Thin Film XRD Training Notebook

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MSE Fee-For-Service Facility

Materials Science and Engineering

University of California, Riverside

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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

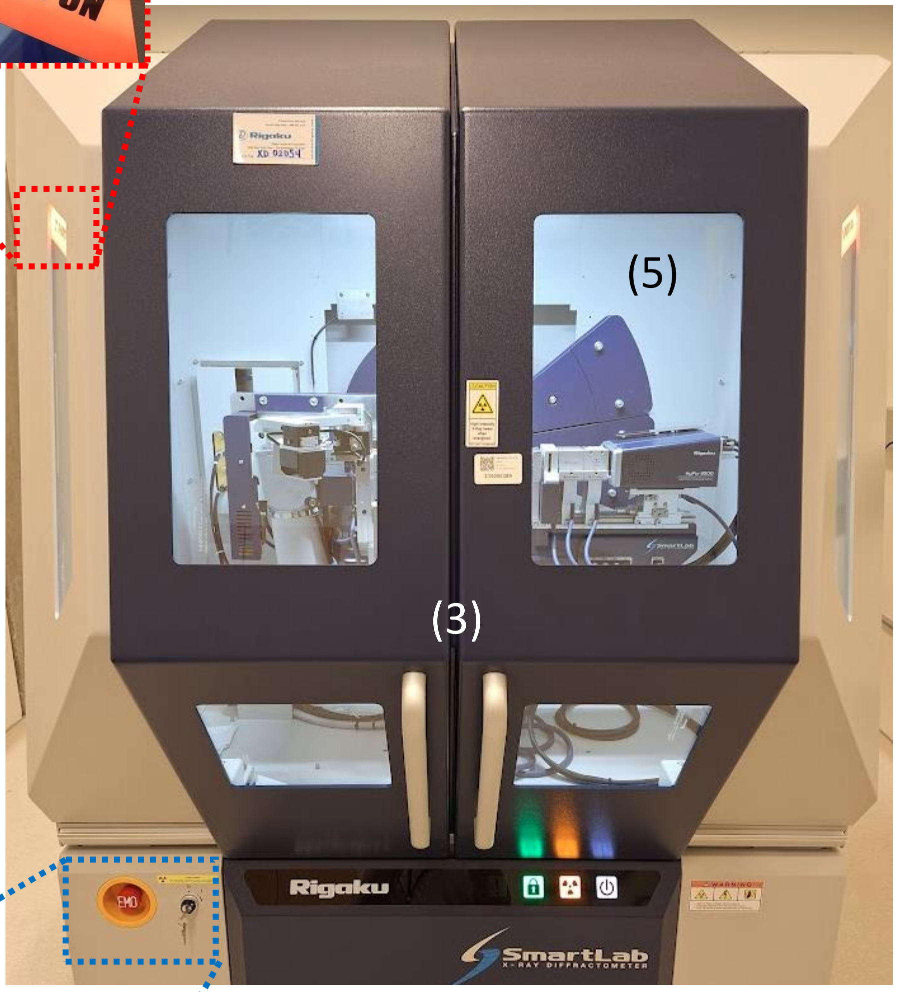
Thin Film XRD (Rigaku SmartLab II) Operation

<u>Troubleshooting</u>

A. XRD Cabinet Overview – 1/2

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

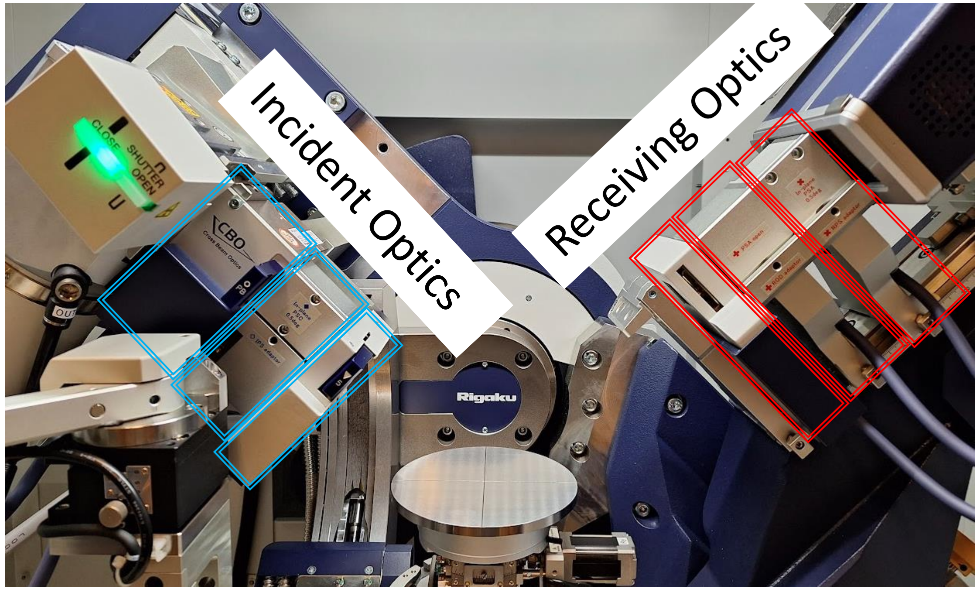
(1)	Power Key:	Power key used to start and stop SmartLab
(2)	Emergency OFF Switch:	Press this switch in the event of an emergency to cut off the power supply to the main unit
(3)	Door:	Opened to change samples and optical devices
(4)	X-Rays ON Lamp:	Lights when X-rays are generated
(5)	Observation Window:	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

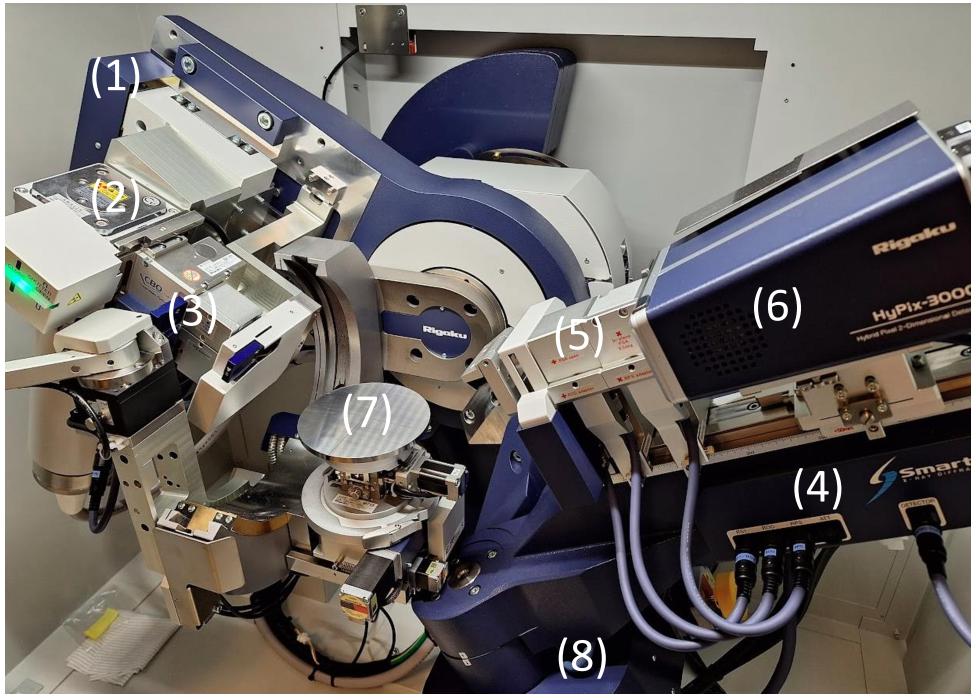


(2)


A. XRD Cabinet Overview – 2/2

(#)	Section	Description
1	Theta_s (θ_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 (θ_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 (θ_χ)	Theta_d arm used for In-Plane measurements





B. Measurement Basics – 1/10

- This summarizes the different Scans and Information obtained

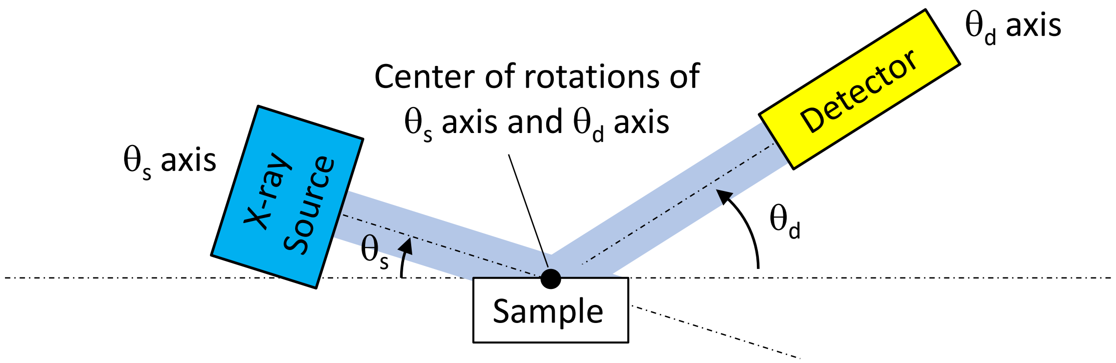
Measurement Technique (Scan)	Information Obtained	Scan Axis
Out-of-Plane (1D)	Information on lattice planes parallel to sample surface → Qualitative analysis and crystal structure	$2\theta/\omega$ (Always $2\theta = 2 \times \omega$)
Thin Film (1D)	Information near sample surface (applies only to unoriented samples) → Qualitative analysis and crystal structure	2θ (Incident angle, ω, 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, ω, is fixed near the critical angle)
Pole Figure (2D)	Information on distribution of specific crystal orientation → Orientation analysis	$\chi(\alpha)$, $\varphi(\beta)$ (2θ or sum of 2θ 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	ω, χ, or φ
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	$2\theta/\omega$, ω (χ or φ) $2\theta_\chi/\varphi$, φ (χ or φ)
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

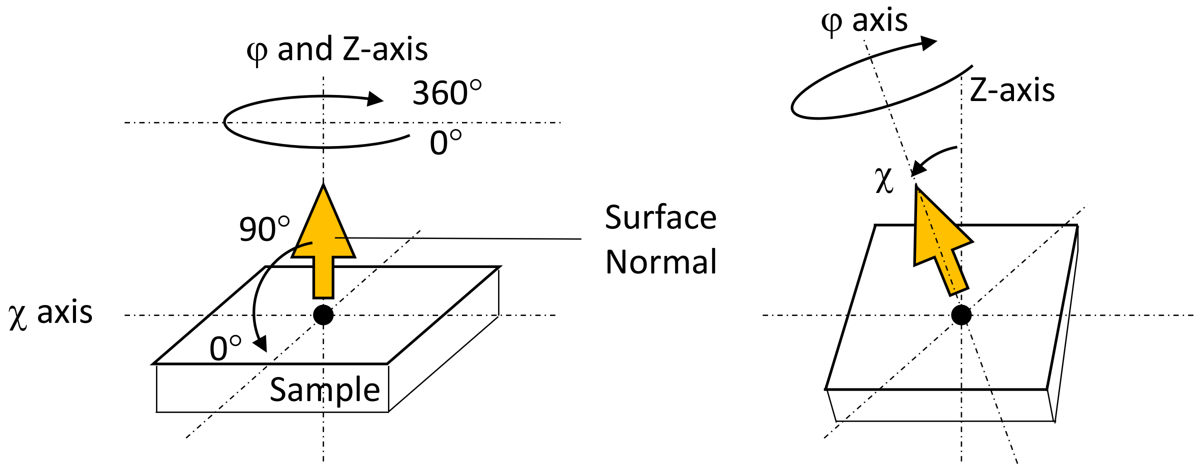
θ_s axis = axis for setting angle of X-ray Source with respect to the Sample Surface (s = "source")

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



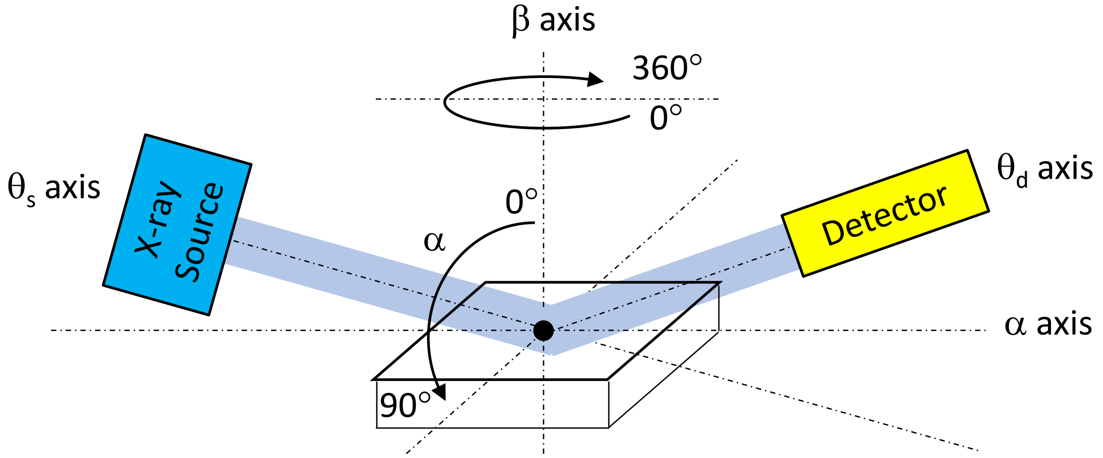
φ axis = sample rotation axis around Surface Normal axis

χ axis = axis for setting tilt of Surface Normal with respect to Goniometer Z-axis



α axis = rotation axis for setting tilt angle with respect to Z-Axis

β axis = sample rotation axis along Surface Normal (see φ axis)



<u>Out-of-Plane Pole Figure Definitions</u>

α tilt = 90° - χ tilt

β rotation angle = φ rotation angle

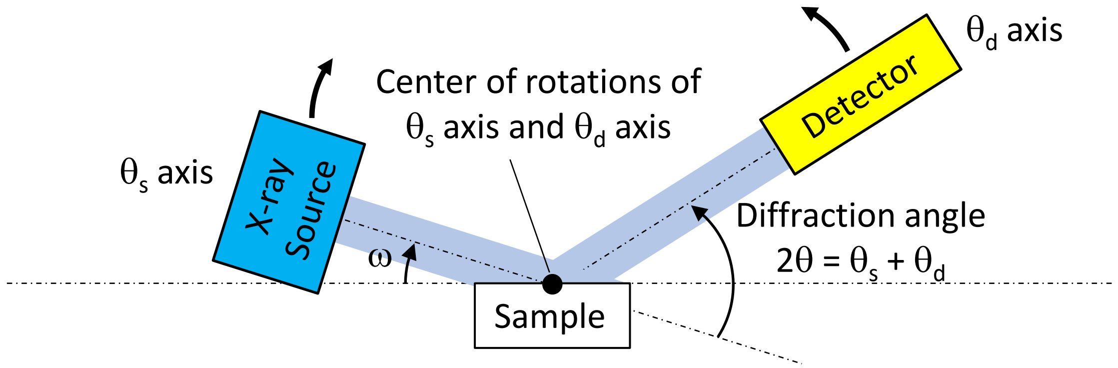
<u>In-Plane Pole Figure Definitions</u>

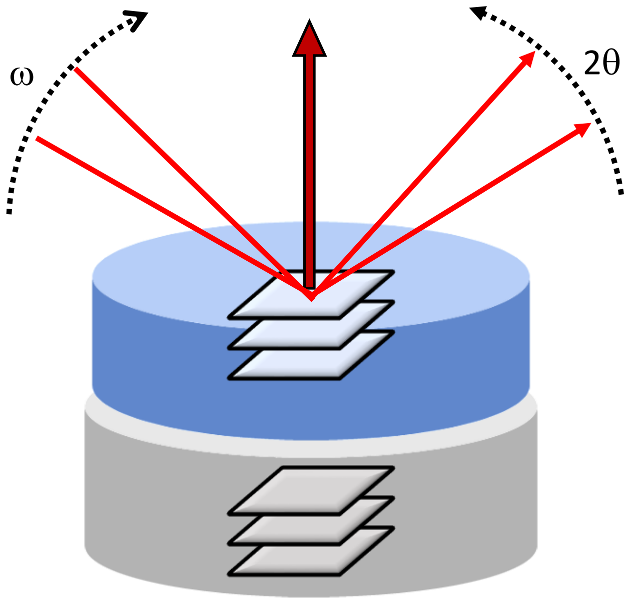
α tilt = χ tilt

β rotation angle = φ rotation angle

B. Measurement Basics – 3/10

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





Movement:

- 2θ is driving arm; 2θ range = -10 to 158°
- ω is slave arm; ω = ½ (2θ)

Pros:

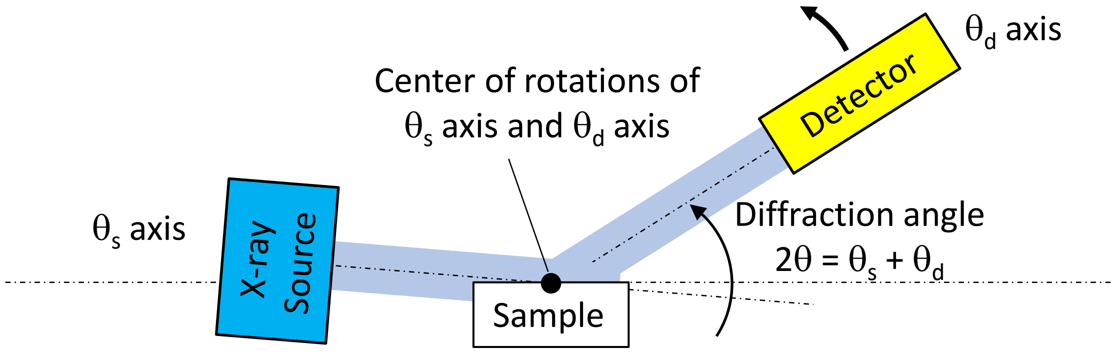
- Used for Qualitative analysis such as:
- Determining presence or absence of a preferred orientation
- Interplanar spacings of lattice planes parallel to surface
- Lattice constants corresponding to these interplanar spacings
- Crystallinity of a crystal lattice parallel to the surface

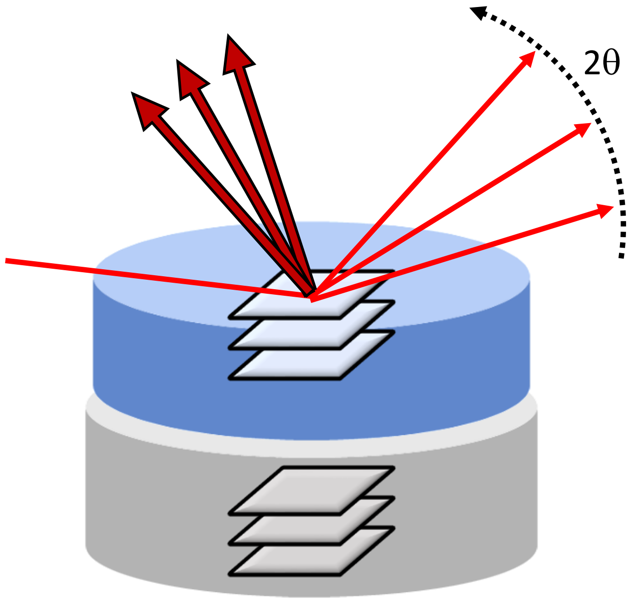
Cons:

- 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:

- 2θ is driving arm; 2θ range = -15 to 120°
- ω is set near a small critical angle usually between 0.1 to 1°

Pros:

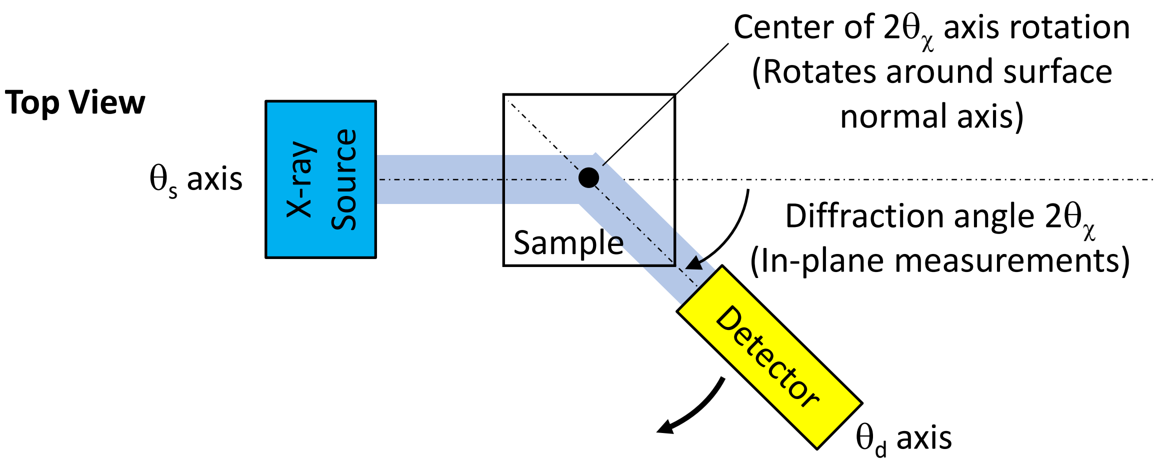
- Avoids scattering from the substrate
- Used for Qualitative analysis such as:
- For unoriented (or weakly oriented) polycrystal samples
- Lattice constants
- Crystallinity of a sample
- Depth dependence of above physical quantities

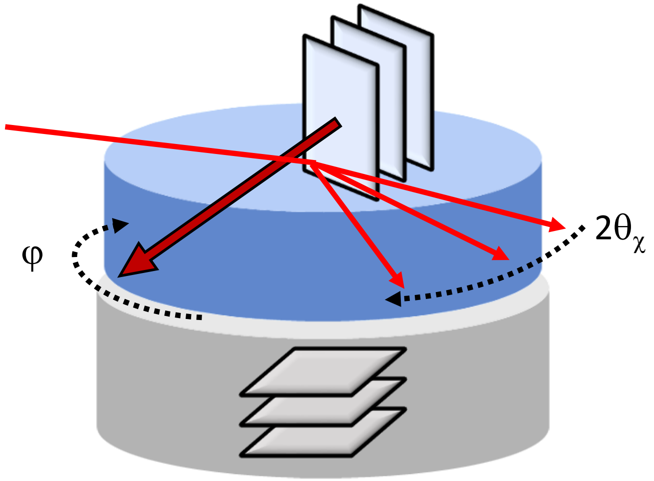
Cons:

- 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 $2\theta_\chi/\varphi$ Measurement





Movement:

- $2\theta_\chi$ is driving arm; $2\theta_\chi$ range = -3 to 89°
- ω is set near a small critical angle usually between 0.1 to 1°
- φ is slave arm; $\varphi = ½\,(2\theta_\chi)$

Pros:

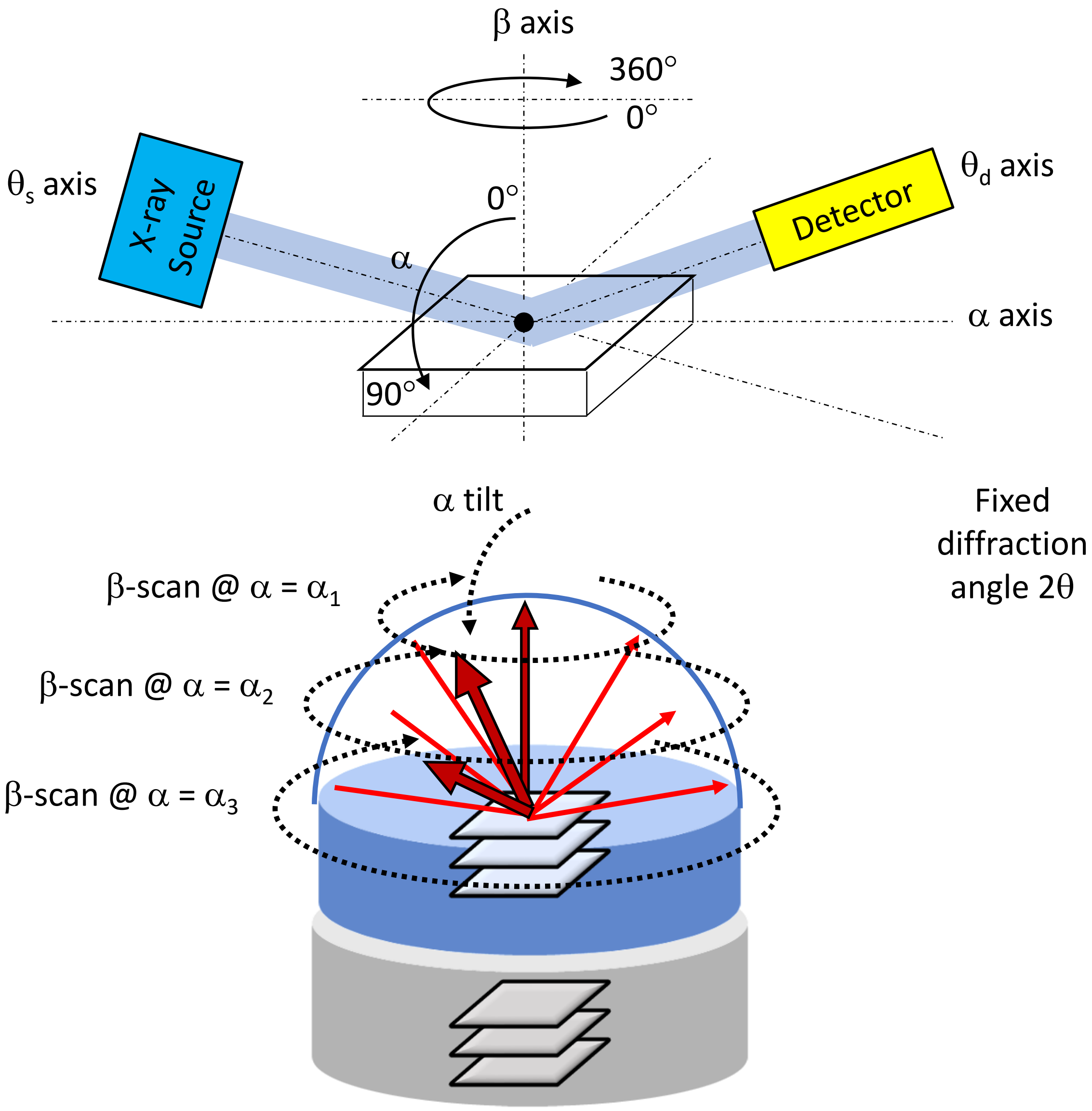
- Similar to GIXRD with respect to depth of analysis
- Used for Qualitative analysis such as:
- Investigating presence or absence of the preferred orientation
- Interplanar spacings of lattice planes perpendicular to the surface
- Lattice constants corresponding to these interplanar spacings
- Crystallinity 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:

- 2θ is kept constant; $\omega = ½ (2\theta)$
- α is stepped; α range = -5 to 95°
- β is continuously rotated; β range = -720 to 720°

Pros:

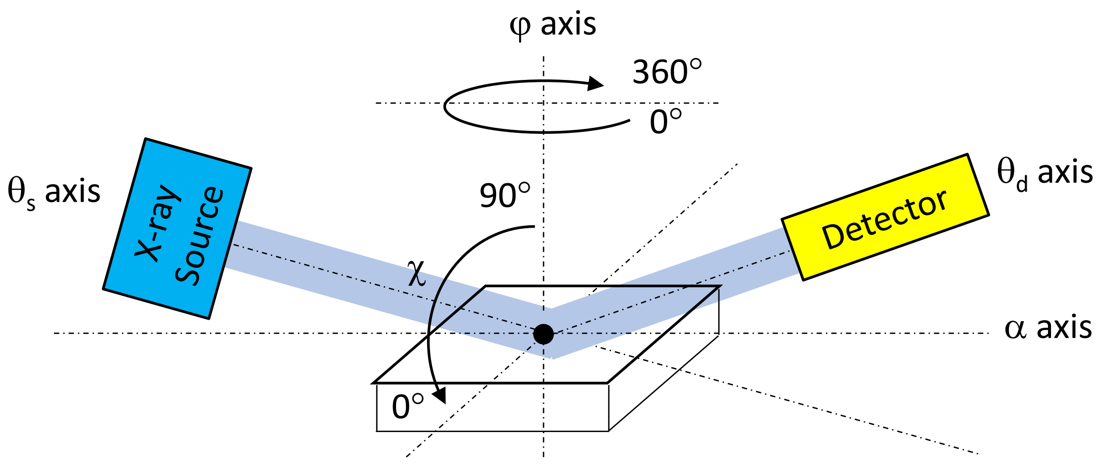
- Measures diffraction intensity distributions by rotating the sample in all directions while keeping the diffraction angle constant
- Direction at high diffraction intensity is observed corresponds to the preferred direction of the pole figure axes – indicating that crystallites with the measurement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

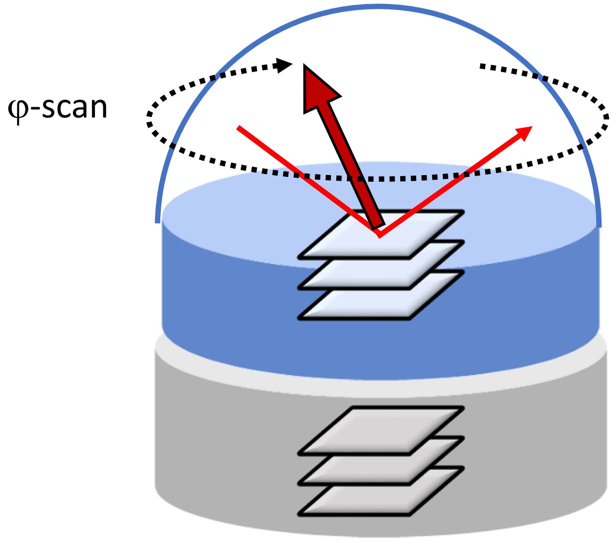
Notes:

