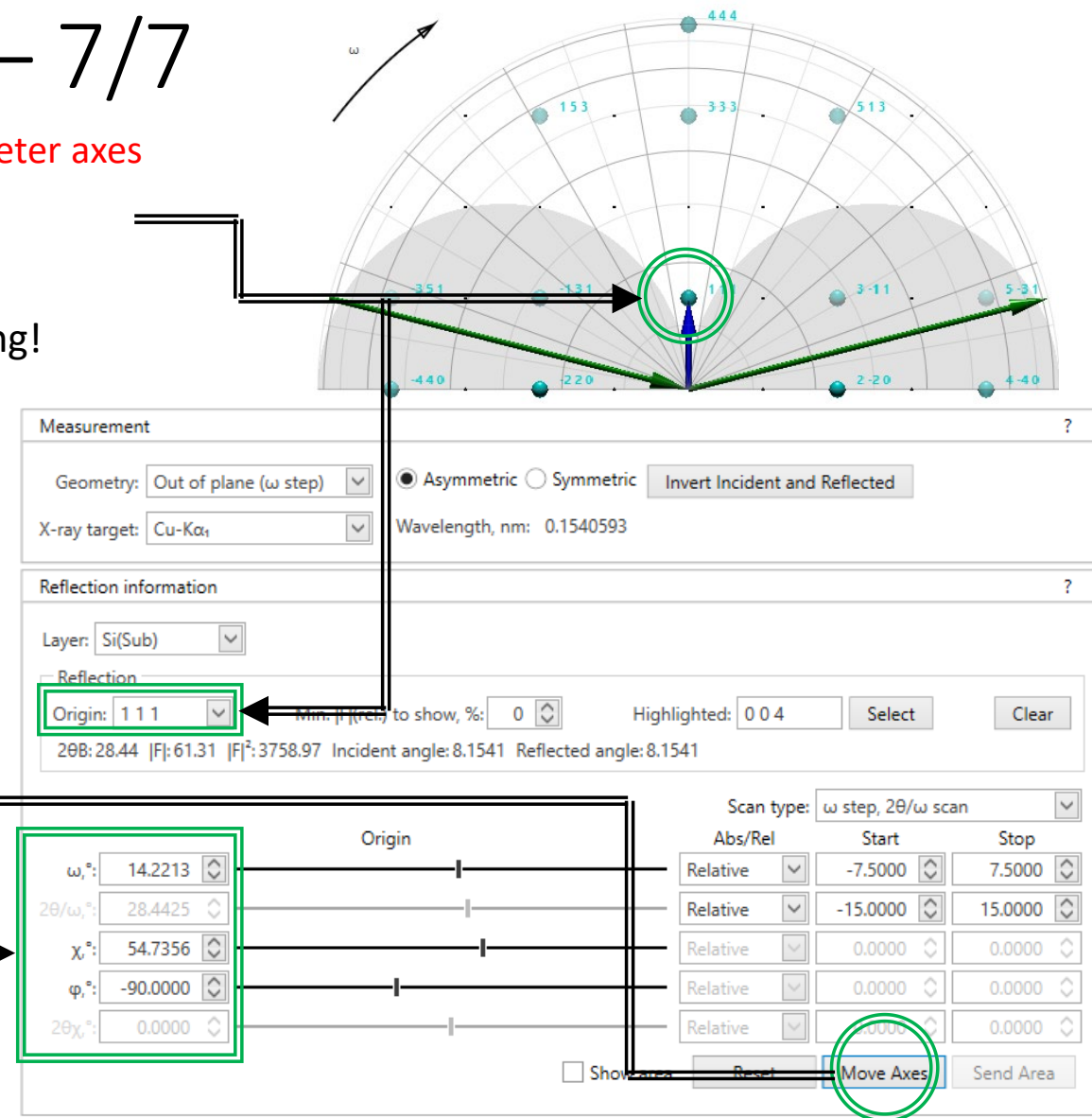
Start, °	Stop, °
27.0000	30.0000

29. Click **Run** (no need to save the scan)

Run

30. Determine if the (111) peak is observed?

31. If the (111) peak is not observed → sample orientation is mismatched with **RS Viewer** via the  $\phi$  position



# VIII. Azimuth or $\varphi$ Scan – 1/3

- This sequence will perform an Azimuth or  $\varphi$  Scan

1. Left-click on **General Measurement**

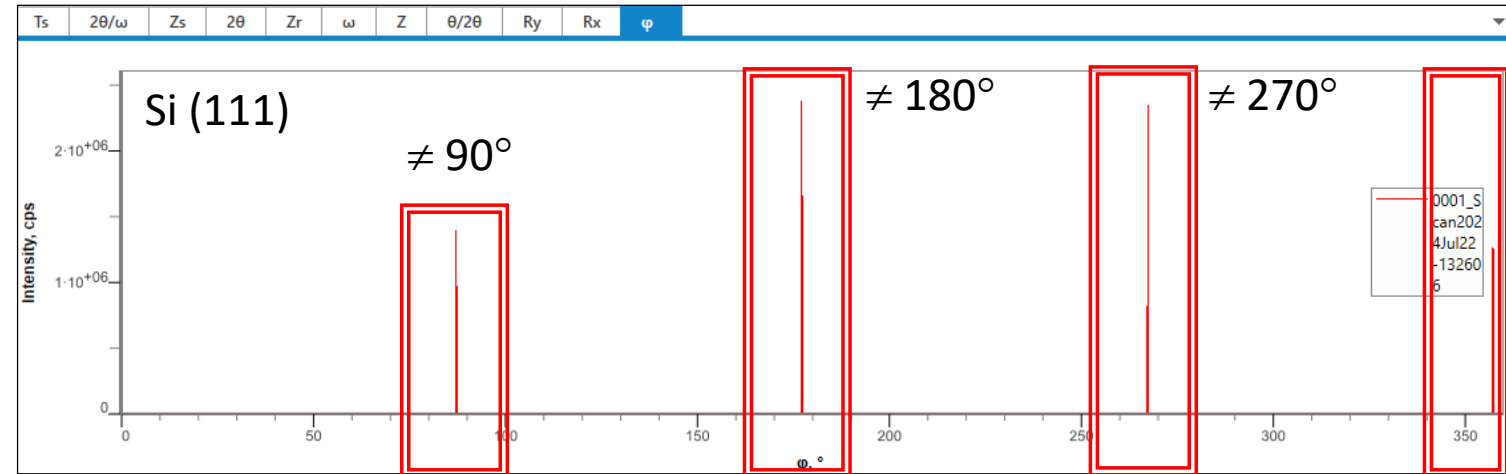
	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator	Comment	Options
1	<input type="checkbox"/>	2 $\theta$ / $\omega$	Absolute	27.0000	30.0000	0.0100	4.000	1.000	1.000	1.100	Open		Set...
2	<input checked="" type="checkbox"/>	$\varphi$	Absolute	0.000	360.000	0.100	120.00	1.000	1.000	1.100	Open		Set...
3	<input type="checkbox"/>	1 $\theta$ / $2\theta$	Absolute	3.0000	80.0000	0.0100	4.000	1.000	1.000	1.100	Open		Set...

2. Select  $\varphi$  for the **Scan Axis** for #2

3. Set **Start** =  $0^\circ$  and **Stop** =  $360^\circ$   
**Step** =  $0.1^\circ$  and **Speed** =  $120^\circ/\text{min}$

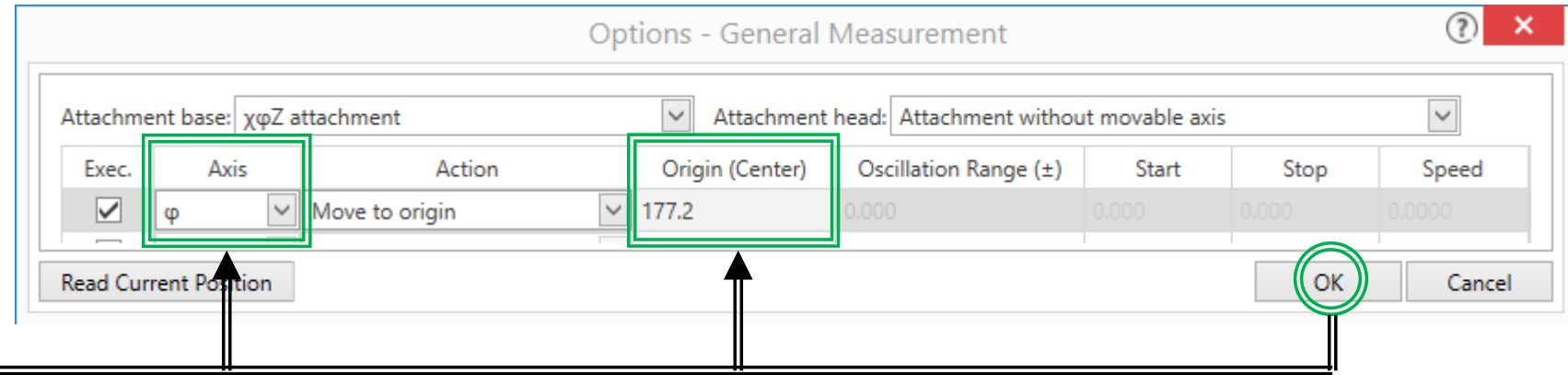
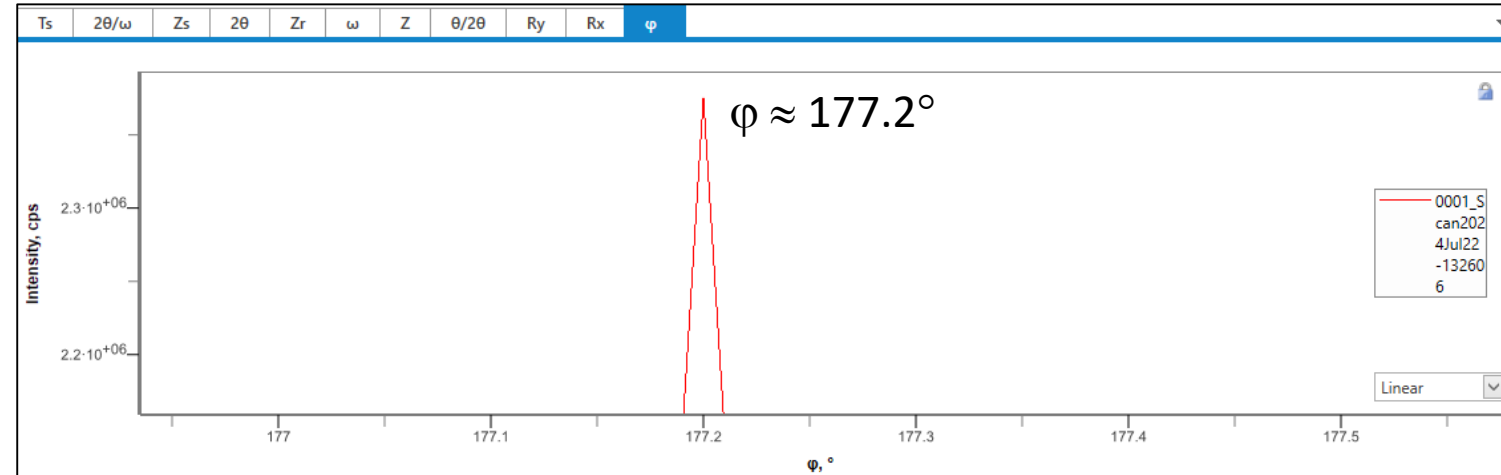
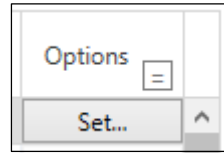
4. Click **Run** (no need to save scan)

5. Your spectra will look similar to this showing the **(111)** peaks are not precisely positioned at  $\varphi = 90^\circ, 180, 270, 360$  due to sample offset!

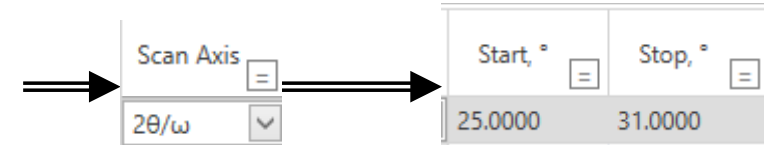


# VIII. Azimuth or $\phi$ Scan – 2/3

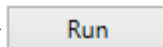
6. Zoom-in one of the peaks...
7. Left-click on **General Measurement** again
8. Click on **Set...** under **Options**
9. Set  $\phi$  to the value of your peak position, and click **OK**



10. Set **Scan Axes** back to **2 $\theta$ / $\omega$**  and input **Start** and **Stop** values back to **27°** and **30°**



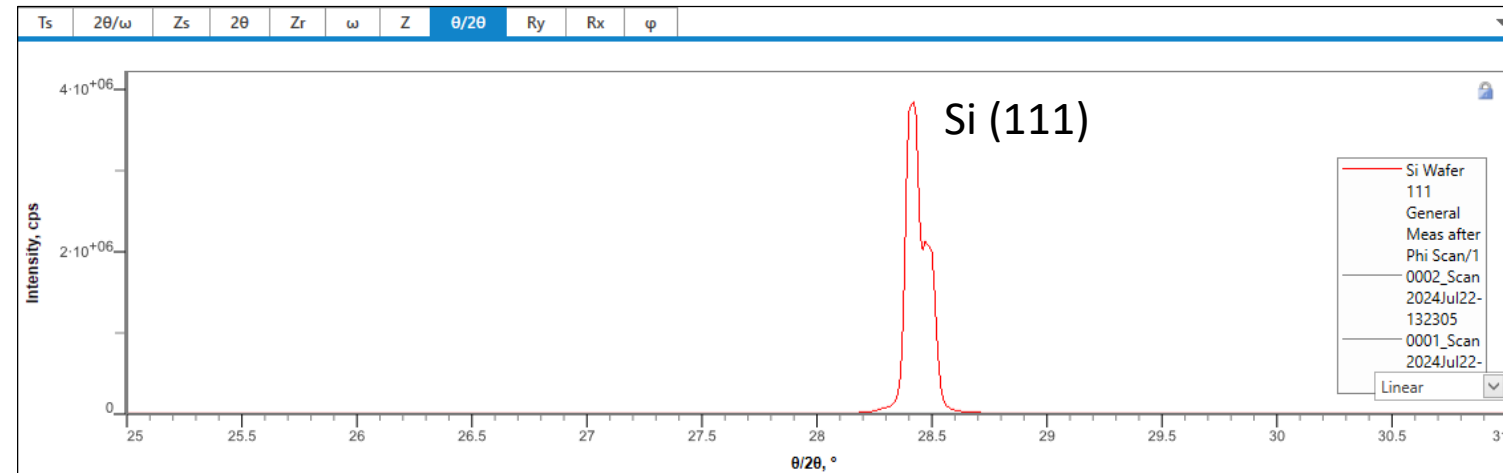
11. Click **Run** again





# VIII. Azimuth or $\phi$ Scan – 3/3

12. You should see a similar spectra showing the **(111)** peak at around  $2\theta_b = 28.4^\circ$  as suggested by the **RS Viewer**



13. Some measurements may perform "**Pre-Measurements**" that will identify and set the goniometer settings (e.g.  $2\theta$ ,  $\omega$ ,  $\phi$ ,  $\theta_\chi$ , etc...) for your scans, but may not always succeed or be available
14. It is still up to the user to be comfortable controlling and moving the **Axes** and setting the **Origin** when appropriate

# IX. Reflectivity – 1/4

○ This sequence will perform a Reflectivity Measurement

1. Select the **Reflectivity** package under **XRD Measurement > Package Activities**

2. Drag the **Reflectivity** package into the **Flow Editor** in **Sequence**

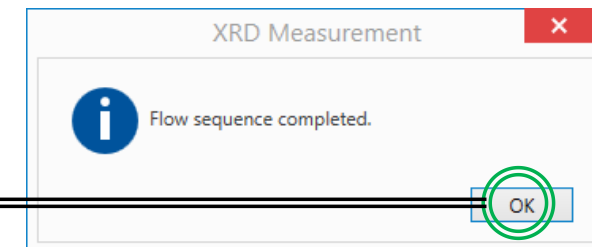
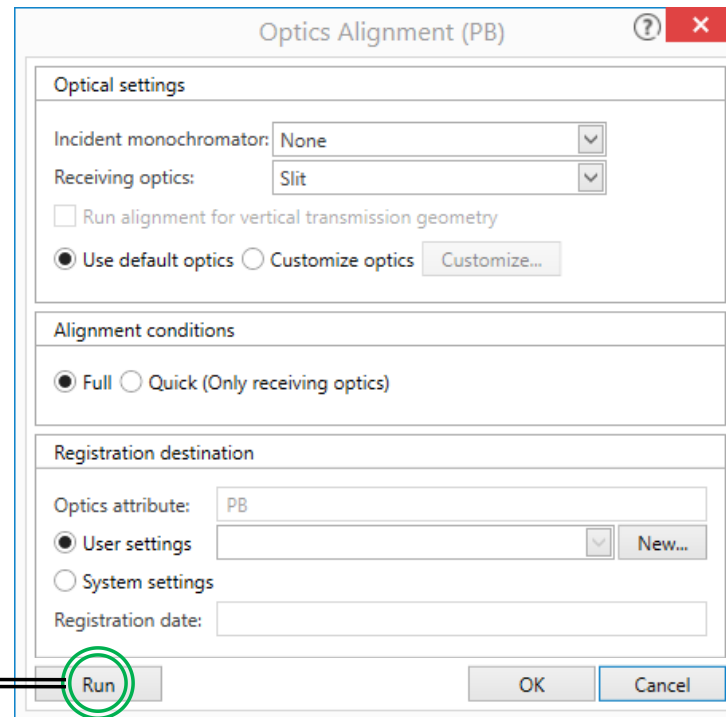
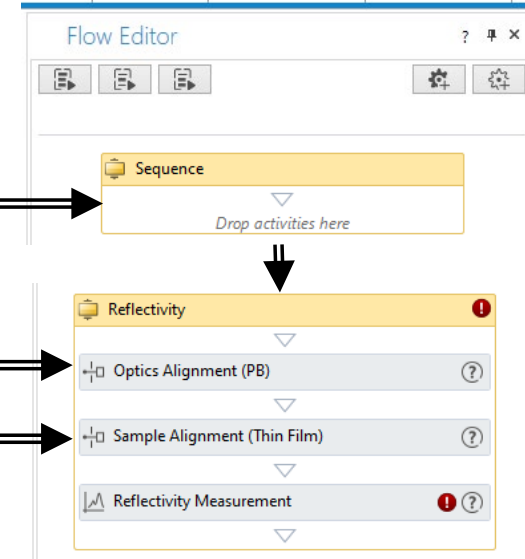
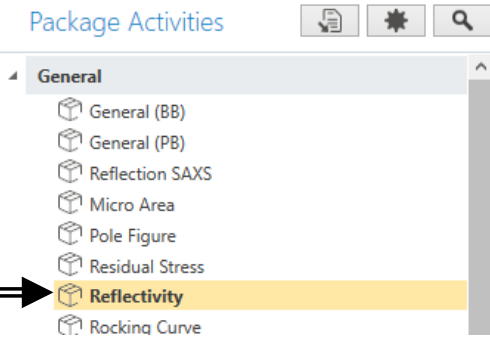
3. If **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** were previously performed, then skip to **Step 11**

4. Left-click on **Optics Alignment (PB)**

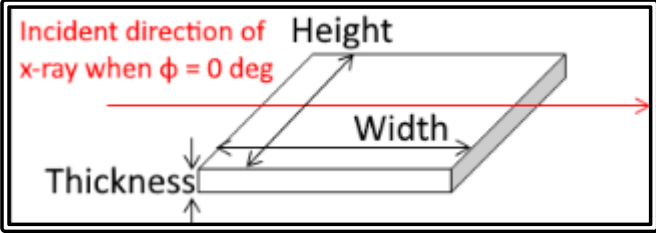
5. Confirm the following are selected:

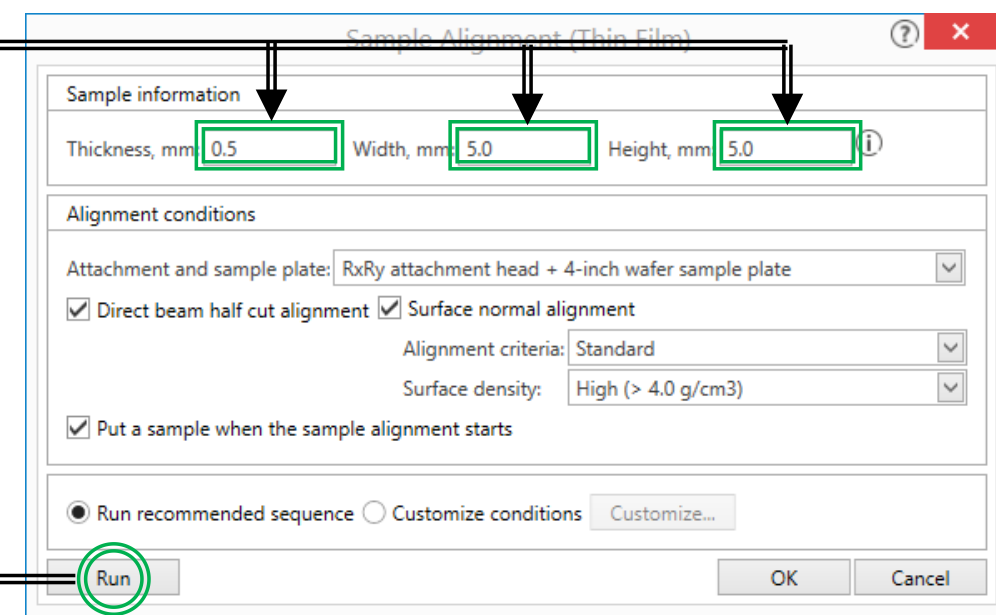
- **Optical settings** → **Use default optics**
- **Alignment conditions** → **Full**
- **Registration destination** → **User settings**

6. Click **Run** and then **OK** when completed



# IX. Reflectivity – 2/4

7. Left-click on **Sample Alignment (Thin Film)**
8. Input your **Sample Info** per the dimensions 
9. Confirm the following are checked:
  - Direct beam half cut alignment
  - Surface normal alignment
  - Put a sample when the sample alignment starts
10. Click **Run** and then **OK** when completed
11. Left-click on **Reflectivity Measurement**
12. Adjust the **Requested scan duration** or use default time  
For training with Silicon: Set duration to 2 min



Sample Alignment (Thin Film)

Sample information

Thickness, mm: 0.5 Width, mm: 5.0 Height, mm: 5.0

Alignment conditions

Attachment and sample plate: RxRy attachment head + 4-inch wafer sample plate

☒ Direct beam half cut alignment ☒ Surface normal alignment

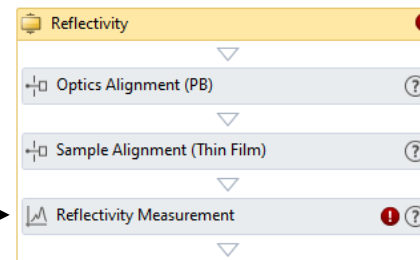
Alignment criteria: Standard

Surface density: High (> 4.0 g/cm<sup>3</sup>)

☒ Put a sample when the sample alignment starts

☒ Run recommended sequence ☐ Customize conditions Customize...