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

B. Measurement Basics – 7/10

- This covers the Preferred Orientation (1D) or Azimuth or φ Scan Measurement





Movement:

- 2θ is kept constant; ω = ½ (2θ)
- φ is continuously rotated; φ range = -720 to 720°

Pros:

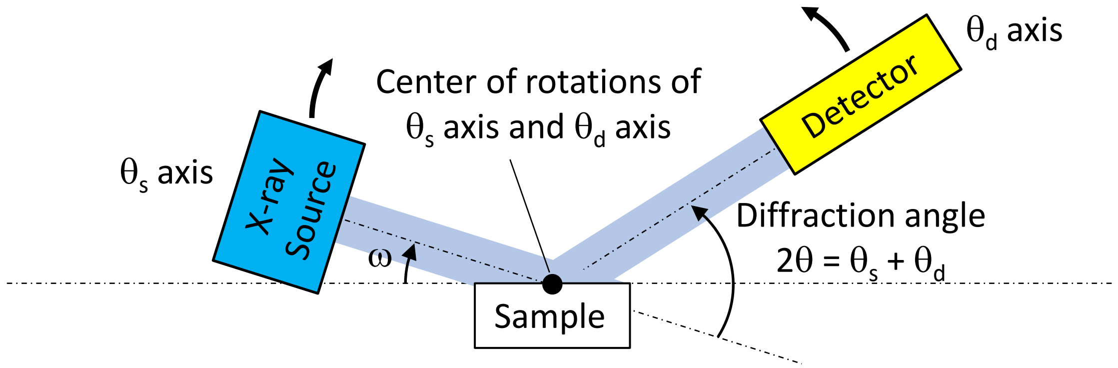
- Same underlying principles for pole figure measurement
- Measures a cross-section of a pole figure measurement
- Measures the spread (width) of diffraction intensity distribution
- Related to degree of preferred orientation (vs randomly orientated sample)
- Related to mosaicity (vs perfect single crystal)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

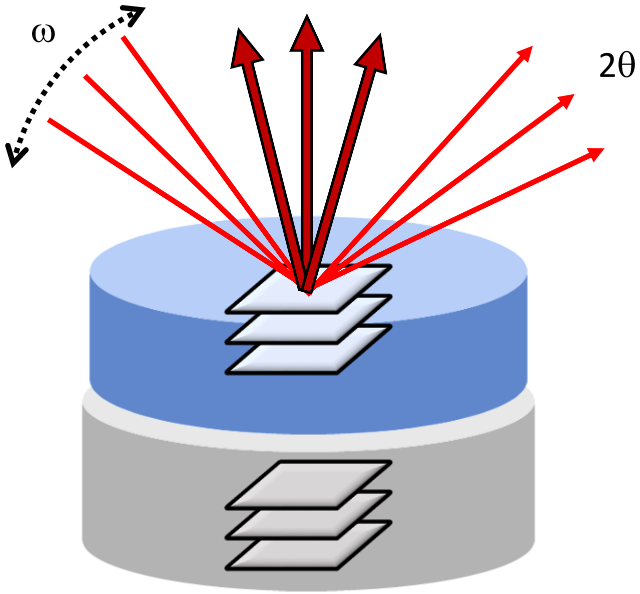
Notes:

- Remember to optimize χ value first!
- Speed of φ scan controls the sign-to-noise ratio of scans

B. Measurement Basics – 8/10

- This covers the Rocking Curve (1D) Measurement





Movement:

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

Pros:

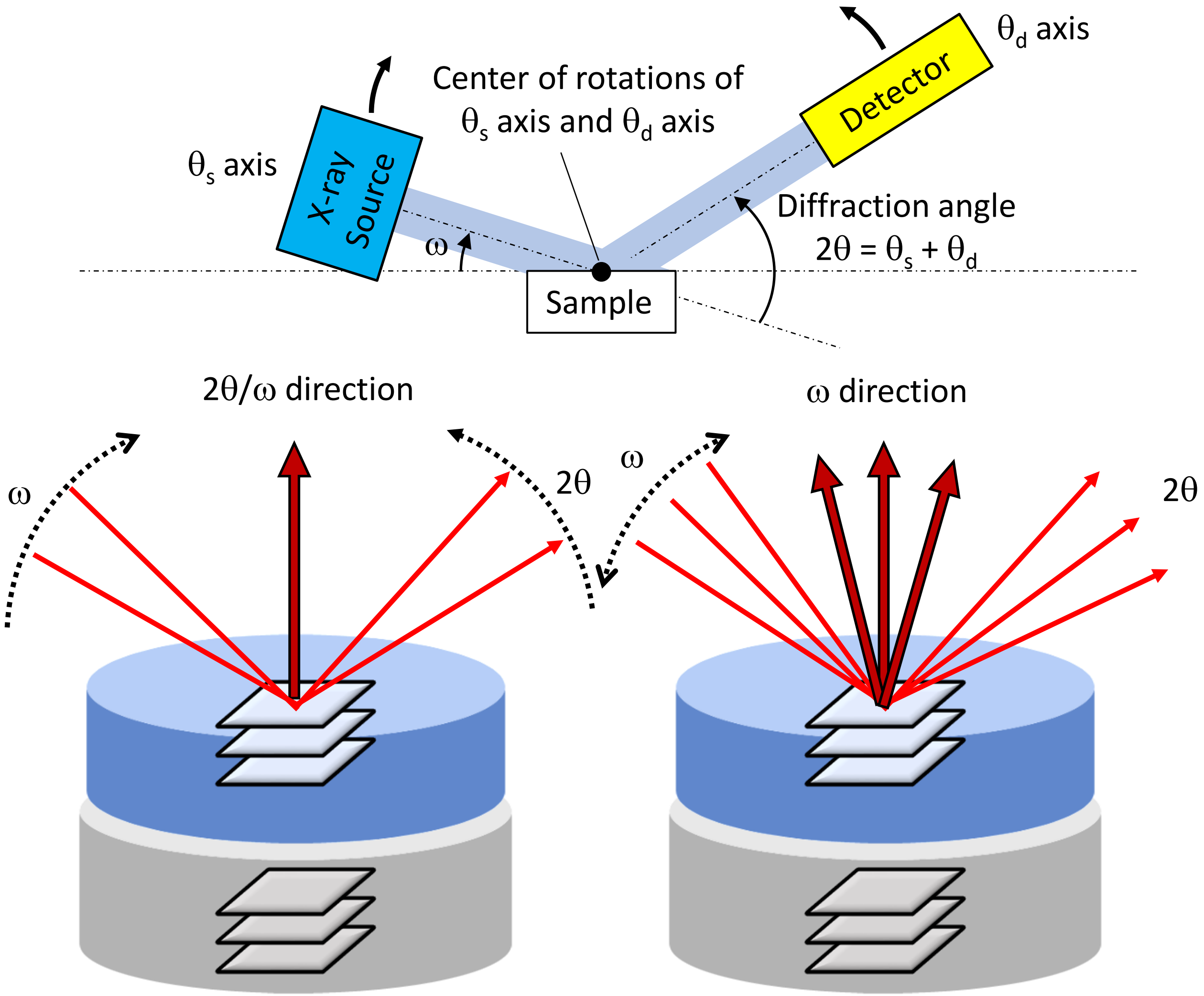
- Measures diffraction intensity distributions along a reciprocal lattice vector
- Planes no longer parallel with sample surface are brought onto the Bragg plane
- Measures changes in interplanar spacing
- Generally used to evaluate the thickness or mixed crystal ratio of an epitaxial film on a sample
- 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
- ω scan is second mapped direction

Pros:

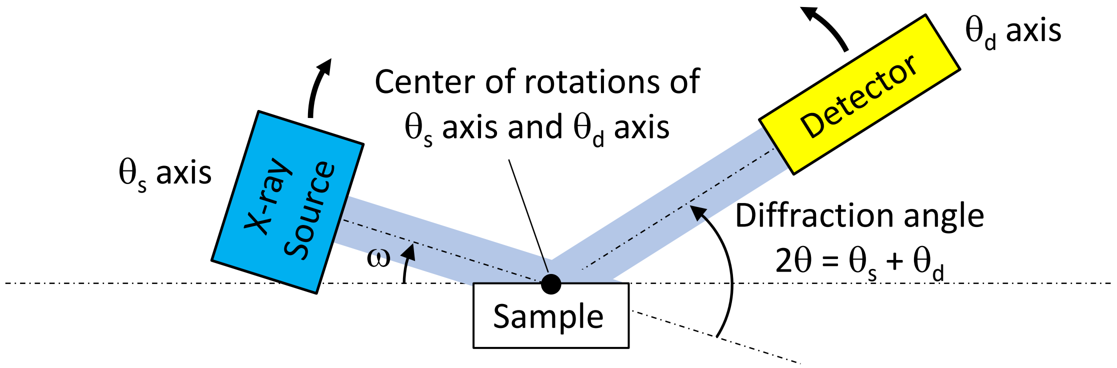
- Measures diffraction intensity distributions and plots result in reciprocal space
- Central coordinates, shapes, and positional relationships of the reciprocal lattice points (film and substrate) appearing in 2D map provides a wide range of info on crystal structure

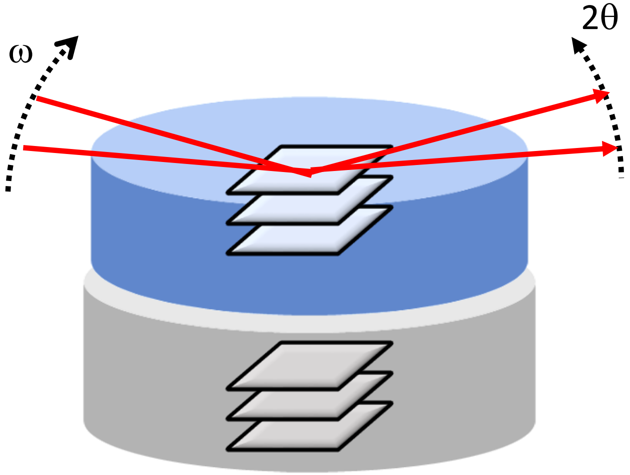
Notes:

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

B. Measurement Basics – 10/10

- This covers the Reflectivity (1D) Measurement





Movement:

- 2θ is driving arm; 2θ range = 0 to 10°
- ω is slave arm; ω = ½ (2θ)

Pros:

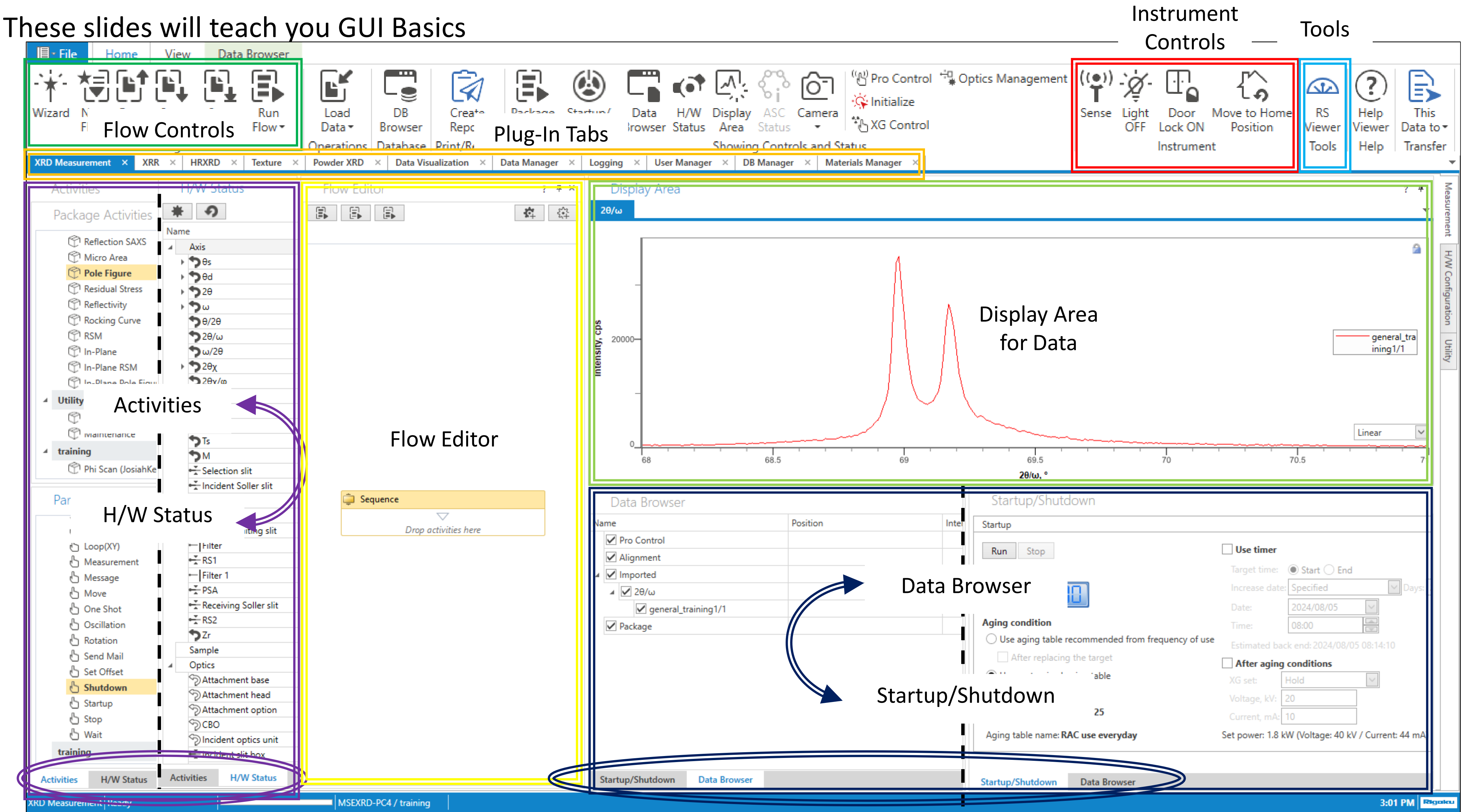
- Can be used for crystalline or amorphous samples
- Evaluates thickness, density, and surface or interface roughness of thin-film materials
- Evaluates structure of a multilayer or single layer film
- Measures samples nondestructively

Cons:

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

C. GUI Basics – 1/3

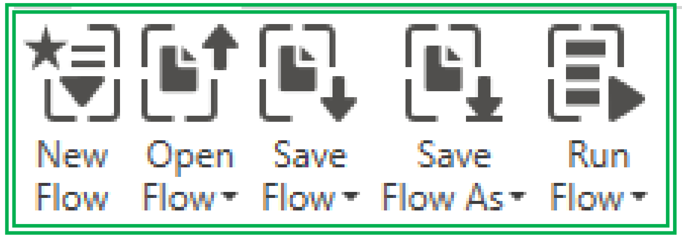
- These slides will teach you GUI Basics



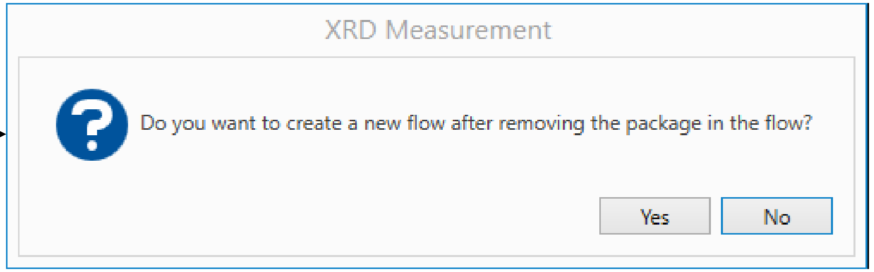
C. GUI Basics – 2/3

- ***Flow Controls*** – used to control your ***Measurement Flow*** (or ***Measurement Program***)

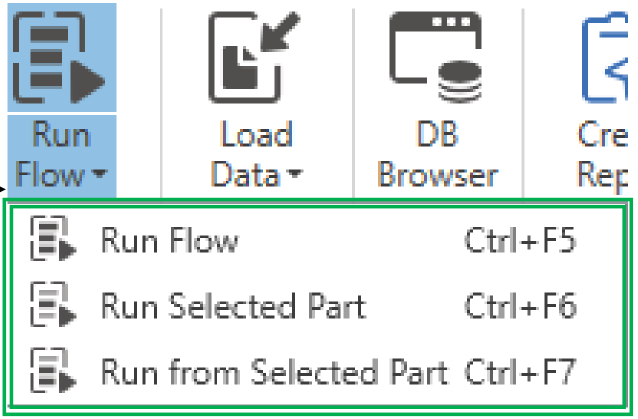
  - ***Wizard*** – will help you selected the recommend packages for desired measurement/analysis



  - ***New Flow*** - remove existing ***Flow*** and create a ***New Flow*** package



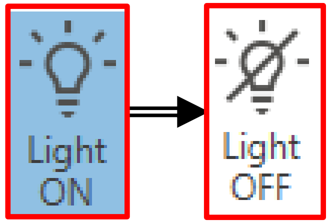
  - ***Open Flow*** - open existing ***Flow*** that you have saved in the ***File System***
  - ***Save Flow*** - saves your edited ***Flow*** into the ***Files System***
  - ***Save Flow As*** - saves your edited ***Flow*** as a new file into the ***Files System***
  - ***Run Flow***



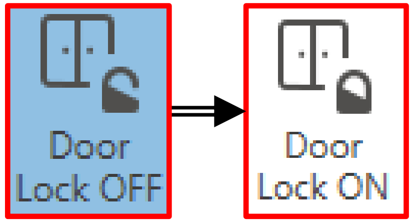
    - ***Run Flow*** – runs your entire ***Flow*** from top to bottom
    - ***Run Selected Part*** – only runs the ***Selected Part***
    - ***Run from Selected Part*** will run your entire – runs your flow from top to bottom starting from ***Selected Part*** (useful for ***Shutdown***)

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***

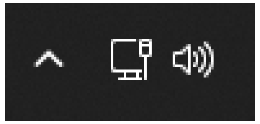
2. First ***Double-click*** on ***SmartLab Studio II*** software icon 

3. Enter your ***Login*** and ***Password***
   - Login: *Faces Login* Password: *Provided by Lab Manager*

4. Confirm that "***XRD Measurement | Ready***" is shown → 

then proceed to ***Step 8***

5. If ***Status*** is "***XRD Measurement |Ready (Not Connected)***", you will need to follow ***Steps 6-7*** 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 8***

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

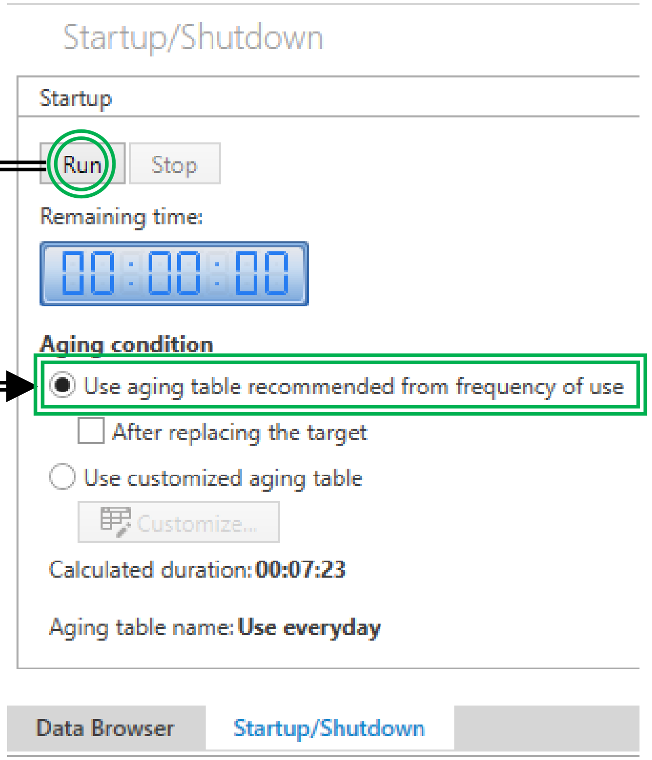
11. Click the ***Run*** button, and confirm with ***Yes***

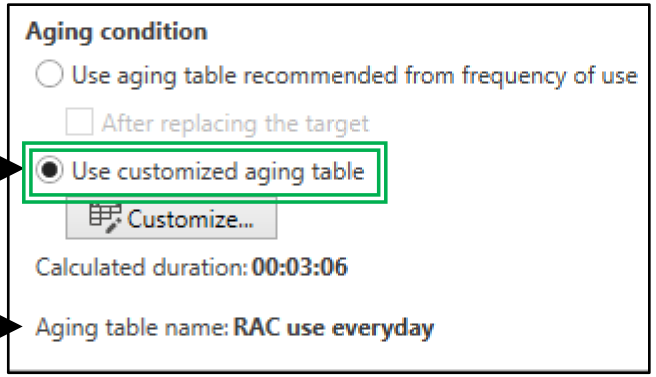
12. Once completed, the ***Standby*** setting should be at ***40 kV*** and ***30 mA*** (1.2 kW)

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***

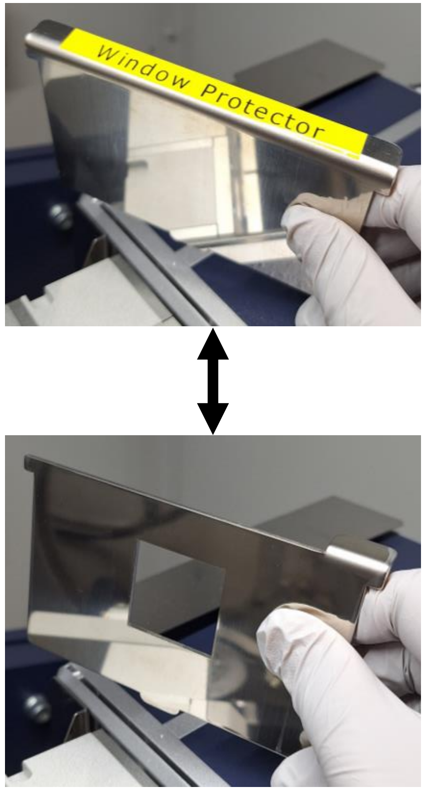
15. The ***Operational Level*** should now be set to ***40 kV*** and ***44 mA*** (1.8 kW) for normal operation

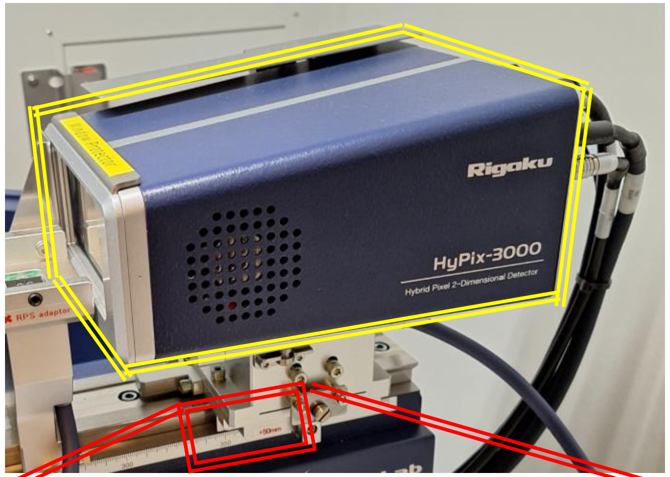
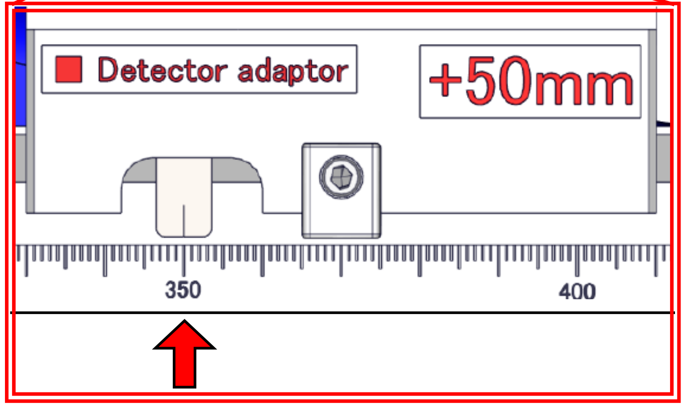
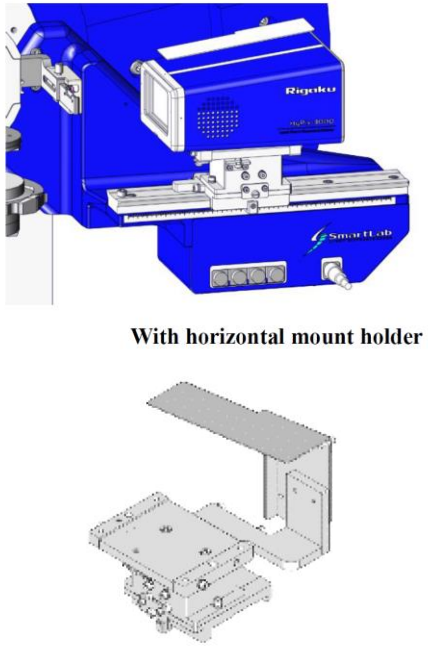
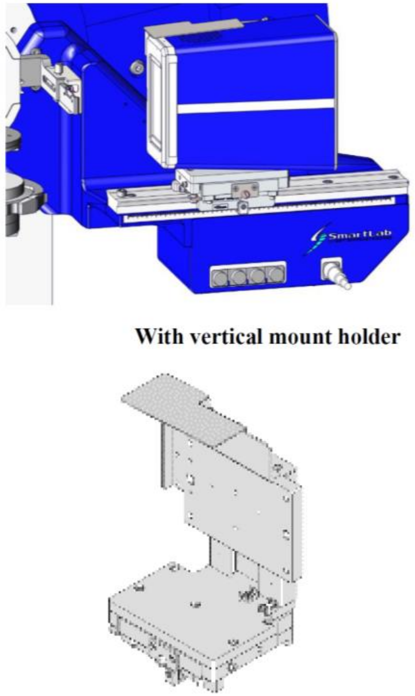




II. XRD Detector – 1/1

- This covers the Detector

<table>
<tr><th>Detector</th><th colspan="2">Orientation (Applications)</th><th colspan="2">Window Protector</th></tr>
<tr><td>HyPix-3000
(2D Detector)</td><td>Horizontal
(Default)</td><td>Vertical
(Micro Area)</td><td rowspan="2"></td><td rowspan="2">Window Protector must be inserted to protect Detector when swapping out Receiving Optics!

$$$$$$</td></tr>
<tr><td rowspan="2"></td><td></td><td></td></tr>
<tr><td colspan="4">Mark indicates position of detector adapter and is +50 mm from the detection plane.
i.e. Detector Plane of 300 mm ⇒ Detector Adaptor Position of 350 mm</td></tr>
</table>

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

<table>
<tr><th colspan="2">Incident Optics Unit #2</th><th colspan="3">Incident Parallel Slits (Aperture) + IPS Adaptor</th></tr>
<tr><td>Incident Parallel Slit (IPS) + Adaptor</td><td>Ge(220) 2-bounce monochromator</td><td>Soller Slit
(Open)</td><td>Soller Slit
(5.0deg)</td><td>In-plane PSC
(0.5deg)</td></tr>
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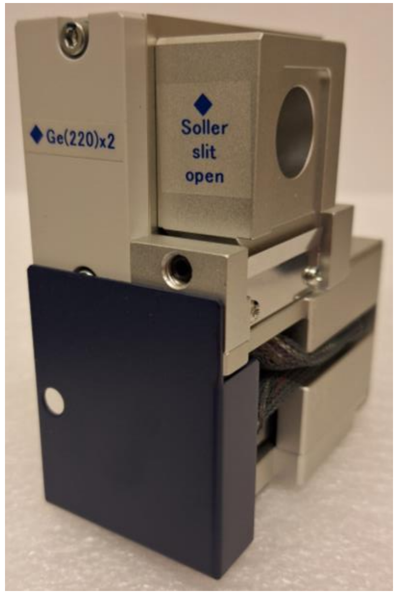
<td>
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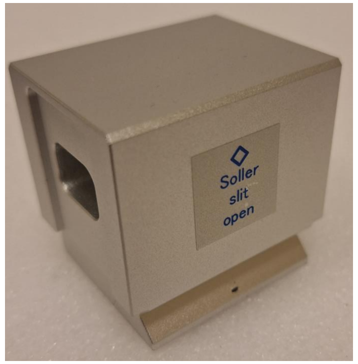
<td>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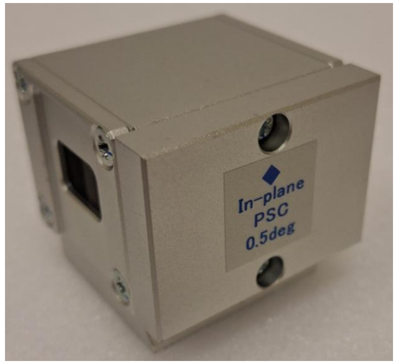
<td>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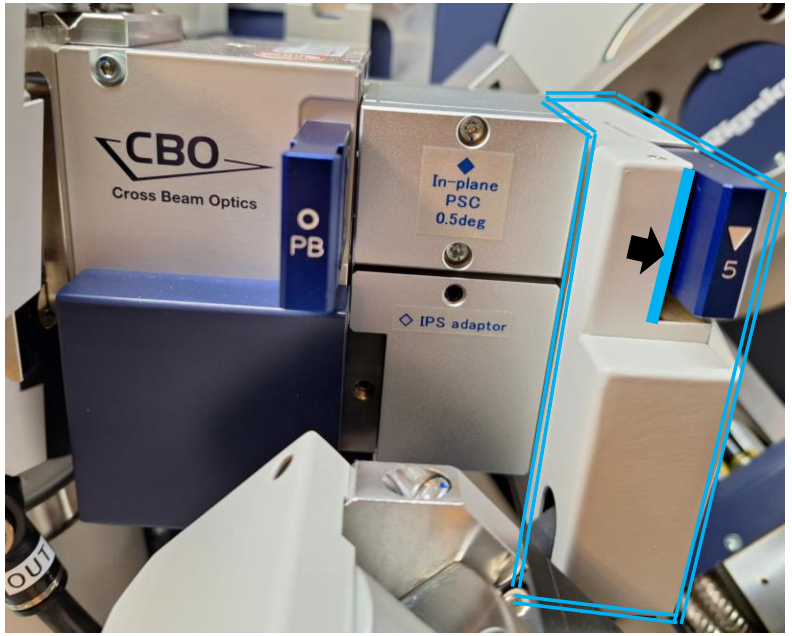
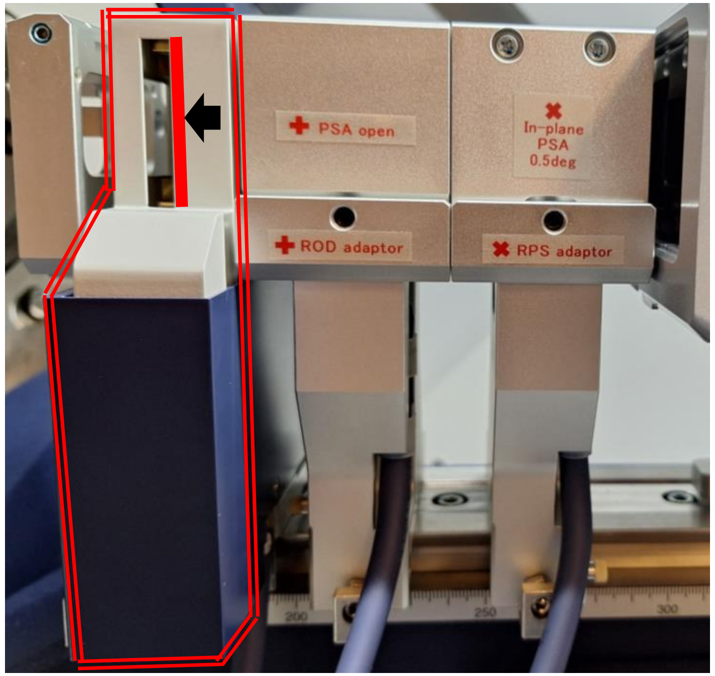
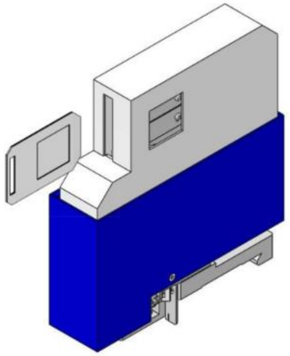
III. XRD Optics – 3/6

- This covers the Incident Slit

Incident Slit	Length-Limiting Slit (Aperture)				
	10 mm	5 mm	2 mm	0.5 mm	0.2 mm
 Insert Slit hugging LEFT edge					

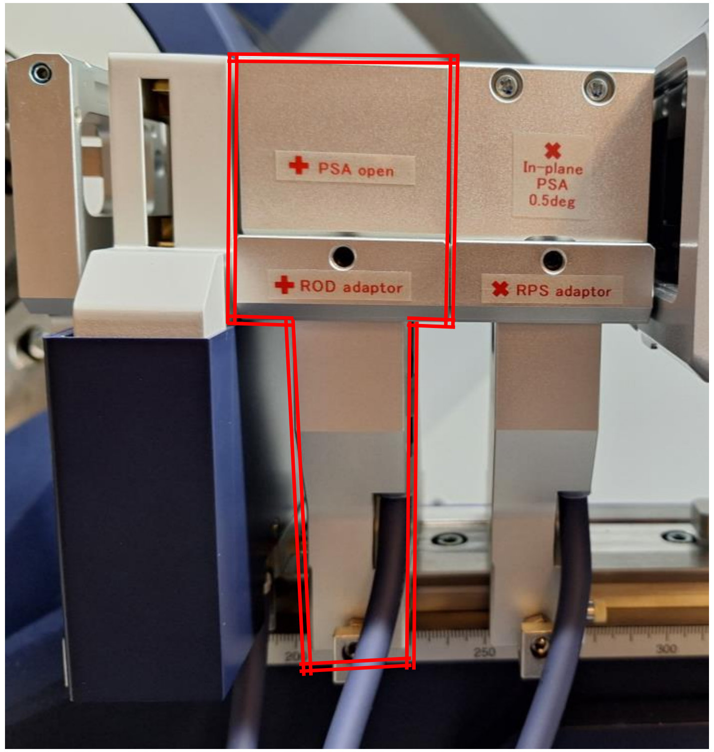
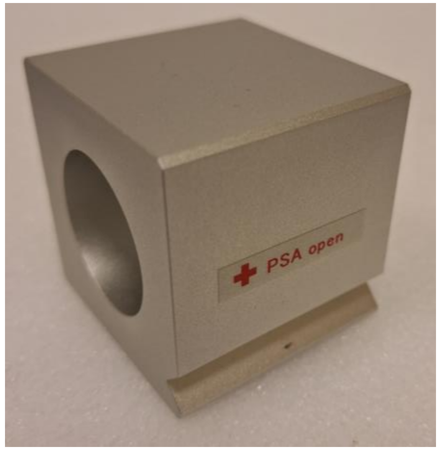
III. XRD Optics – 4/6

- This covers the Receiving Slit

Receiving Slit	Insertion Diagram	kβ Filters (Thickness)		
Receiving Slit Box		CuK β 1D (23 μm)	CuK β 15 μm (15 μm)	9 kW filter
 Insert Card hugging RIGHT edge				

III. XRD Optics – 5/6

- This covers the Receiving Optics Unit #1

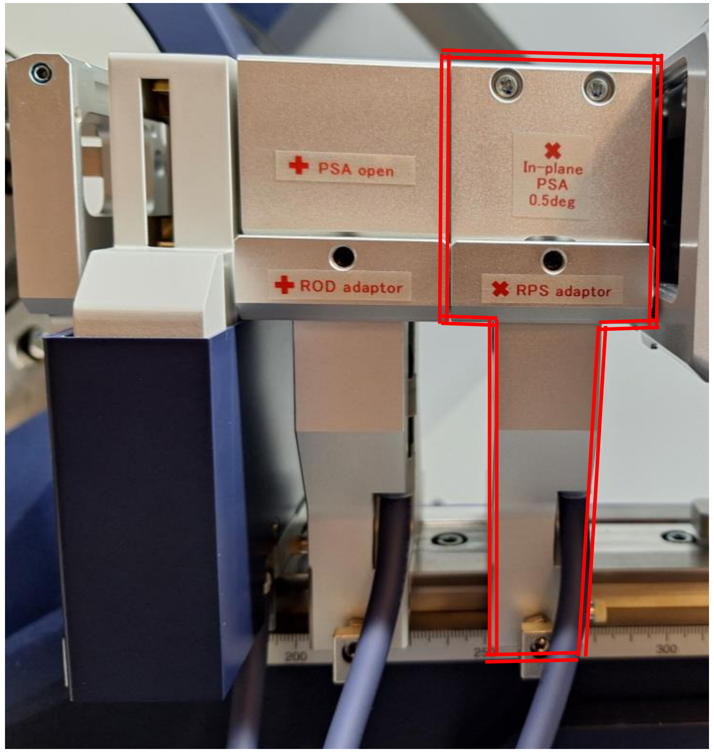
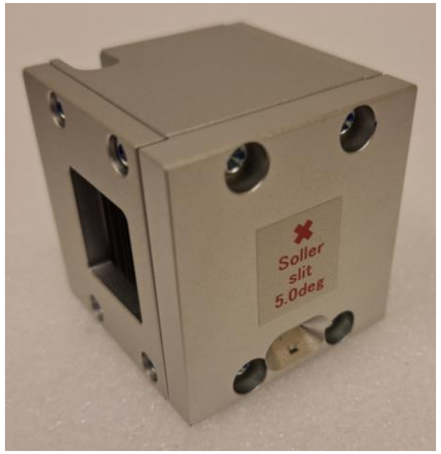
<table>
<tr><th>Receiving Optics Unit #1</th><th colspan="2">Parallel Slits Analyzers (Aperture) + ROD Adaptor</th></tr>
<tr><td>Parallel Slit Analyzer (PSA) + Adaptor</td><td>PSA
(Open)</td><td>PSA
(0.5deg)</td></tr>
<tr><td>
</td><td>


</td><td>


</td></tr>
</table>

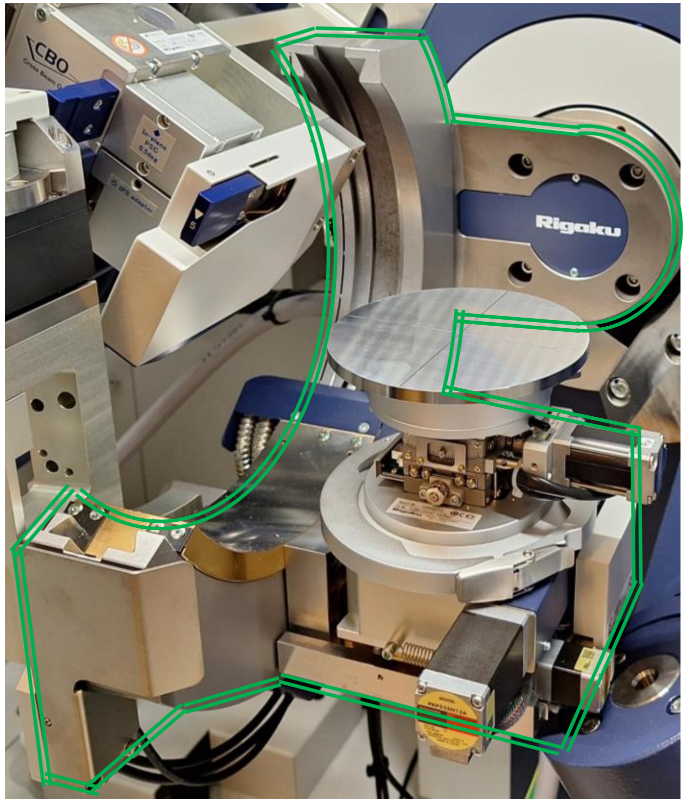
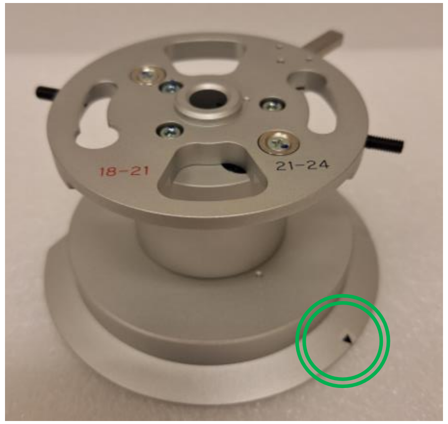
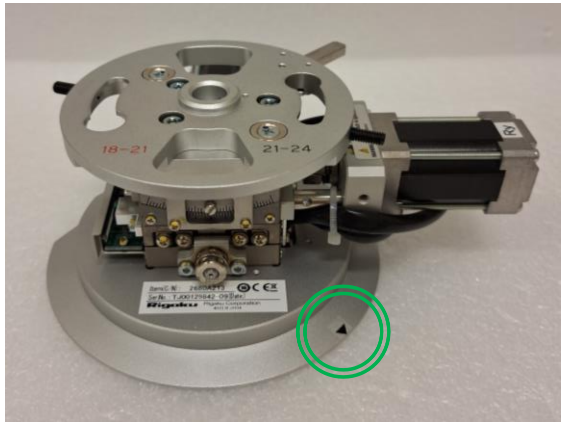
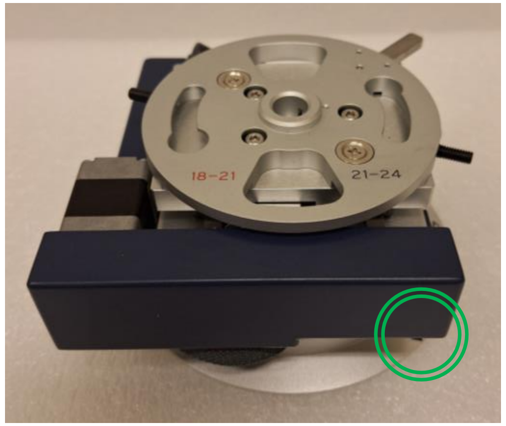
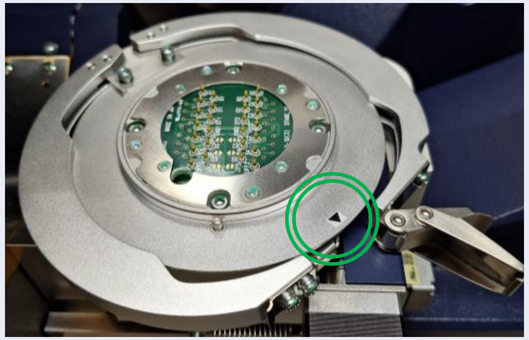
III. XRD Optics – 6/6

- 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

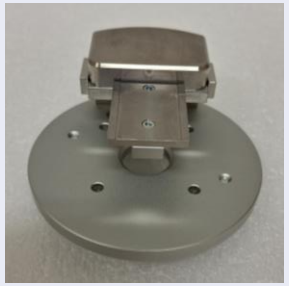
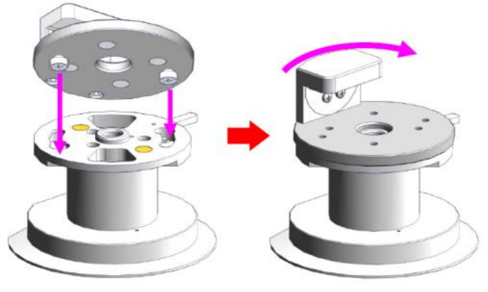
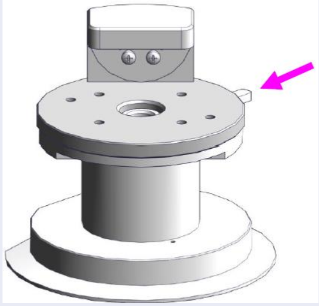
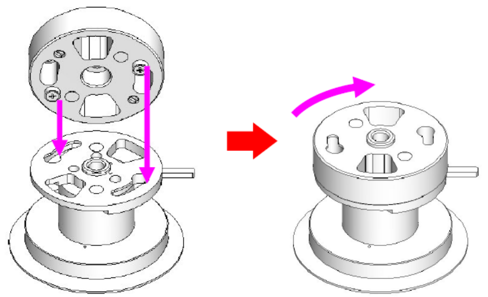
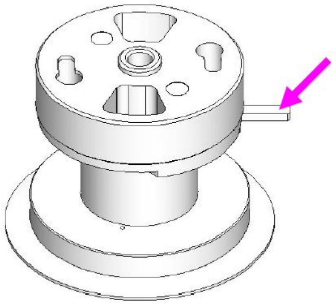
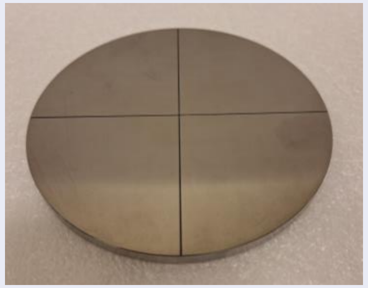
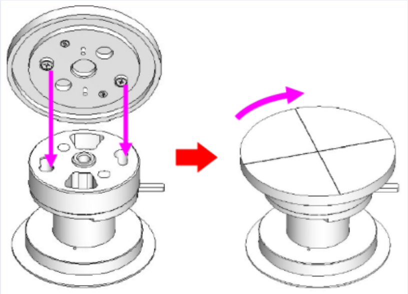
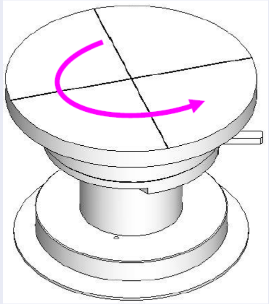
<table>
<tr><th>Attachment Platform</th><th colspan="3">Attachment Heads (Applications)</th></tr>
<tr><td>χφZ Attachment Platform</td><td>Standard
(Alignment, Bulk Sample)</td><td>RxRy
(Reflectivity, RSM, In-Plane)</td><td>XY-20 mm
(Micro-area)</td></tr>
<tr><td rowspan="2">
</td><td>
</td><td>
</td><td>
</td></tr>
<tr><td></td><td colspan="2">Carefully align Attachment Head to the Attachment Platform via the black triangle ▼ indicator.

DO NOT BEND OR DAMAGE THE CONNECTOR PINS!

Secure in place by closing the Clasps and Front Latch.</td></tr>
</table>

IV. XRD Sample Attachment – 2/2

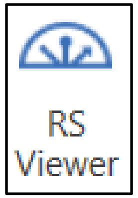
- This covers the Sample Plates (Note: Does not have recognition chips – you must remove even if not instructed!)

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

V. RS Viewer – 1/2

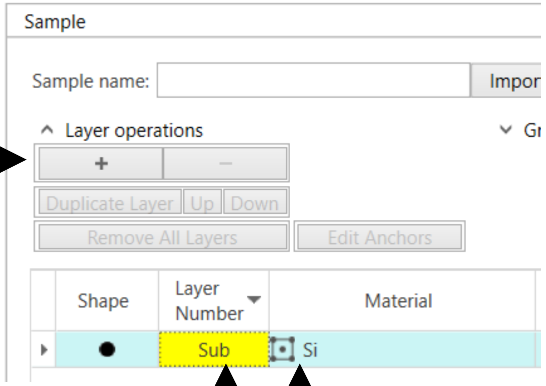
- These slides will teach you about the Reciprocal Space (RS) Viewer

1. Click on the ***RS Viewer*** icon at the top of the ***XRD Measurement*** plugin

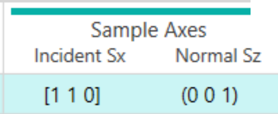


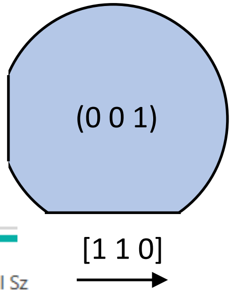
2. Click on the **?** icon to open up the ***RSViewer_UserManual_en.pdf*** to review the tutorial

3. Add and edit layers to build up your substrate + films

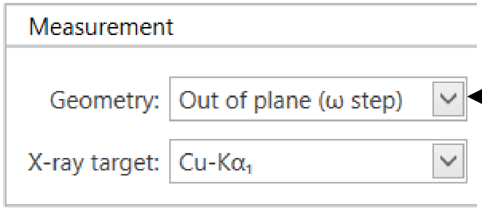


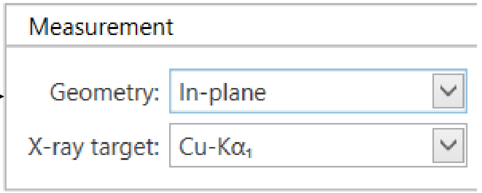
4. Edit the ***Samples Axes*** (if known) to correlate actual sample to reciprocal lattice (e.g. Si wafer)



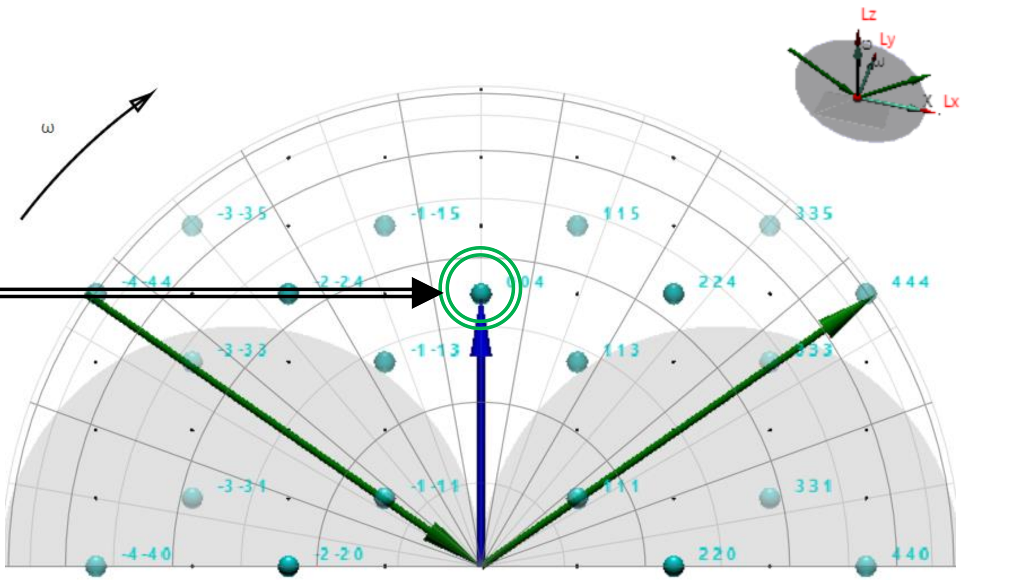


5. Set ***Geometry*** for your scans: ***Out of plane*** or ***In-Plane***



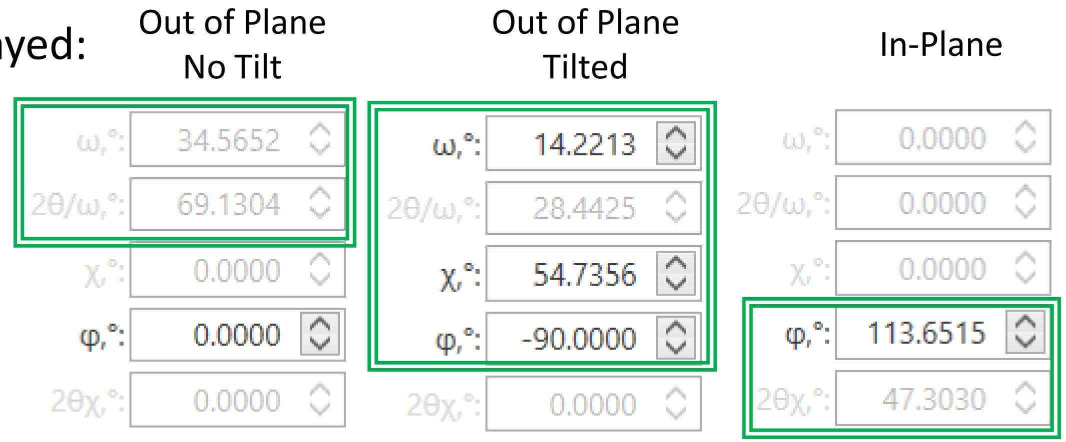


6. Enter in desired reflection plane in ***Origin*** or select the ***Shape Icon***



V. RS Viewer – 2/2

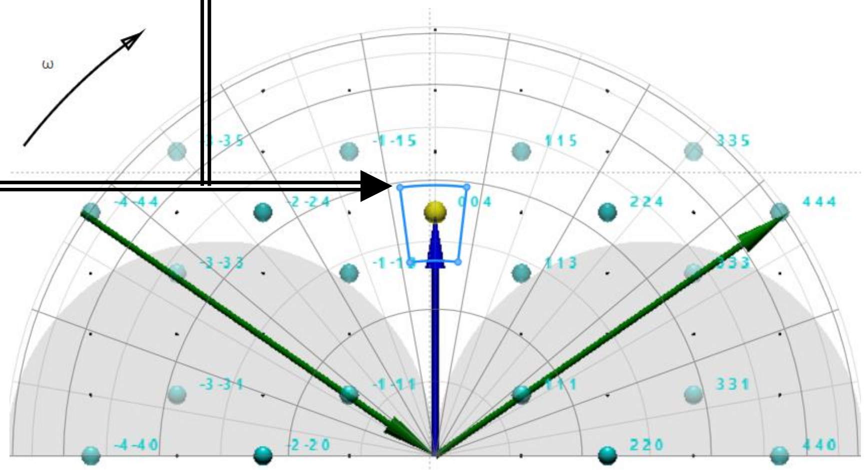
7. The goniometer settings to achieve desired diffraction will be displayed:
   - Out-of-Plane – No Tilt Scans: χ and φ are both zero
   - Out-of-Plane – Tilted Scans: χ and/or φ are non-zero values
   - In-Plane Scans: $2\theta_\chi$ is diffraction angle



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

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***

Accessing COD Data

Browse
Search
Search by structural formula
JSME search

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

Search

(For more information on search see the hints and tips)

Search by COD ID:	Search
OpenBabel FastSearch:	**Enter SMILES:** Search

Note: substructure search by SMILES is currently available in a subset of COD containing 225655 structures.

text (1 or 2 words)
journal

14. Identify the desired sample information you want to import

COD ID ▲	Links	Formula ▲	Space group ▲	Cell parameters	Cell volume ▲	Bibliography
1001452	CIF	Ba2 Cu3 O7 Y	P m m m	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 *Europhysics Letters*, **1987**, 3, 1301-1307

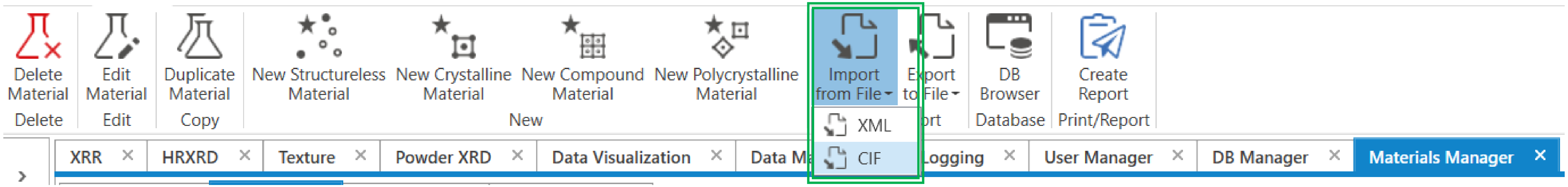
15. Click on the ***CIF*** link and download the file into your ***CIF Folder***

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

Materials Manager ✕

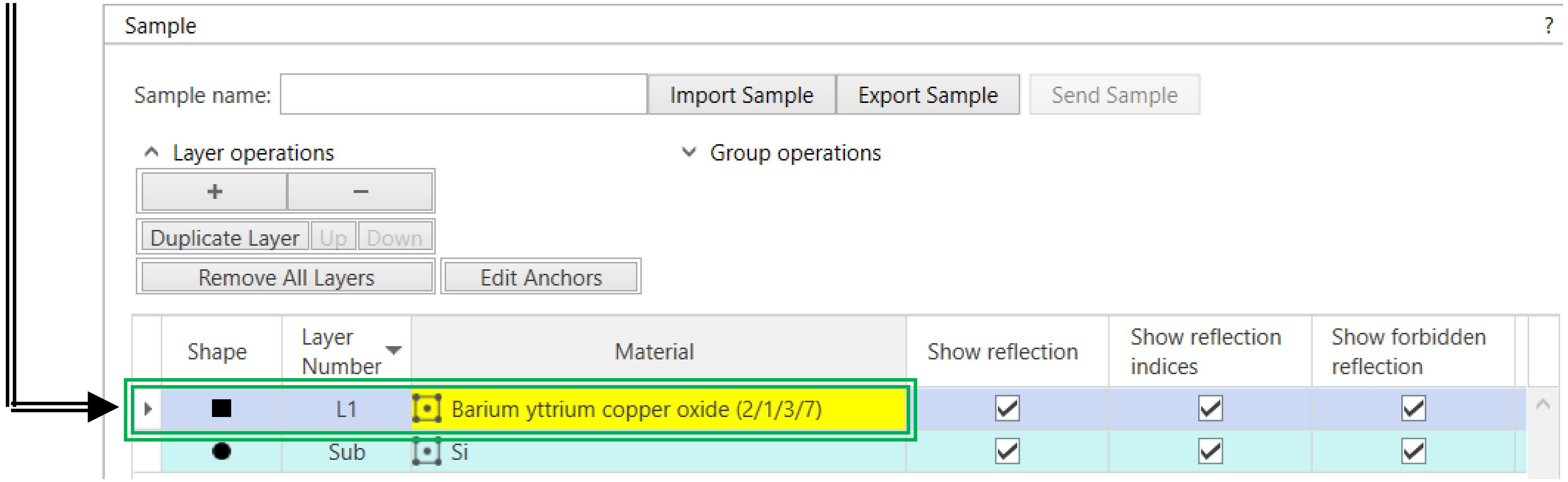
V. RS Viewer – 4/4

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



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

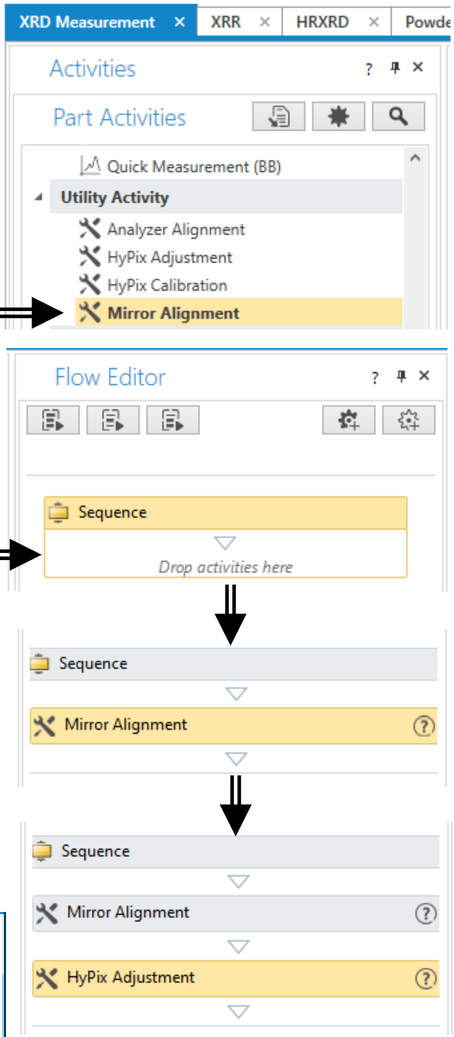
19. The sample information should now be available for you in the ***Material*** selection in ***RS Viewer***