Run OK Cancel

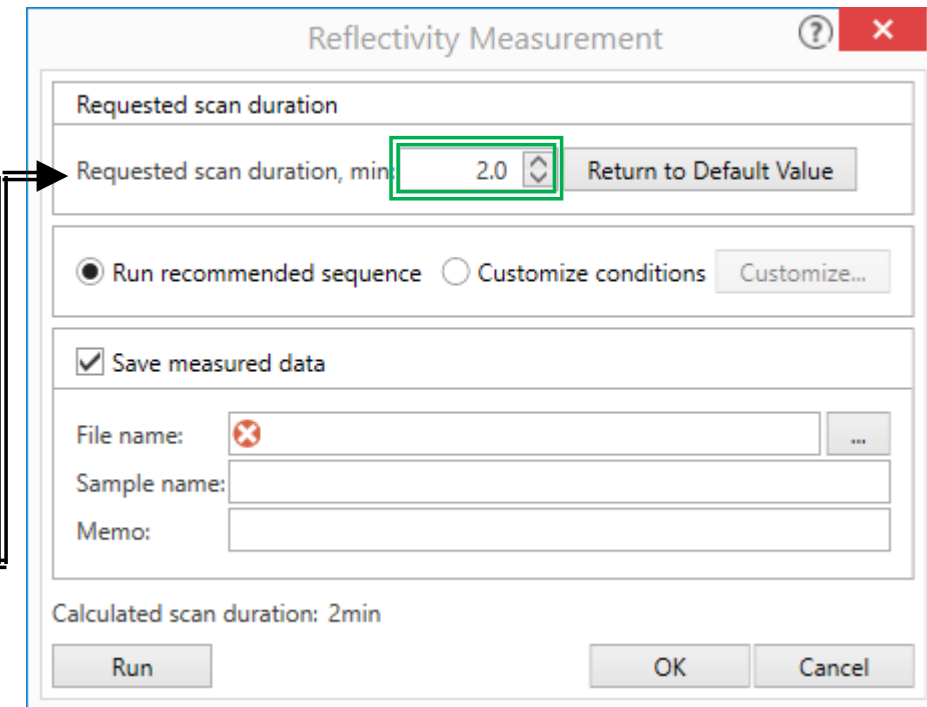


Reflectivity

Optics Alignment (PB)

Sample Alignment (Thin Film)

Reflectivity Measurement



Reflectivity Measurement

Requested scan duration

Requested scan duration, min: 2.0 Return to Default Value

☒ Run recommended sequence ☐ Customize conditions Customize...

☒ Save measured data

File name:

Sample name:

Memo:

Calculated scan duration: 2min

Run OK Cancel

# IX. Reflectivity – 3/4

13. Input your desired **File name** and **File location** here

14. Select **Customize conditions** if you wish

15. You may include different scan parameters such as the **Step** and **Speed** for different **2θ** ranges

13. Click **OK** then click **Run**

☒ Run recommended sequence

☐ Customize conditions

Customize...

☒ Save measured data

File name:

Sample name:

Memo:

Calculated scan duration: 15min

Run

OK

Cancel

Customize - Reflectivity Measurement

Requested scan duration

Requested scan duration, min: 5

Return to Default Value

☐ Manual exchange slit conditions:

Incident Soller slit: Soller slit 5.0°

Receiving optics: Slit

Length-limiting slit: 10 mm

Receiving Soller slit: Soller slit 5.0°

Read Current Optics

Scan conditions

Scan mode: 0D(continuous)

Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator
<input checked="" type="checkbox"/>	2θ/ω	Absolute	0.0000	5.0000	0.0100	12.000	0.050	0.250	0.300	Auto
<input checked="" type="checkbox"/>	2θ/ω	Absolute	5.0000	10.0000	0.0100	6.000	0.050	0.250	0.300	Auto
<input type="checkbox"/>	2θ/ω	Absolute	0.0000	10.0000	0.0100	0.667	0.050	0.250	0.300	Auto
<input type="checkbox"/>	2θ/ω	Absolute	0.0000	10.0000	0.0100	0.667	0.050	0.250	0.300	Auto
<input type="checkbox"/>	2θ/ω	Absolute	0.0000	10.0000	0.0100	0.667	0.050	0.250	0.300	Auto

Calculated scan duration: 1min 15s

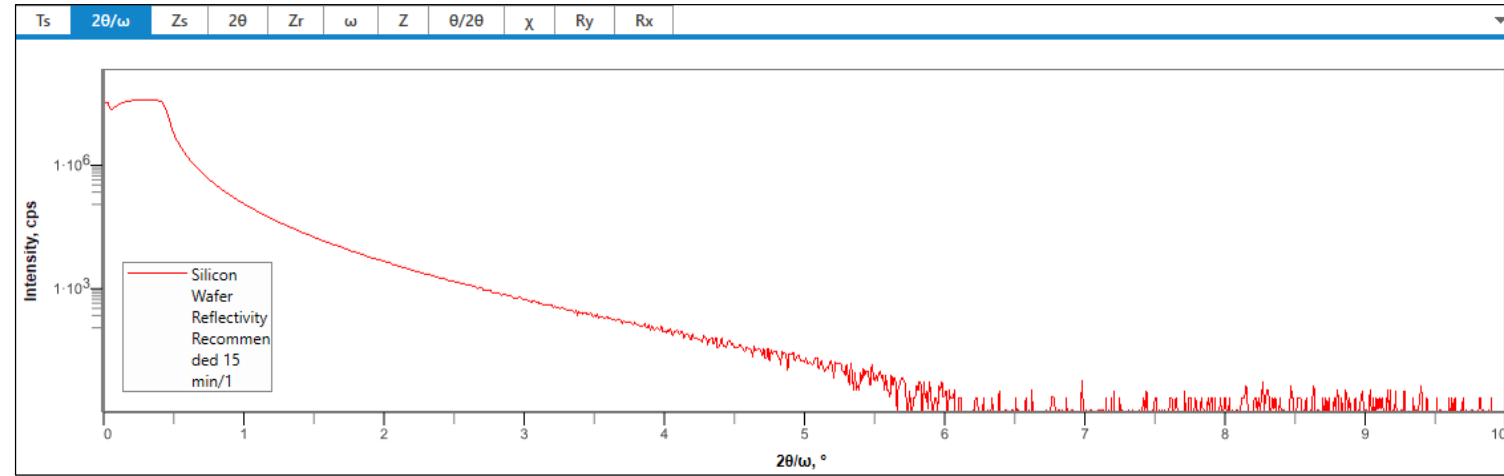
Set Recommended Values

OK

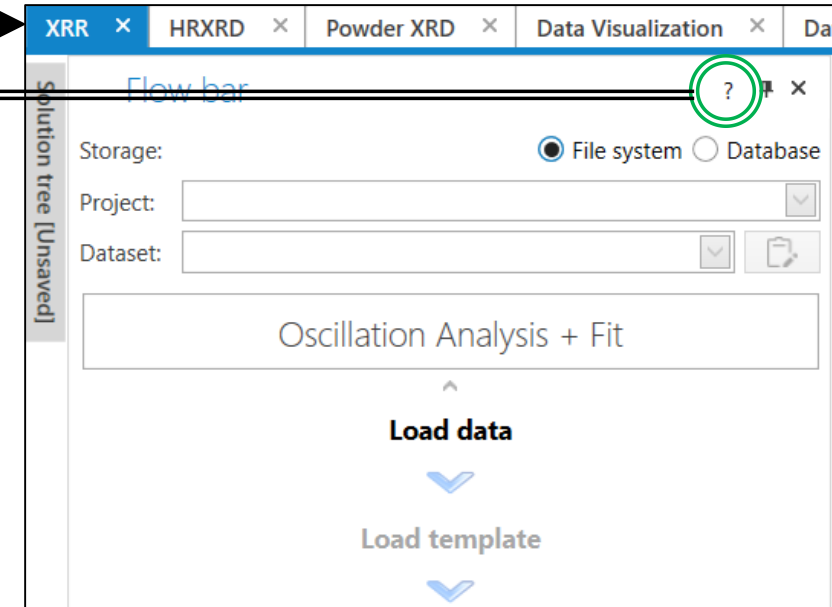
Cancel

# IX. Reflectivity – 4/4

17. The **XRR Plugin** allows you to analyze data



18. Click on the ? icon to open up the **XRR\_UserManual\_en.pdf** to review the tutorial



# X. Pole Figure – 1/5

○ This sequence will perform a Pole Figure using Parallel Beam optics

1. Select the **Pole Figure** package under **XRD Measurement > Package Activities**

2. Drag the **Pole Figure** package into the **Flow Editor** in **Sequence**

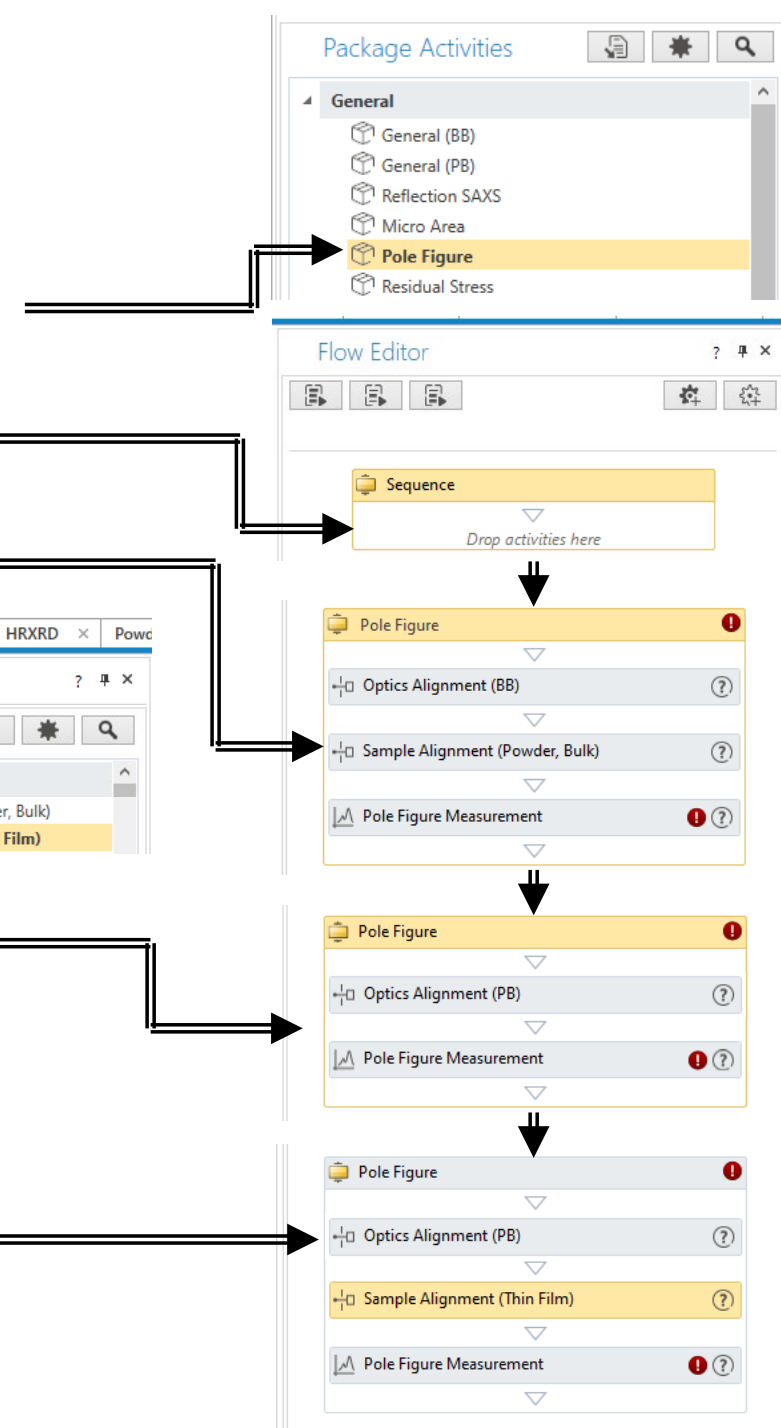
3. Right-click on **Optics Alignment (BB)** and **Sample Alignment (Powder, Bulk)** and select “Delete”

4. Find **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** under **Part Activities**

5. Drag **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** above the **Pole Figure Measurement**

6. If **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** were previously performed, then skip to **Step 14**

7. Left-click on **Optics Alignment (PB)**



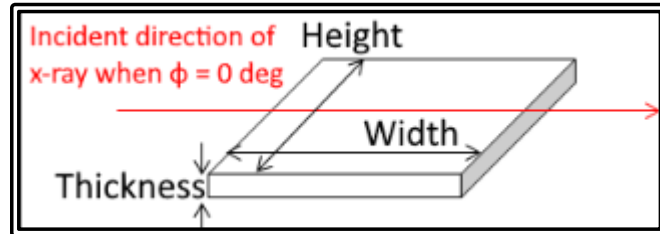
# X. Pole Figure – 2/5

8. Confirm the following are selected:
- **Optical settings** → *Use default optics*
  - **Alignment conditions** → *Full*
  - **Registration destination** → *User settings*

9. Click **Run** and then **OK** when completed

10. Left-click on **Sample Alignment (Thin Film)**

11. Input your **Sample Info** per the dimensions



12. Confirm the following are checked:
- Direct beam half cut alignment
  - Surface normal alignment
  - Put a sample when the sample alignment starts

The screenshot shows the XRD Measurement software interface. The 'Optics Alignment (PB)' dialog is open, showing 'Optical settings' (Incident monochromator: None, Receiving optics: Slit), 'Alignment conditions' (Full selected), and 'Registration destination' (User settings selected). The 'Run' button is circled in green. A message box titled 'XRD Measurement' says 'Flow sequence completed.' with an 'OK' button circled in green. The 'Sample Alignment (Thin Film)' dialog is also open, showing 'Sample information' (Thickness: 0.5 mm, Width: 5.0 mm, Height: 5.0 mm) and 'Alignment conditions' (Direct beam half cut alignment, Surface normal alignment, and Put a sample when the sample alignment starts are all checked). The 'Sample Alignment (Thin Film)' button in the main interface is highlighted in yellow.



# X. Pole Figure – 3/5

13. Click **Run** and then **OK** when completed

14. Left-click on **Pole Figure Measurement**

15. Remember to input the following parameters!

- **Sample information**
- **Crystal system** (i.e. Cubic)
- **Index** (i.e. 111)
- **Measurement angle** (i.e.  $2\theta_b$ )

16. Choose if any **Background measurements** will be run if desired

17. Choose to **Run recommended sequence** or **Customize conditions**

The screenshot shows the XRD Measurement software interface. At the top, there is a 'Run' button circled in green. Below it, a dialog box titled 'XRD Measurement' shows 'Flow sequence completed.' with an 'OK' button circled in green. On the right, a sidebar titled 'Pole Figure' lists 'Optics Alignment (PB)', 'Sample Alignment (Thin Film)', and 'Pole Figure Measurement' (which is highlighted with a red icon). The main 'Pole Figure Measurement' dialog box is open, showing the following fields:

- Sample information:** Thickness, mm: 0.50000; Width, mm: 5.0; Crystal system: Cubic; Linear absorption coefficient, 1/cm: 0.000.
- Pole figure measurement conditions:** Index: 1 1 1; Measurement angle, °: 28.7480; Transmission: Not run; Reflection: Schulz; Background measurement: Not run; Step: Fine, Standard, Coarse (Coarse is selected); Speed: Slow, Standard, Fast (Fast is selected).
- Run/Customize section:** Run recommended sequence (selected), Customize conditions, Customize...

Arrows from the numbered instructions point to the following elements:

- 13. 'Run' button and 'OK' button in the 'XRD Measurement' dialog box.
- 14. 'Pole Figure Measurement' in the sidebar.
- 15. 'Sample information' section.
- 16. 'Thickness, mm', 'Width, mm', 'Crystal system', 'Index', 'Measurement angle, °', and 'Background measurement' fields.
- 17. 'Run recommended sequence' radio button.

# X. Pole Figure – 4/5

18. Depending on your **Step** chosen, be aware that it may not be sufficient so choose the following carefully for  $\alpha$  scan axes!

- **Start**
- **Stop**
- **Step**
- **Speed**

Data measurement conditions

2 $\theta$  angle, ° (Transmission): 28.4664    2 $\theta$  angle, ° (Reflection): 28.4664     $\gamma$  axis oscillation: Not run

Geometry	Step Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Incident Slit	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator
Transmission	$\alpha$	OD(step)	Absolute	0.000	15.264	5.000	0.1 mm	10.000	9.900	Open
Reflection	$\alpha$	OD(step)	Absolute	15.264	55.264	5.000	1/6°	10.000	9.900	Open

Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min
$\beta$	OD(continuous)	Absolute	0.000	360.000	5.000	200.00

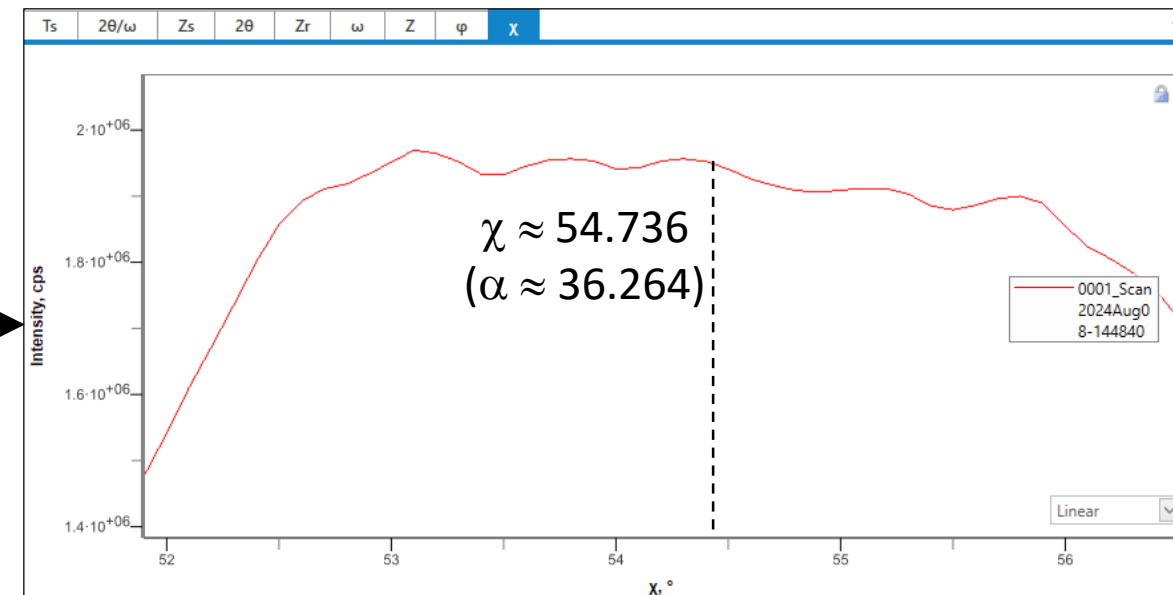
Remember that  $\alpha = 90^\circ - \chi$

(Note: Will the peaks appear for  $\alpha$  if arbitrarily chosen?)

19. If not performed, identify the optimal  $\chi$  tilt for your peak

↓

	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min
1	<input checked="" type="checkbox"/>	$\chi$	Absolute	50.000	60.000	0.100	15



# X. Pole Figure – 5/5

18. If **Background measurements** were selected, determine the desired conditions

☒ Background measurement conditions

		Background #1				Background #2			
Geometry	Step Axis	Step, °	2 $\theta$ Angle, °	Receiving Slit #1, mm	Receiving Slit #2, mm	2 $\theta$ Angle, °	Receiving Slit #1, mm	Receiving Slit #2, mm	
Transmission	$\alpha$	5	25.7480	10.000	9.900	31.7480	10.000	9.900	
Reflection	$\alpha$	5.000	25.7480	10.000	9.900	31.7480	10.000	9.900	

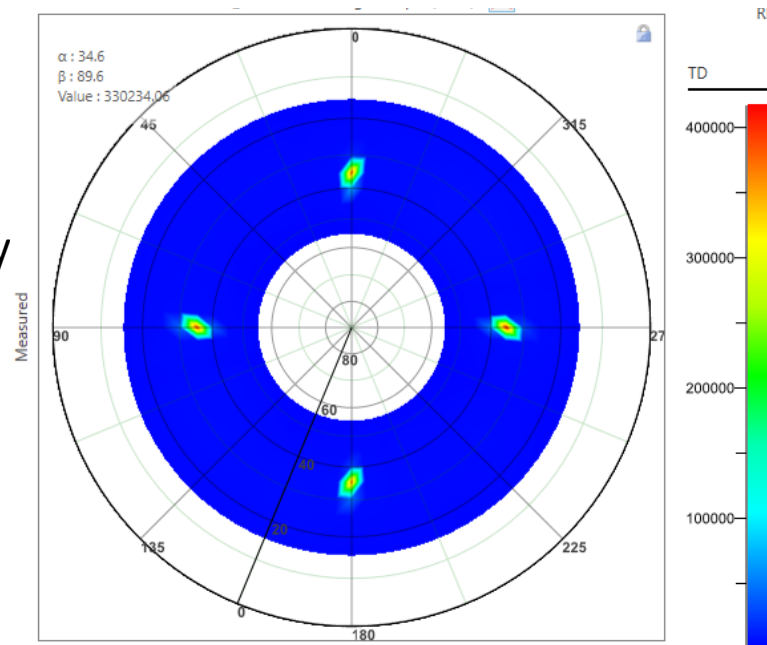
Scan Axis	Background Data Acquisition Method	Scan Mode	Range	Start, °	Stop, °	Step, °	Duration, s
$\beta$	1 point ( $\beta$ = Minimum intensity)	Fixed time	Absolute	177.200	537.200	5.000	1.5

20. Input your desired **File name** and **File location** here

21. Click **Run** then **OK** when completed

For training with Silicon: Do not Run

22. If the parameters were chosen properly, you should eventually see intensity peaks appear at the appropriate  $\alpha$  and  $\beta$  positions



☐ Run recommended sequence ☒ Customize conditions Customize...

☒ Save measured data

File name:

Sample name:

Memo:

Calculated scan duration: 34min 15s

Run OK Cancel

XRD Measurement

i Flow sequence completed.