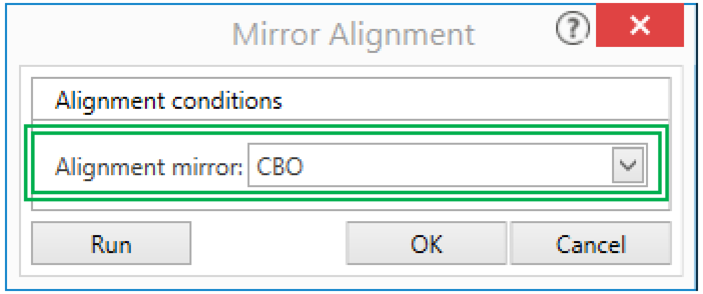


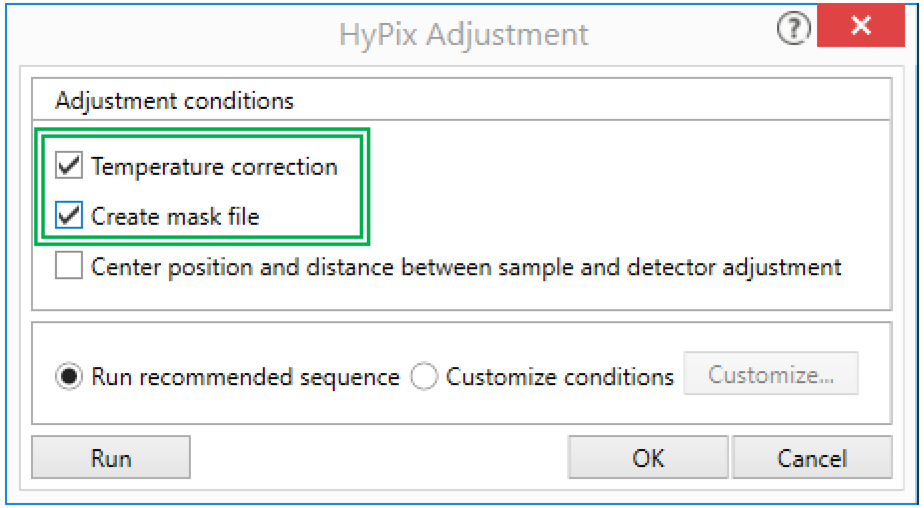
VI. Utility Activity – 1/2

- This sequence will perform a Mirror Alignment and HyPix Adjustment **BEFORE** and **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***







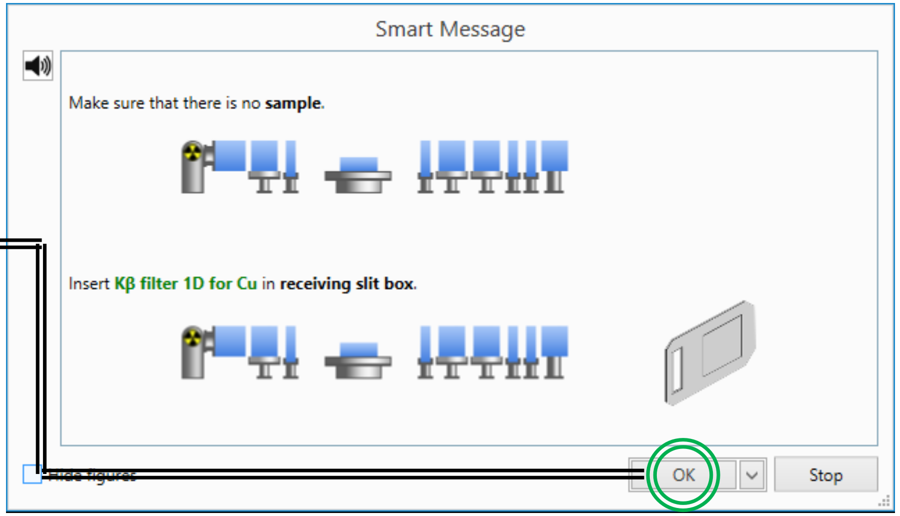
VI. Utility Activity – 2/2

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

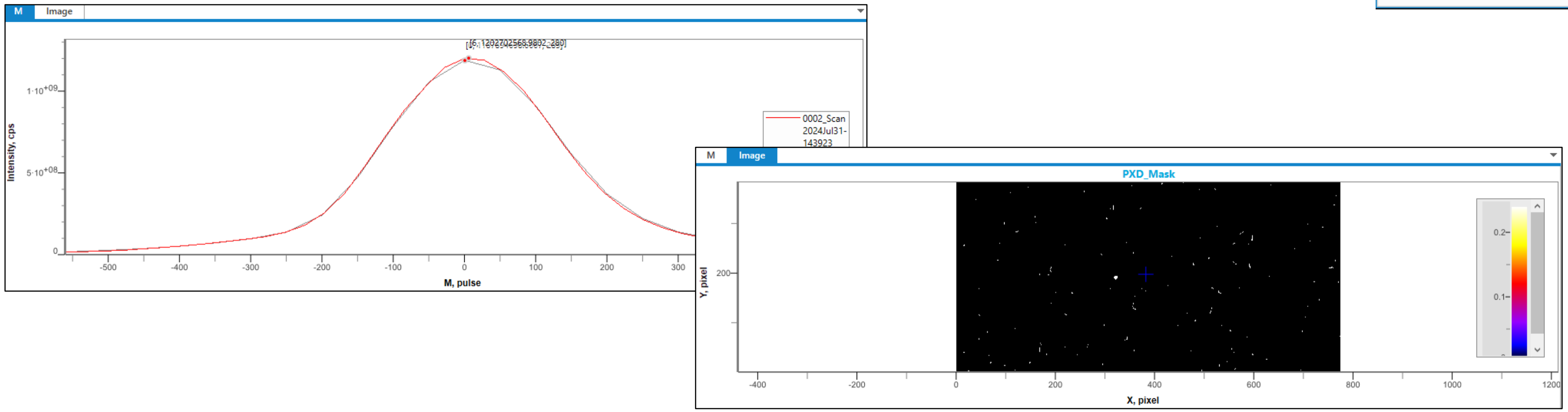
7. A ***Smart Message*** will appear indicating all the optics and attachments that need to be ***removed*** (indicated in **RED**) and those that need to be ***installed*** (indicated in **GREEN**)

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

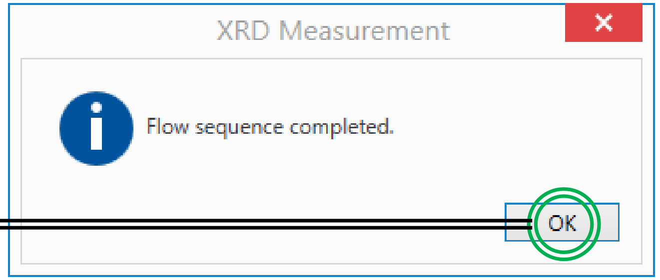
   Note: Your ***Smart Message*** may differ from example shown



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



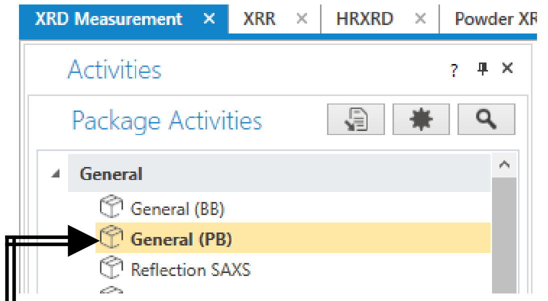
10. Wait for the ***Flow sequence completed*** prompt to continue by clicking ***OK***

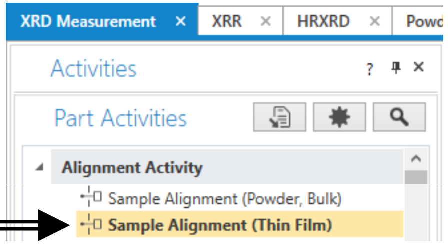


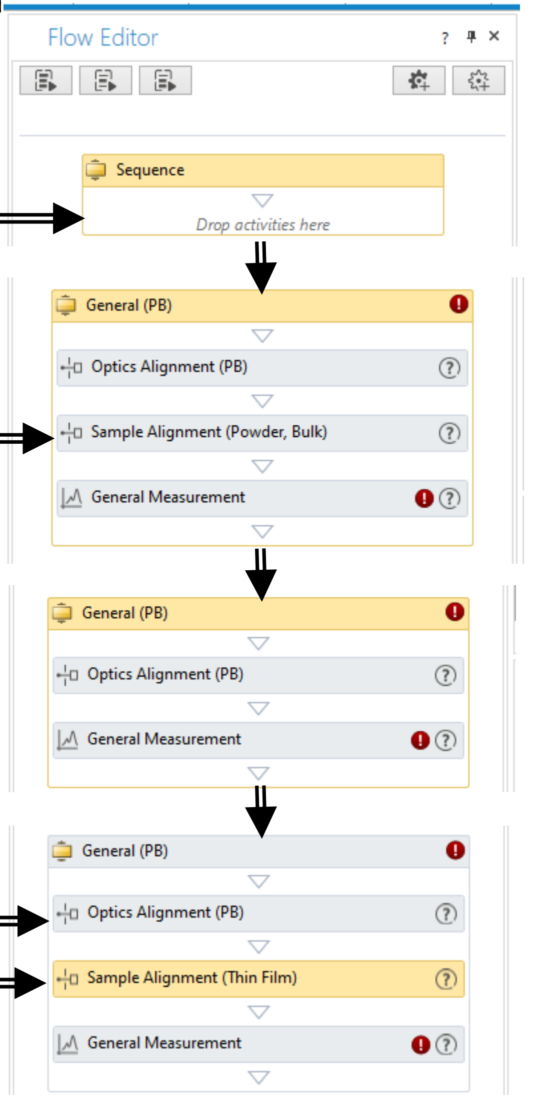
VII. General (PB) or 2θ/ω Scan – 1/7

- This sequence will perform a General 2θ/ω 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





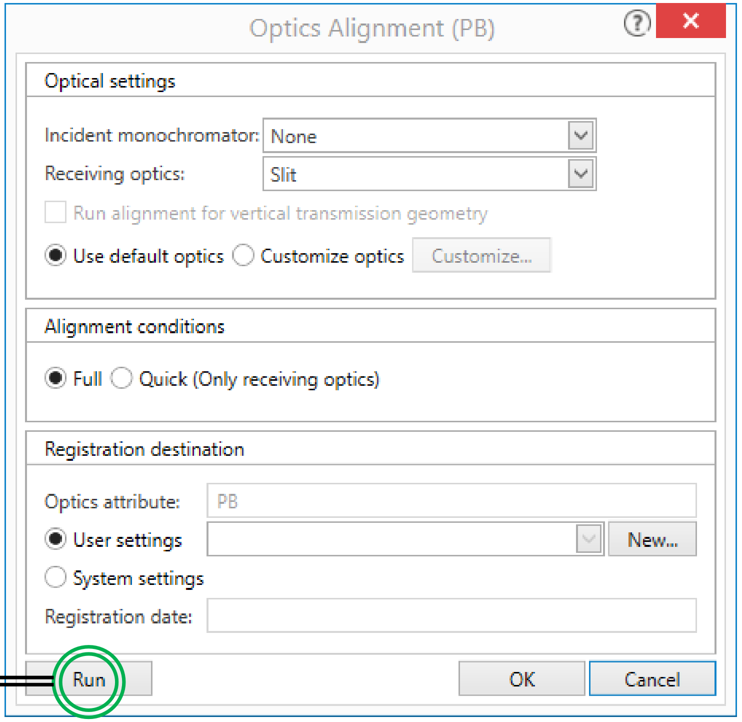


VII. General (PB) or 2θ/ω Scan – 2/7

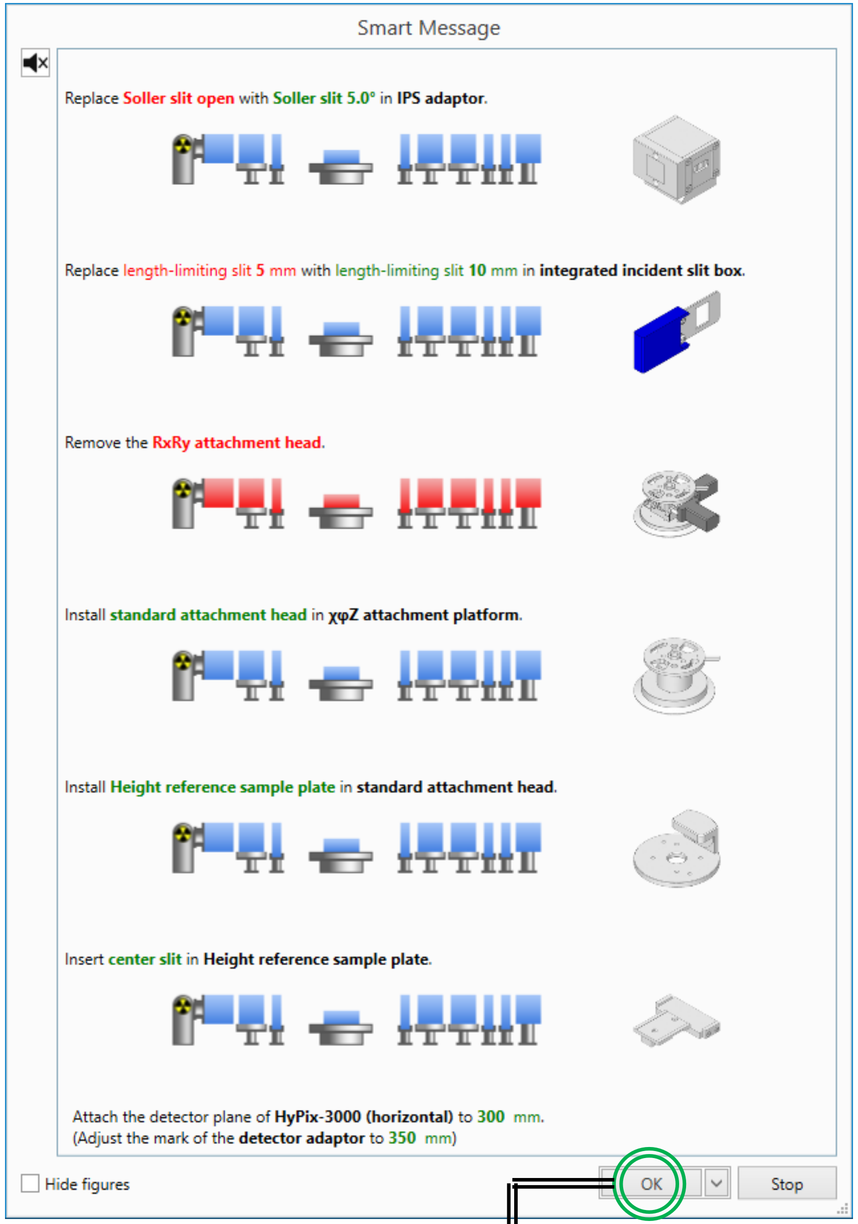
7. Confirm ***Use default optics*** is selected under ***Optical settings***

8. Confirm ***Full*** is selected under ***Alignment conditions***

9. Confirm ***User settings*** is selected, then click ***Run***



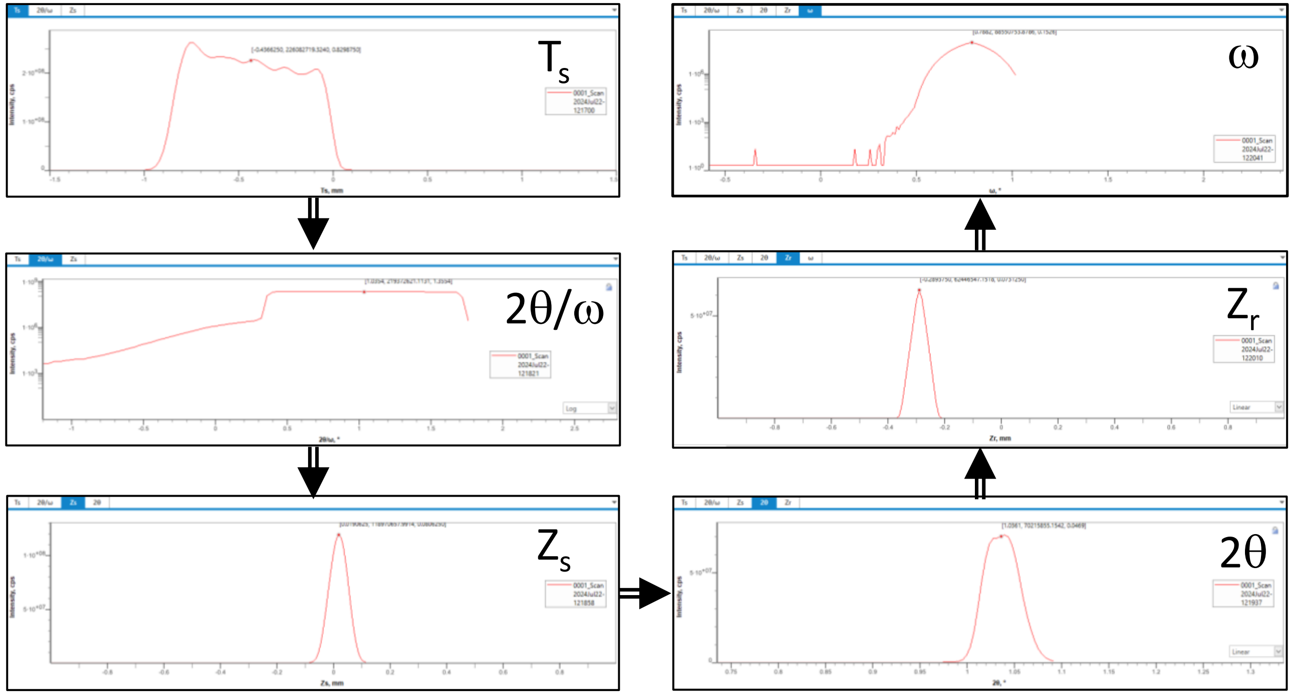
10. A ***Smart Message*** may appear indicating all the optics and attachments that need to be ***removed*** (indicated in **RED**) and those that need to be ***installed*** (indicated in **GREEN**)



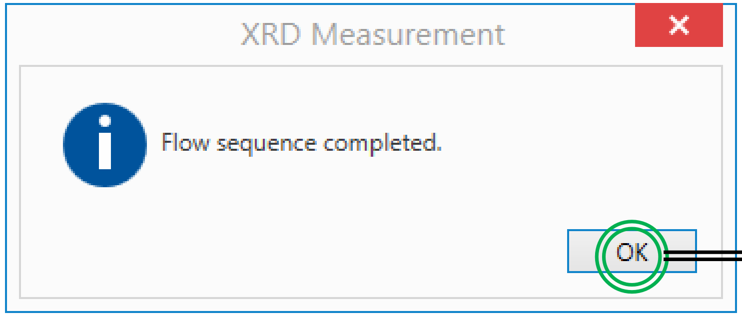
11. Click ***OK*** when completed

VII. General (PB) or 2θ/ω Scan – 3/7

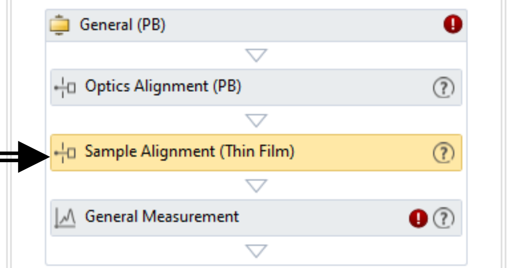
12. System will perform ***Optics Alignment*** on various axes (Average time ≈ 4 minutes)



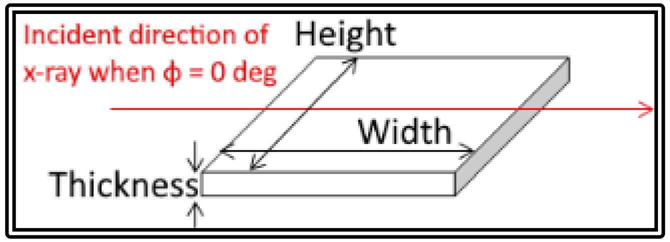
13. Wait for the ***Flow sequence completed*** prompt to continue by clicking ***OK***

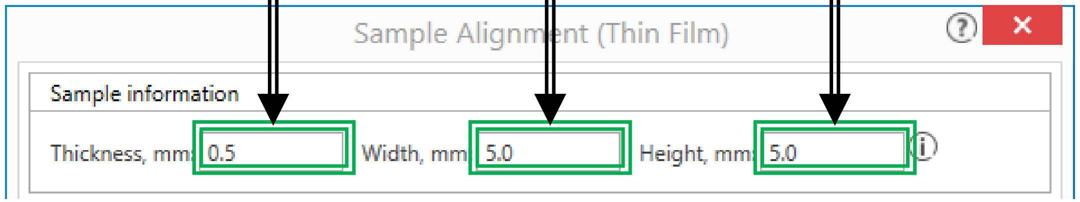


14. Left-click on ***Sample Alignment (Thin Film)*** to set ***Sample Info***



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



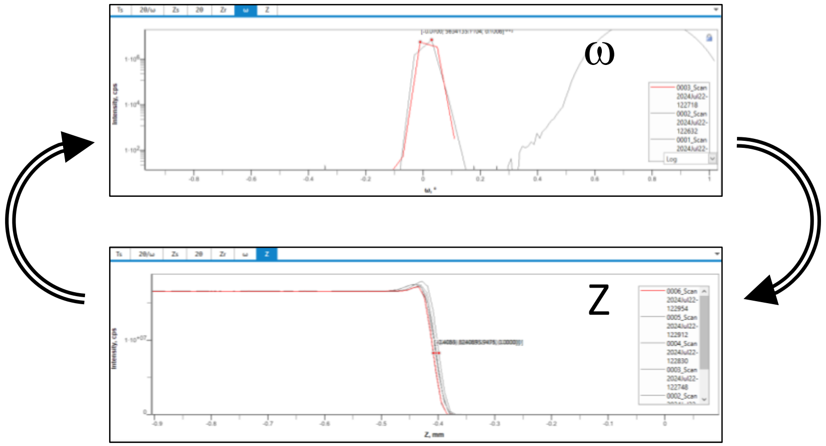


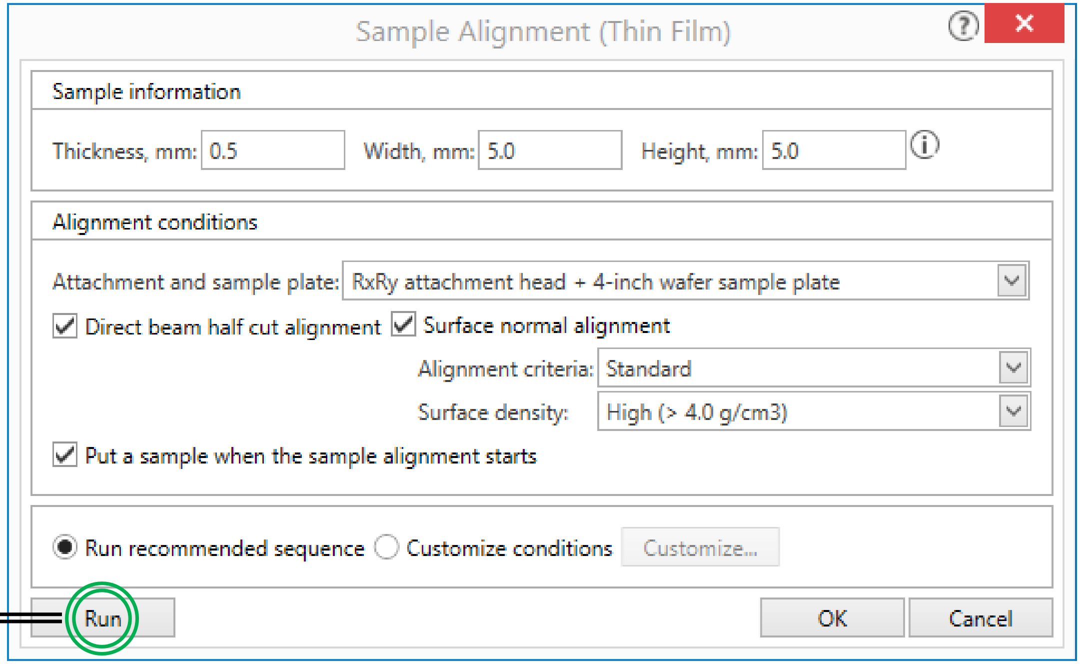
- For training with Silicon: Thickness = 0.5 mm; Width & Height = 5 mm

VII. General (PB) or 2θ/ω 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

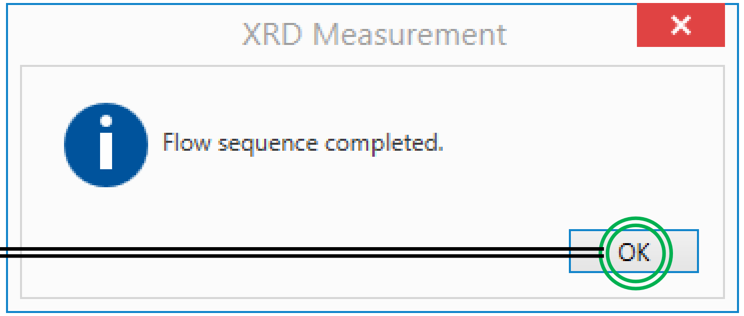
17. Click ***Run*** (Average time ≈ 6 minutes)





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

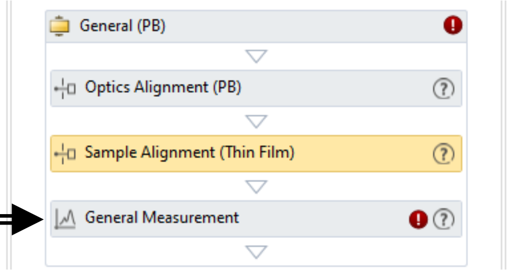
- For training with Silicon: Surface density should be set to High

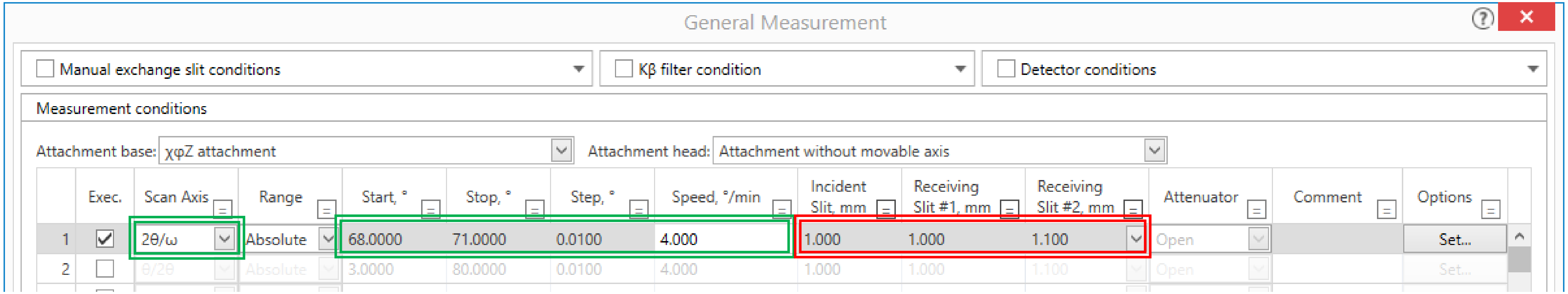


19. Wait for the ***Flow sequence completed*** prompt to continue by clicking ***OK***

VII. General (PB) or 2θ/ω Scan – 5/7

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





21. Select **2θ/ω** for the ***Scan Axis***

22. Adjust the following parameters based on your desired scan conditions
    - **Start, °**: Enter starting scan position for 2θ angle (e.g. 68°)
    - **Stop, °**: Enter ending scan position for 2θ angle (e.g. 71°)
    - **Step, °**: Enter scan step size for 2θ angle (e.g. 0.01°) – controls resolution or spacing of data points
    - **Speed, °/min**: Enter the scan speed (e.g. 4°/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

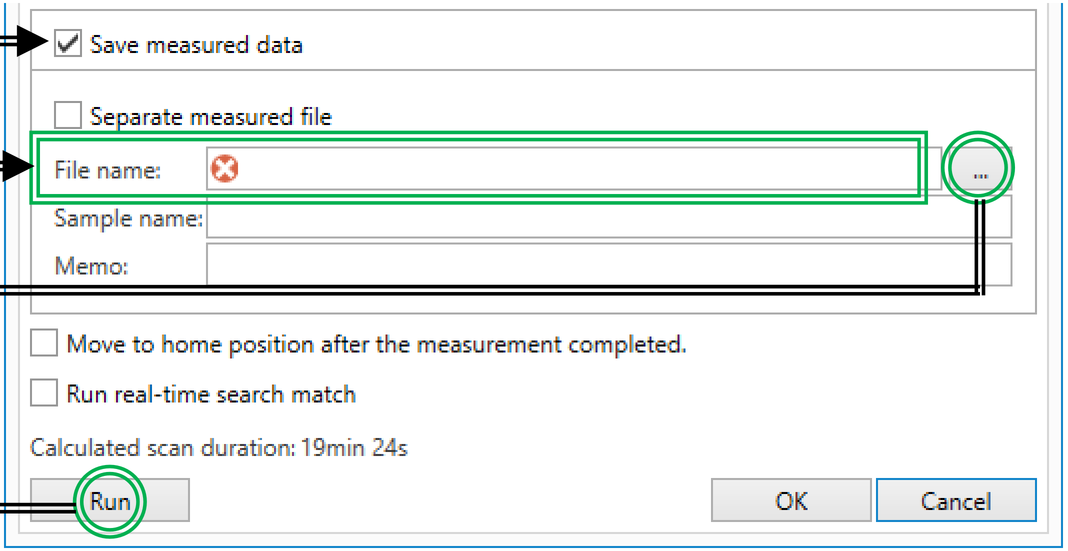
    } Default values are automatically chosen based on sample dimensions

VII. General (PB) or 2θ/ω Scan – 6/7

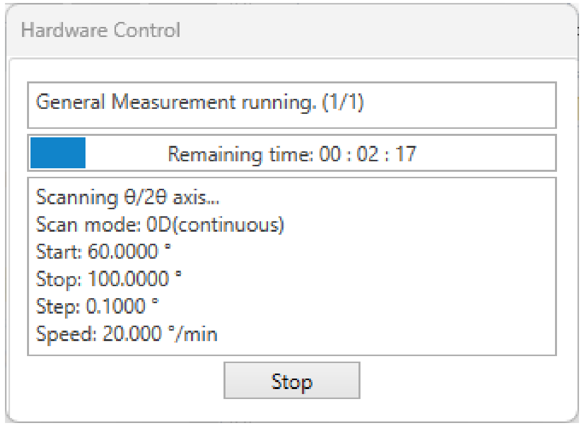
24. If you wish to save your data, click on the ***Save measured data*** box (Note: You can always save your data after collection!)