OK

# XI. Rocking Curve – 1/5

○ This sequence will perform a Rocking Curve using Parallel Beam optics

1. Select the **Rocking Curve** package under **XRD Measurement > Package Activities**

2. Drag the **Rocking Curve** package into the **Flow Editor** in **Sequence**

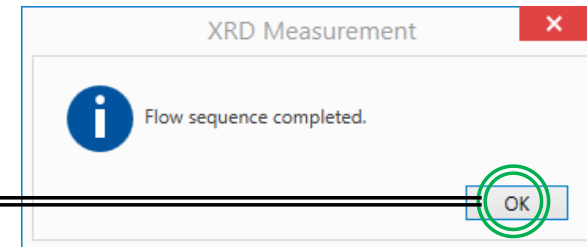
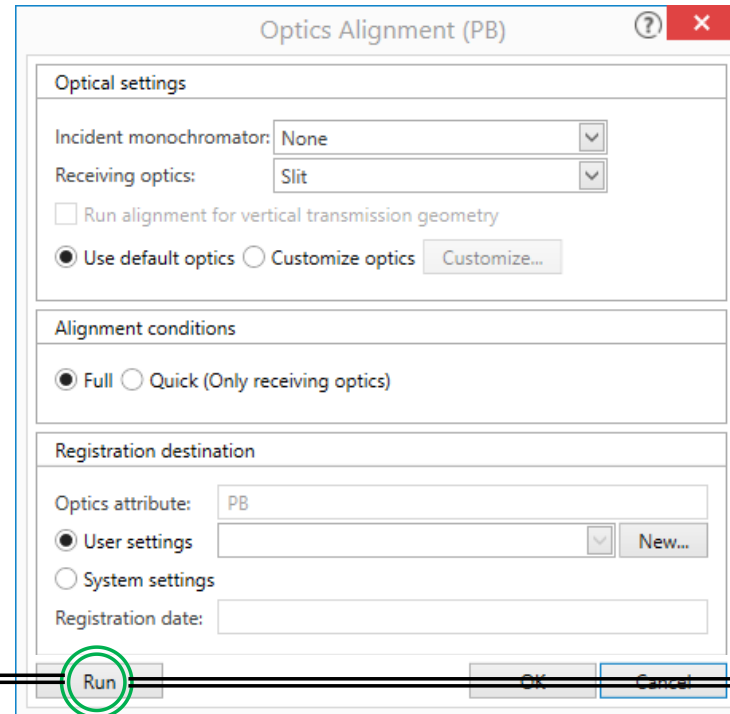
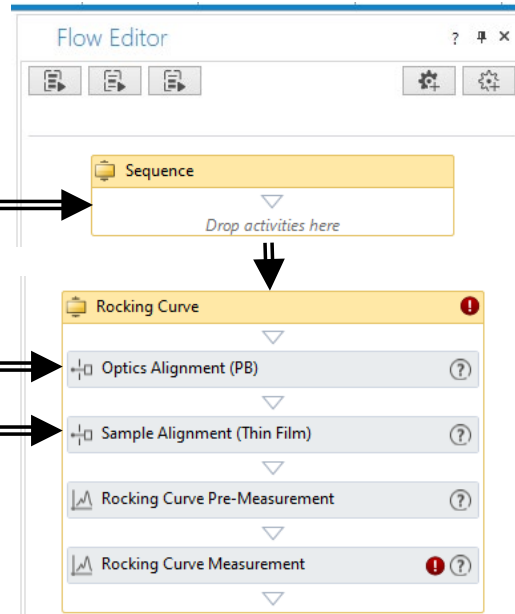
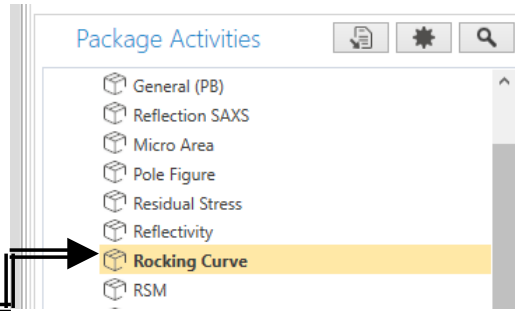
3. If **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** were previously performed, then skip to **Step 11**

4. Left-click on **Optics Alignment (PB)**

5. Confirm the following are selected:

- **Optical settings** → **Use default optics**
- **Alignment conditions** → **Full**
- **Registration destination** → **User settings**

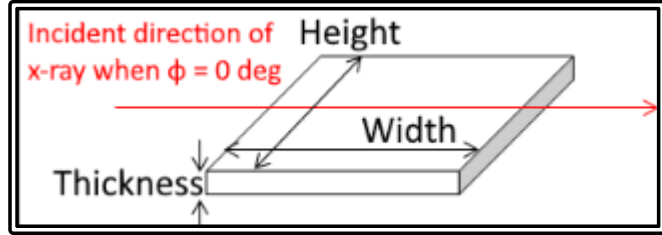
6. Click **Run** and then **OK** when completed



# XI. Rocking Curve – 2/5

7. Left-click on **Sample Alignment (Thin Film)**

8. Input your **Sample Info** per the dimensions



Sample Alignment (Thin Film)

Sample information

Thickness, mm: 0.5 Width, mm: 5.0 Height, mm: 5.0

Alignment conditions

Attachment and sample plate: RxRy attachment head + 4-inch wafer sample plate

☒ Direct beam half cut alignment ☒ Surface normal alignment

Alignment criteria: Standard

Surface density: High (> 4.0 g/cm<sup>3</sup>)

☒ Put a sample when the sample alignment starts

☒ Run recommended sequence ☐ Customize conditions Customize...

Run OK Cancel

9. Confirm the following are checked:

- Direct beam half cut alignment
- Surface normal alignment
- Put a sample when the sample alignment starts

10. Click **Run** and then **OK** when completed

11. Left-click on **Rocking Curve Pre-Measurement**

12. Determine the desired **Alignment method** (see next page)

Rocking Curve

Optics Alignment (PB)

Sample Alignment (Thin Film)

Rocking Curve Pre-Measurement

Rocking Curve Measurement

Rocking Curve Pre-Measurement

Rocking curve pre-measurement conditions

Alignment method: Without axis alignment

☐ Optimize 2θ

☐ Axis realignment after 2θ optimization

Scanning sequence: From ω to χ(φ)

☒ 2θ, °: 69.1280

☒ ω, °: 34.5640

☒ χ, °: 0.000

☒ φ, °: 0.000

Read Current Positions

☒ Run recommended sequence ☐ Customize conditions Customize...

☒ Save measured data

File name:

Sample name:

Memo:

Post measurement

☐ Change ω offset so that ω position becomes a half of 2θ position

Calculated scan duration: 0s

Run OK Cancel



# XI. Rocking Curve – 3/5

Alignment Method	Description
Without alignment	Drive each axis to the reflection position specified in the <b>Move origin</b> section. An additional alignment will not be performed.
Quick axis alignment	Drive each axis to the reflection position specified in the <b>Move origin</b> section, and align the $\omega$ and $\chi$ (or Rx) axes (for symmetric reflection) or $\phi$ axis (for asymmetric reflection).
Recursive axis alignment	Drive each axis to the reflection position specific in the <b>Move origin</b> section, and perform the $\omega$ scan as driving the $\chi$ (or Rx) axis (for symmetric reflection) or $\phi$ axis (for asymmetric reflection) step-by-step. Plot the peak intensity vs. the $\chi$ (or Rx) or $\phi$ axis to the optimized position, then align the $\omega$ axis.
Standard axis alignment	Drive each axis to the reflection position specified in the <b>Move origin</b> section, and perform the Rx, Ry scan to face the $\phi$ axis to the normal of the lattice plane. Then, align the $\omega$ axis.
Precise axis alignment	Drive each axis to the reflection position specified in the <b>Move origin</b> section, and perform the $\omega$ scan at four positions ( $\phi = 0^\circ, 180^\circ, 90^\circ, -90^\circ$ ) to face the $\phi$ axis to the normal of the lattice plane. Then, align the $\omega$ (and $\chi$ ) axes.

13. Depending on the **Alignment method** chosen, it will perform additional alignment – recommend **Quick axis alignment**

14. If available, also recommend performing:
- Optimize 2 $\theta$
  - Axis realignment after 2 $\theta$  optimization

15. Before proceeding, check that your **Origin Position** is set to desired plane using **RS Viewer** before proceeding

The screenshot shows the 'Rocking Curve Pre-Measurement' dialog box. It has a title bar with a question mark and a close button. The main area is divided into two panes. The left pane, titled 'Rocking curve pre-measurement conditions', contains the following settings: 'Alignment method' is set to 'Quick axis alignment' (indicated by an arrow from step 13); 'Optimize 2 $\theta$ ' is checked (indicated by an arrow from step 14); 'Axis realignment after 2 $\theta$  optimization' is checked (indicated by an arrow from step 14); and 'Scanning sequence' is set to 'From  $\omega$  to  $\chi(\phi)$ '. The right pane contains a 'Move to origin' checkbox which is checked, and four input fields for axis positions: '2 $\theta$ , °' (69.1280), ' $\omega$ , °' (34.5640), ' $\chi$ , °' (0.000), and ' $\phi$ , °' (0.000). Below these fields is a 'Read Current Positions' button. At the bottom of the dialog, there are three buttons: 'Reset', 'Move Axes' (circled in green with an arrow from step 15), and 'Send Area'. A green circle is also drawn around the 'Read Current Positions' button, with an arrow pointing to it from the 'Move Axes' button.

# XI. Rocking Curve – 4/5

16. Proceed to check the Post measurement setting
  - Change  $\omega$  offset so that  $\omega$  position becomes a half of  $2\theta$  position
17. Click on **Run**
18. Axes will be aligned per the chosen **Alignment method**

☒ Run recommended sequence ☐ Customize conditions [Customize...](#)

☐ Save measured data

File name:

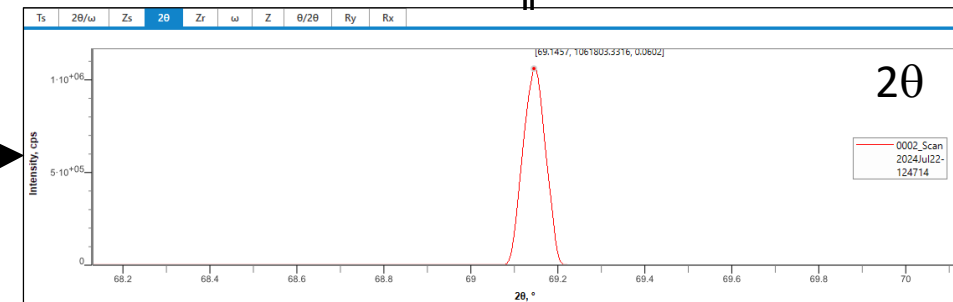
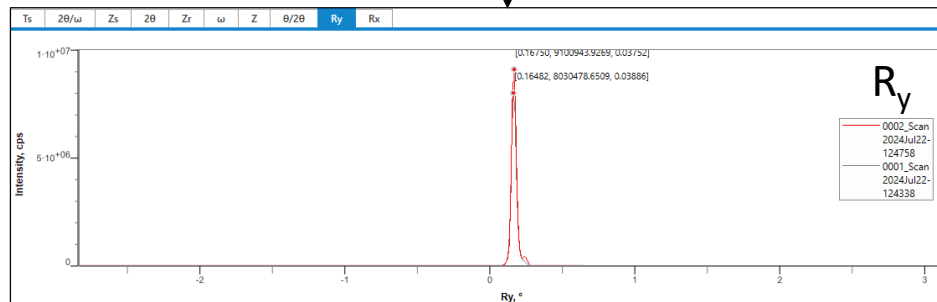
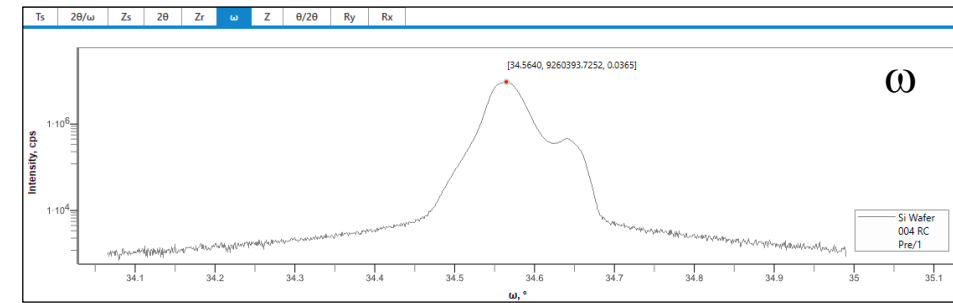
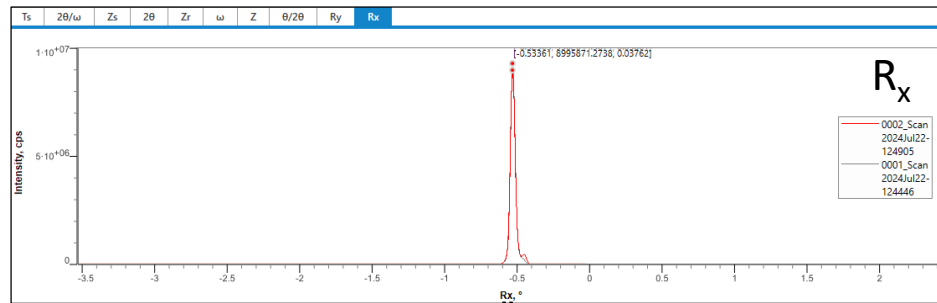
Sample name:

Memo:

Post measurement

☒ Change  $\omega$  offset so that  $\omega$  position becomes a half of  $2\theta$  position

Calculated scan duration: 8min 6s



# XI. Rocking Curve – 5/5

19. Left-click on **Rocking Curve Measurement**

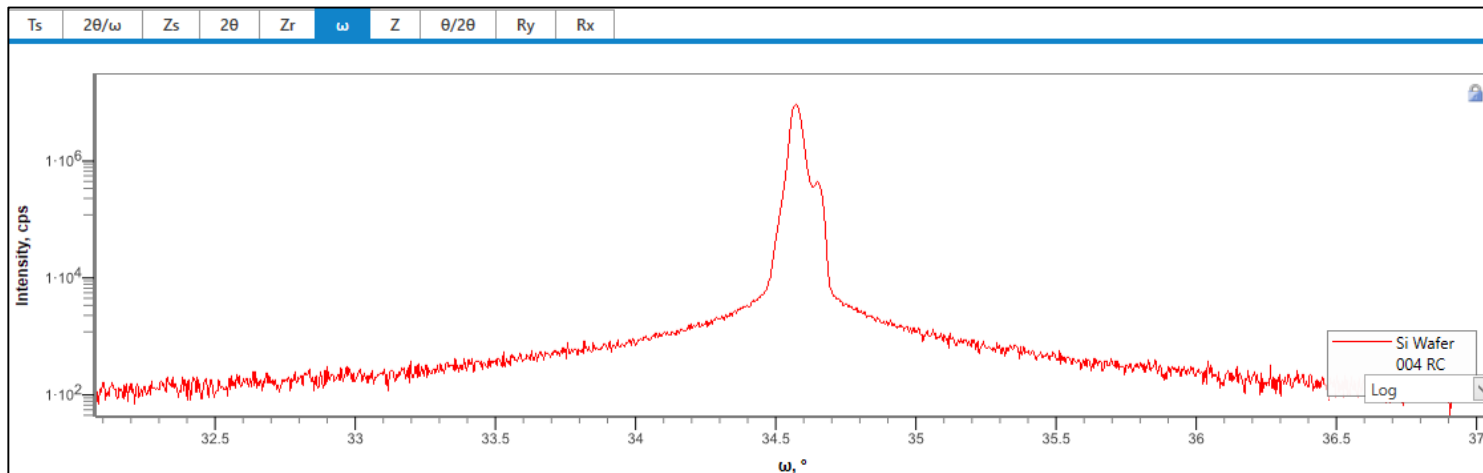
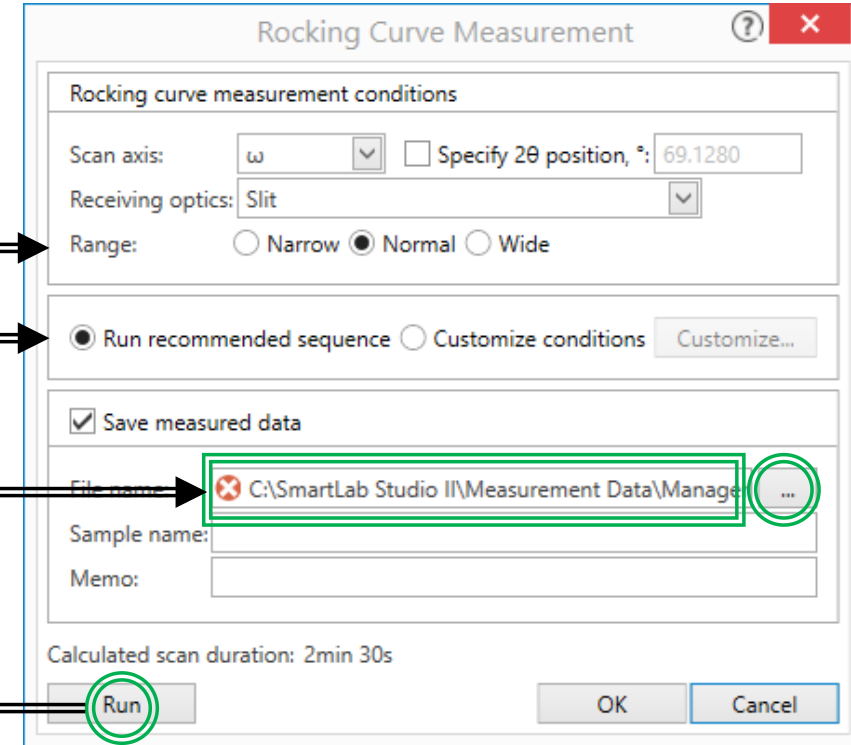
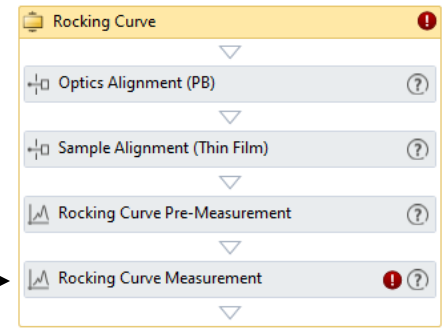
20. Determine the **Range** if choosing to **Run recommended sequence**:

- Narrow
- Normal
- Wide

21. Select **Customize conditions** if you wish

22. Input your desired **File name** and **File location** here

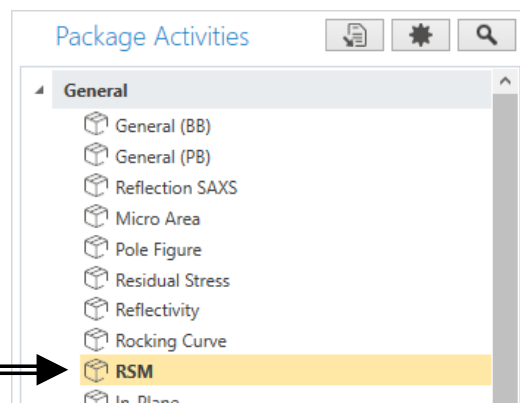
23. Click **OK** then click **Run**



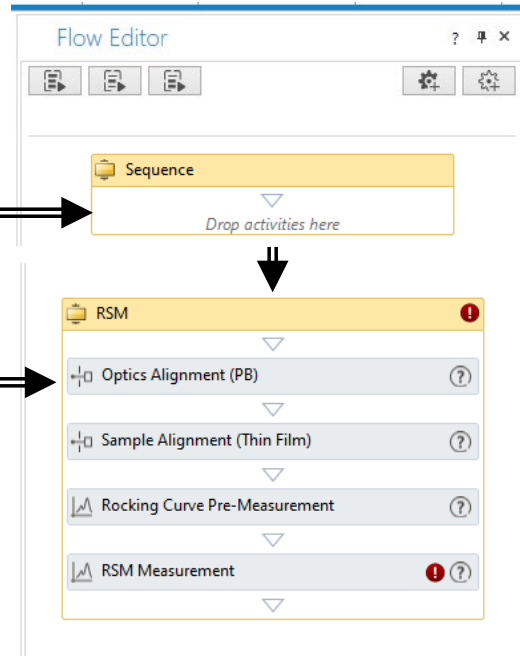
# XII. Reciprocal Space Map (RSM) – 1/5

○ This sequence will perform a Reciprocal Space Map or RSM using Parallel Beam optics

1. Select the **RSM** package under **XRD Measurement > Package Activities**

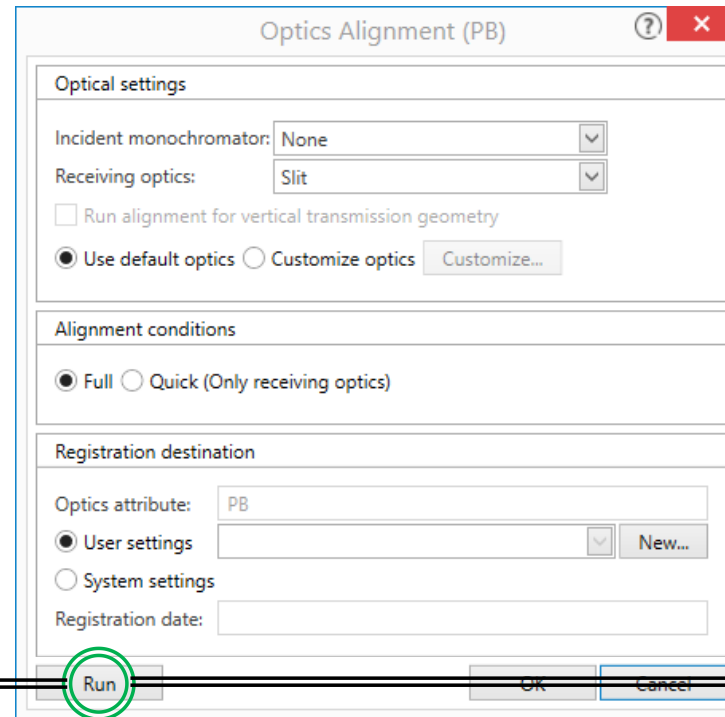


2. Drag the **RSM** package into the **Flow Editor** in **Sequence**



3. If **Optics Alignment (PB)** and **Sample Alignment (Thin Film)** were previously performed, then skip to **Step 11**

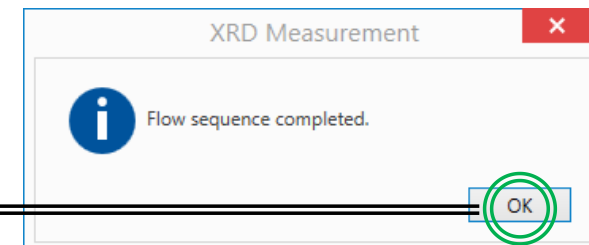
4. Left-click on **Optics Alignment (PB)**



5. Confirm the following are selected:

- **Optical settings** → **Use default optics**
- **Alignment conditions** → **Full**
- **Registration destination** → **User settings**

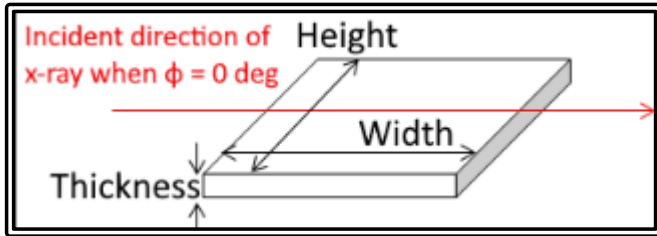
6. Click **Run** and then **OK** when completed



## XII. Reciprocal Space Map (RSM) – 2/5

7. Left-click on **Sample Alignment (Thin Film)**

8. Input your **Sample Info** per the dimensions

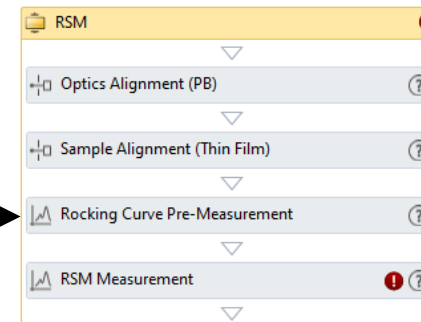
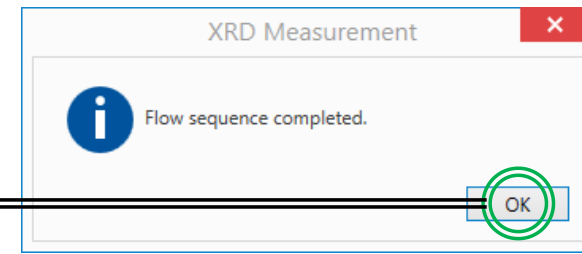
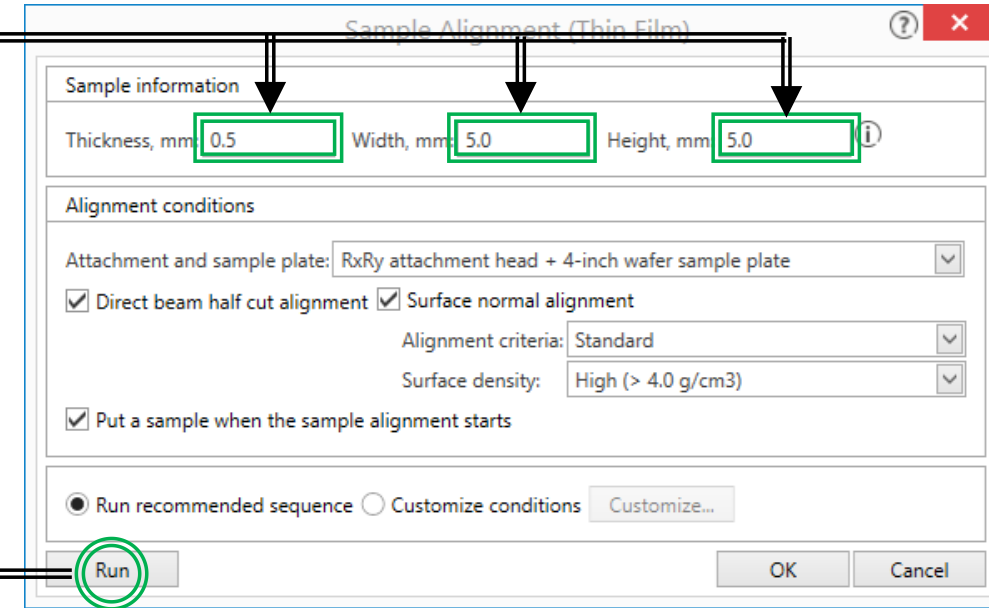
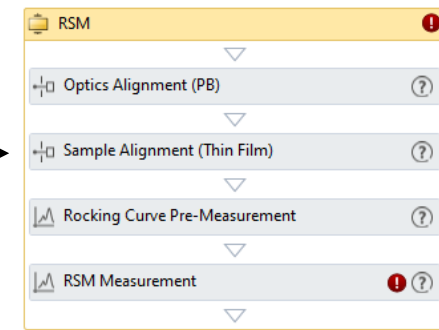


9. Confirm the following are checked:

- Direct beam half cut alignment
- Surface normal alignment
- Put a sample when the sample alignment starts

10. Click **Run** and then **OK** when completed

11. Left-click on **Rocking Curve Pre-Measurement**





# XII. Reciprocal Space Map (RSM) – 3/5

12. Determine the desired **Alignment method** (see **XI. Rocking Curve** for review)

13. Before proceeding, check that your **Origin Position** is set to desired plane using **RS Viewer** before proceeding

14. Click **Run** and then **OK** when completed

Customize – Rocking Curve Pre-Measurement

Rocking curve pre-measurement conditions

Alignment method: **Quick axis alignment**

☒ Optimize 2 $\theta$

☒ Axis realignment after 2 $\theta$  optimization

Scanning sequence: From  $\omega$  to  $\chi(\varphi)$

☐ Clear previous alignment results

☒ Move to origin

☒ 2 $\theta$ , °: 28.4426

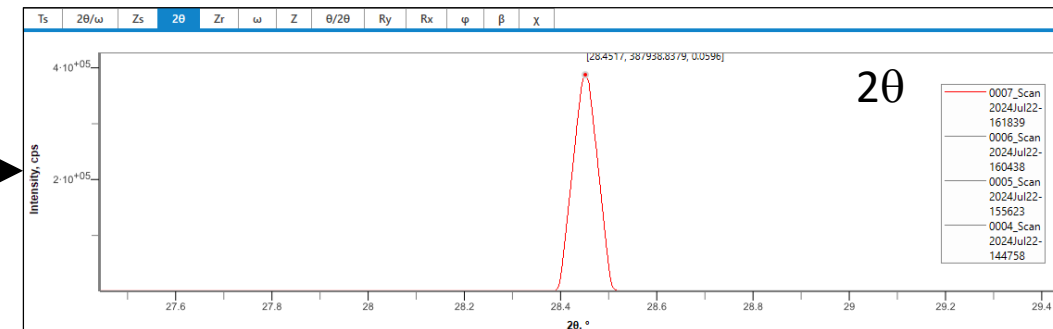
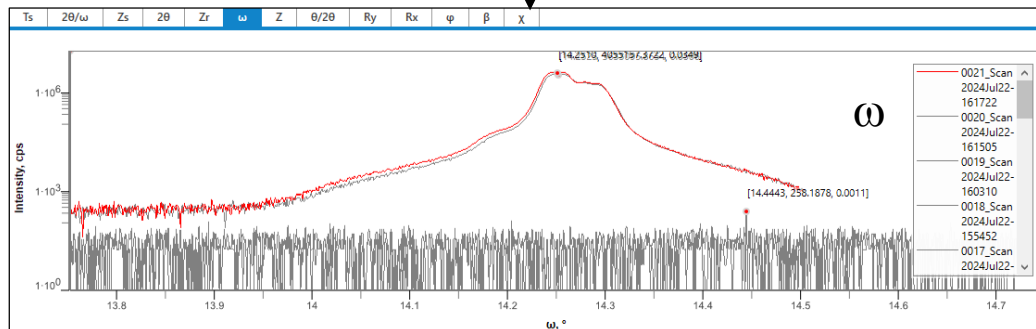
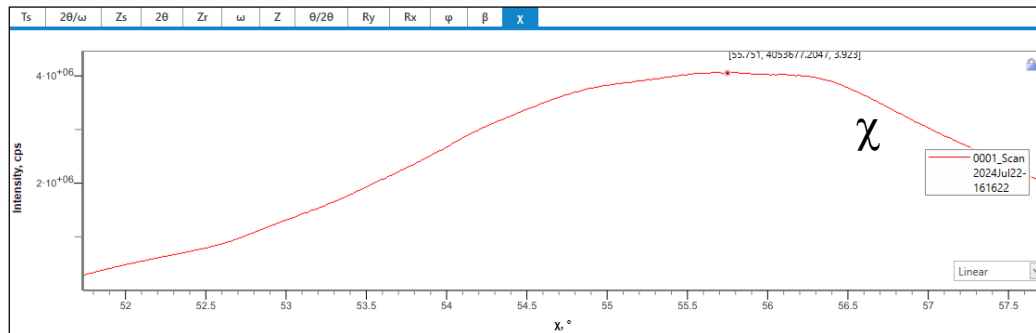
☒  $\omega$ , °: 14.2213

☒  $\chi$ , °: 54.736

☒  $\varphi$ , °: 177.200

Read Current Positions

$\varphi$  needs to be optimized separately!



XRD Measurement

Flow sequence completed.

OK

# XII. Reciprocal Space Map (RSM) – 4/5

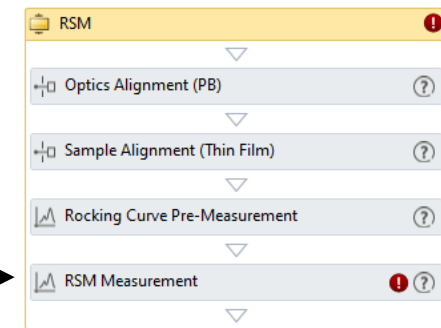
15. Left-click on **RSM Measurement**

16. Click on **Read Current Positions** to align axis positions from **Pre-Measurement**

17. Use the **Run recommended sequence** or click **Customize conditions**

18. Adjust the following parameters if desired:

- **Start**
- **Stop**
- **Step**
- **Speed**



RSM Measurement

RSM measurement conditions

Detector: HyPix-3000 (horizontal)

Detector mode: OD(continuous)

Data acquisition method:  $\omega$  step,  $2\theta/\omega$  scan

Receiving optics: Slit

Detector distance, mm: 150

Range: ☐ Narrow ☒ Normal ☐ Wide

Move to origin ☒

☒  $2\theta$ , °: 28.4517

☒  $\omega$ , °: 14.2512

☒  $\chi$ , °: 55.737

☒  $\phi$ , °: 177.2

Read Current Positions

Scan range simulation

Sample information: ... DB Launch RS Viewer...

☒ Run recommended sequence ☐ Customize conditions Customize...

Scan conditions

Incident slit, mm: 1.000 Receiving slit #1, mm: 1.000 Receiving slit #2, mm: 1.100

Step Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Number of Steps
$\omega$	---	Relative	-3.0000	3.0000	0.5000	13

Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min	Attenuator
$2\theta/\omega$	OD(continuous)	Relative	-3.0000	3.0000	0.5000	100.000	Auto

# XII. Reciprocal Space Map (RSM) – 5/5

19. Input your desired **File name** and **File location** here

20. Click **Run** then **Ok** then OK when completed

For training with Silicon: Do not Run

☒ Save measured data


File name:

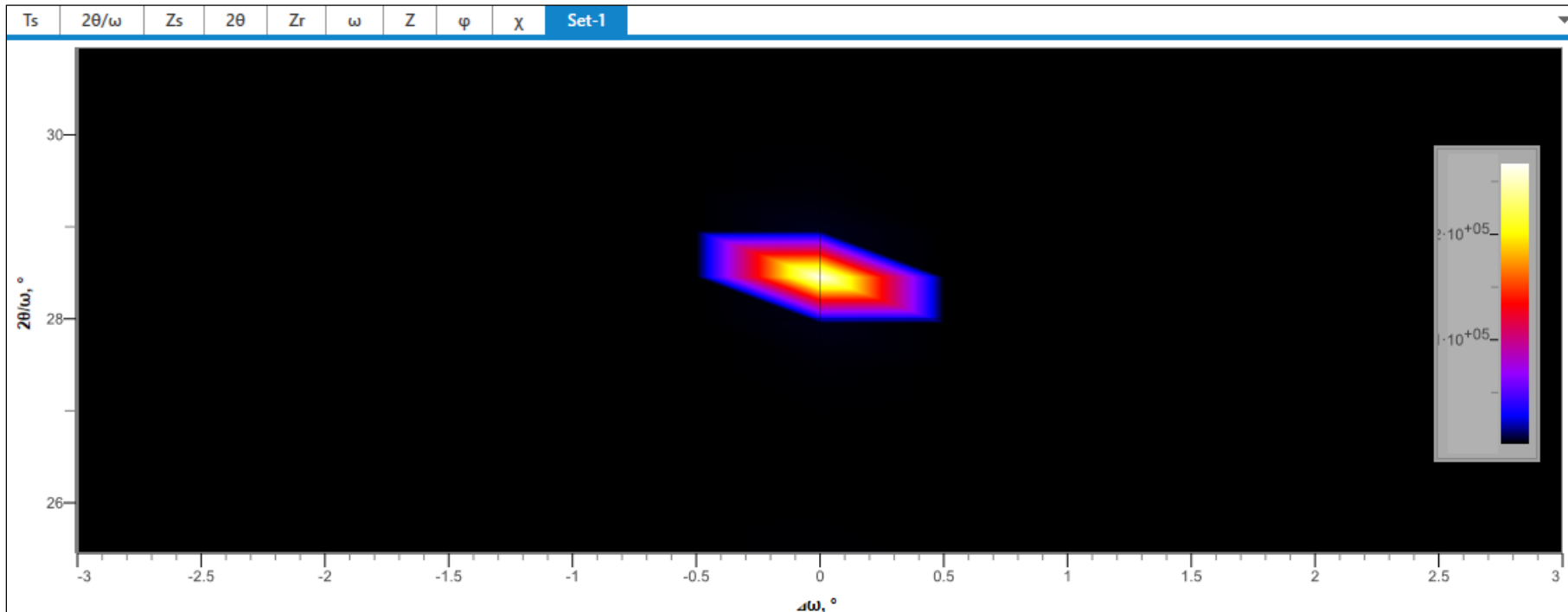
Sample name:

Memo:

Calculated scan duration: 7h 25min 25s

XRD Measurement

 Flow sequence completed.



# XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi - 1/9$

○ This sequence will perform an In-Plane Measurement or  $2\theta_{\chi}/\varphi$  using Parallel Beam optics

1. Select the **In-Plane** package under **XRD Measurement > Package Activities**

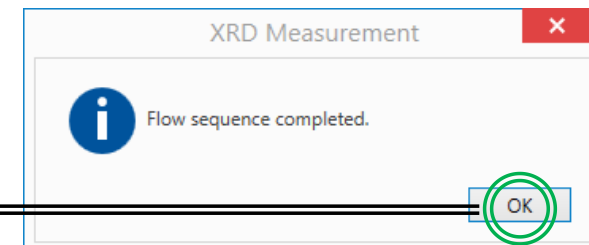
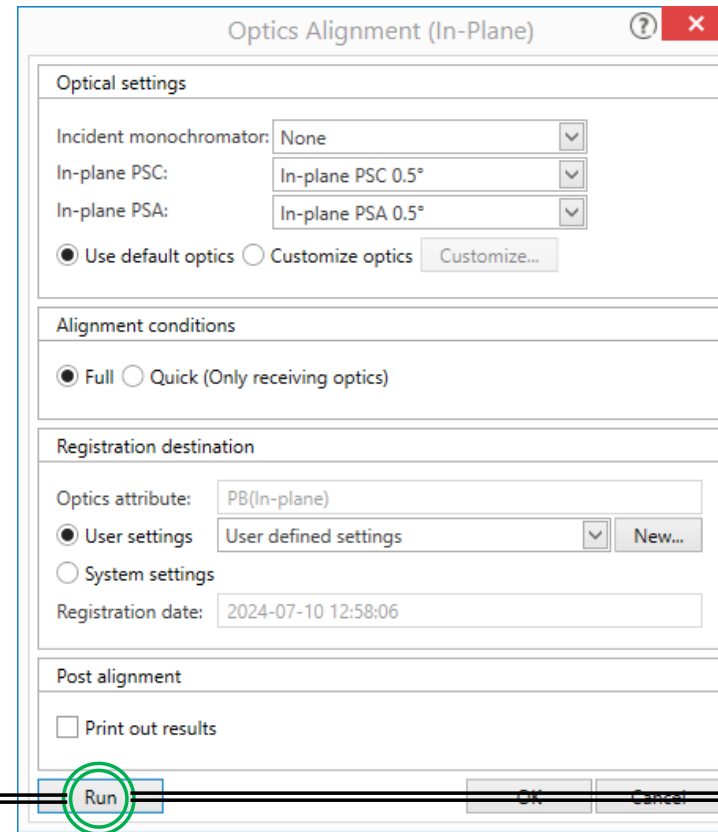
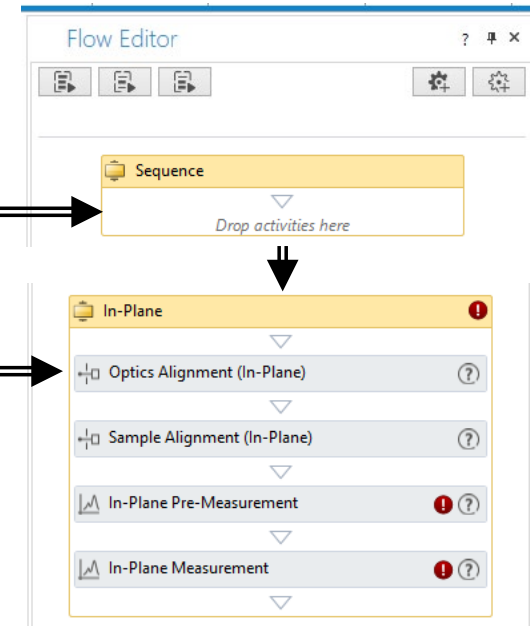
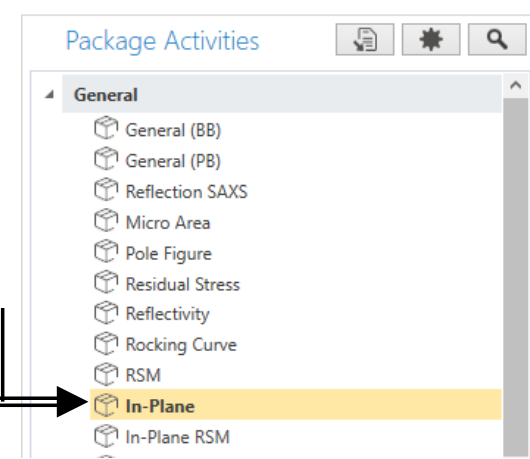
2. Drag the **In-Plane** package into the **Flow Editor** in **Sequence**

3. Left-click on **Optics Alignment (In-Plane)**

4. Confirm the following are selected:

- **Optical settings** → **Use default optics**
- **Alignment conditions** → **Full**
- **Registration destination** → **User settings**

5. Click **Run**  
and then **OK** when completed



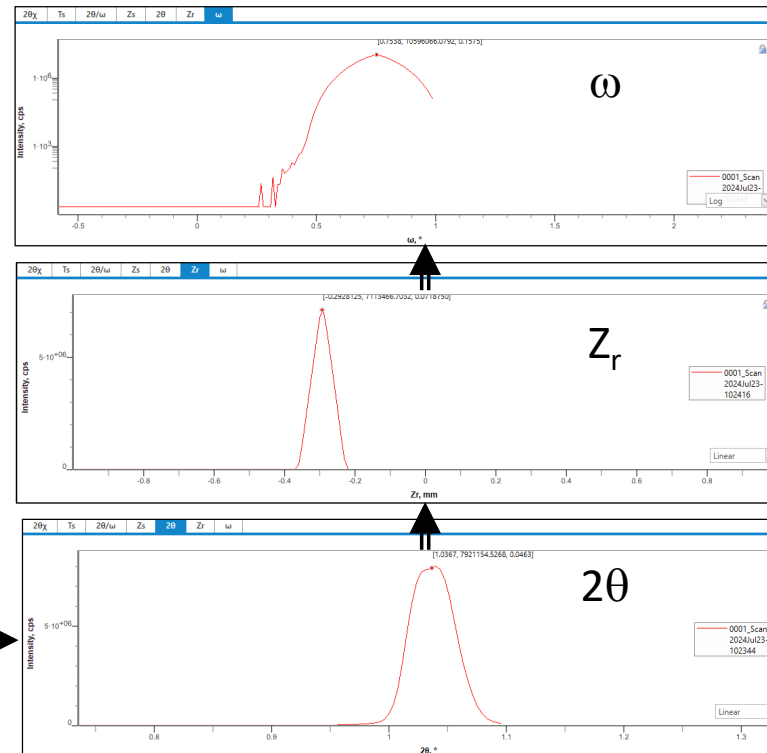
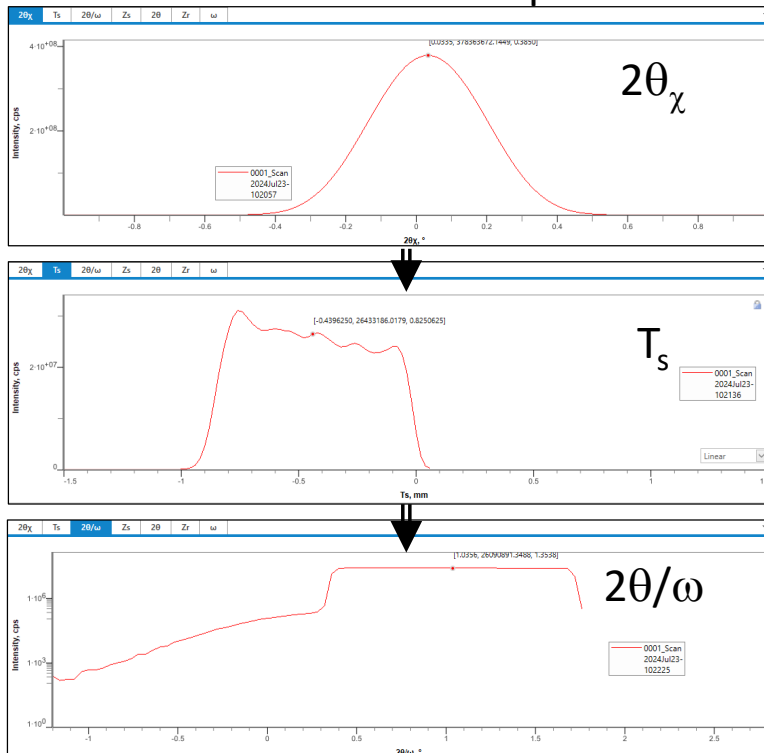
# XIII. In-Plane Measurement or $2\theta_\chi/\varphi - 2/9$

6. A **Smart Message** may appear indicating all the optics and attachments that need to be **removed** (indicated in **RED**) and those that need to be **installed** (indicated in **GREEN**)

**Reminder:** **Window Protector** must be inserted to protect **Detector** when swapping out **Receiving Optics**!



7. Click **OK** when completed



Smart Message

Replace **Soller slit open** with **In-plane PSC 0.5°** in IPS adaptor.

Replace **length-limiting slit 2 mm** with **length-limiting slit 10 mm** in integrated incident slit box.

Remove the **sample spacer 0-3 mm**.

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

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

Replace **Soller slit 5.0°** with **In-plane PSA 0.5°** in RPS adaptor.