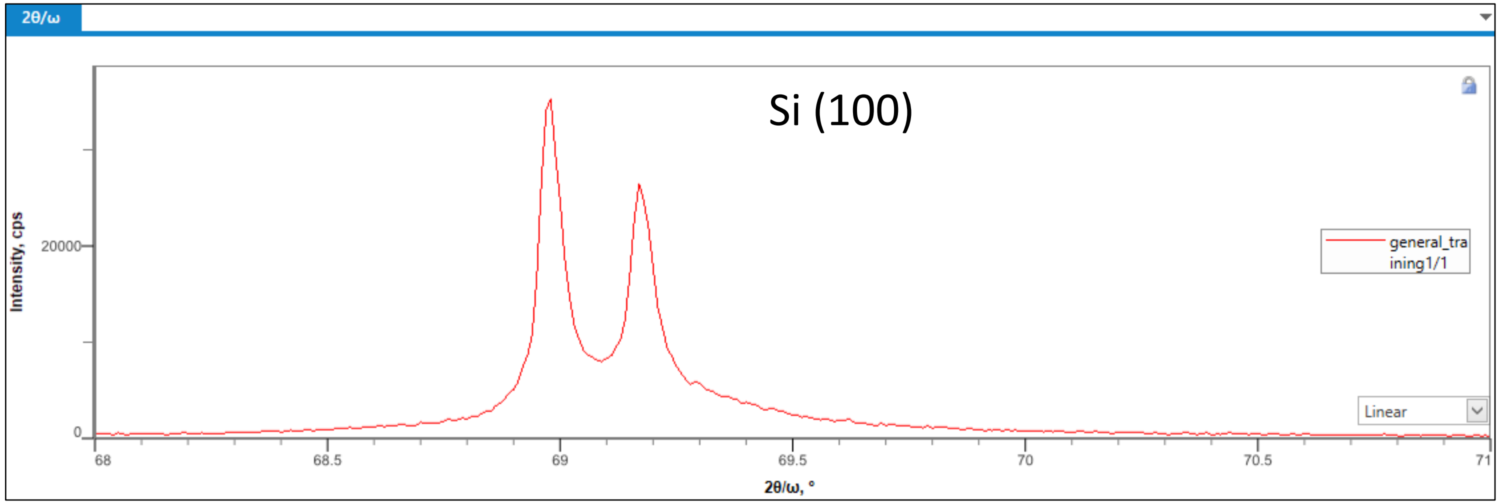
25. Input your desired ***File name*** and ***File location*** here

26. Click ***Run***

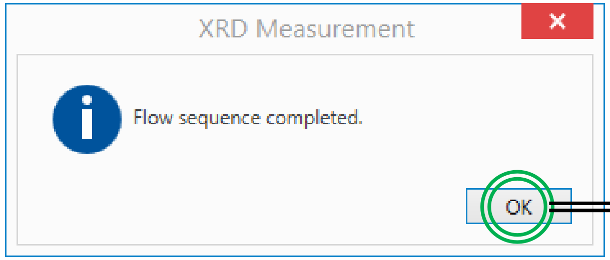


27. The ***Remaining time*** left in the scan will be displayed





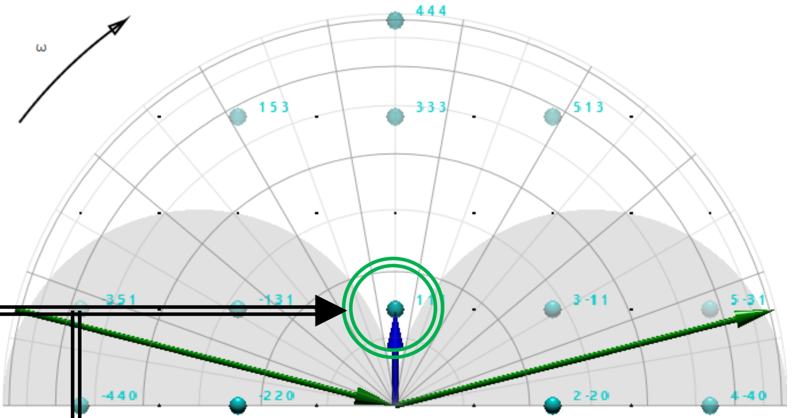
28. Click ***OK*** when complete



VII. General (PB) or 2θ/ω Scan – 7/7

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

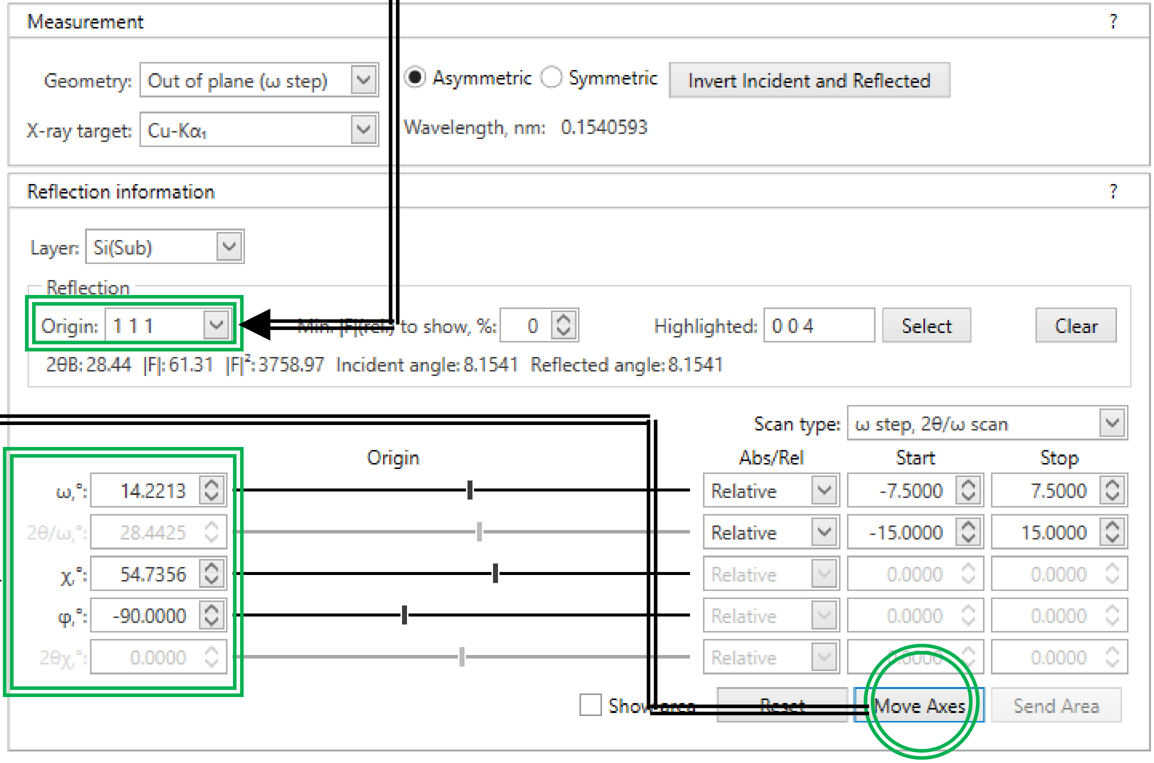
24. Open the ***RS Viewer*** and find the Si **(111)** plane for diffraction



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?

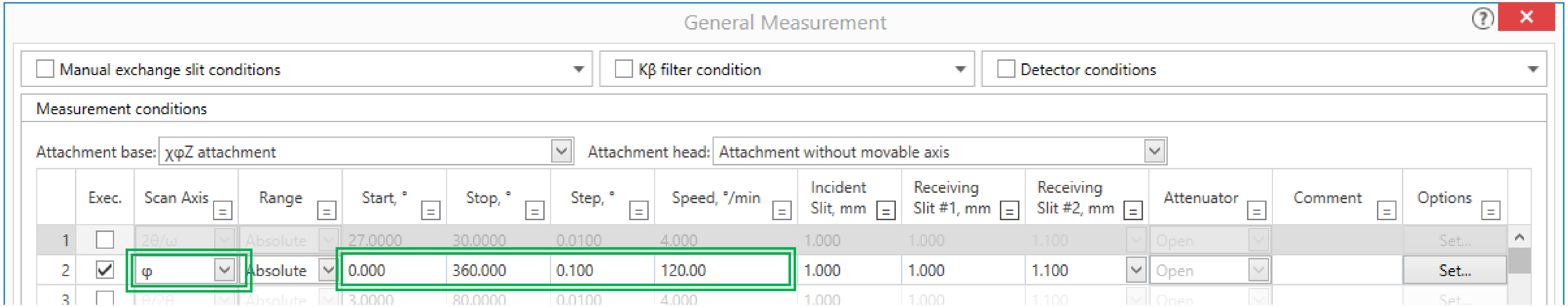


31. If the **(111)** peak is not observed → sample orientation is mismatched with ***RS Viewer*** via the φ position

VIII. Azimuth or φ Scan – 1/3

○ This sequence will perform an Azimuth or φ Scan

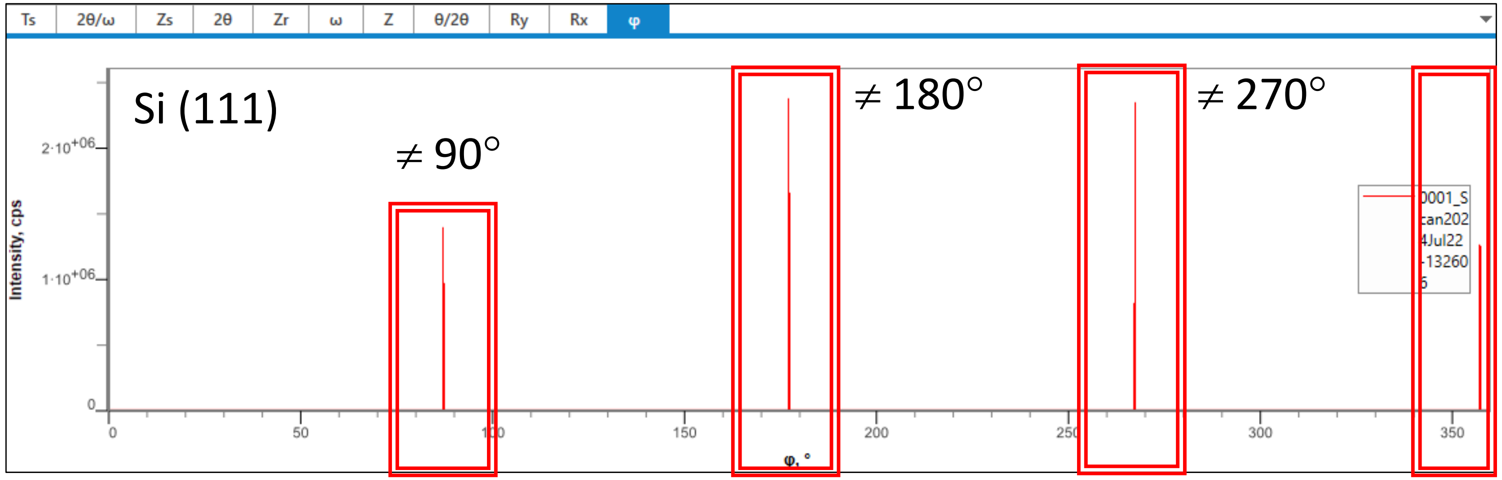
1. Left-click on ***General Measurement***



2. Select φ for the ***Scan Axis*** for ***#2***

3. Set ***Start* = 0°** and ***Stop* = 360°**
***Step* = 0.1°** and ***Speed* = 120°/min**

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

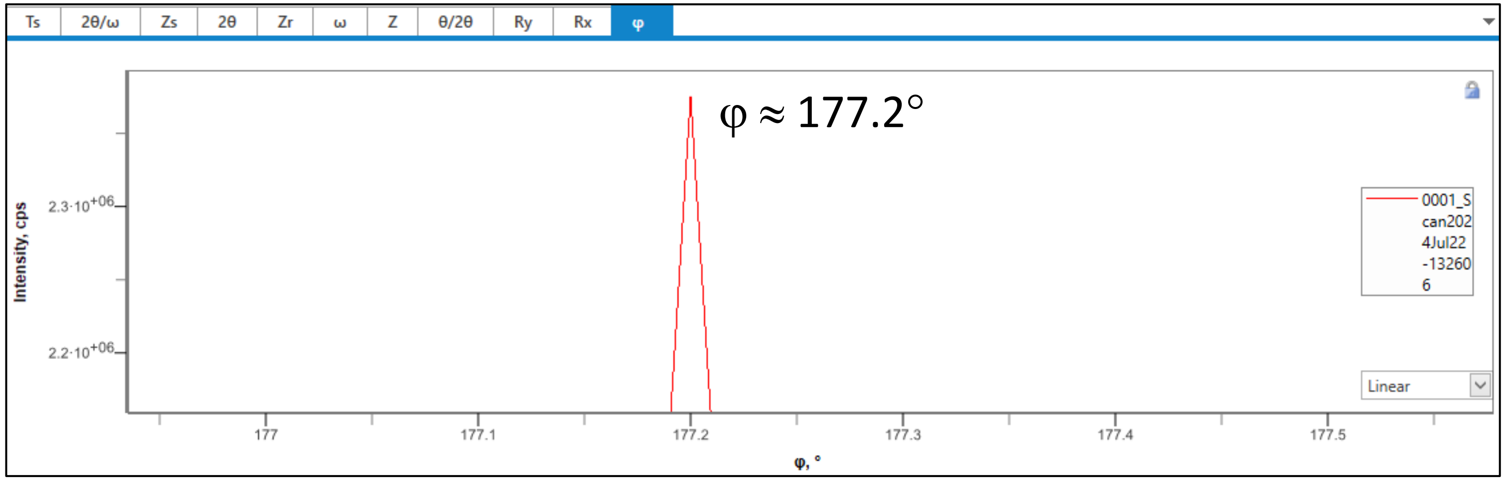


≠ 0 or 360°

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

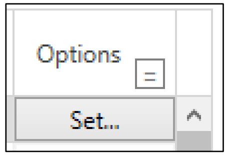
VIII. Azimuth or φ Scan – 2/3

6. Zoom-in one of the peaks…

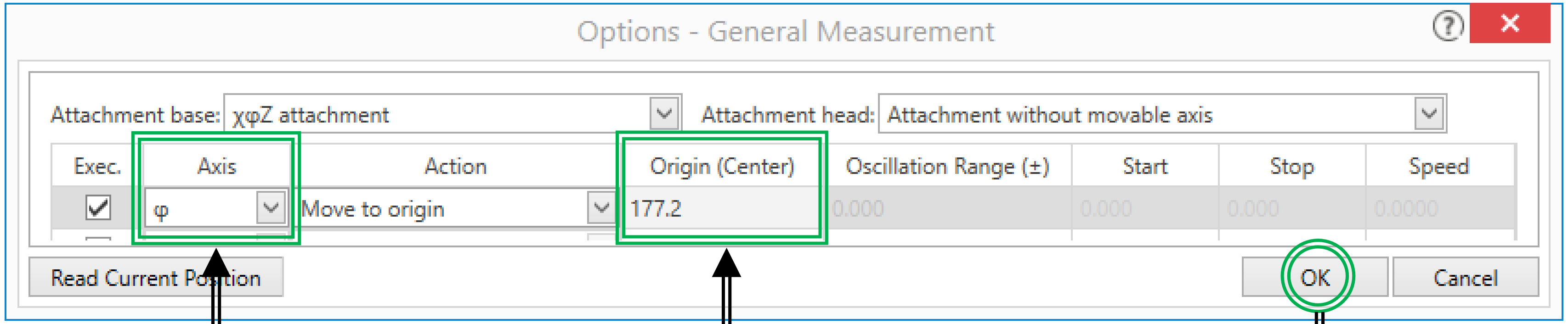


7. Left-click on ***General Measurement*** again

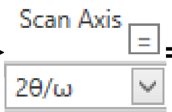
8. Click on ***Set…*** under ***Options***



9. Set φ to the value of your peak position, and click ***OK***



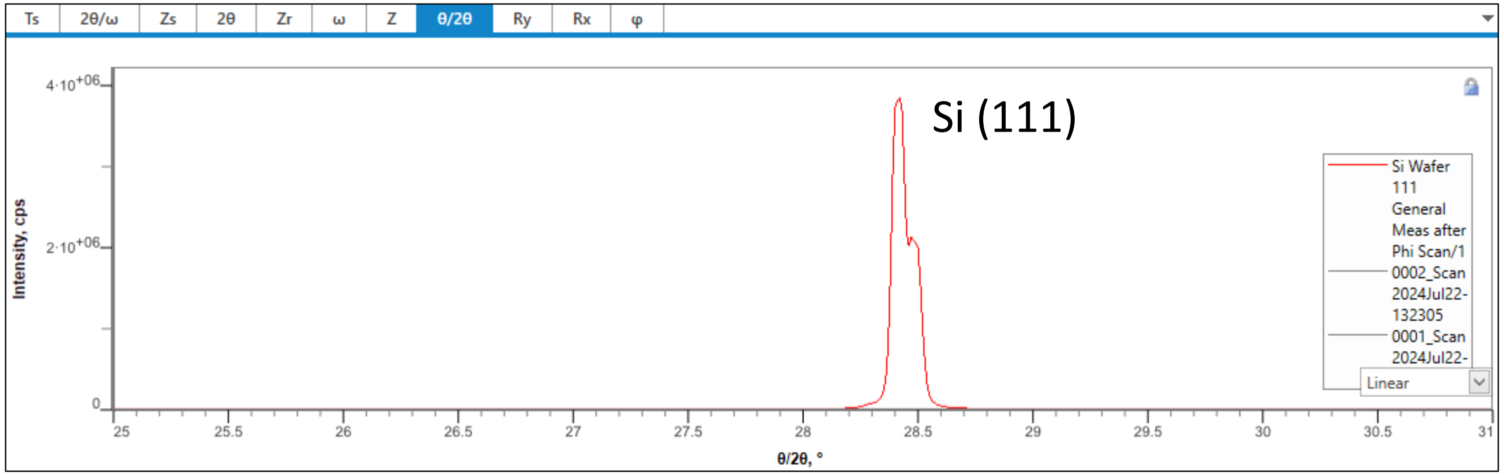
10. Set ***Scan Axes*** back to **2θ/ω** and input ***Start*** and ***Stop*** values back to **27°** and **30°**



11. Click ***Run*** again

VIII. Azimuth or φ Scan – 3/3

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



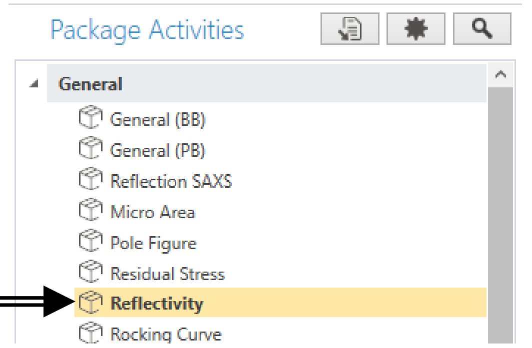
13. Some measurements may perform ***"Pre-Measurements"*** that will identify and set the goniometer settings (e.g. **2θ**, ω, φ, θ_χ, etc…) for your scans, but may not always succeed or be available

14. It is still up to the user to be comfortable controlling and moving the ***Axes*** and setting the ***Origin*** when appropriate

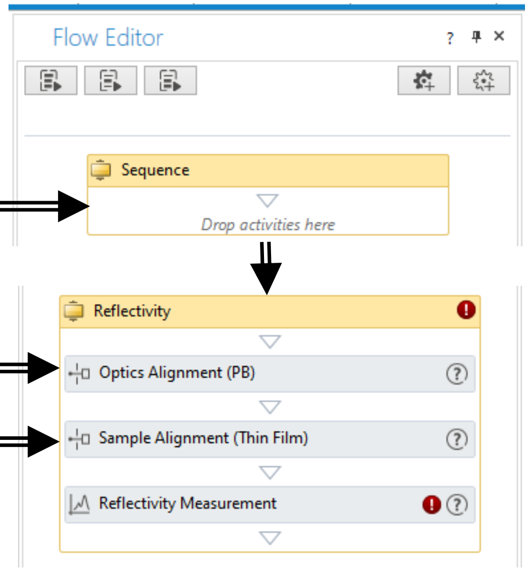
IX. Reflectivity – 1/4

- 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***

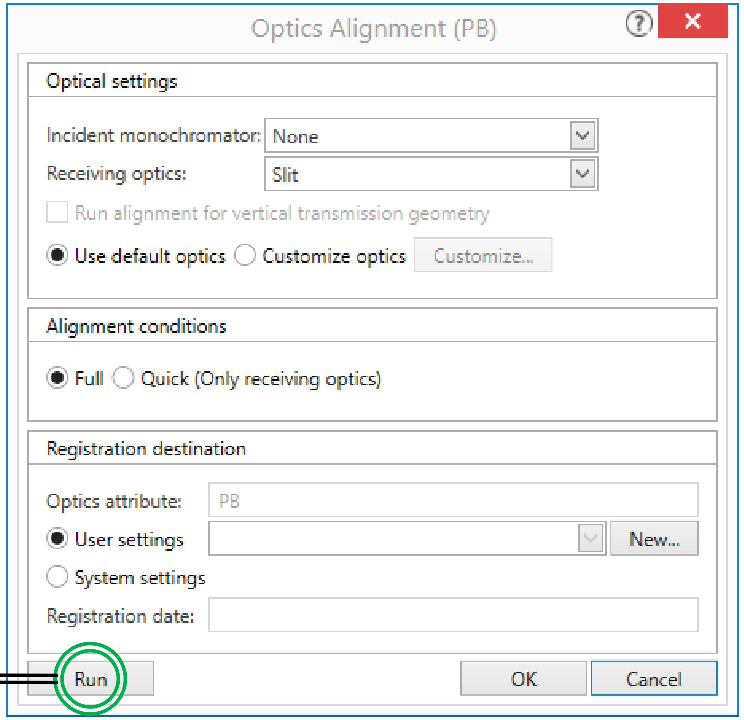


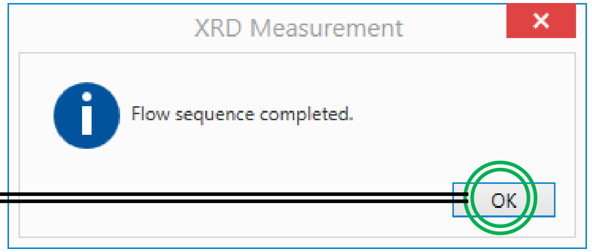
3. If ***Optics Alignment (PB)*** and ***Sample Alignment (Thin Film)*** were previously performed, then skip to ***Step 11***

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***

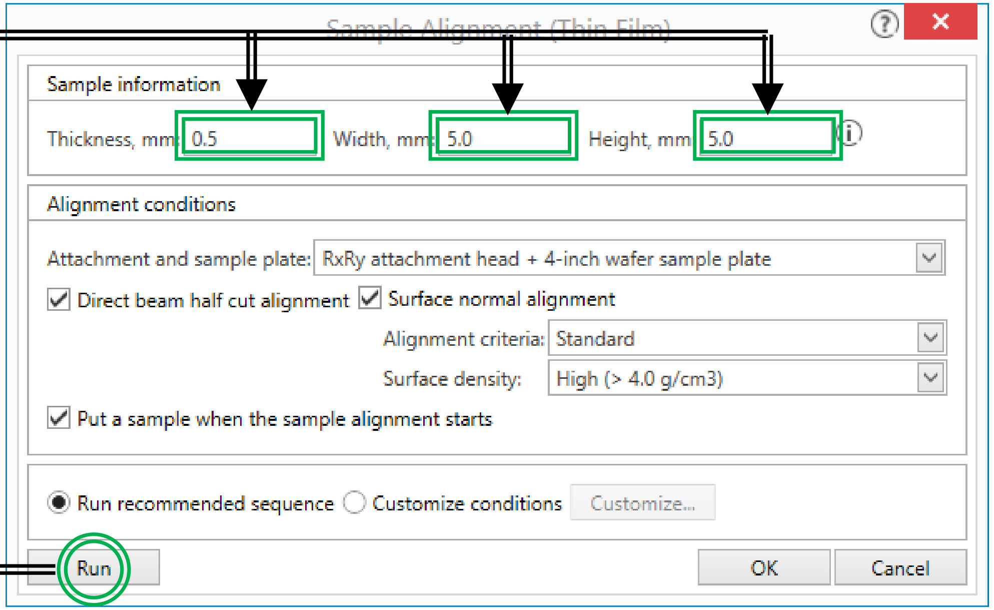
6. Click ***Run*** and then ***OK*** when completed



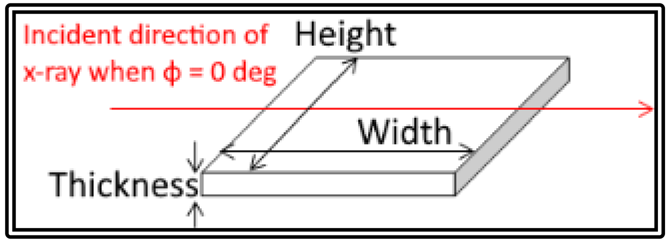


IX. Reflectivity – 2/4

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

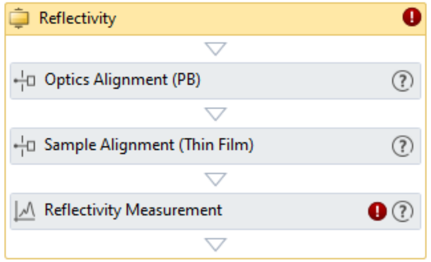


8. Input your ***Sample Info*** per the dimensions



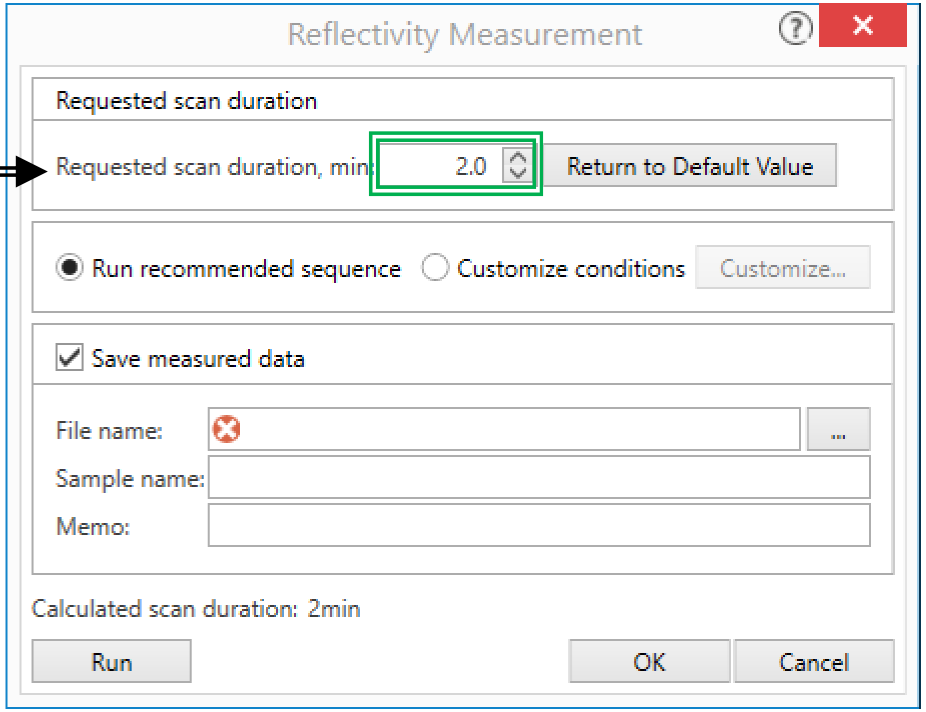
9. Confirm the following are checked:
   - Direct beam half cut alignment
   - Surface normal alignment
   - Put a sample when the sample alignment starts

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

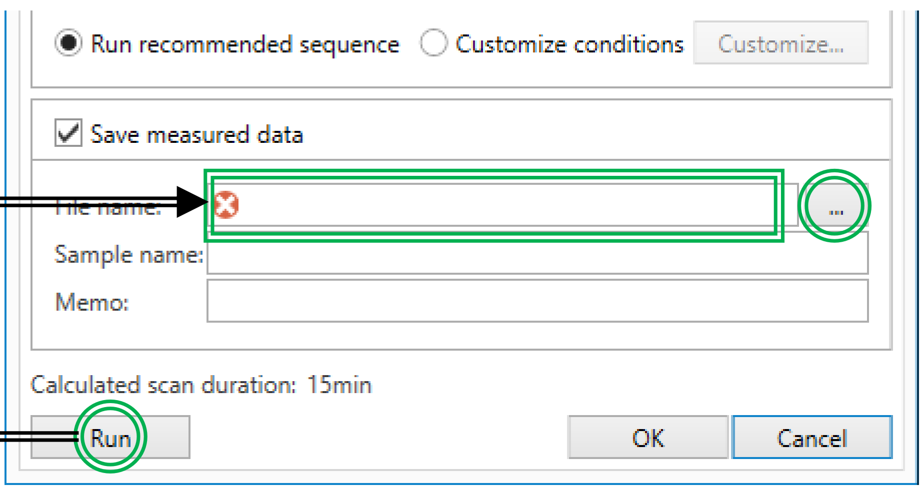
IX. Reflectivity – 3/4

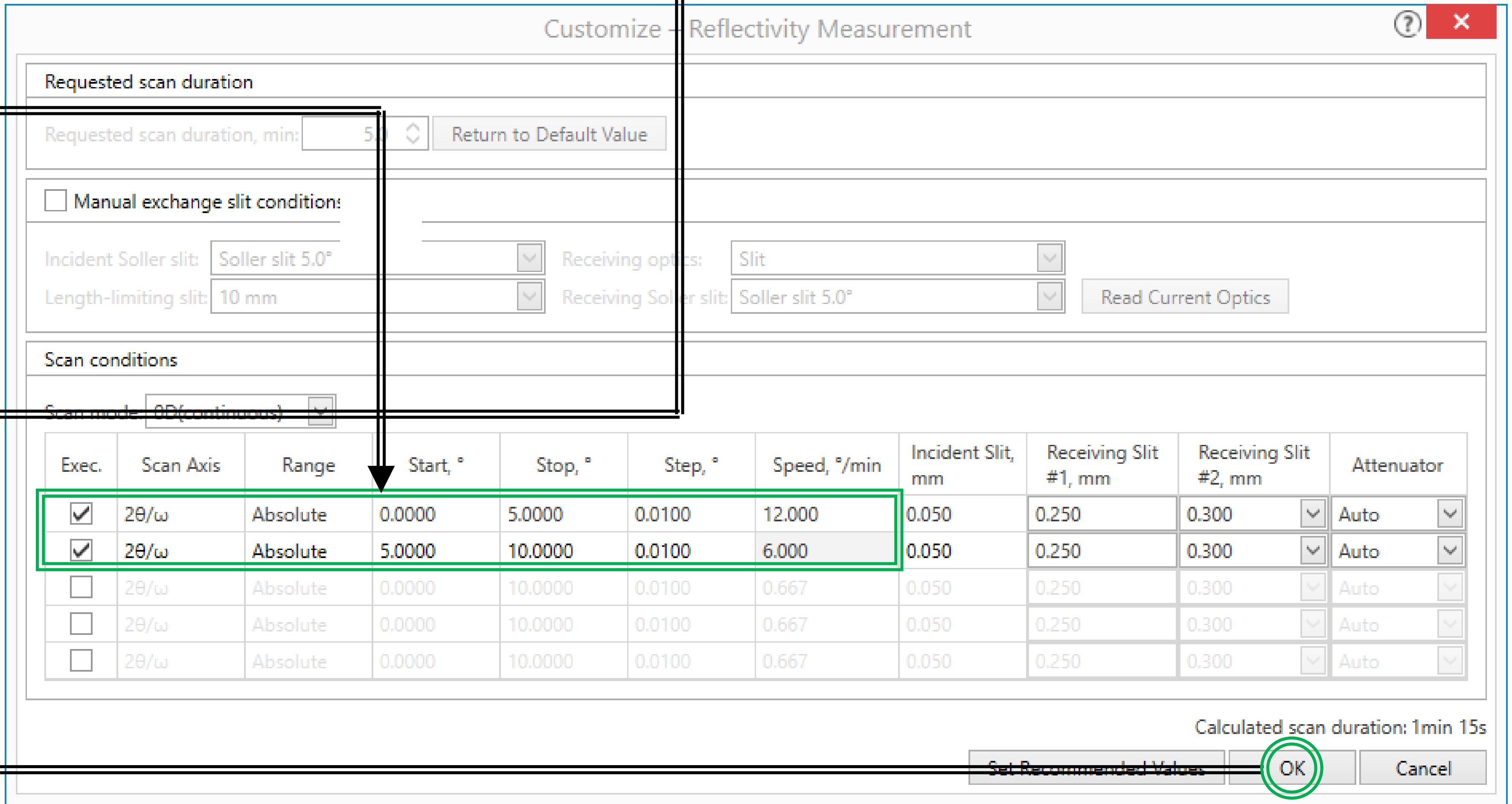
13. Input your desired ***File name*** and ***File location*** here

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

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

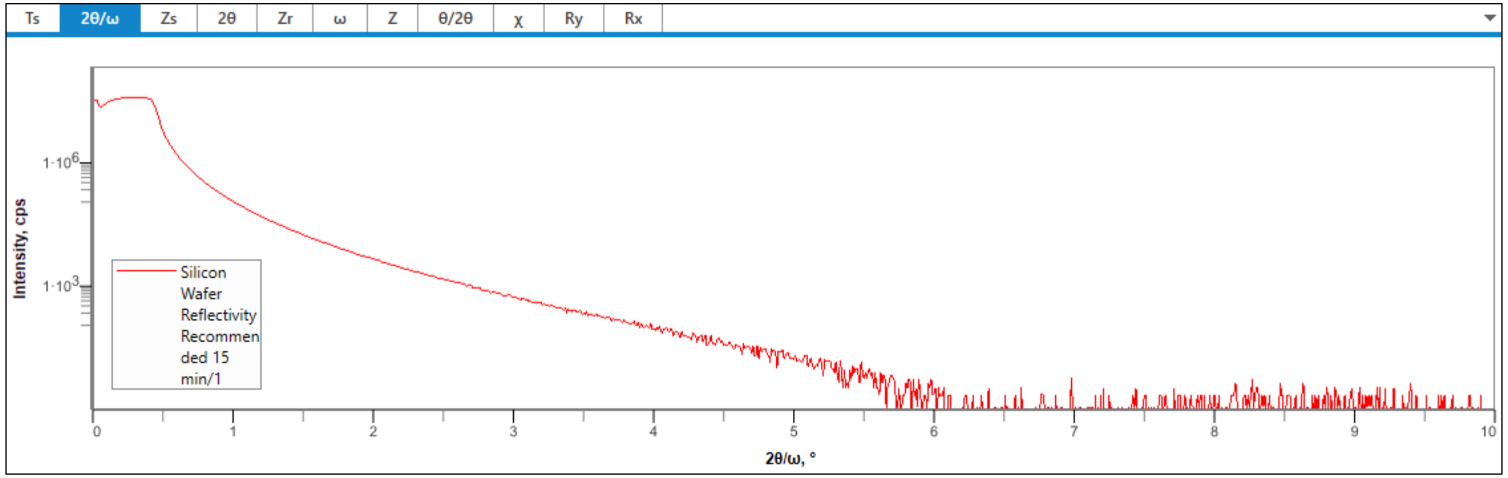
13. Click ***OK*** then click ***Run***



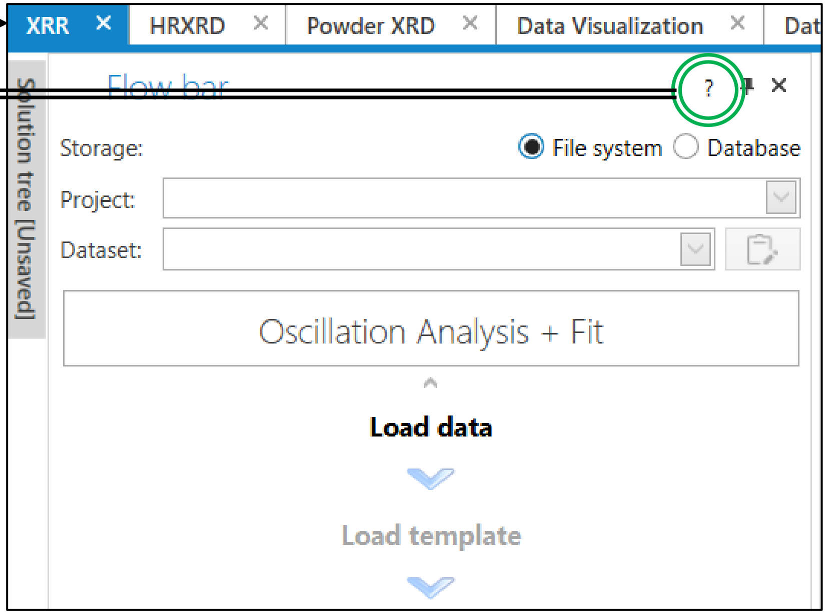


IX. Reflectivity – 4/4

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



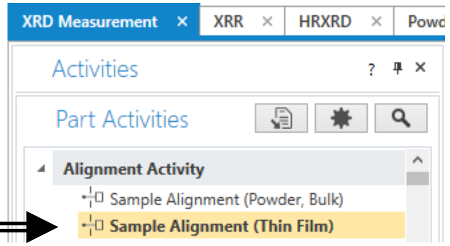
18. Click on the **?** icon
to open up the ***XRR_UserManual_en.pdf*** to review the tutorial

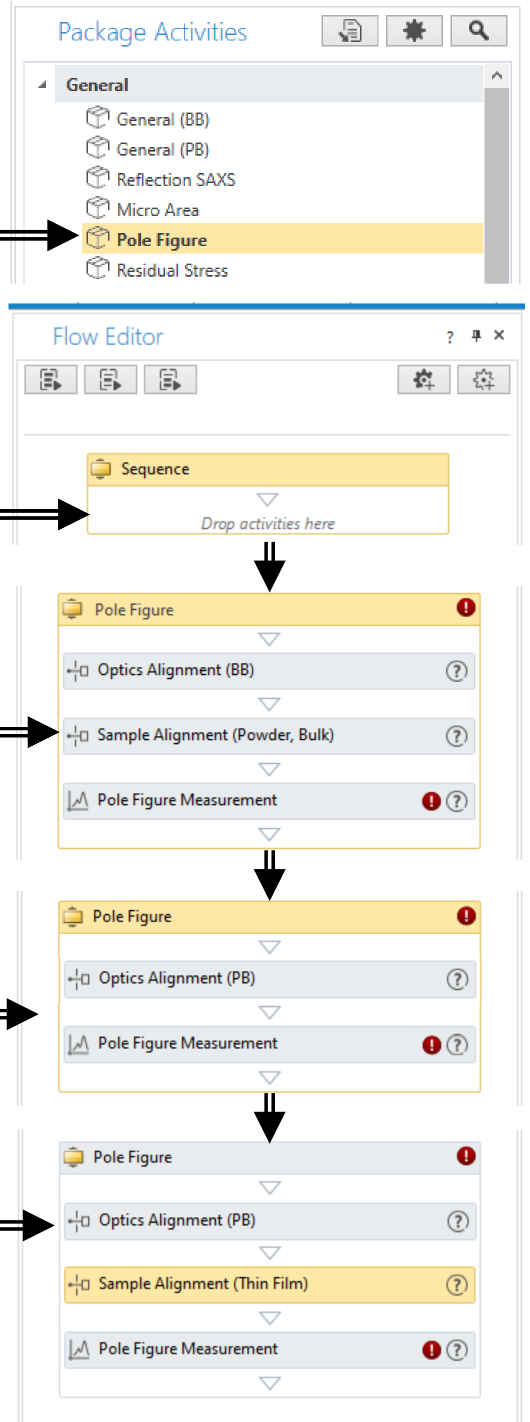


X. Pole Figure – 1/5

- 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)***

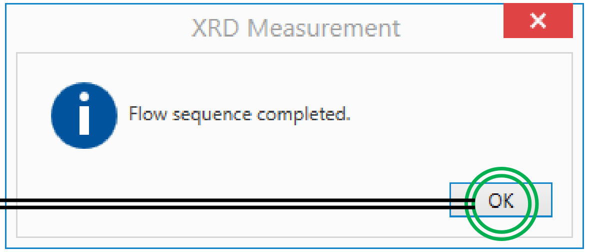


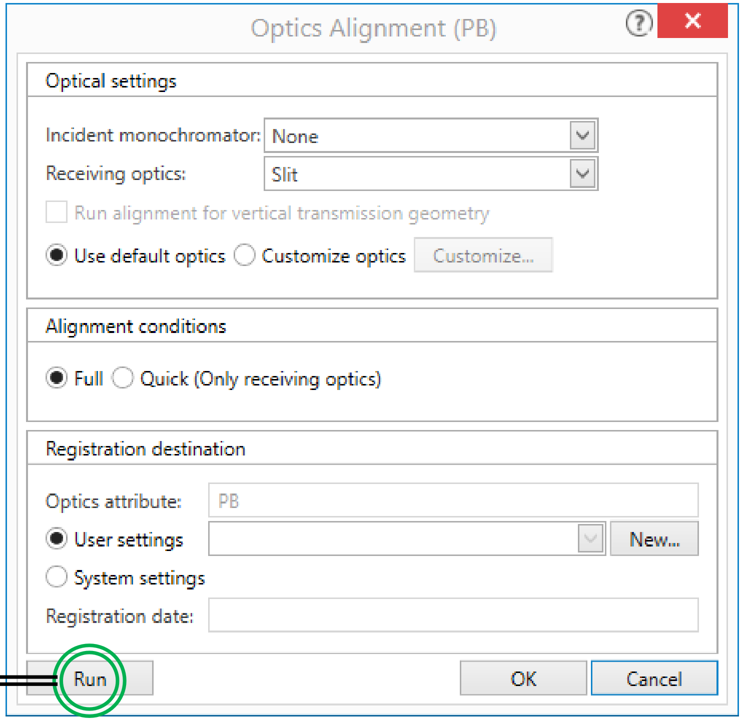


X. Pole Figure – 2/5

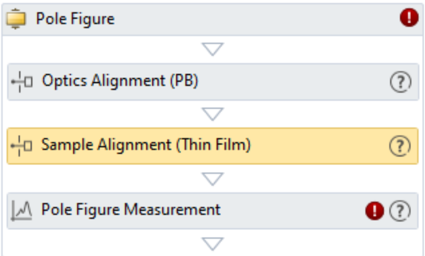
8. Confirm the following are selected:
   - ***Optical settings → Use default optics***
   - ***Alignment conditions → Full***
   - ***Registration destination → User settings***

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

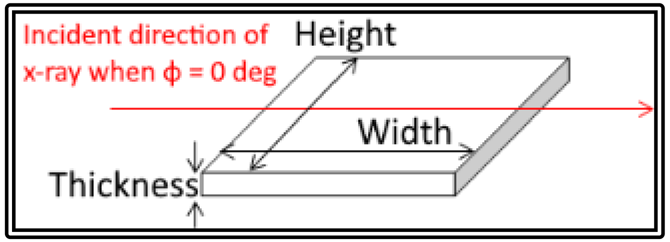


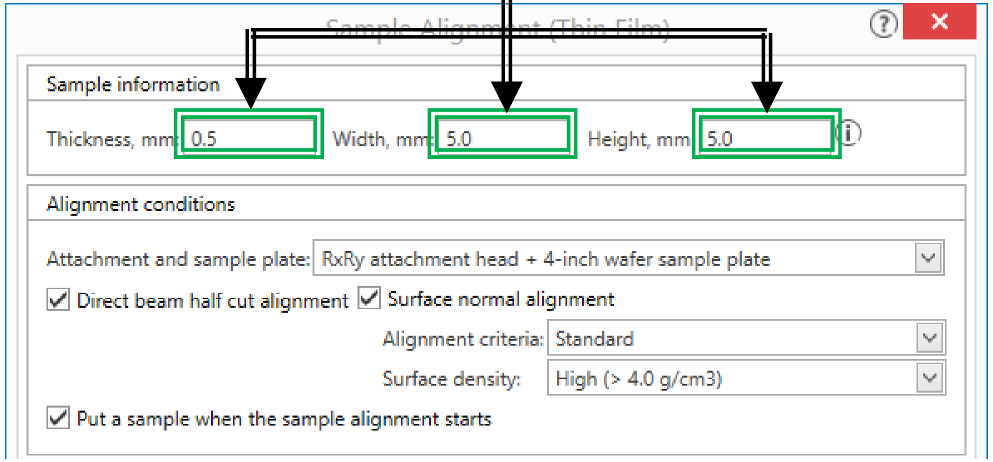


10. Left-click on ***Sample Alignment (Thin Film)***



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

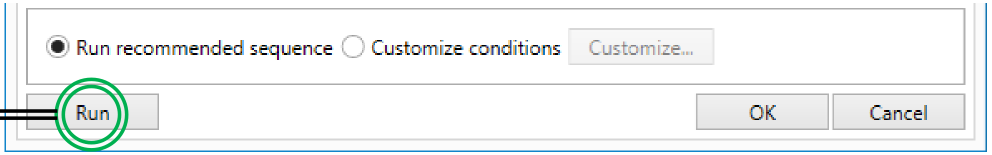


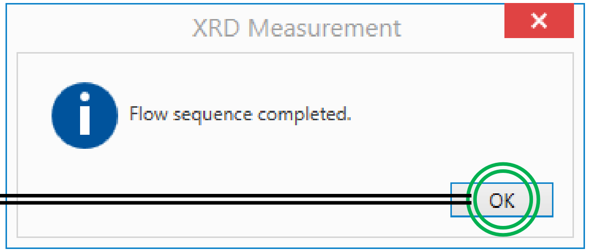


12. Confirm the following are checked:
   - Direct beam half cut alignment
   - Surface normal alignment
   - Put a sample when the sample alignment starts

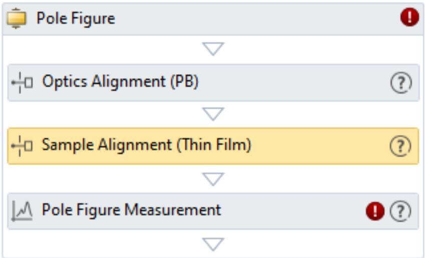
X. Pole Figure – 3/5

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

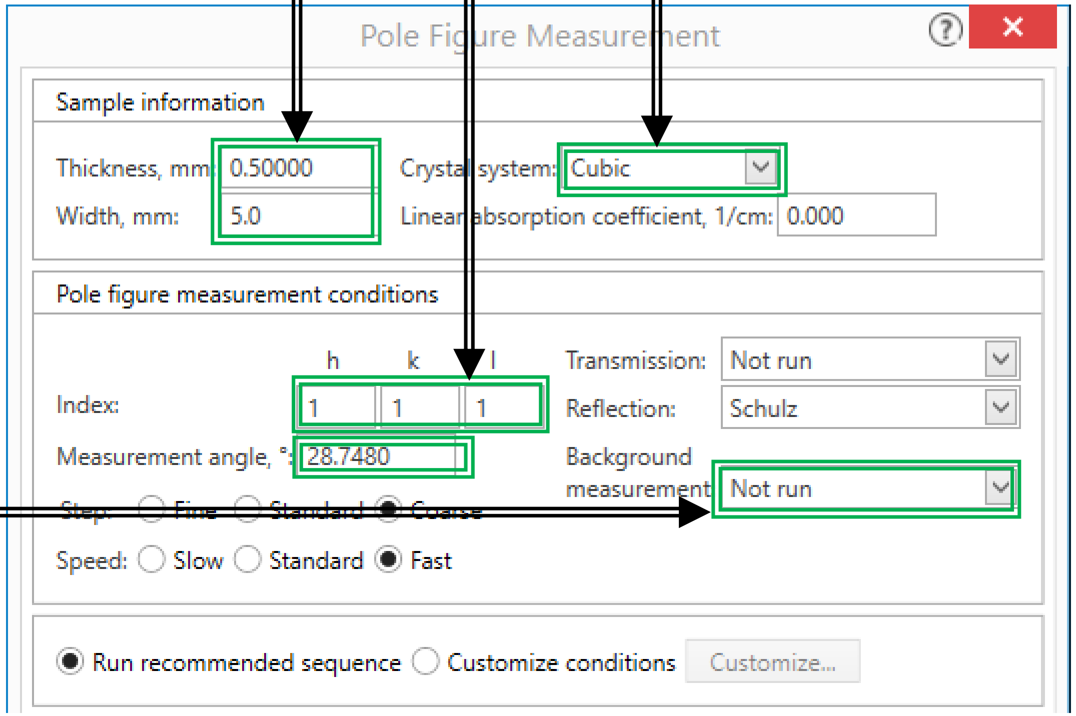




14. Left-click on ***Pole Figure Measurement***



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***

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 α scan axes!
    - ***Start***
    - ***Stop***
    - ***Step***
    - ***Speed***

Data measurement conditions

2θ angle, ° (Transmission): 28.4664 | 2θ angle, ° (Reflection): 28.4664 | χ 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	α	0D(step)	Absolute	0	15	5	0.1 mm	10.000	9.900	Open
Reflection	α	0D(step)	Absolute	15.264	55.264	5.000	1/6°	10.000	9.900	Open

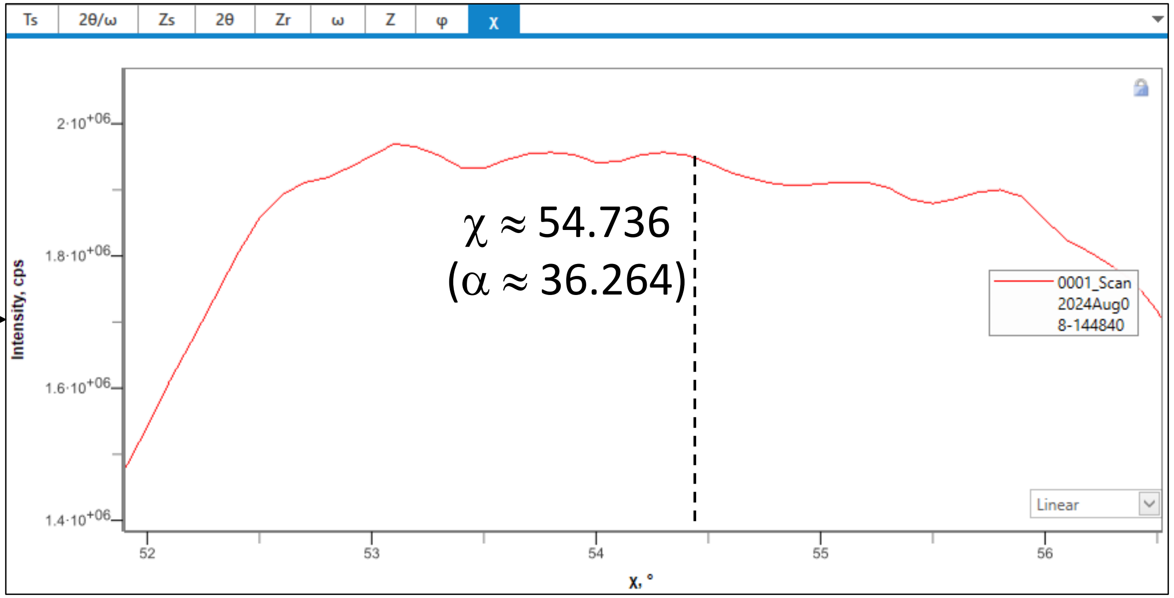
Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min
β	0D(continuous)	Absolute	0.000	360.000	5.000	200.00

Remember that $\alpha = 90° - \chi$

(Note: Will the peaks appear for α if arbitrarily chosen?)

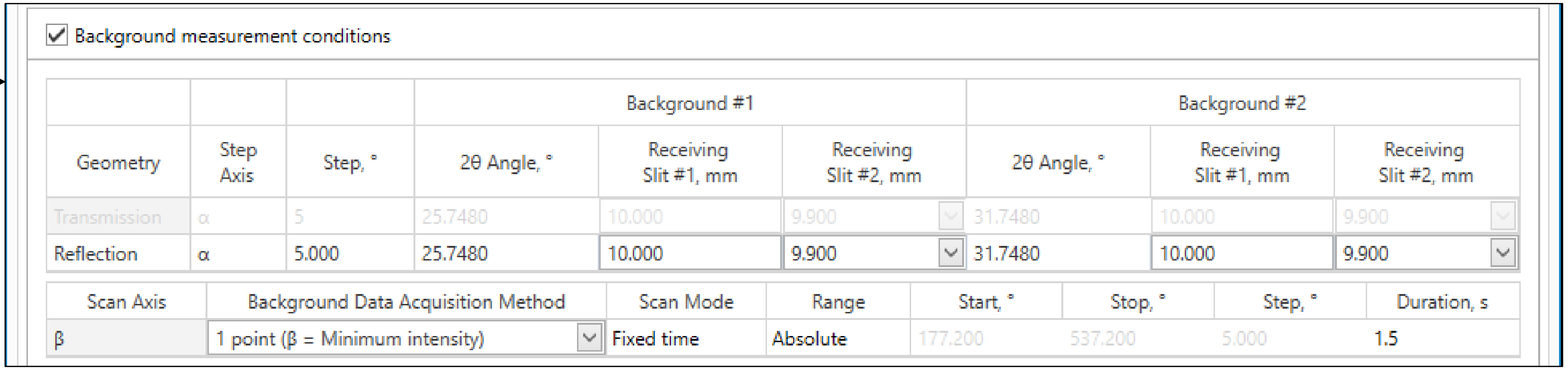
19. If not performed, identify the optimal χ tilt for your peak

	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min
1	☑	χ	Absolute	50.000	60.000	0.100	15



X. Pole Figure – 5/5

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



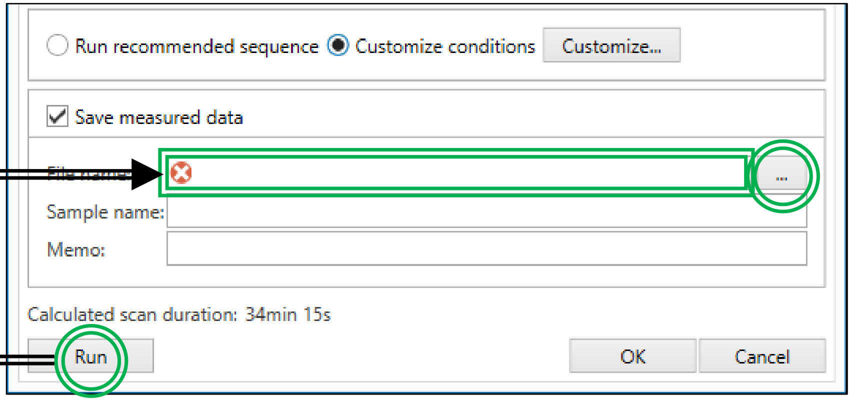
☑ Background measurement conditions

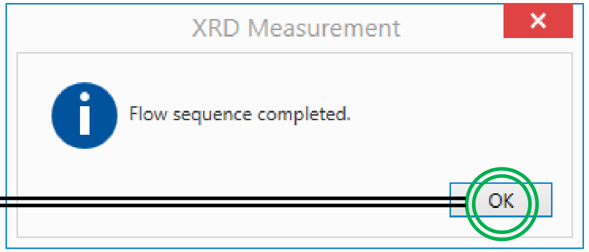
Geometry	Step Axis	Step, °	Background #1 2θ Angle, °	Background #1 Receiving Slit #1, mm	Background #1 Receiving Slit #2, mm	Background #2 2θ Angle, °	Background #2 Receiving Slit #1, mm	Background #2 Receiving Slit #2, mm
Transmission	α	5	25.7480	10.000	9.900	31.7480	10.000	9.900
Reflection	α	5.000	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
β	1 point (β = Minimum intensity)	Fixed time	Absolute	177.200	537.200	5.000	1.5

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

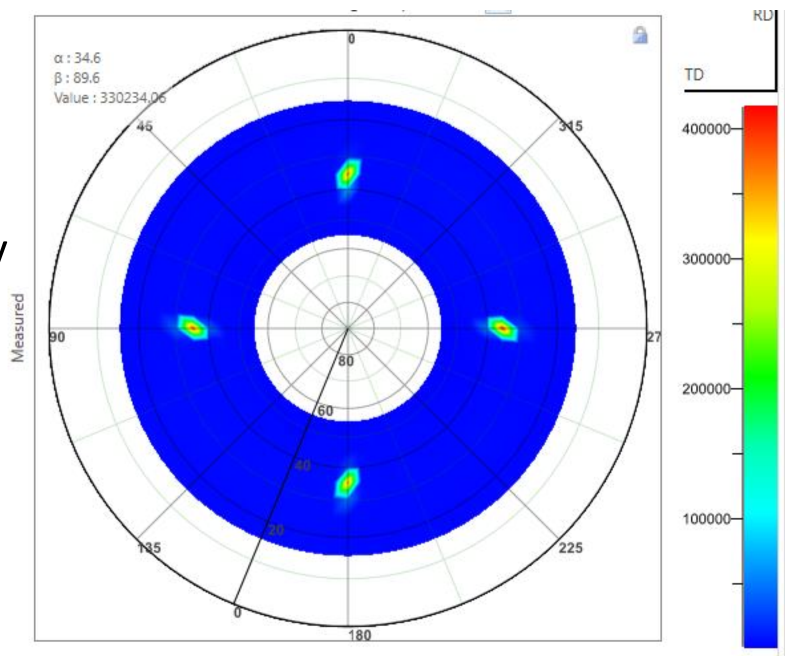
21. Click ***Run*** then ***OK*** when completed





For training with Silicon: Do not Run

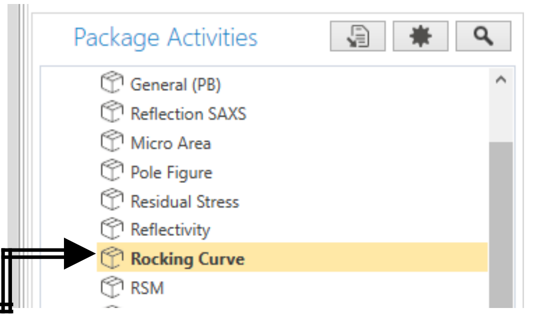
22. If the parameters were chosen properly, you should eventually see intensity peaks appear at the appropriate α and β positions