Attach the detector plane of **HyPix-3000 (horizontal)** to **300 mm**. (Adjust the mark of the **detector adaptor** to **350 mm**)

Hide figures

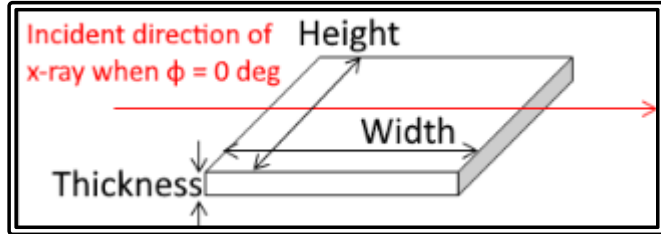
OK

Stop

# XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi - 3/9$

8. Left-click on **Sample Alignment (In-Plane)** to set **Sample Info**

9. Input your **Sample Info** per the dimensions

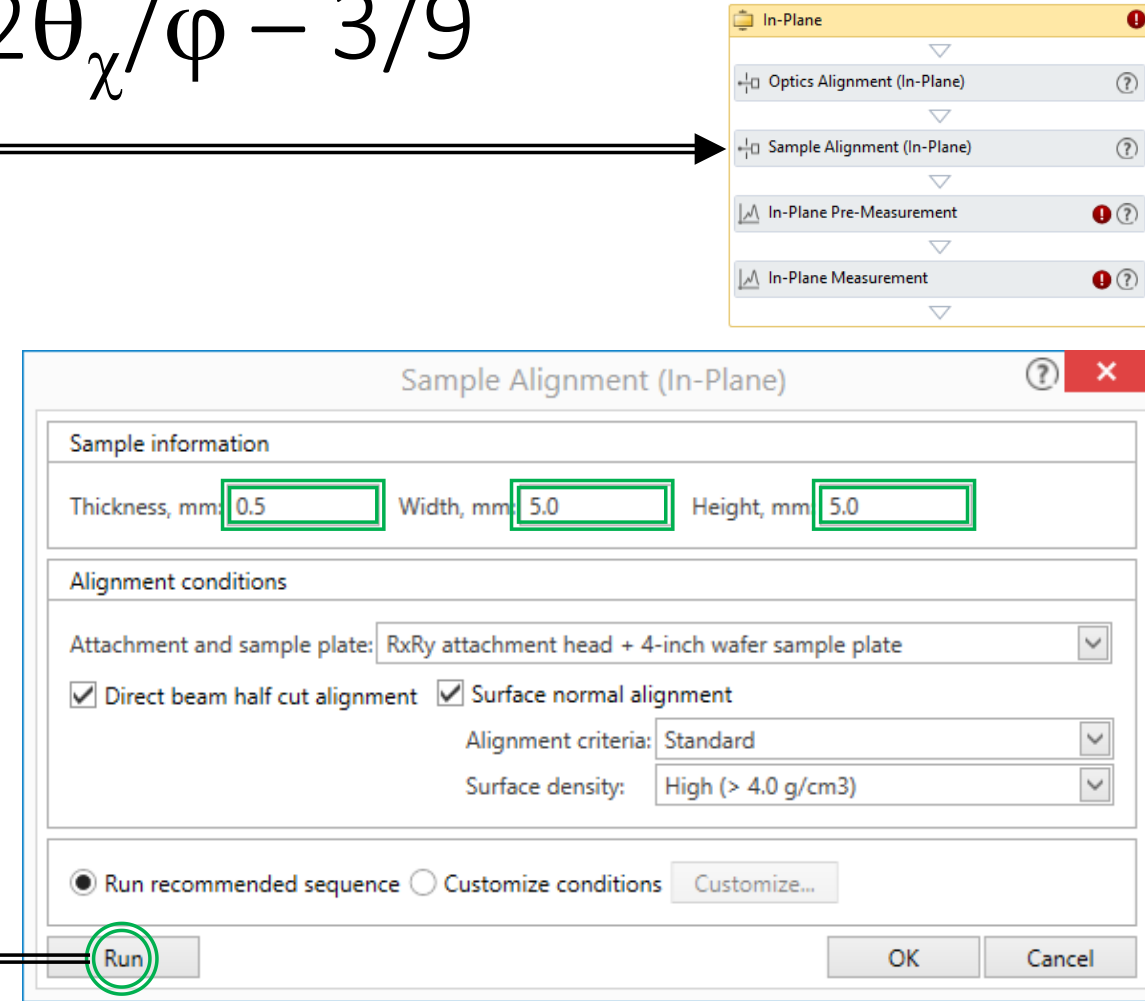


For training with Silicon: Thickness = 0.5 mm; Width & Height = 5 mm

10. Confirm the following are checked:

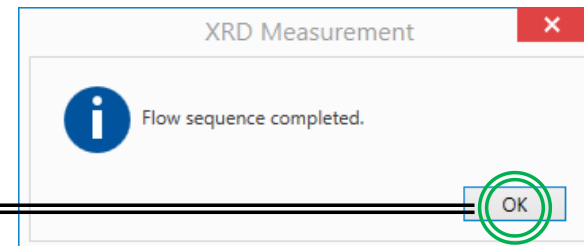
- Direct beam half cut alignment
- Surface normal alignment
- Put a sample when the sample alignment starts

11. Click **Run**



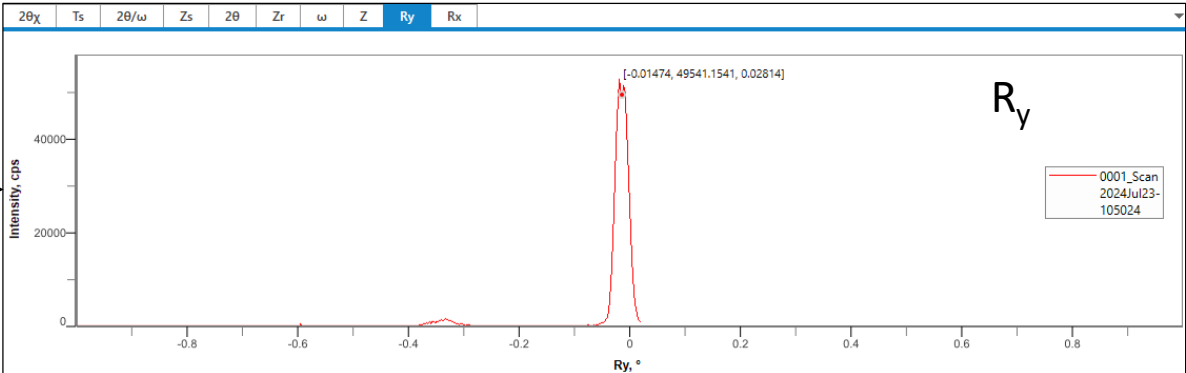
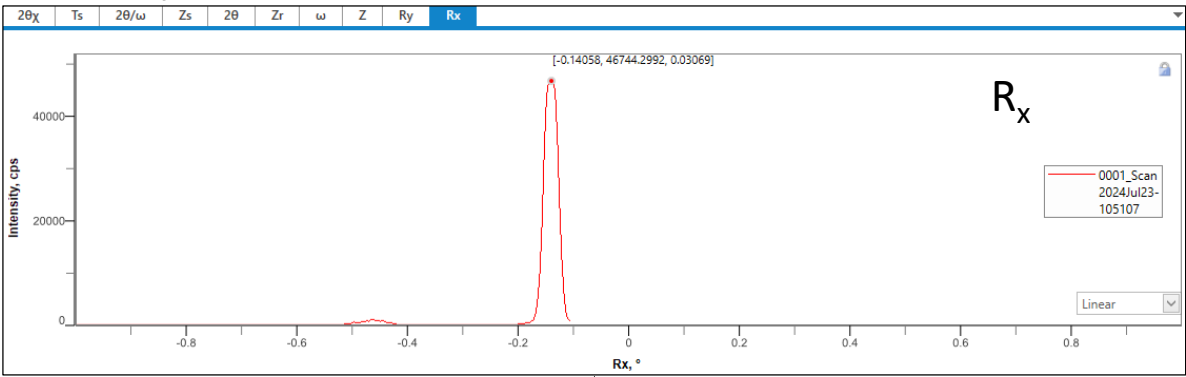
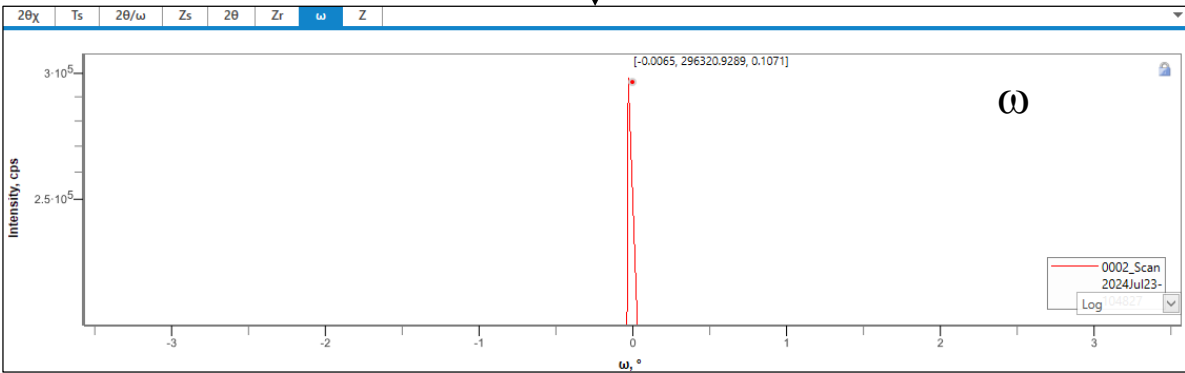
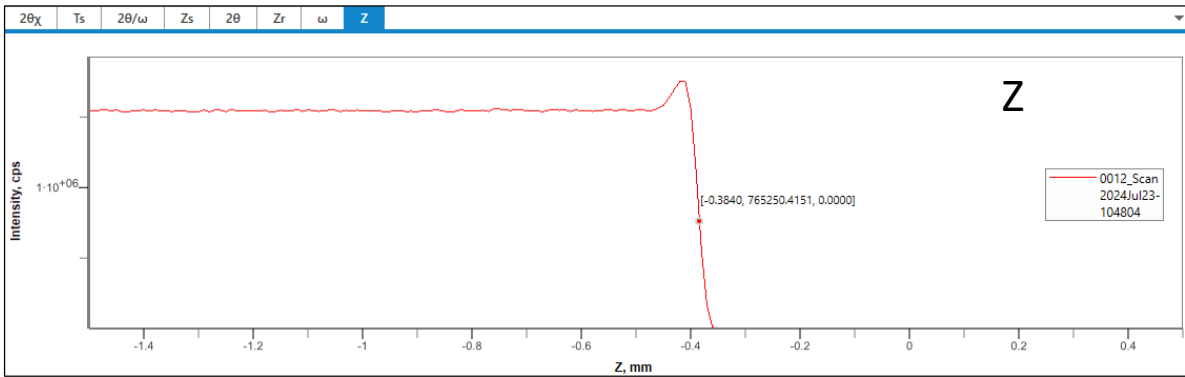
12. If **Sample Alignment (In-Plane)** fails, try changing surface density (e.g. High)

13. Wait for the **Flow sequence completed** prompt to continue by clicking **OK**





# XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi - 4/9$



14. Left-click on ***In-Plane Pre-Measurement***

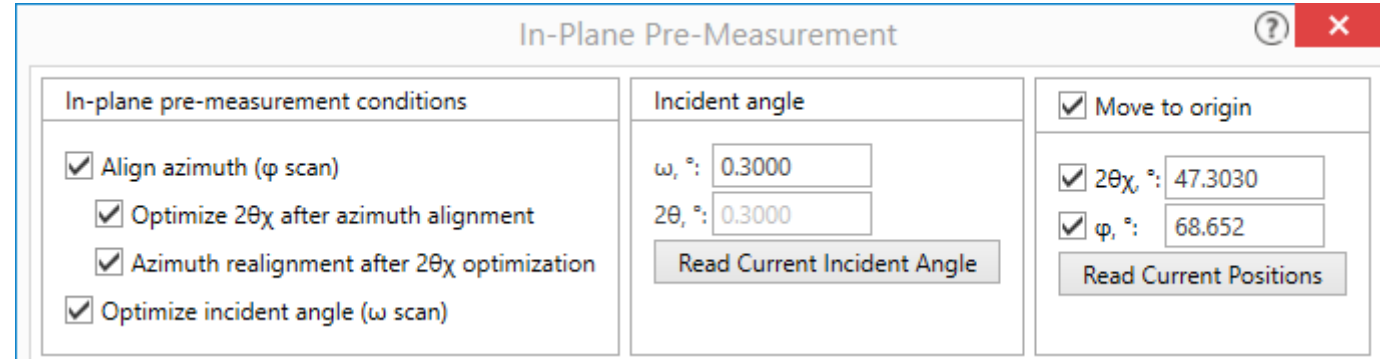
**In-Plane**

- Optics Alignment (In-Plane)
- Sample Alignment (In-Plane)
- In-Plane Pre-Measurement**
- In-Plane Measurement**

# XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 5/9

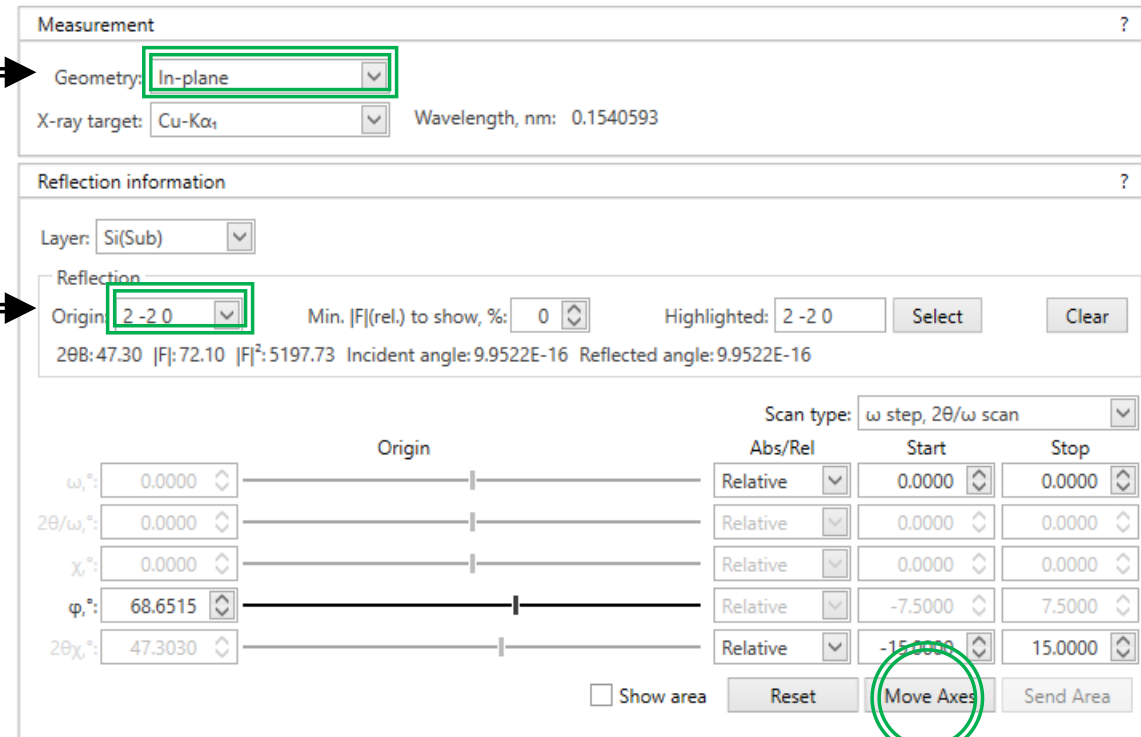
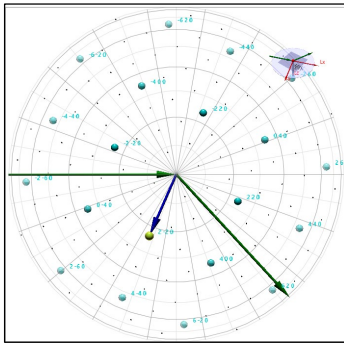
15. Recommend the following is checked:

- Align azimuth ( $\varphi$  scan)
- Optimize  $2\theta_\chi$  after azimuth alignment
- Azimuth realignment after  $2\theta_\chi$  optimization
- Optimize incident angle ( $\omega$  scan)



○ Note: The following is for training purposes to learn how to move goniometer axes for In-Plane

16. Open the **RS Viewer** and find the Si (2 -2 0) plane



17. Remember to select **In-plane** for Geometry

18. Click on **Move Axes**

19. The **Goniometer** will now reposition the axes per the values described here

# XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi$ – 6/9

20. Once the axes have moved, proceed to click **Read Current Incident Angle** and **Read Current Positions**

21. Use the **Run recommended sequence** or click **Customize conditions**

22. Adjust the following parameters if desired:

- **Start**
- **Stop**
- **Step**
- **Speed**

23. Click **Run** and then **OK** when completed

In-Plane Pre-Measurement

In-plane pre-measurement conditions

- ☒ Align azimuth ( $\varphi$  scan)
- ☒ Optimize  $2\theta_{\chi}$  after azimuth alignment
- ☒ Azimuth realignment after  $2\theta_{\chi}$  optimization
- ☒ Optimize incident angle ( $\omega$  scan)

Incident angle

$\omega$ , °: 0.3000

$2\theta$ , °: 0.3000

Read Current Incident Angle

☒ Move to origin

☒  $2\theta_{\chi}$ , °: 47.3030

☒  $\varphi$ , °: 68.652

Read Current Positions

☒ Run recommended sequence ☐ Customize conditions Customize...

Axis alignment conditions

Incident slit, mm: 0.100 Attenuator: Auto

Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Peak Search Method
<input checked="" type="checkbox"/>	$\varphi$ (Coarse)	Relative	-50.000	50.000	1.000	180.00	Maximum intensity
<input checked="" type="checkbox"/>	$\varphi$ (Fine)	Relative	-3.000	3.000	0.100	10.00	Sequential center of gravity
<input checked="" type="checkbox"/>	$2\theta_{\chi}$	Relative	-3.0000	3.0000	0.040	5.000	Sequential center of gravity
<input checked="" type="checkbox"/>	$\omega$	Absolute	0.0000	1.0000	0.0200	1.0000	Maximum intensity

XRD Measurement

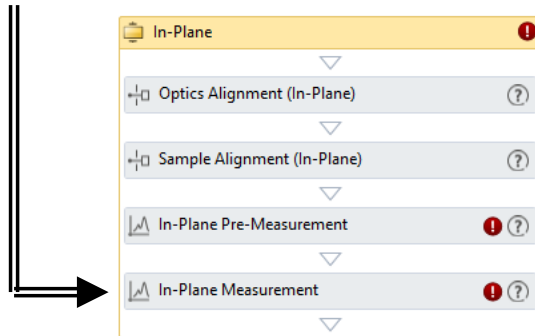
Flow sequence completed.

Run

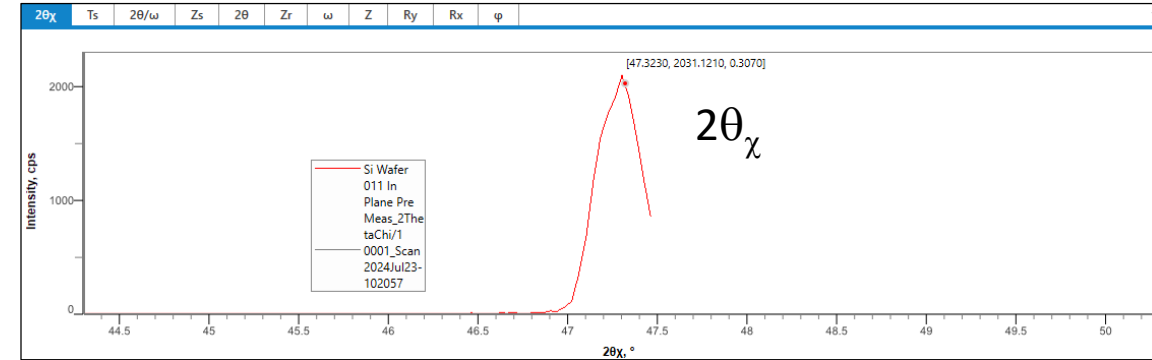
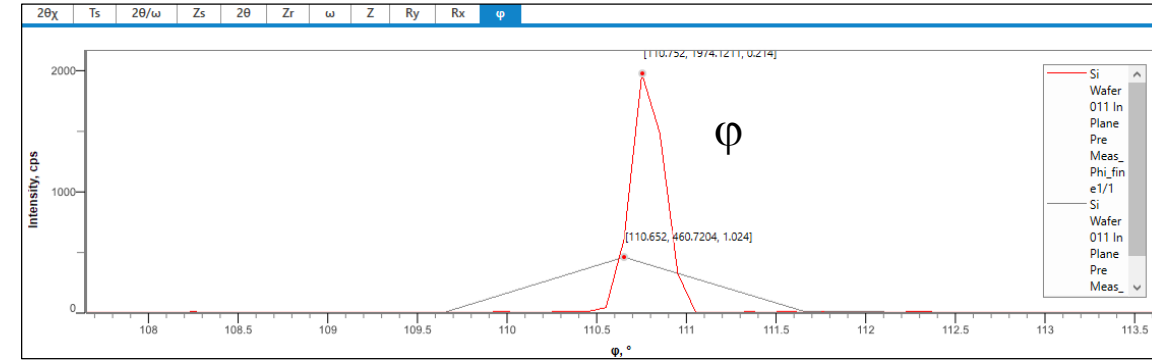
OK

# XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 7/9

20. Once completed, the optimal  $\varphi$  and  $2\theta_\chi$  parameters should be determined
21. If the parameters are not clearly optimized, check if the search is wide enough or if the initial parameters were appropriate
22. Left-click on ***In-Plane Measurement***



23. Use the ***Run recommended sequence*** or click ***Customize conditions***



# XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 8/9

24. Click on **Read Current Positions** so that both the  $2\theta_\chi$  and  $\varphi$  are updated here if **Move to origin** is checked

25. Adjust the following parameters if desired:

- **Start**
- **Stop**
- **Step**
- **Speed**

For training with Silicon: See example

Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min	Attenuator
$2\theta_\chi/\varphi$	OD(continuous)	Absolute	42.000	52.000	0.096	5.00	Auto

26. Input your desired **File name** and **File location** here

File name:

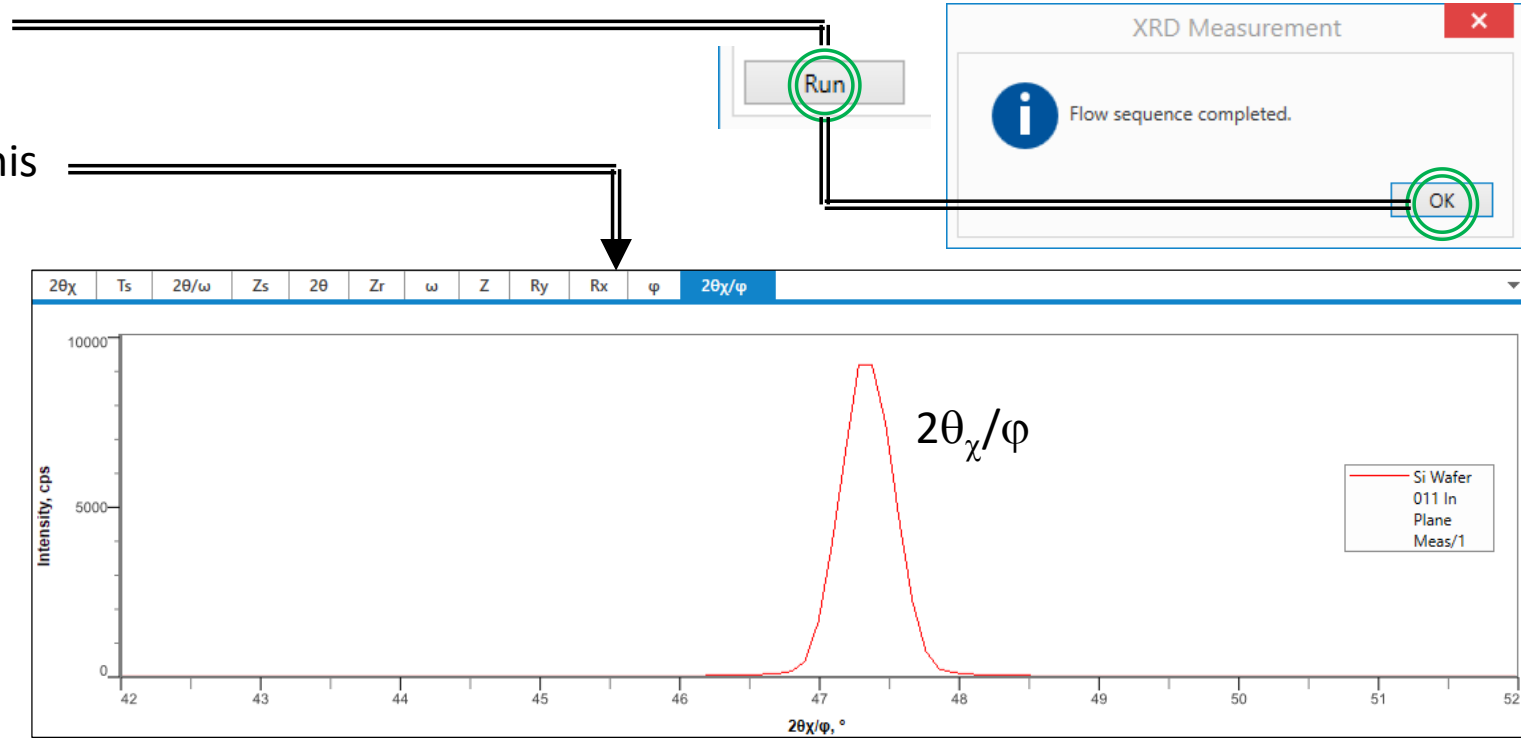
Sample name:

Memo:

# XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi$ – 9/9

27. Click **Run** and then **OK** when completed

28. The  $2\theta_{\chi}/\varphi$  measurement should look like this

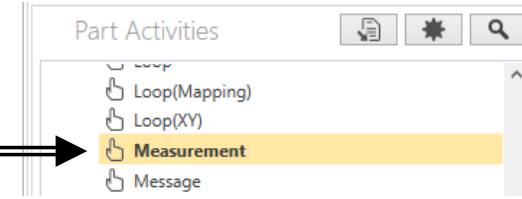




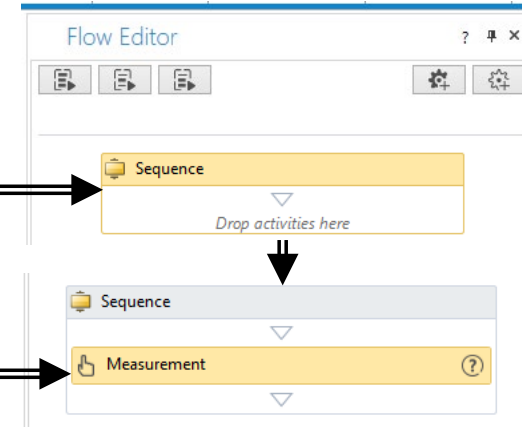
# XIV. In-Plane Azimuth or Phi( $\phi$ ) Scan – 1/2

- This sequence will perform an In-Plane Azimuth or Phi( $\phi$ ) Scan using Parallel Beam optics
- 1. The following assumes that the ***In-Plane Pre-Measurement*** has already been performed

2. Select the ***Measurement*** under ***XRD Measurement > Part Activities***



3. Drag the ***Measurement*** into the ***Flow Editor*** in ***Sequence***

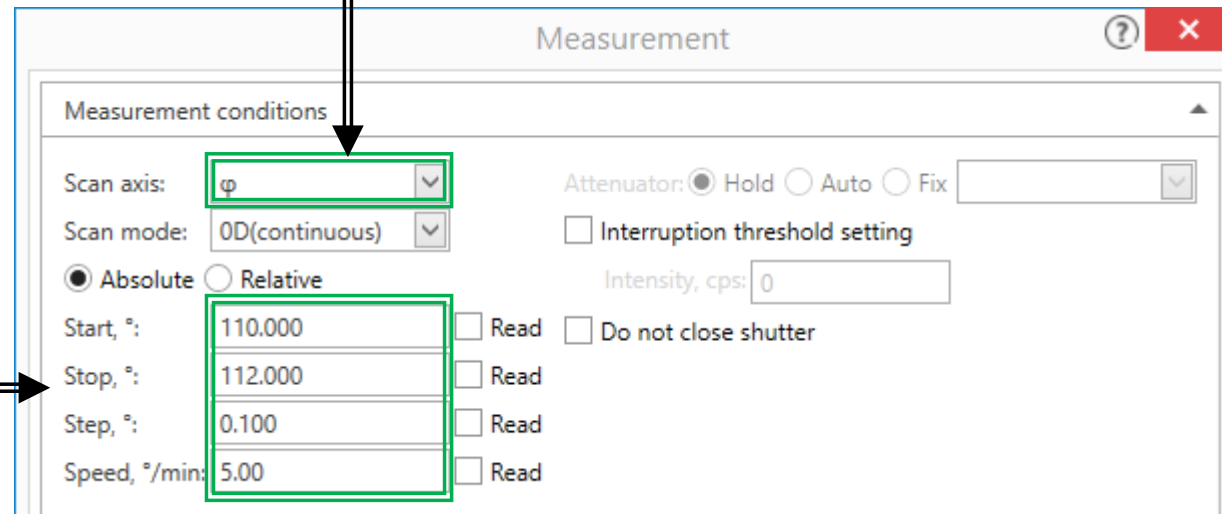


4. Left-click on ***Measurement***

5. Select  $\phi$  for ***Scan axis***

6. Configure the following:

- ***Start***
- ***Stop***
- ***Step***
- ***Speed***



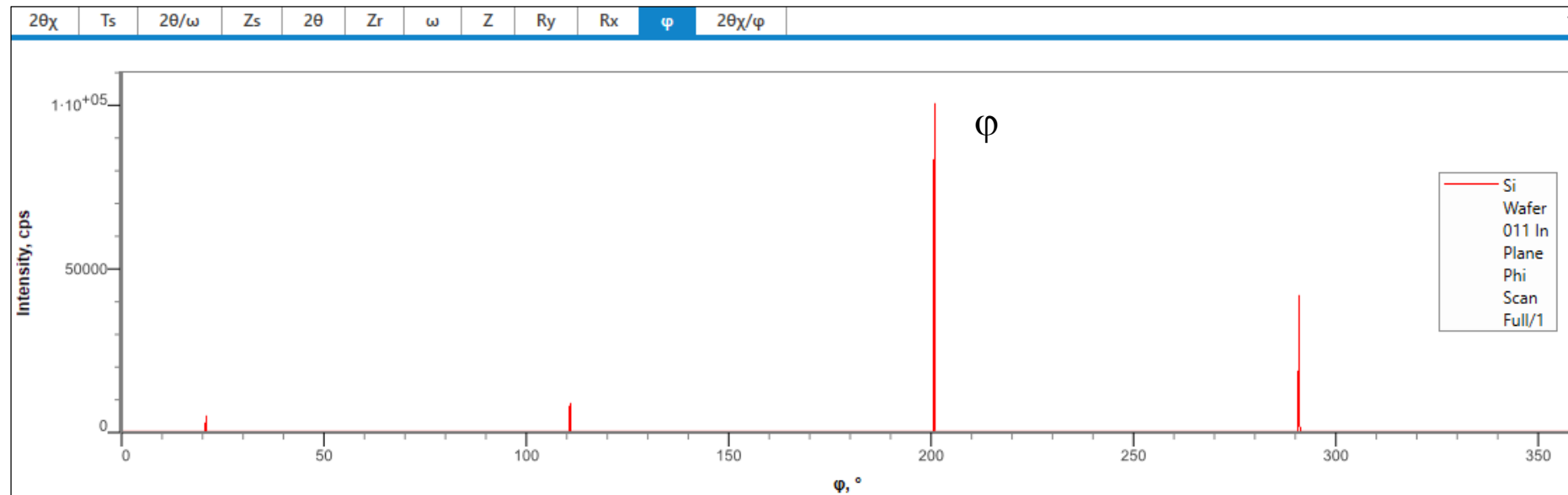
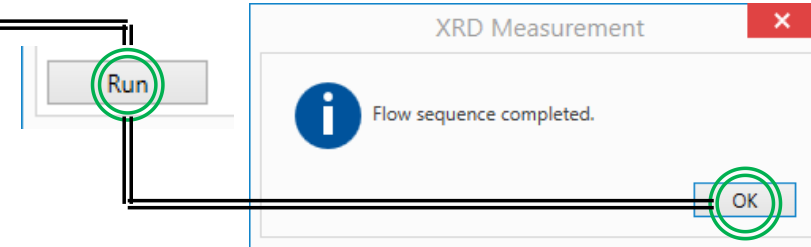
# XIV. In-Plane Azimuth or Phi( $\phi$ ) Scan – 2/2

7. Input your desired **File name** and **File location** here
8. Click **Run** and then **OK** when completed
9. If the parameters were chosen properly, you should eventually see a series of peaks appear at the appropriate  $\phi$  positions

☐ Save measured data

Post measurement

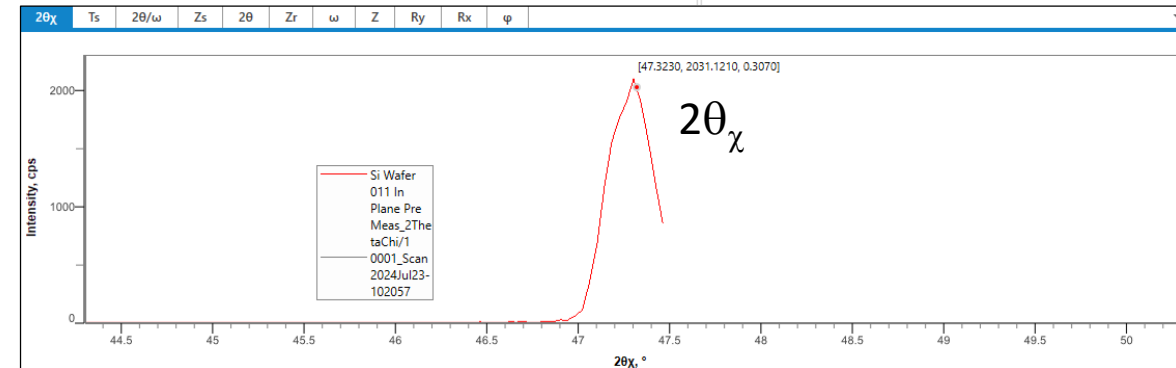
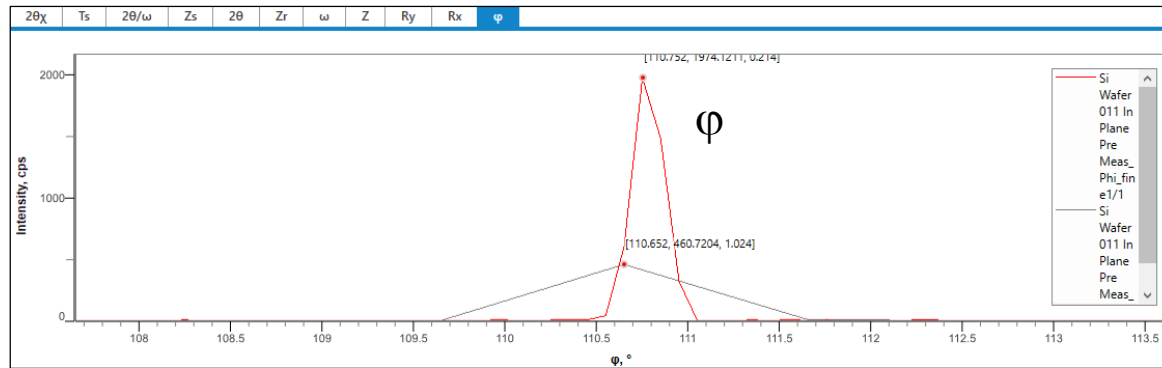
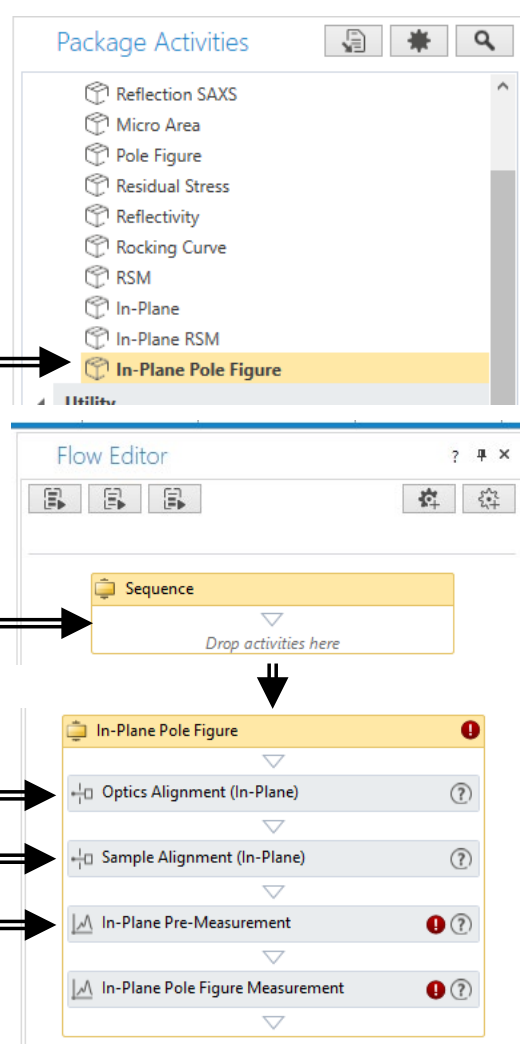
Run OK Cancel



# XV. In-Plane Pole Figure – 1/4

○ This sequence will perform an In-Plane Pole Figure using Parallel Beam optics

1. Select the ***In-Plane Pole Figure*** under ***XRD Measurement > Package Activities***
2. Drag the ***In-Plane Pole Figure*** into the ***Flow Editor*** in ***Sequence***
3. If ***Optics Alignment (In-Plane)*** and ***Sample Alignment (In-Plane)*** were previously performed, then skip to ***Step 4*** (see ***XIII. In-Plane Measurement*** for review)
4. Left-click on ***In-Plane Pre-Measurement***
5. Perform the ***In-Plane Pre-Measurement*** (see ***XIII. In-Plane Measurement*** for review) – optimized  $\phi$  and  $2\theta_\chi$  should be found



# XV. In-Plane Pole Figure – 2/4

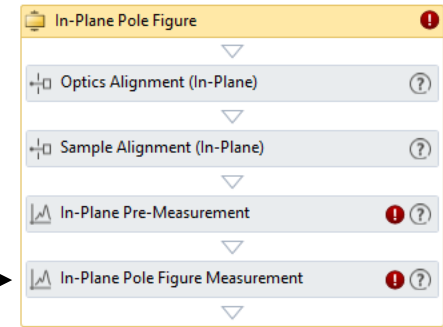
6. Left click ***In-Plane Pole Figure Measurement***

7. Remember to input the following parameters!

- ***Crystal system*** (i.e. Cubic)
- ***Index*** (i.e. 2 -2 0)
- ***Measurement angle*** (i.e.  $2\theta_{\chi}$ )

8. Choose if any ***Background measurements*** will be run if desired

9. Choose to ***Run recommended sequence*** or ***Customize conditions***



# XV. In-Plane Pole Figure – 3/4

10. Depending on your **Step** chosen, be aware that it may not be sufficient so choose the following carefully for  $\alpha$  scan axes!

- **Start**
- **Stop**
- **Step**
- **Speed**

Data measurement conditions						
Minimum $\omega$ angle, °:		0.5000	Receiving slit #1, mm:		1.000	Attenuator: Auto
Incident slit, mm:		1.000	Receiving slit #2, mm:		2.100	
Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min
$\alpha$	OD(step)	Absolute	0.00	20.00	5.00	
$\beta$	OD(continuous)	Absolute	0.000	360.000	3.000	150.000

Remember that  $\alpha = \chi$  for in-plane!

(Note: Will the peaks appear for  $\alpha$  and  $\beta$  if arbitrarily chosen?)

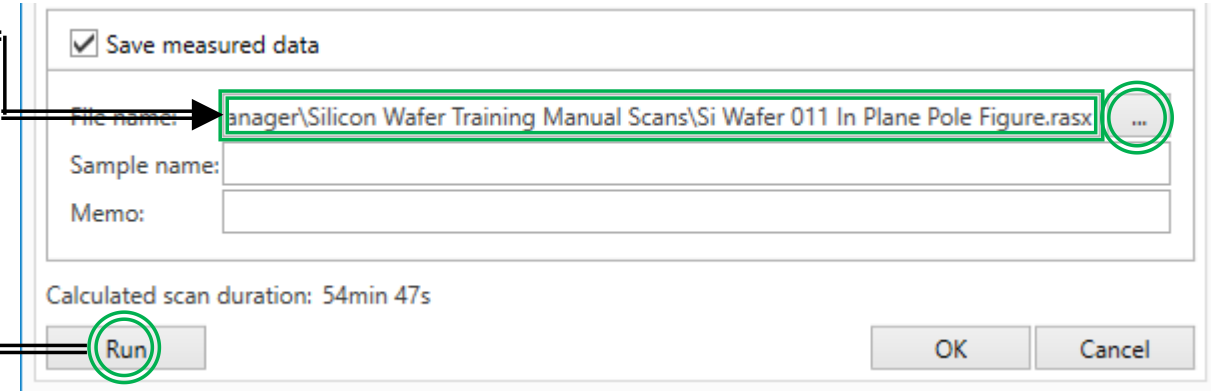
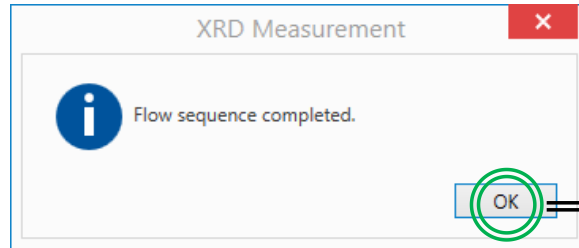
11. If **Background measurements** were selected, determine the desired conditions

<input checked="" type="checkbox"/> Background measurement conditions								
			Background #1			Background #2		
Geometry	Step Axis	Step, °	2 $\theta$ Angle, °	Receiving Slit #1, mm	Receiving Slit #2, mm	2 $\theta$ Angle, °	Receiving Slit #1, mm	Receiving Slit #2, mm
Transmission	$\alpha$	5	25.7480	10.000	9.900	31.7480	10.000	9.900
Reflection	$\alpha$	5.000	25.7480	10.000	9.900	31.7480	10.000	9.900
Scan Axis	Background Data Acquisition Method		Scan Mode	Range	Start, °	Stop, °	Step, °	Duration, s
$\beta$	1 point ( $\beta$ = Minimum intensity)		Fixed time	Absolute	177.200	537.200	5.000	1.5

# XV. In-Plane Pole Figure – 4/4

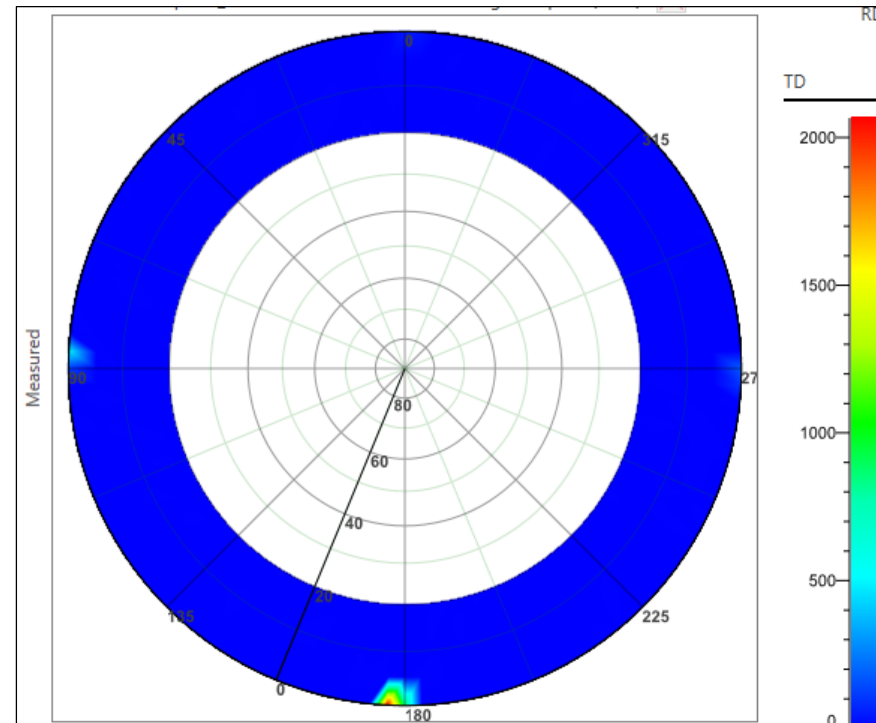
12. Input your desired **File name** and **File location** here

13. Click **Run** then **OK** when completed



For training with Silicon: Do not Run

14. If the parameters were chosen properly, you should eventually see a series of peaks appear at the appropriate  $\alpha$  and  $\beta$  positions

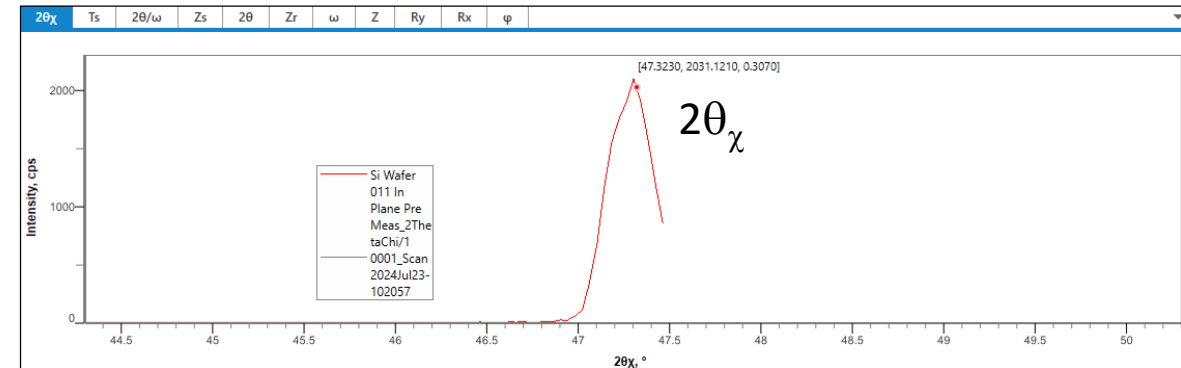
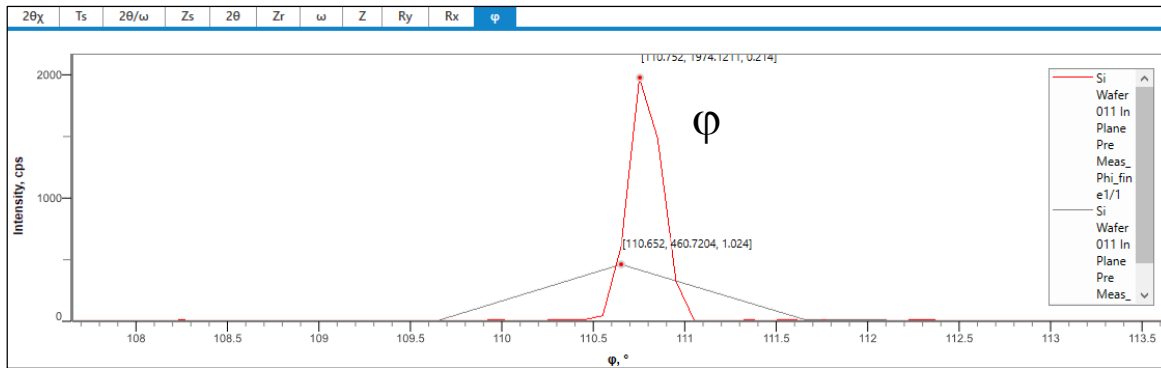
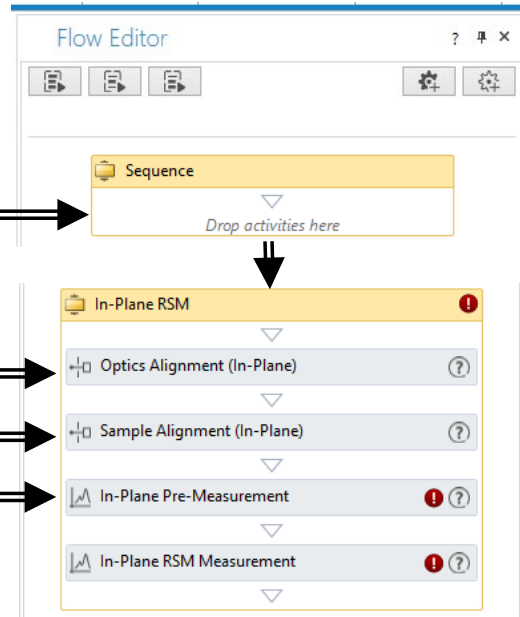
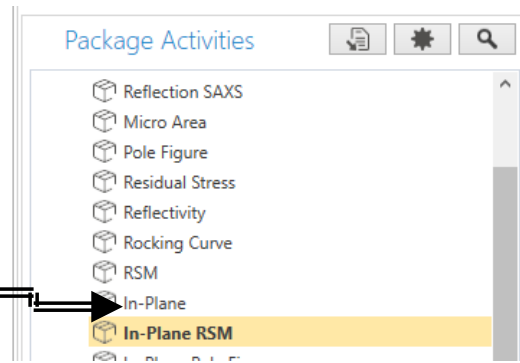




# XVI. In-Plane RSM – 1/3

- This sequence will perform an In-Plane RSM using Parallel Beam optics

1. Select the ***In-Plane RSM*** under ***XRD Measurement > Package Activities***
2. Drag the ***In-Plane RSM Figure*** into the ***Flow Editor*** in ***Sequence***
3. If ***Optics Alignment (In-Plane)*** and ***Sample Alignment (In-Plane)*** were previously performed, then skip to ***Step 4*** (see ***XIII. In-Plane Measurement*** for review)
4. Left-click on ***In-Plane Pre-Measurement***
5. Perform the ***In-Plane Pre-Measurement*** (see ***XIII. In-Plane Measurement*** for review) – optimized  $\phi$  and  $2\theta_\chi$  should be found



# XVI. In-Plane RSM – 2/3

6. Left click ***In-Plane RSM Measurement***

7. Confirm ***Move to origin*** is checked

8. Click ***Read Current Positions*** and ***Read Current Incident Angle*** to set the proper ***Origin***

9. Configure the following:

- ***Start***
- ***Stop***
- ***Step***
- ***Speed***

The screenshot shows the 'Customize - In-Plane RSM Measurement' dialog box. Annotations include arrows from the text instructions to specific UI elements: an arrow from step 6 points to the 'In-Plane RSM Measurement' option in the top right menu; an arrow from step 7 points to the 'Move to origin' checkbox; an arrow from step 8 points to the 'Read Current Incident Angle' and 'Read Current Positions' buttons; and an arrow from step 9 points to the 'Incident slit' field and the scan tables.