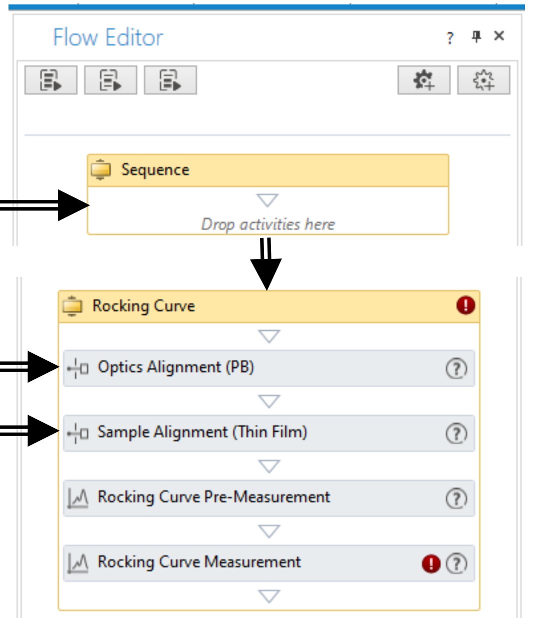
XI. Rocking Curve – 1/5

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

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



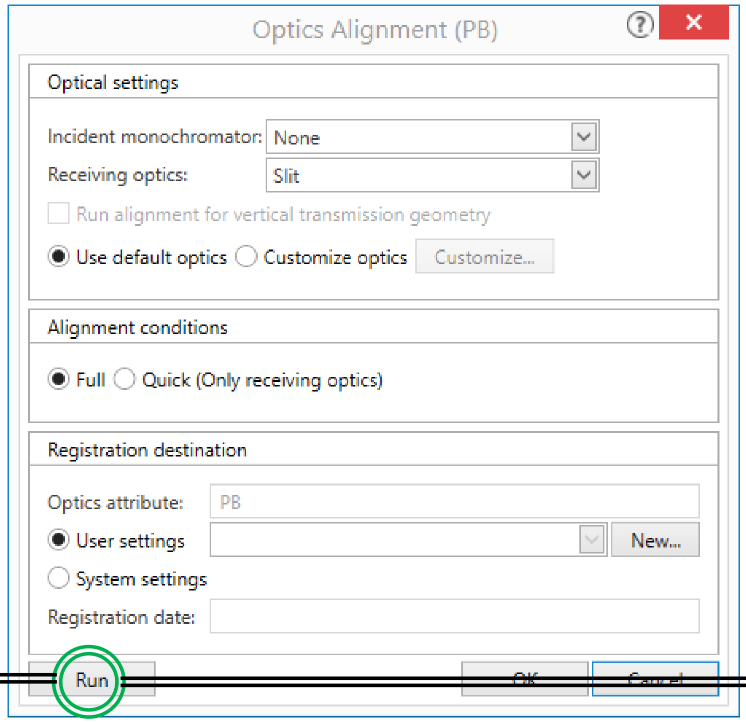
2. Drag the ***Rocking Curve*** package into the ***Flow Editor*** in ***Sequence***



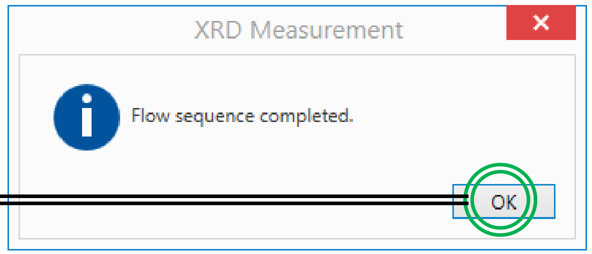
3. If ***Optics Alignment (PB)*** and ***Sample Alignment (Thin Film)*** were previously performed, then skip to ***Step 11***

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***



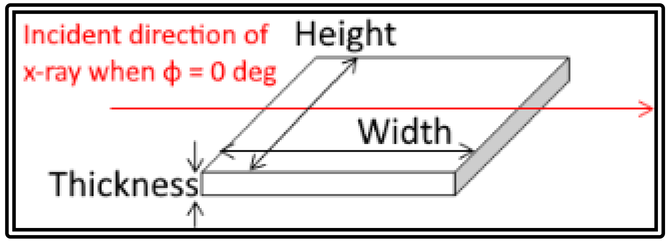
6. Click ***Run***
   and then ***OK*** when completed

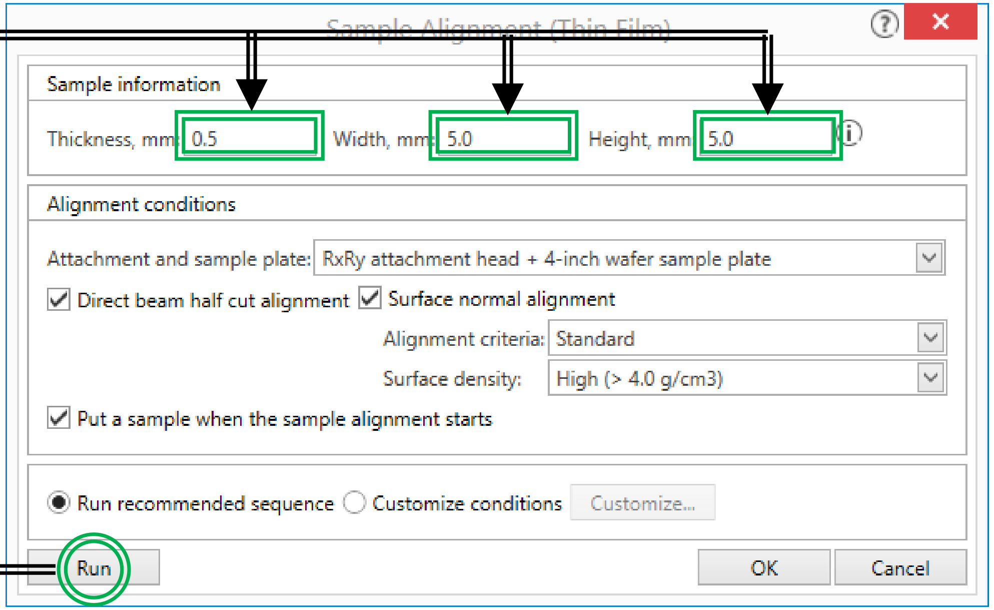


XI. Rocking Curve – 2/5

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

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

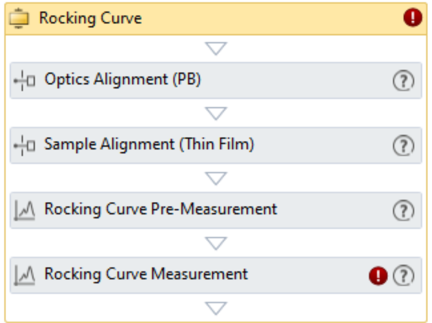




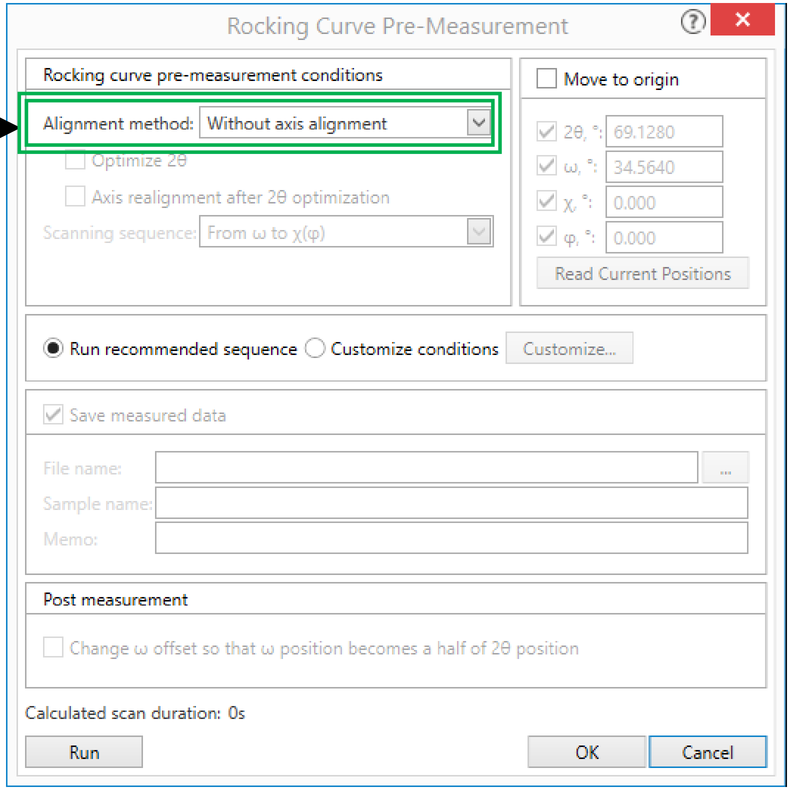
9. Confirm the following are checked:
   - Direct beam half cut alignment
   - Surface normal alignment
   - Put a sample when the sample alignment starts

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)



XI. Rocking Curve – 3/5

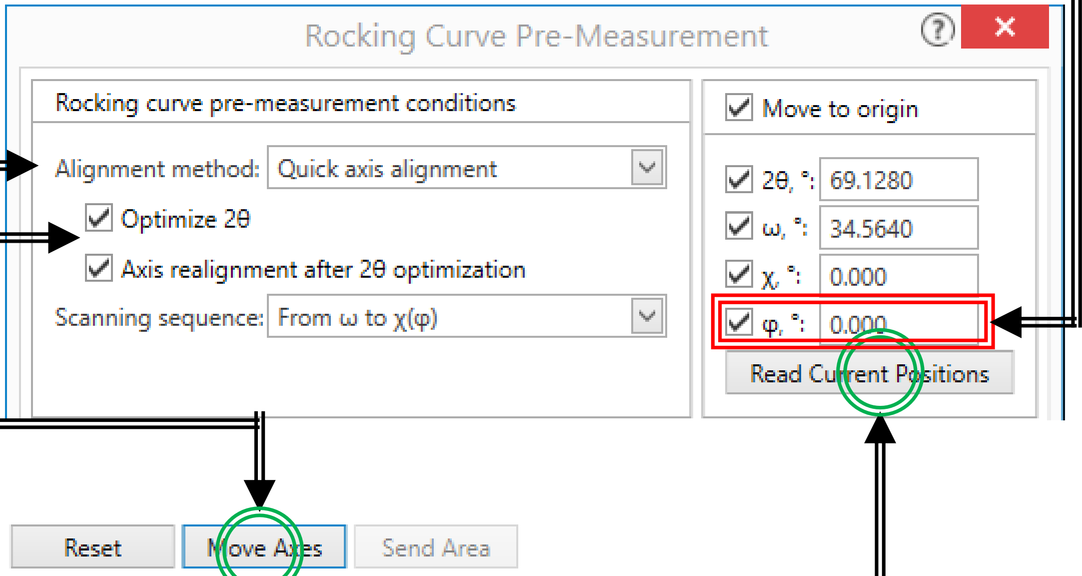
φ needs to be optimized separately!

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

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

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

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



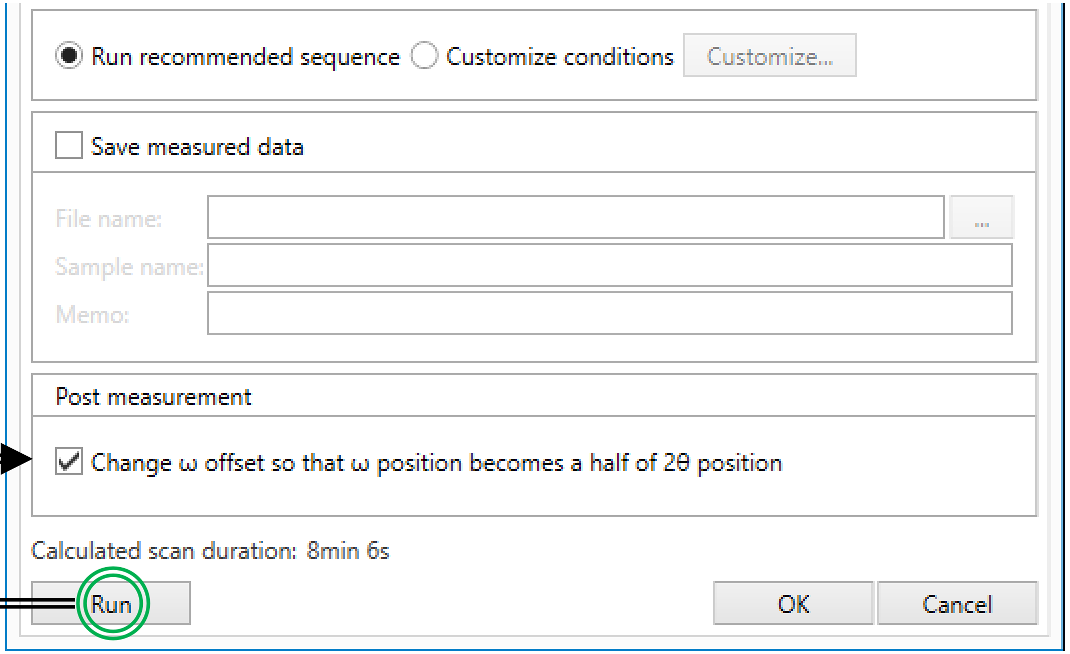
NOTE: Remember to check if your φ needs to be optimized first

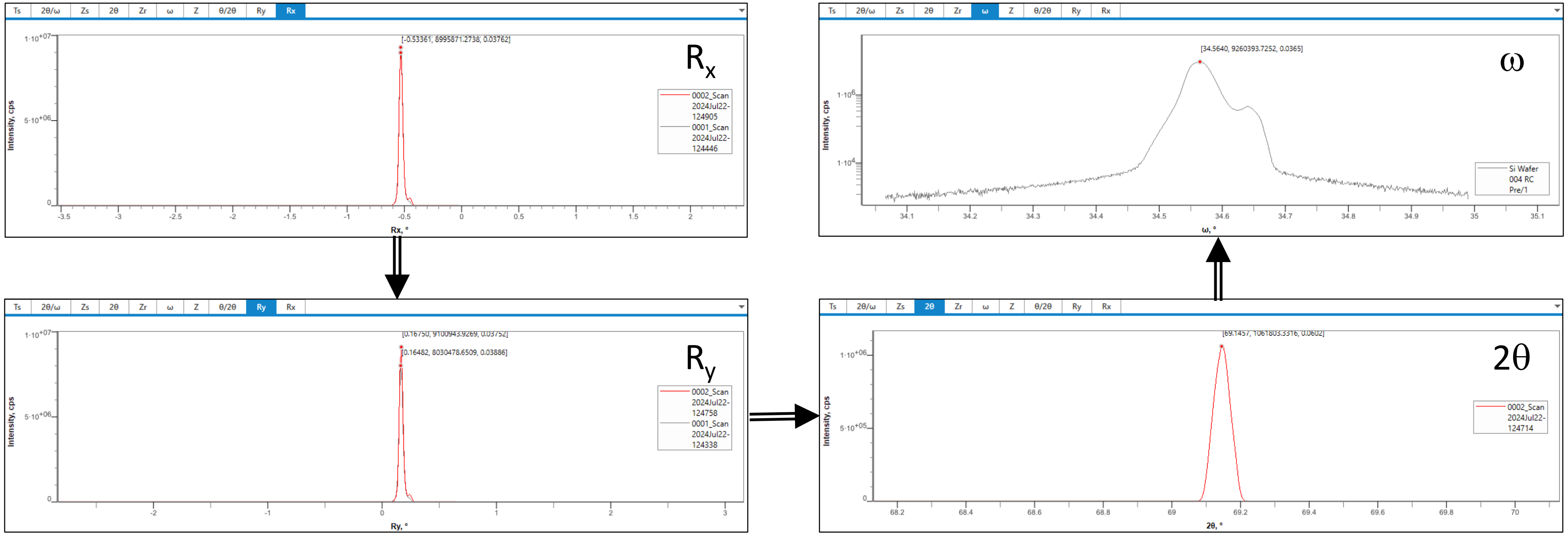
XI. Rocking Curve – 4/5

16. Proceed to check the Post measurement setting
    - Change ω offset so that ω position becomes a half of 2θ position

17. Click on ***Run***

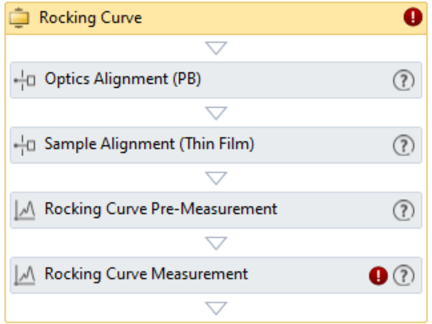
18. Axes will be aligned per the chosen ***Alignment method***





XI. Rocking Curve – 5/5

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

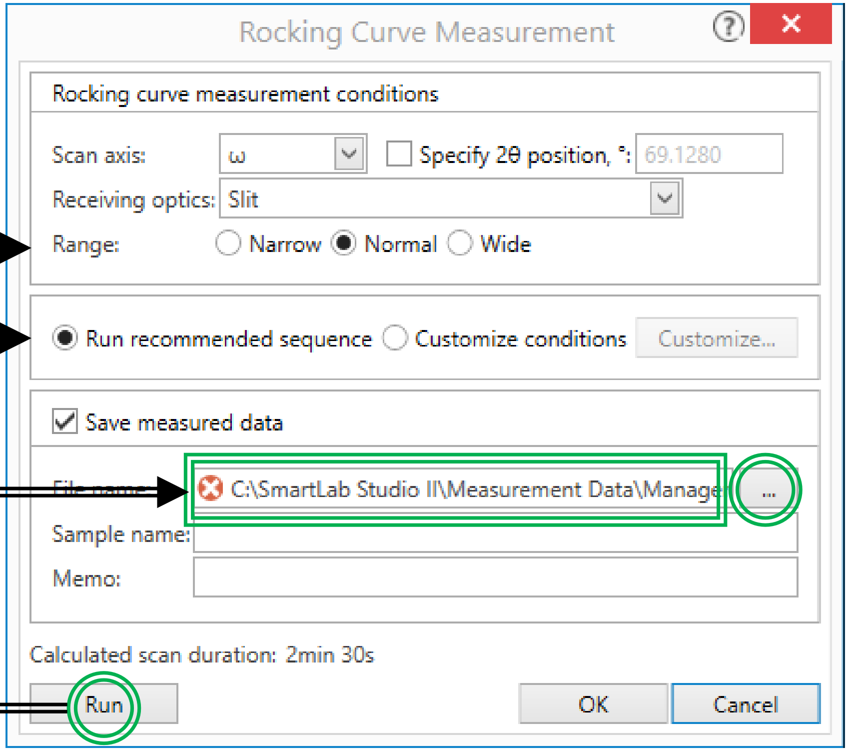


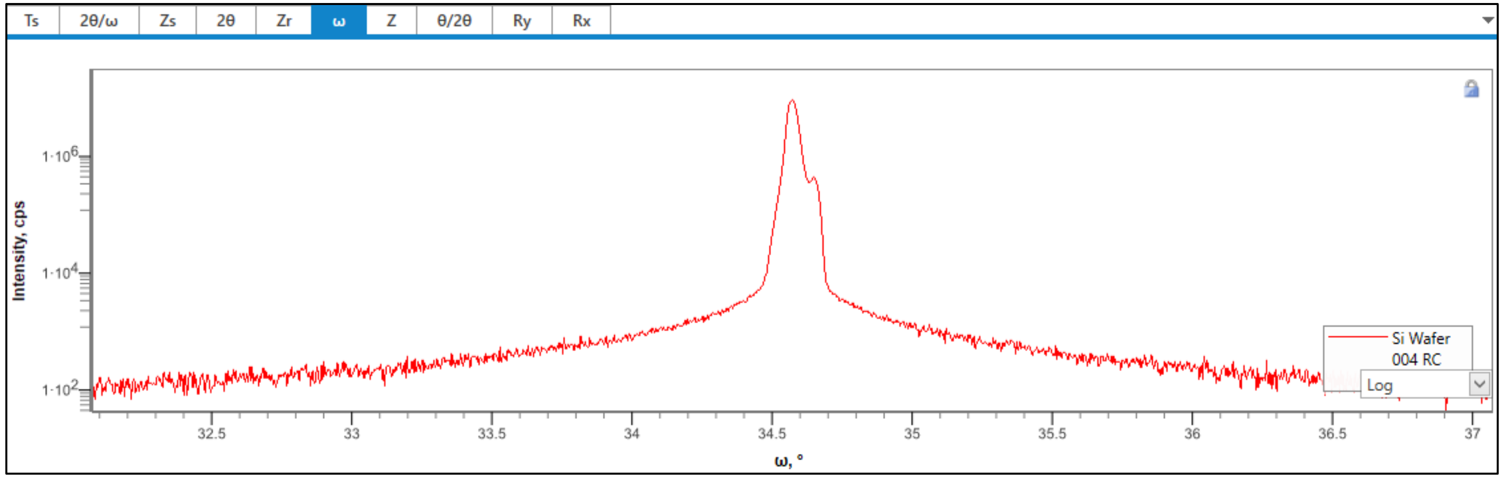
20. Determine the ***Range*** if choosing to ***Run recommended sequence***:
    - Narrow
    - Normal
    - Wide

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

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

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

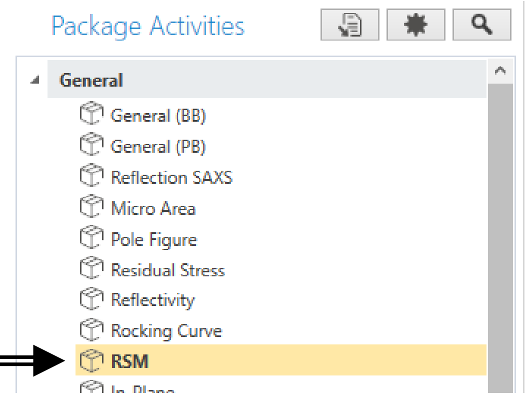




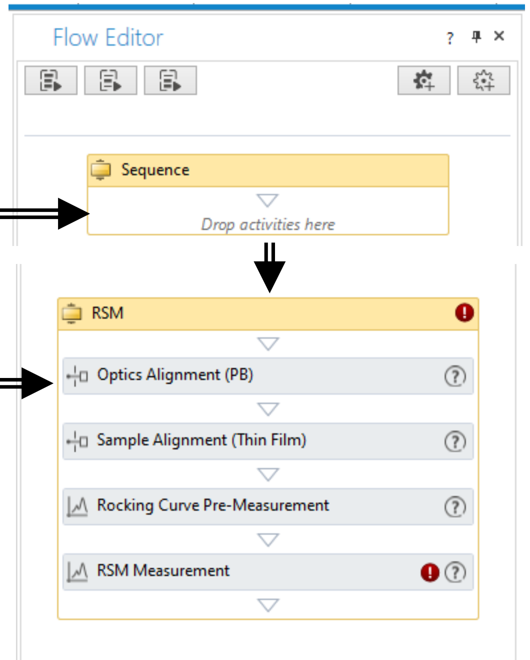
XII. Reciprocal Space Map (RSM) – 1/5

- 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***

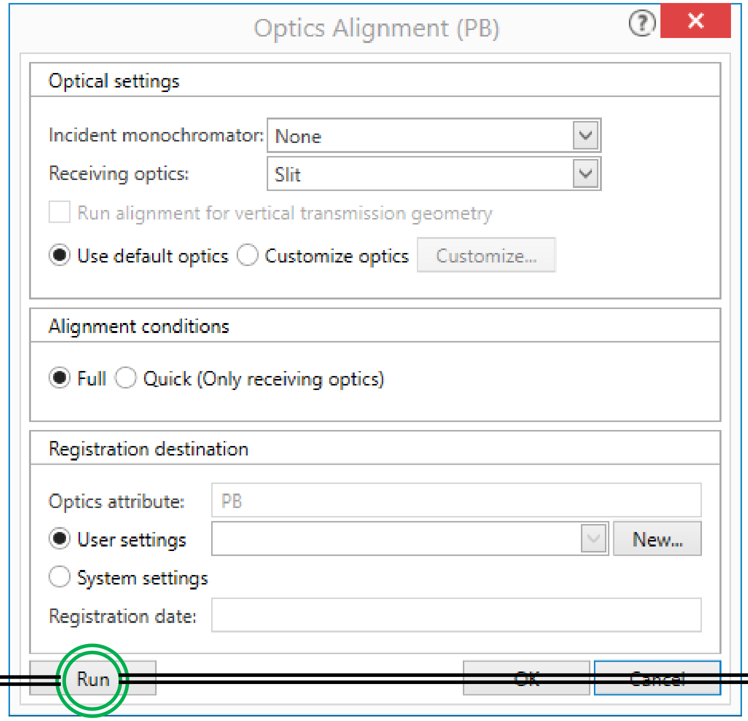


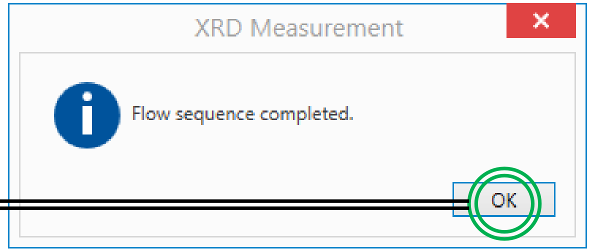
3. If ***Optics Alignment (PB)*** and ***Sample Alignment (Thin Film)*** were previously performed, then skip to ***Step 11***

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

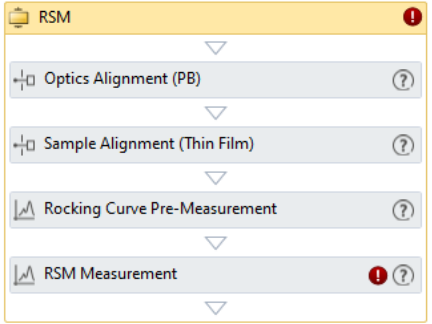
5. Confirm the following are selected:
   - ***Optical settings → Use default optics***
   - ***Alignment conditions → Full***
   - ***Registration destination → User settings***

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



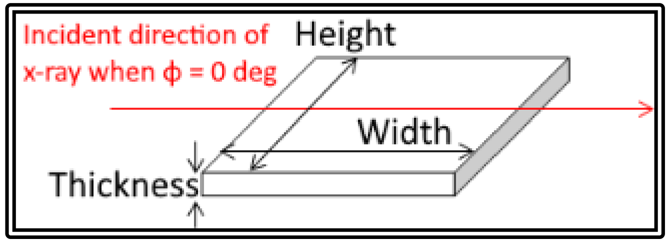


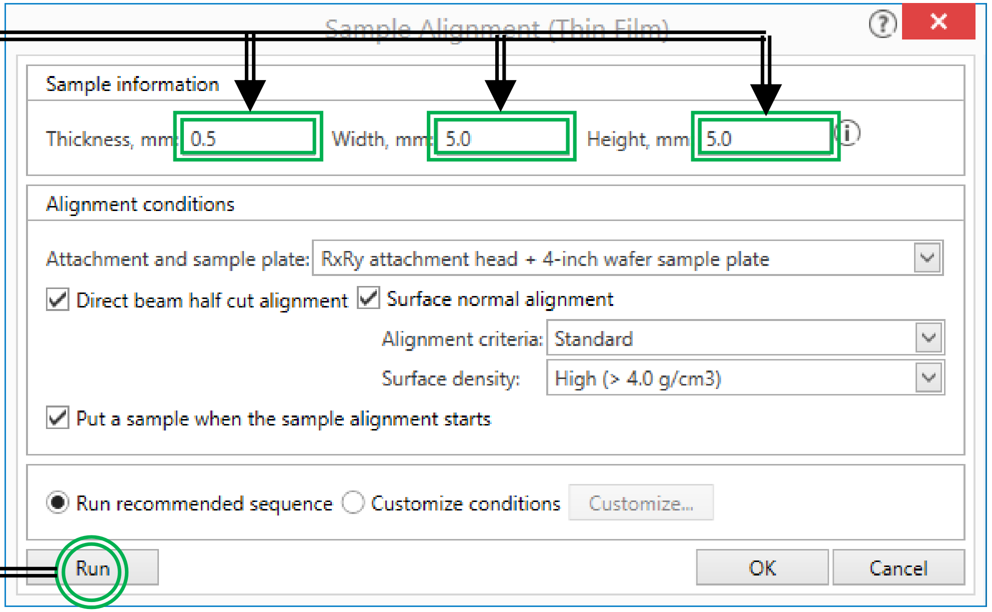
XII. Reciprocal Space Map (RSM) – 2/5



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

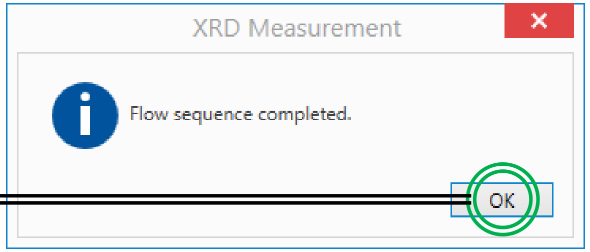
8. Input your ***Sample Info*** per the dimensions

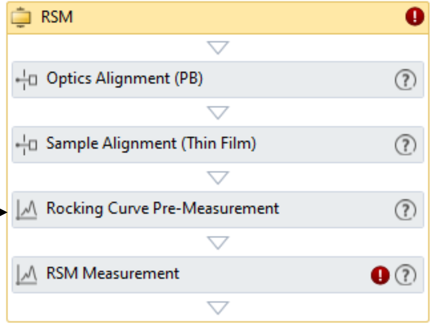




9. Confirm the following are checked:
   - Direct beam half cut alignment
   - Surface normal alignment
   - Put a sample when the sample alignment starts

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





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

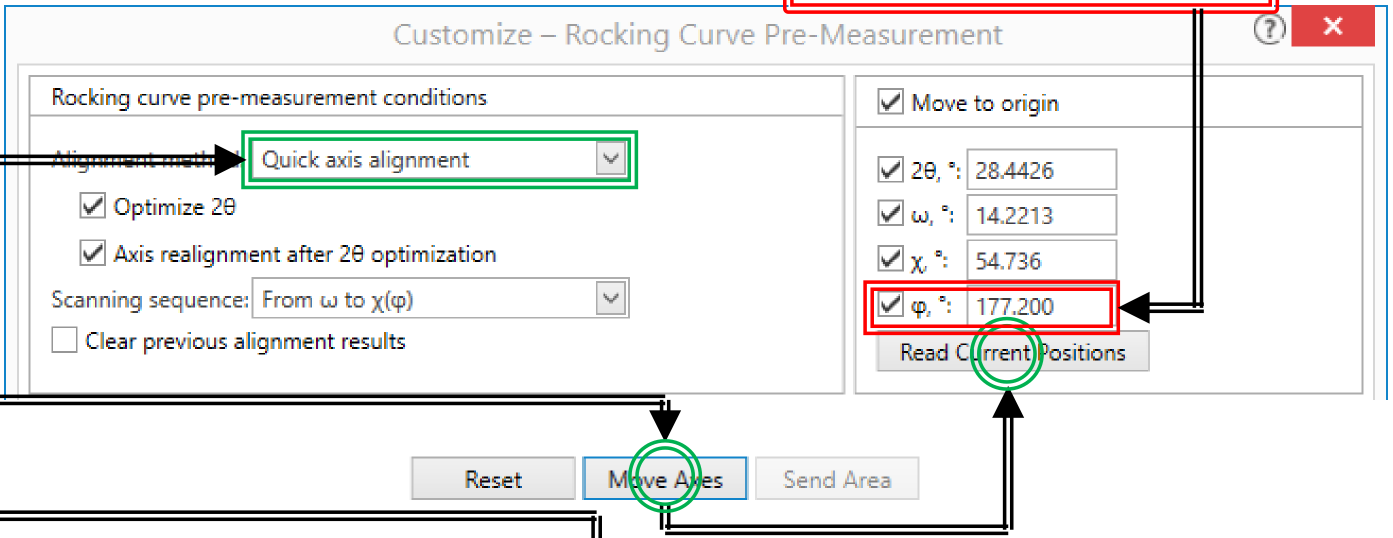
XII. Reciprocal Space Map (RSM) – 3/5

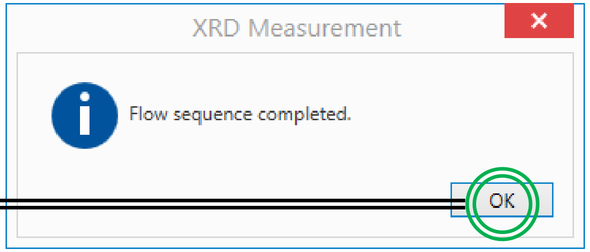
φ needs to be optimized separately!