**In-plane RSM measurement conditions**

Data acquisition method:  $\varphi$  step,  $2\theta\chi/\varphi$  scan

Range: ☐ Narrow ☒ Normal ☐ Wide

☒ Incident angle

$\omega$ , °: 0.2400

$2\theta$ , °: 0.2400

☒ Move to origin

☒  $2\theta\chi$ , °: 47.3015

☒  $\varphi$ , °: 111.052

**Manual exchange slit conditions**

☐ Manual exchange slit conditions

In-plane PSA: In-plane PSA 0.5°

Length-limiting slit: 10 mm

**Scan conditions**

Incident slit, mm: 0.100

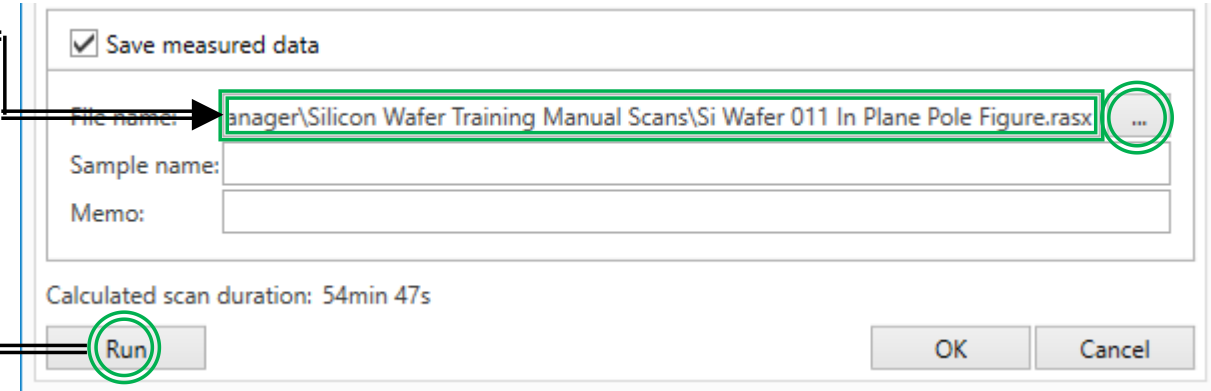
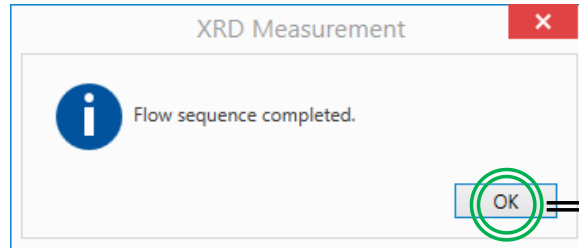
Step Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Number of Steps
$\varphi$	OD(step)	Relative	-3.000	3.000	0.100	61

Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min	Attenuator
$2\theta\chi/\varphi$	OD(continuous)	Relative	-3.000	3.000	0.096	5.00	Auto

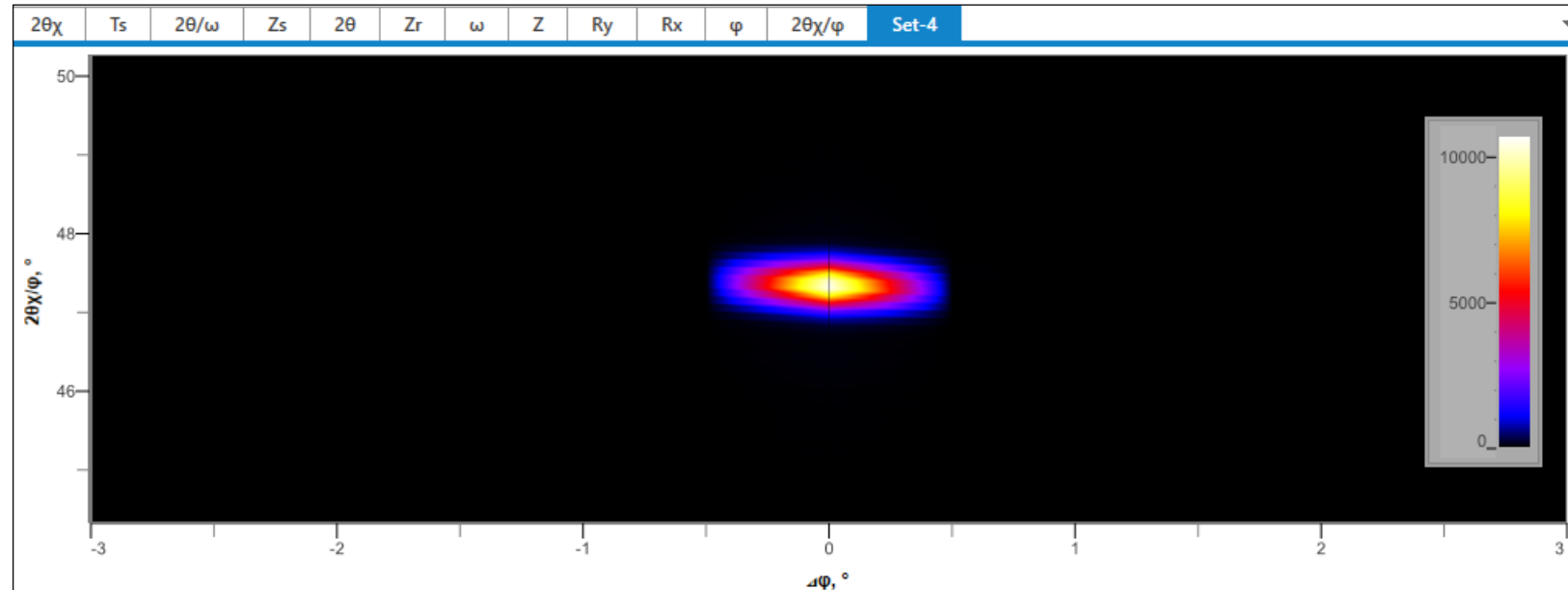
# XVI. In-Plane RSM – 3/3

10. Input your desired **File name** and **File location** here

11. Click **Run** then **OK** when completed



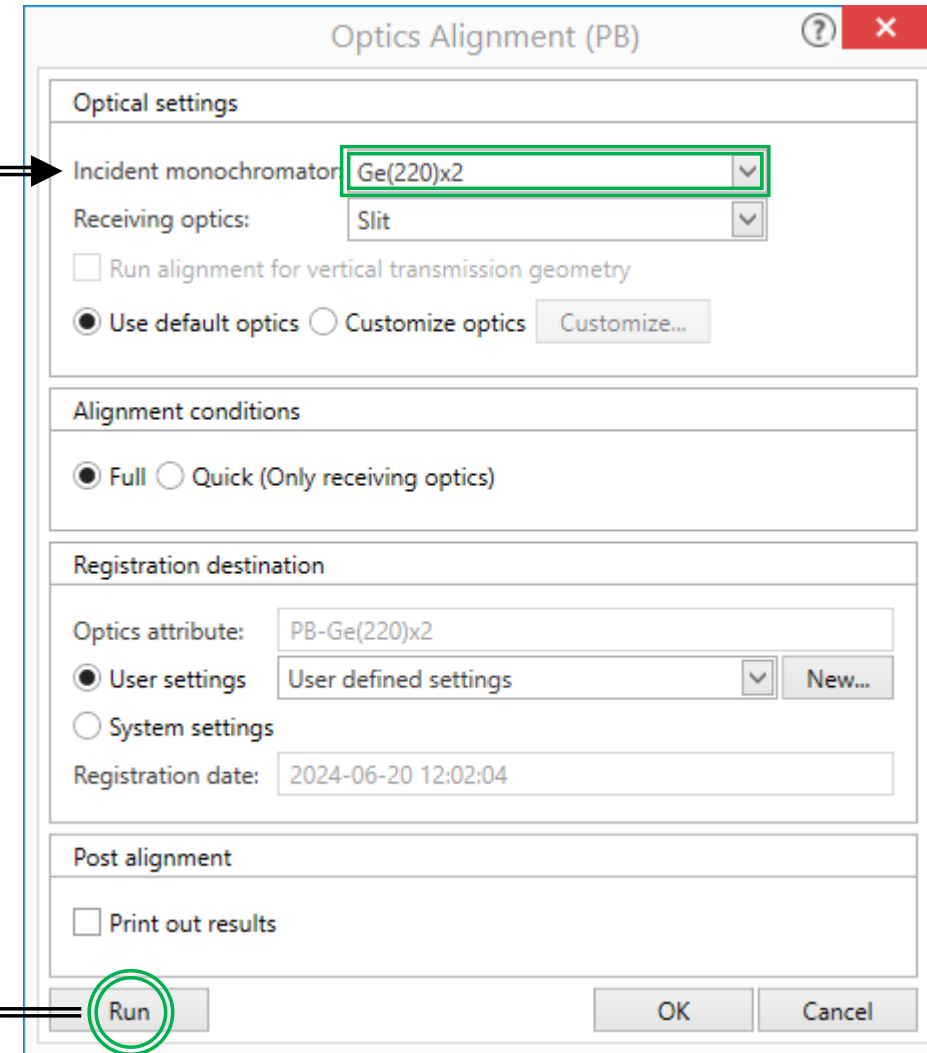
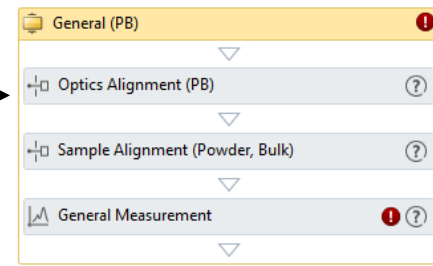
For training with Silicon: Do not Run



# XVII. Monochromator Ge(220)x2 – 1/2

- This sequence will show how to use Monochromator Ge(220)x2 using Parallel Beam optics

1. Left-click on **Optics Alignment (PB)** tab to select optics
2. Select **Ge(220)x2** for the **Incident monochromator**
3. Confirm **Use default optics** is selected under **Optical settings**
4. Confirm **Full** is selected under **Alignment conditions**
5. Confirm **User settings** is selected, then click **Run**
6. A **Smart Message** will appear indicating all the optics and attachments that need to be **removed** (indicated in **RED**) and those that need to be **installed** (indicated in **GREEN**)



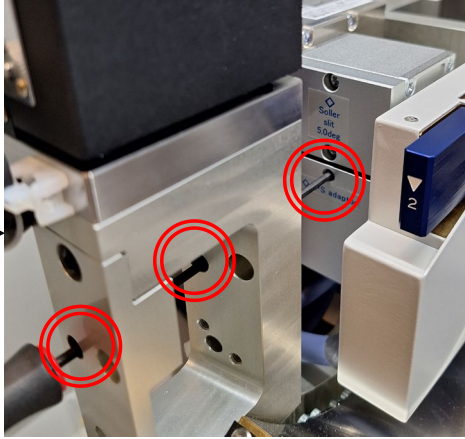
# XVII. Monochromator Ge(220)x2 – 2/2

7. To remove the **IPS adaptor** and install the **Ge(220)x2** perform the following:

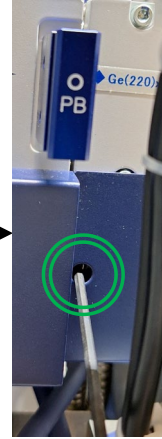
Identify right  
**Connector Cables** first



Loosen set screw to  
remove **IPS**



Loosen set screw  
**IPS Adaptor**



Detach the **Connector  
Cables** for **IPS adaptor**



Remove the **IPS adaptor**.



Install 2-bounce monochromator **Ge(220)x2** in incident side of diffractometer.



Install **Soller slit open (short)** in 2-bounce monochromator **Ge(220)x2**.



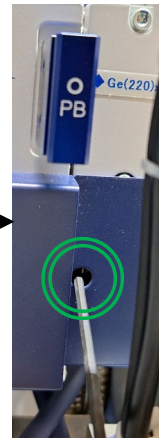
Carefully remove the  
**IPS adaptor** - watch the **Cables**



Carefully install **Ge(220)x2**  
– align along the slot



Gently tighten set  
screws – just **Snug**



Identify the **Connector** pin  
locations to fit properly



Connect the **Connector  
Cables** for **Ge(220)x2**



# XVIII. Grazing Incidence XRD or GIXRD – 1/5

○ This sequence will perform a GIXRD measurement using Parallel Beam optics

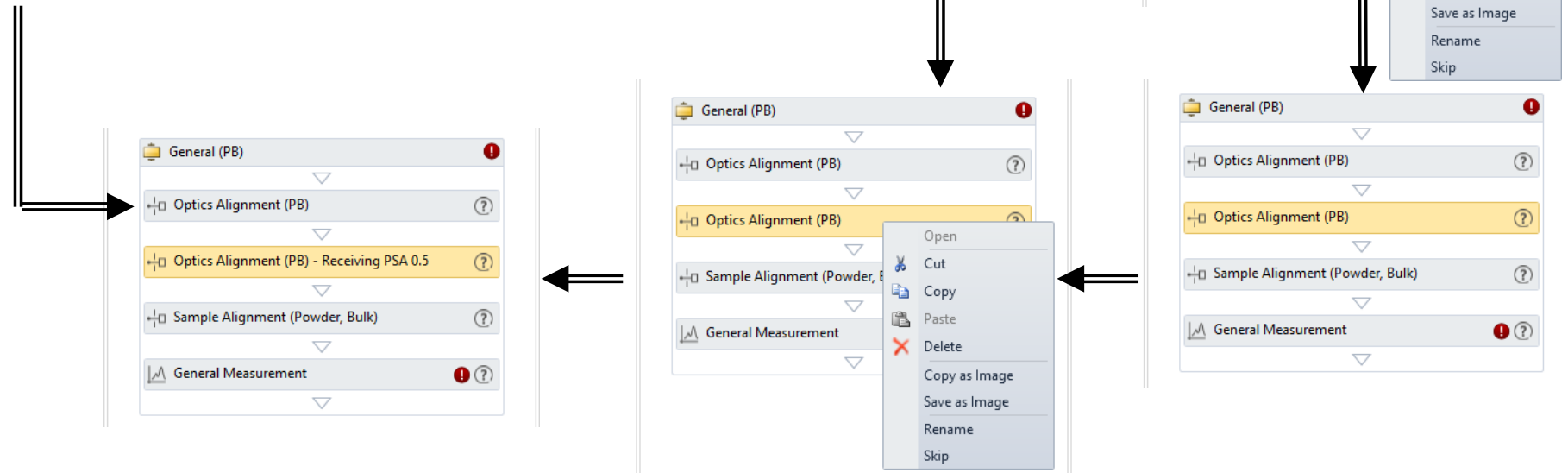
1. Select the **General (PB)** package under **XRD Measurement > Package Activities**

2. Drag the **General (PB)** package into the **Flow Editor** in **Sequence**

3. Right-click on **Optics Alignment** and **Copy** and **Paste** underneath

4. Right-click on the **2<sup>nd</sup> Optics Alignment** and **Rename** it to “**Receiving PSA 0.5**”

5. Left-click on **Optics Alignment (PB)** tab to confirm default parameters





# XVIII. Grazing Incidence XRD or GIXRD – 2/5

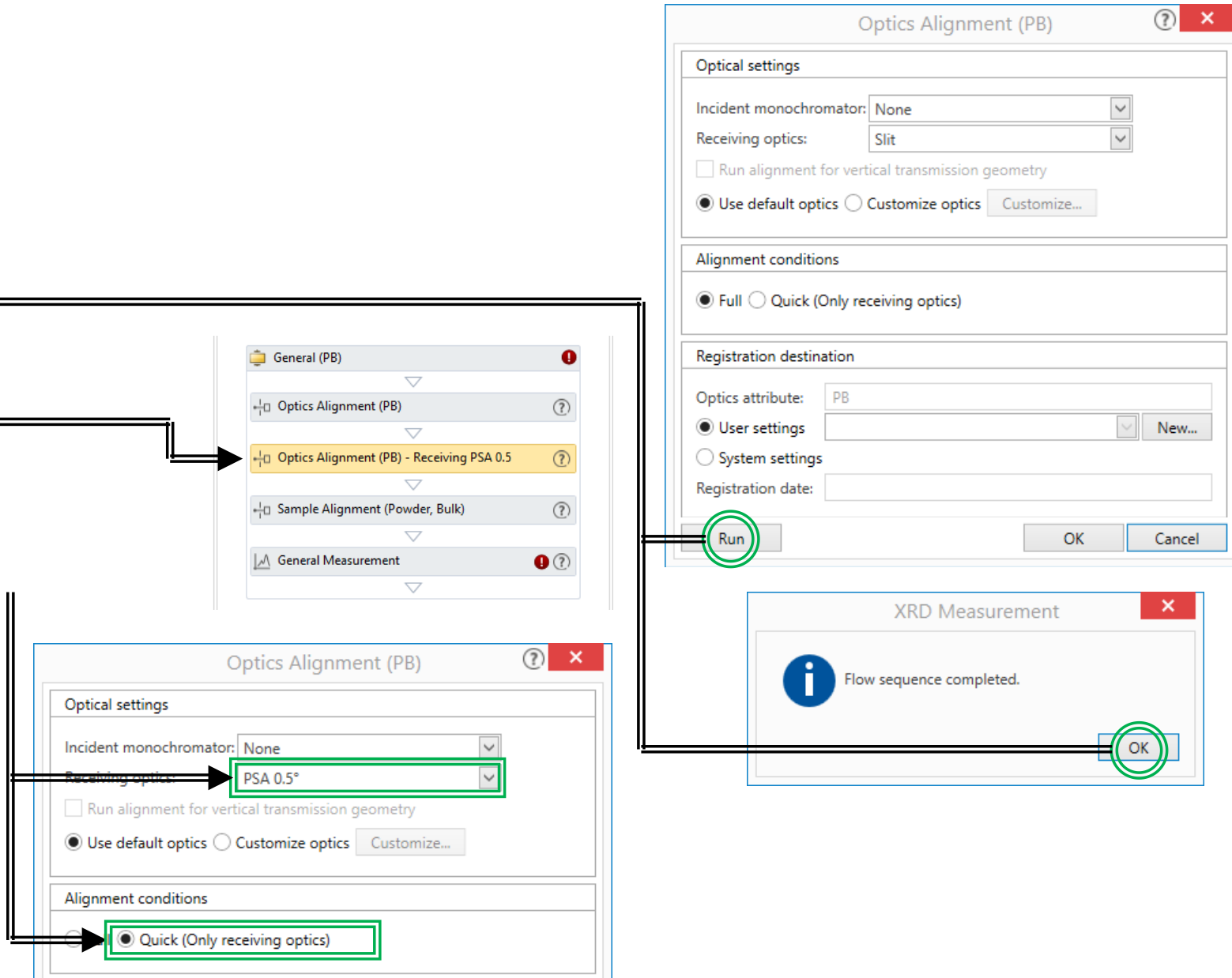
6. Confirm the following are selected:
- **Optical settings** → *Use default optics*
  - **Alignment conditions** → *Full*
  - **Registration destination** → *User settings*

7. Click **Run**  
and then **OK** when completed

8. Left-click on **Optics Alignment (PB)**  
– **Receiving PSA 0.5** tab

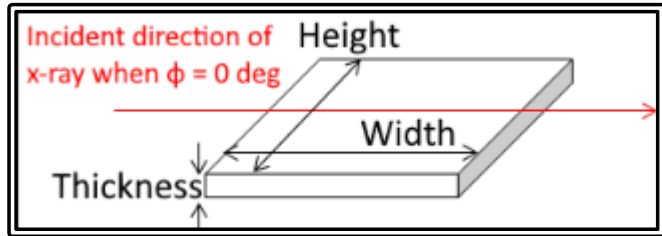
9. Change the **Receiving optics** to **PSA 0.5°**  
and check **Quick (Only receiving optics)**

10. Click **Run**  
and then **OK** when completed as well



# XVIII. Grazing Incidence XRD or GIXRD – 3/5

11. Left-click on **Sample Alignment (Powder, Bulk)** to set **Sample Info**
12. Set the **Attachment and sample plate** to **RxRy attachment head + 4-inch wafer sample plate**
13. Select **Flat sample**
14. Input your **Sample Info** per the dimensions
15. Click **Run** and then **OK** when completed



Sample Alignment (Powder, Bulk)

Sample alignment conditions

Attachment and sample plate: RxRy attachment head + 4-inch wafer sample plate

☐ No height alignment

☐ Set registered position without alignment

☐ Curved sample (Z scan only)

☒ Flat sample

Sample height, mm: 5.0

Sample thickness, mm: 0.5

☒ Run recommended sequence ☐ Customize conditions [Customize...](#)

☒ Put a sample every time

[Run](#) [OK](#) [Cancel](#)

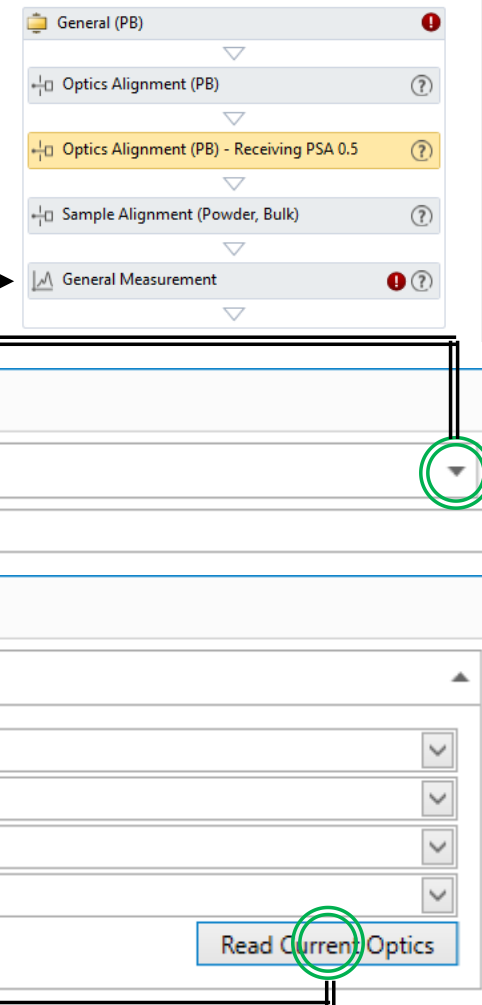
XRD Measurement

[i](#) Flow sequence completed.

[OK](#)

# XVIII. Grazing Incidence XRD or GIXRD – 4/5

16. Left-click on **General Measurement**
17. Left-click on the drop down for **Manual exchange slit conditions**
18. Click on the box for **Manual exchange slit conditions**
19. Click on **Read Current Optics**
20. Select **2 $\theta$**  for the **Scan Axis**
21. Input desired **Start**, **Stop**, **Step**, and **Speed** values
22. Set both the **Receiving Slit #1** and **#2** to **20** and **20.1 mm**; respectively



Measurement conditions													
Attachment base: $\chi\phi Z$ attachment				Attachment head: Attachment without movable axis									
	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator	Comment	Options
1	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
2	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
3	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...

# XVIII. Grazing Incidence XRD or GIXRD – 4/5

23. Left-click on **Set...**

Measurement conditions

Attachment base:  $\chi\phi Z$  attachment Attachment head: Attachment without movable axis

	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator	Comment	Options
1	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
2	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
3	<input checked="" type="checkbox"/>	2 $\theta$	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...

24. Set  $\omega$  as the **Axis**

25. Set the **Origin** to desired value typically ranging from **0.1 - 1°**

Options - General Measurement

Attachment base:  $\chi\phi Z$  attachment Attachment head: Attachment without movable axis

Exec.	Axis	Action	Origin (Center)	Oscillation Range ( $\pm$ )	Start	Stop	Speed
<input checked="" type="checkbox"/>	$\omega$	Move to origin	0.1000	0.0000	0.0000	0.0000	0.0000

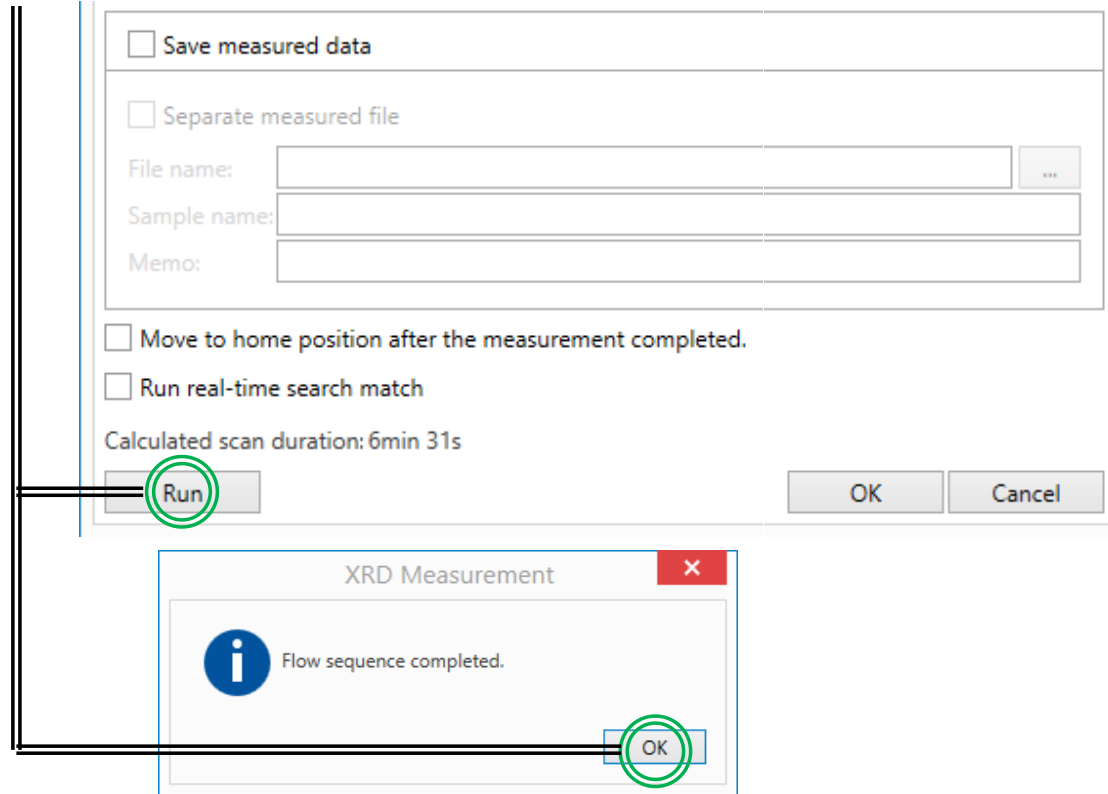
26. You may wish to vary the  $\omega$  values to see the impact on the angle to your desired peak

27. Clicking on the  box will **Copy** the value from the top row if you choose to keep the values the same for each row

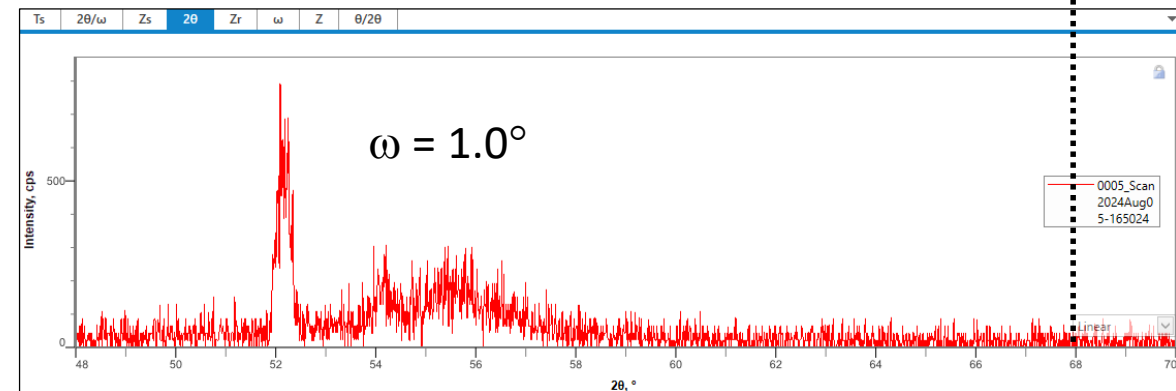
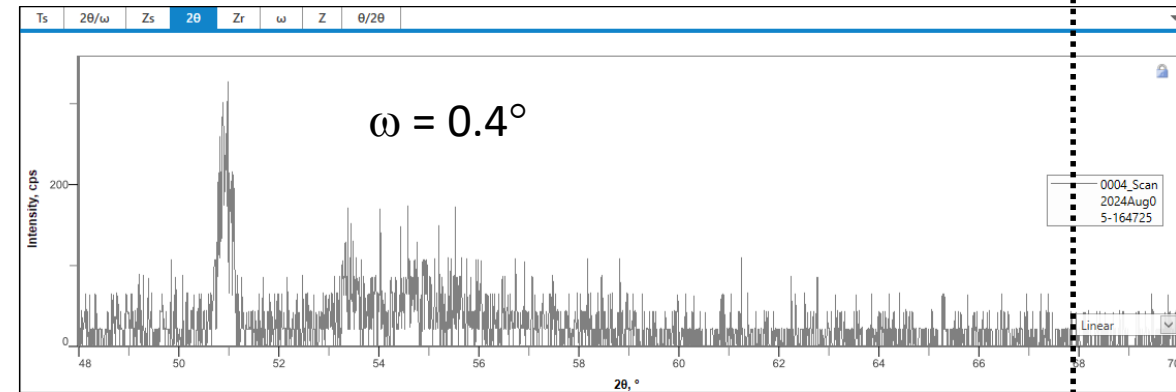
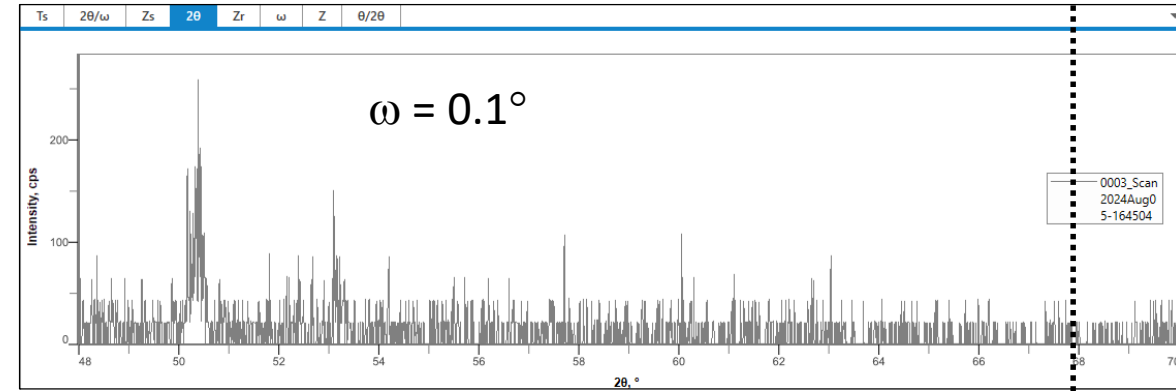
# XVIII. Grazing Incidence XRD or GIXRD – 5/5

Substrate Peak

28. Click on **Run** then **OK** when completed



29. You may wish to run different values of  $\omega$  for comparison



# XIX. Clean-up and Shutdown – 1/1

- This sequence is for cleaning up your work and shutting down equipment

1. Click on the **Startup/Shutdown** tab at the bottom right panel

2. Confirm **XG off** is selected

3. Click the **Run** button, and confirm with **Yes**

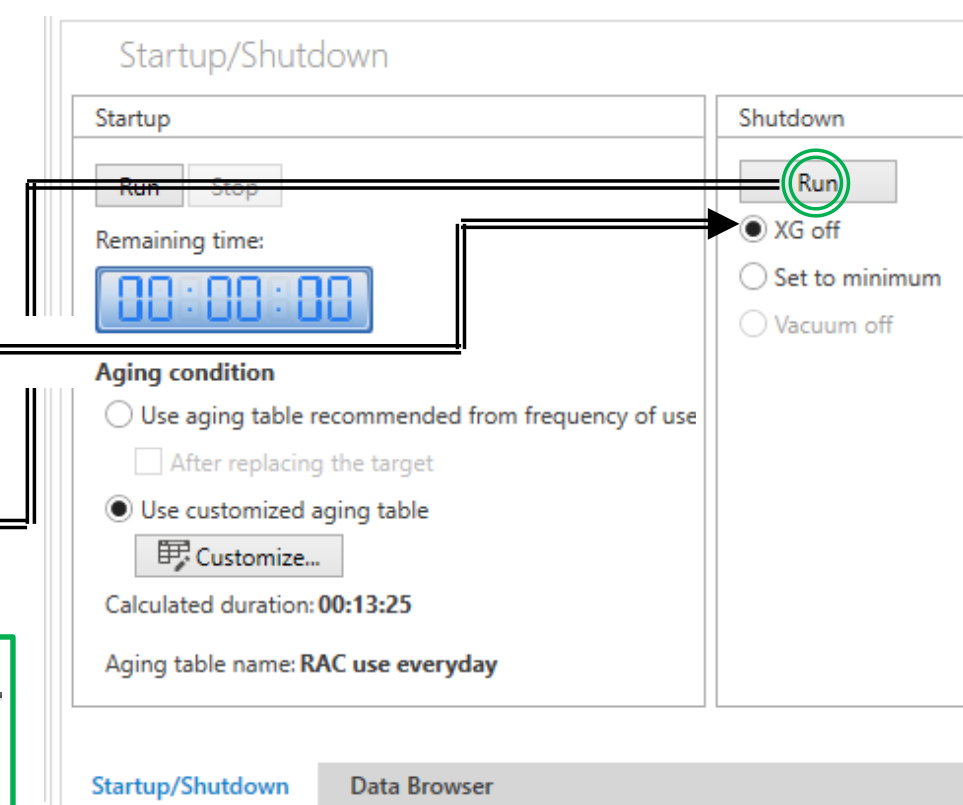
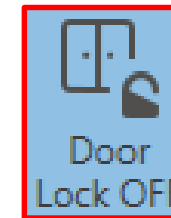
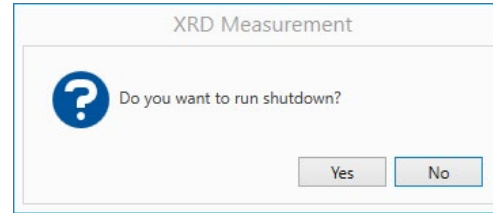
4. Turn off **Light** to cabinet by clicking on **Light ON** button

5. Return the system back to the **Home Position**

6. Close the **Doors** to cabinet, and lock doors by clicking on **Door Lock OFF** button

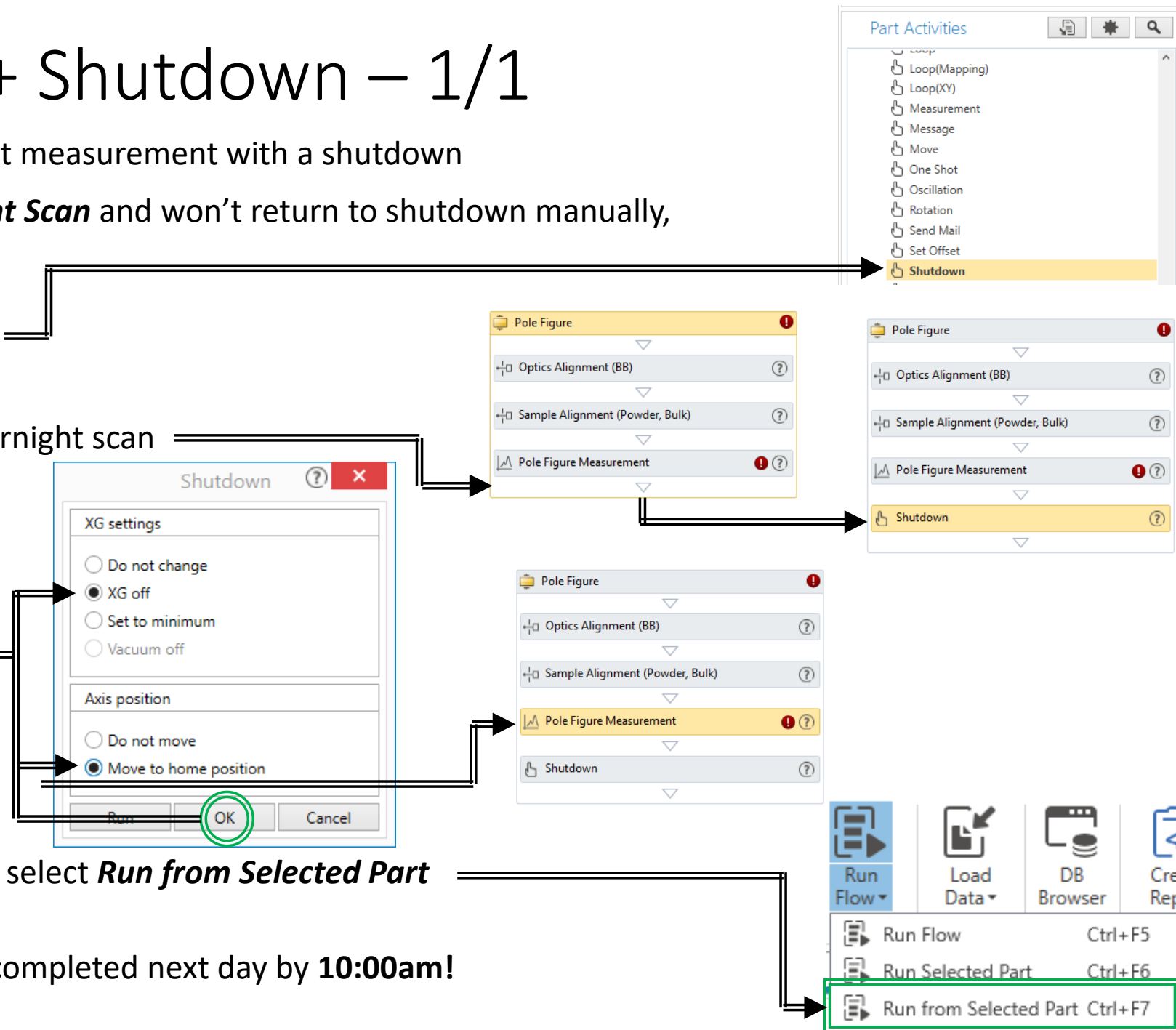
7. Clean up any materials, samples, or waste from work area

8. Record your time and make any notes in the **Sign-In Sheet**



# XX. Overnight Scan + Shutdown – 1/1

- This sequence is for planning an overnight measurement with a shutdown
- 1. If you plan on performing an **Overnight Scan** and won't return to shutdown manually, you may perform the following steps
- 2. Find **Shutdown** under **Part Activities**
- 3. Drag **Shutdown** under the desired overnight scan (e.g. **Pole Figure Measurement**)
- 4. Left-click on **Shutdown** tab to confirm **XG off** and **Move to home position** are checked and click **OK**
- 5. Left-click on the selected **Step**
- 6. Left-click on **Run Flow** drop-down and select **Run from Selected Part**
- 7. The **Cleanup** of your sample must be completed next day by **10:00am!**



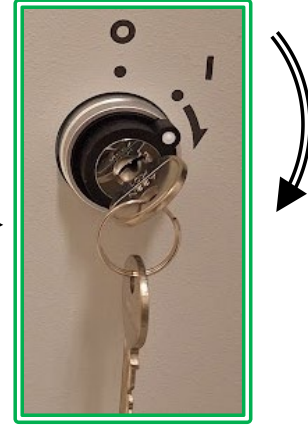
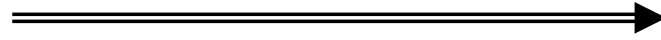


# TS-A. Initial Power Up

○ This sequence is only used for Initial Power Up (power completely off)

1. The following should **ONLY** be performed if instructed by the **Lab Manager**

2. Toggle the **Safety Key** to the right and release



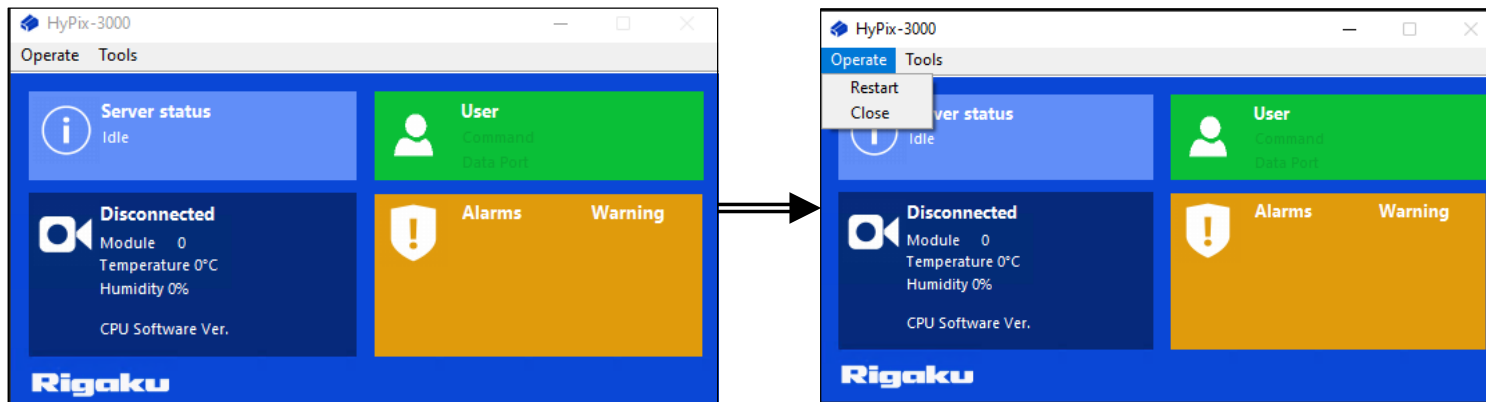
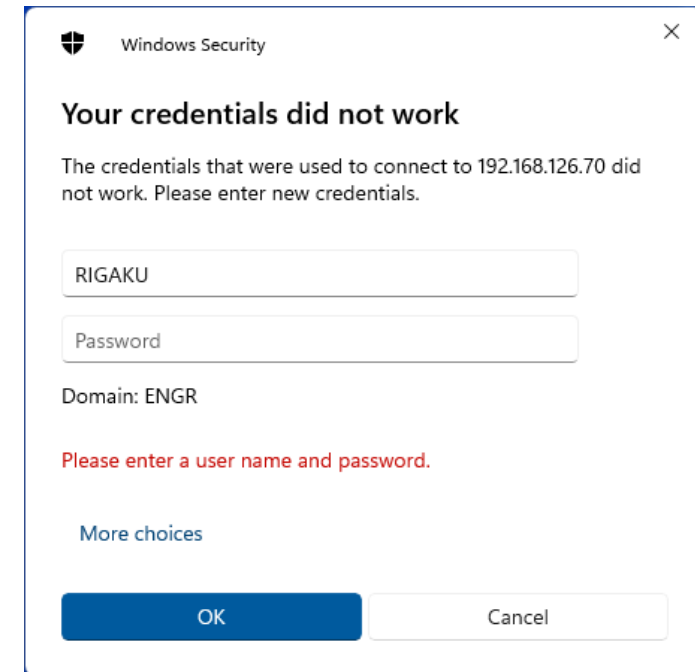
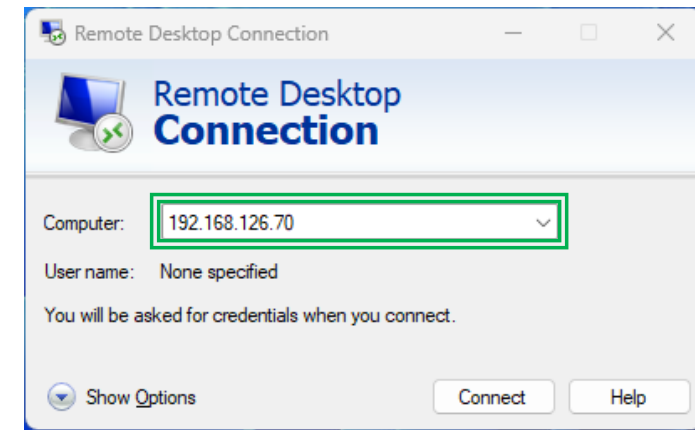
3. Cabinet will perform **Power Up** sequence

4. The **Power ON** lights will illuminate



# TS-B. Hypix Detector Troubleshooting

- This sequence is only used for troubleshooting the Hypix Detector
- 1. If the **Hypix Detector** needs to be remotely connected to troubleshoot...
- 2. Click on **Remote Desktop Connection** and connect to **192.168.126.70**
- 3. No password needed to access
- 4. Provides status of detector
- 5. Click on **Operate -> Restart** to reset **Alarms** if necessary



END OF SLIDES