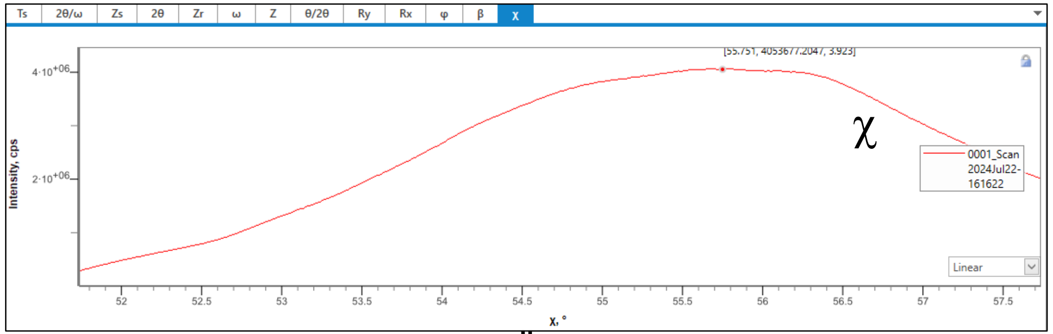
12. Determine the desired ***Alignment method*** (see ***XI. Rocking Curve*** for review)

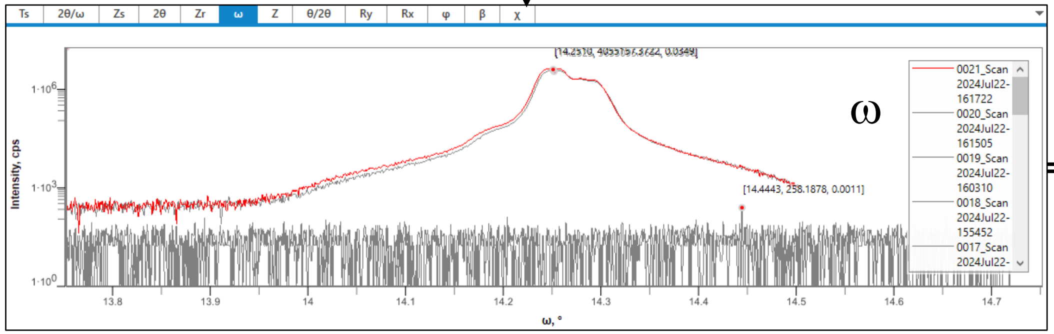
13. Before proceeding, check that your ***Origin Position*** is set to desired plane using ***RS Viewer*** before proceeding

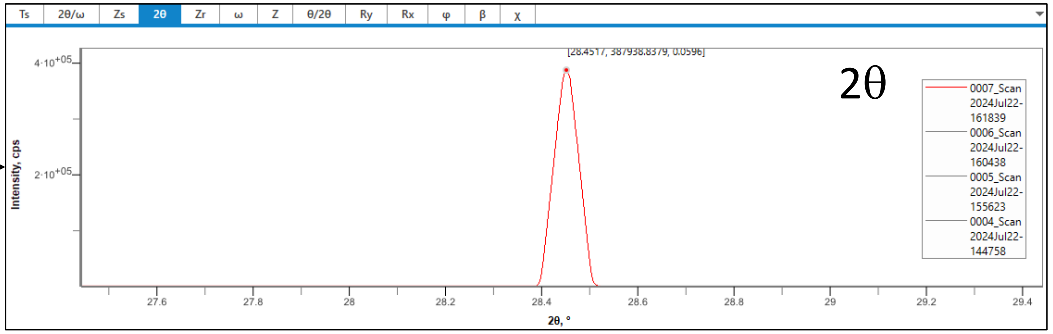
14. Click ***Run*** and then ***OK*** when completed





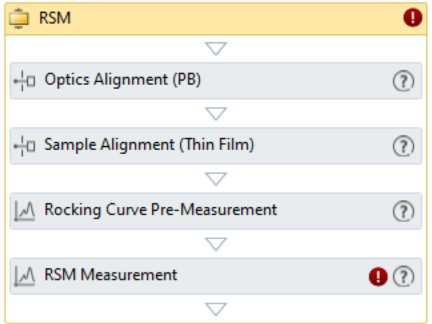






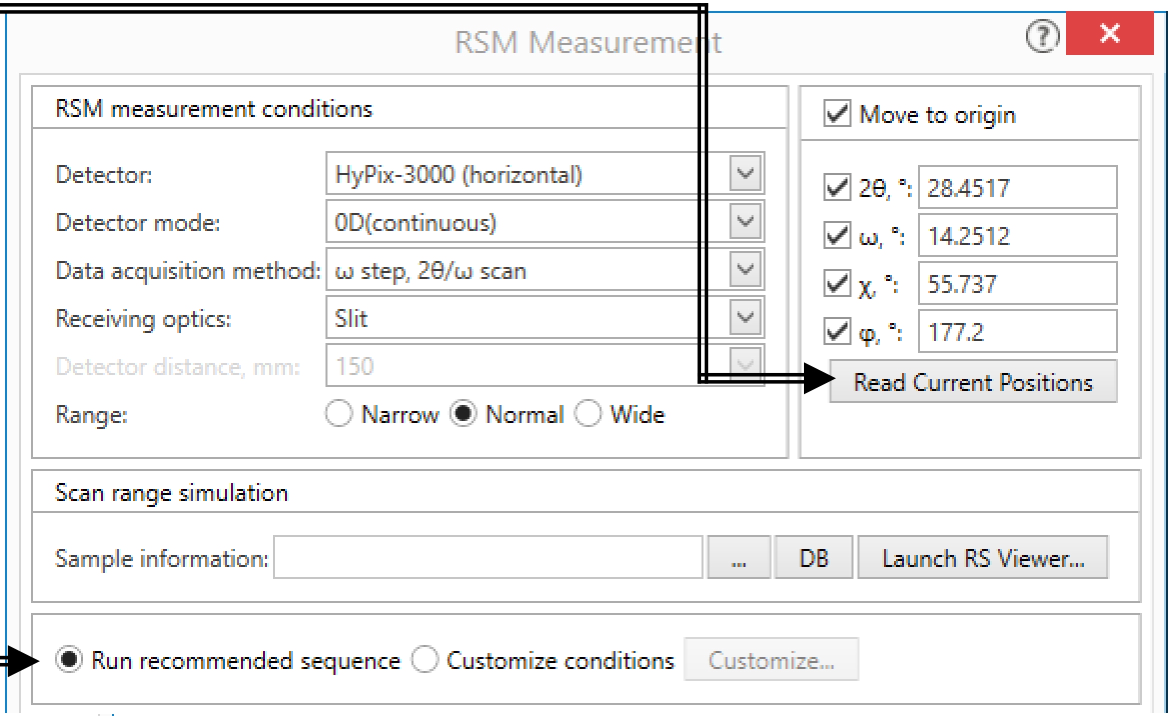
XII. Reciprocal Space Map (RSM) – 4/5

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

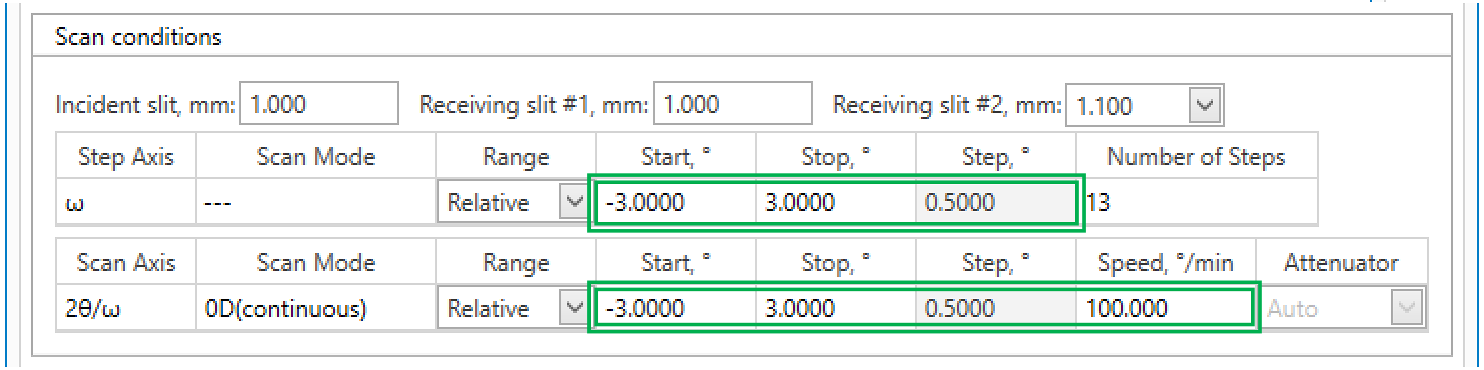


16. Click on ***Read Current Positions*** to align axis positions from ***Pre-Measurement***

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



18. Adjust the following parameters if desired:
    - ***Start***
    - ***Stop***
    - ***Step***
    - ***Speed***

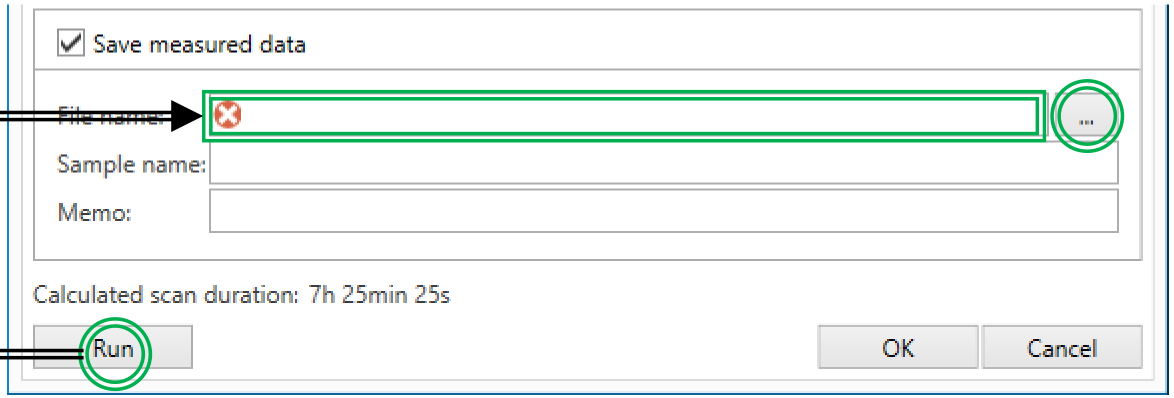


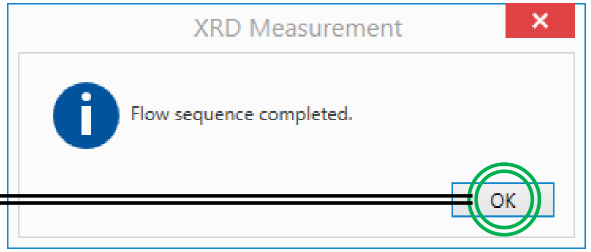
XII. Reciprocal Space Map (RSM) – 5/5

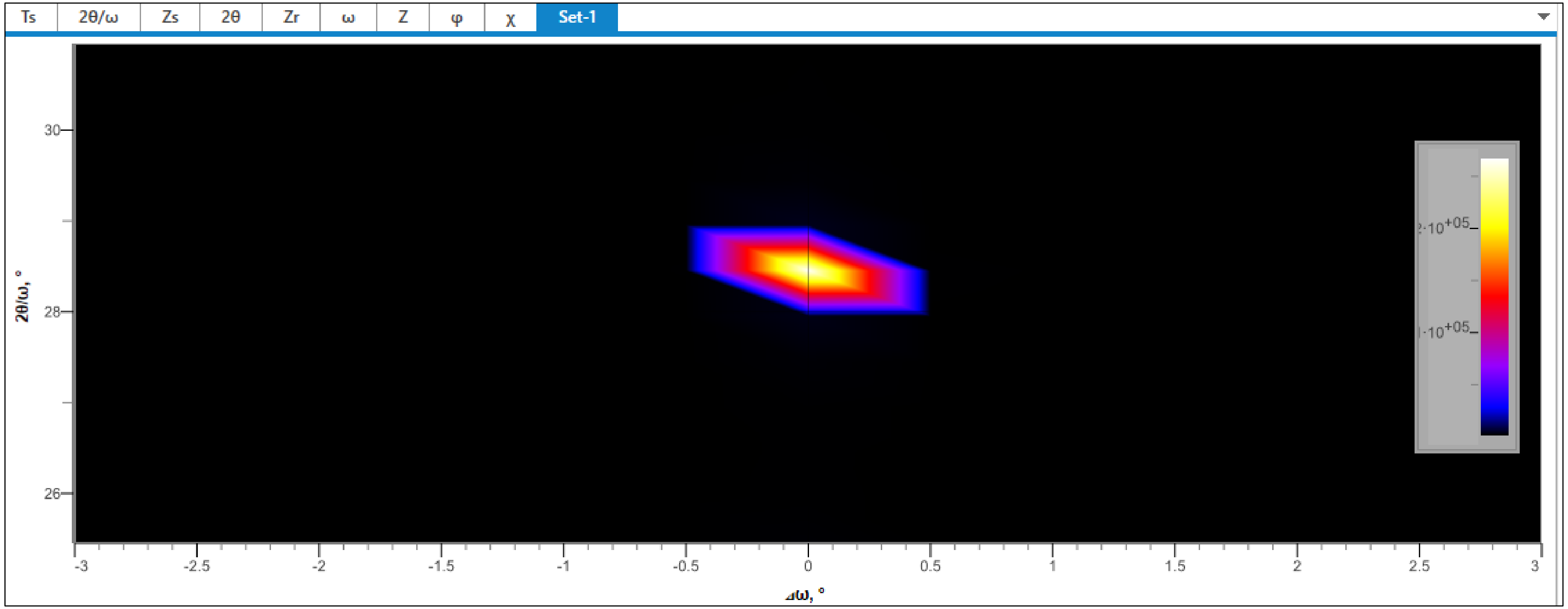
19. Input your desired ***File name*** and ***File location*** here

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

    For training with Silicon: Do not Run



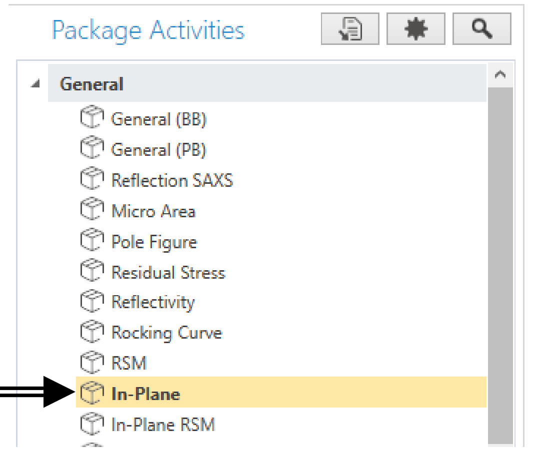


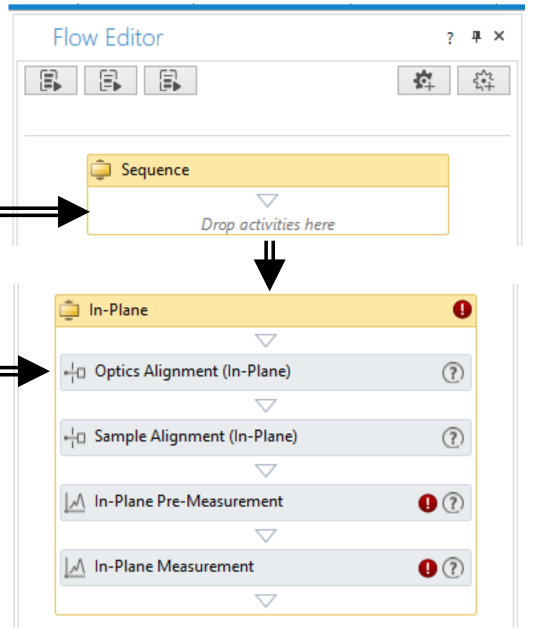


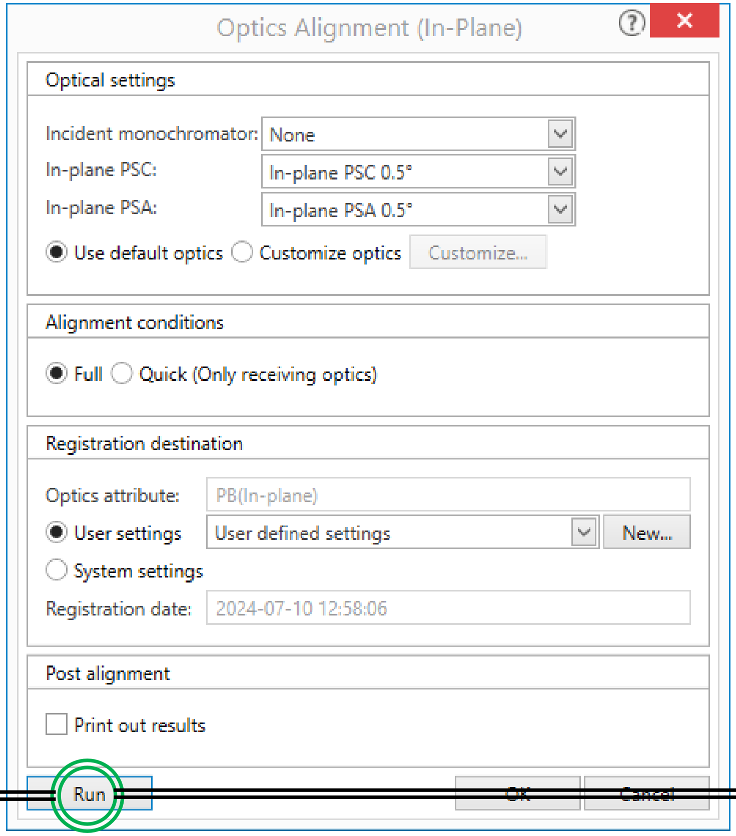
XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 1/9

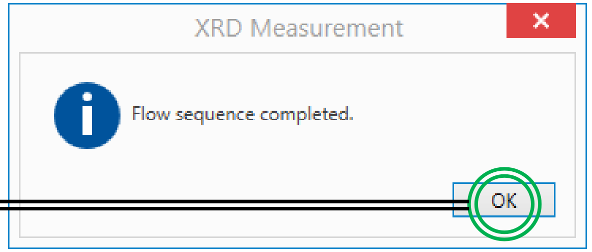
- This sequence will perform an In-Plane Measurement or $2\theta_\chi/\varphi$ using Parallel Beam optics

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







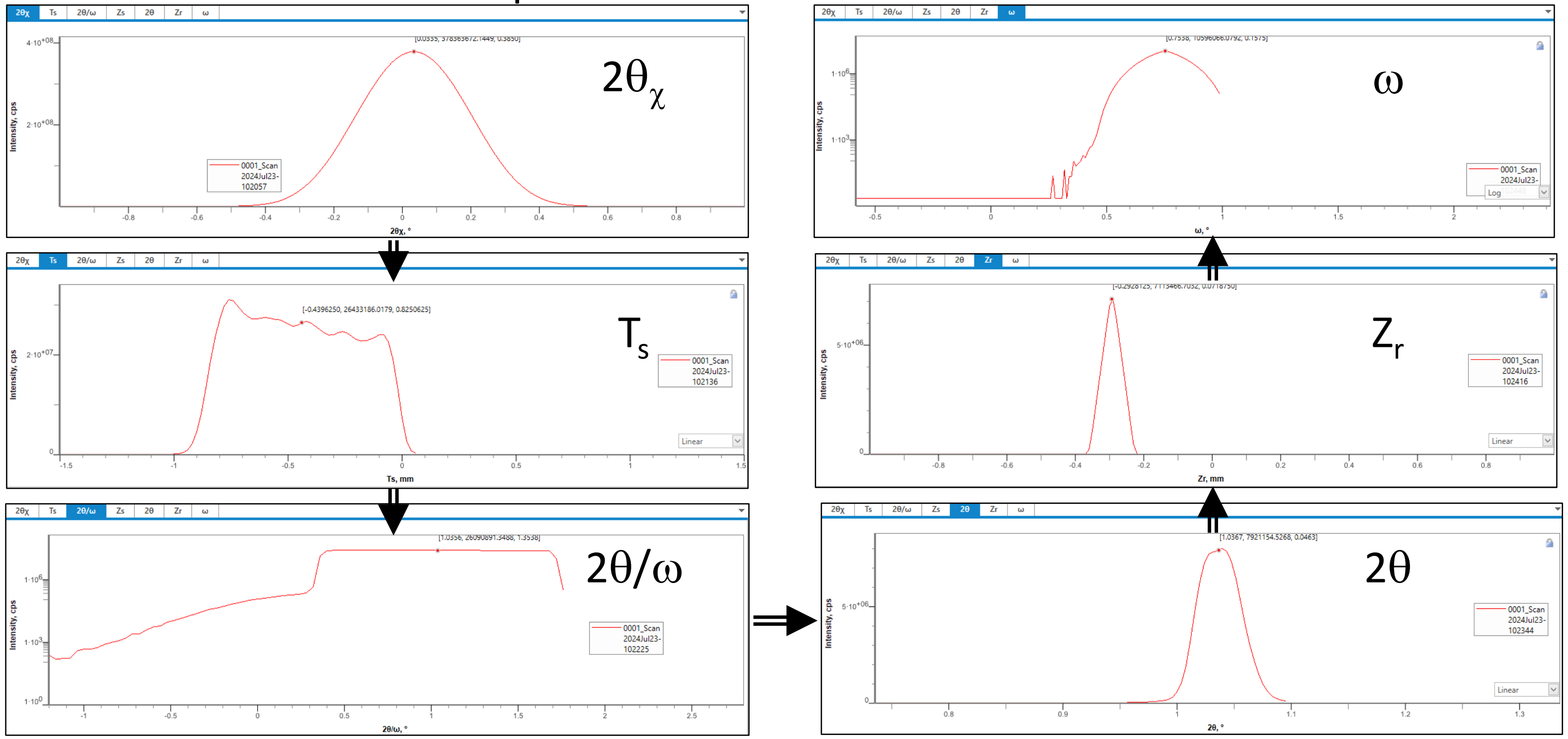


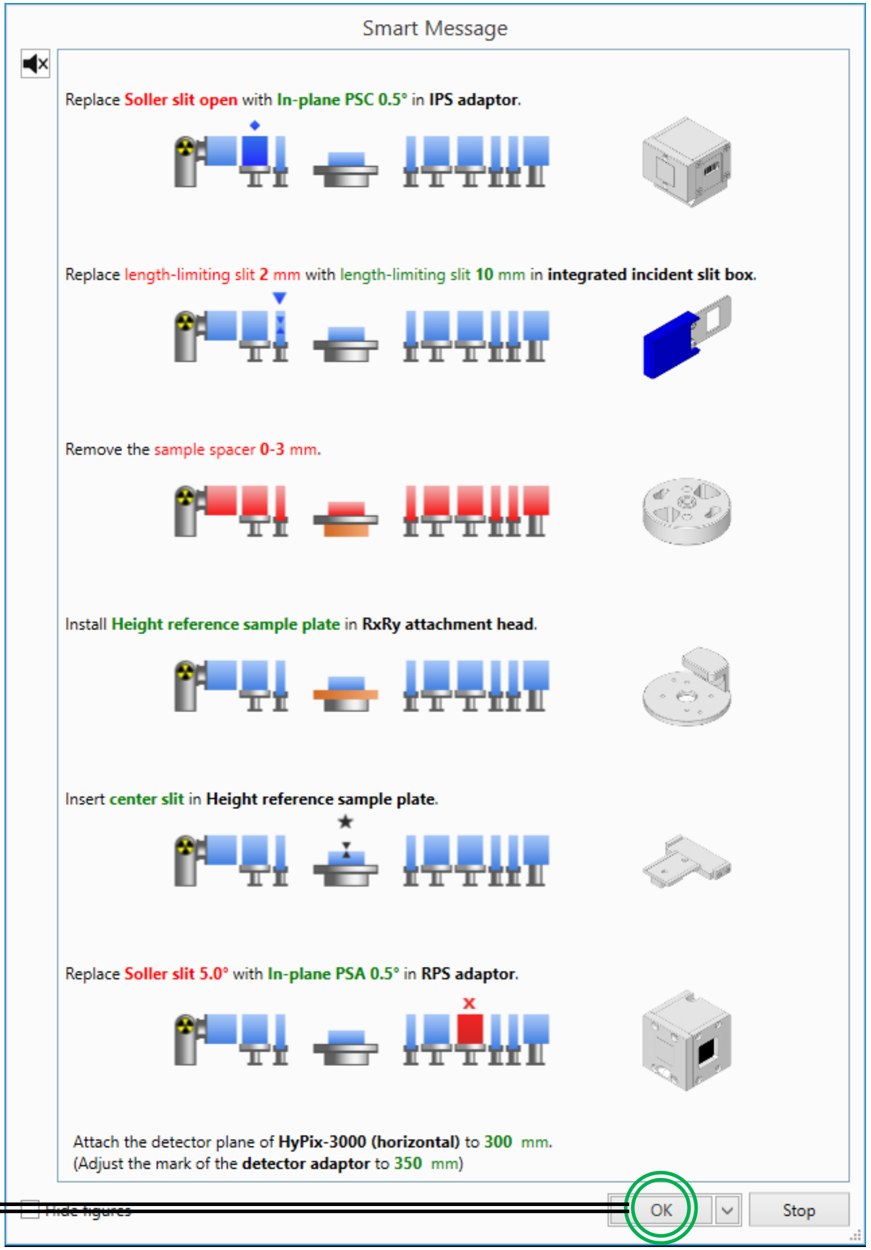
XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 2/9

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**)

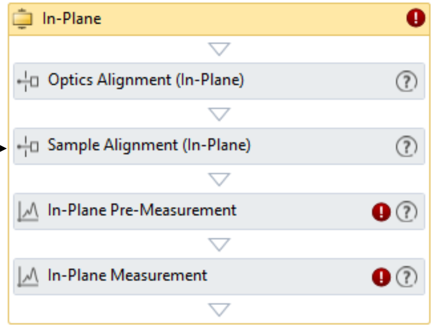
Reminder: ***Window Protector*** must be inserted to protect ***Detector*** when swapping out ***Receiving Optics***!

7. Click ***OK*** when completed



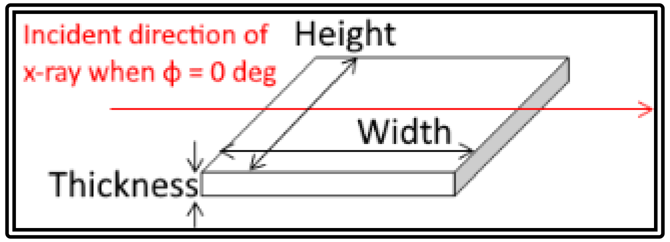


XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi$ – 3/9

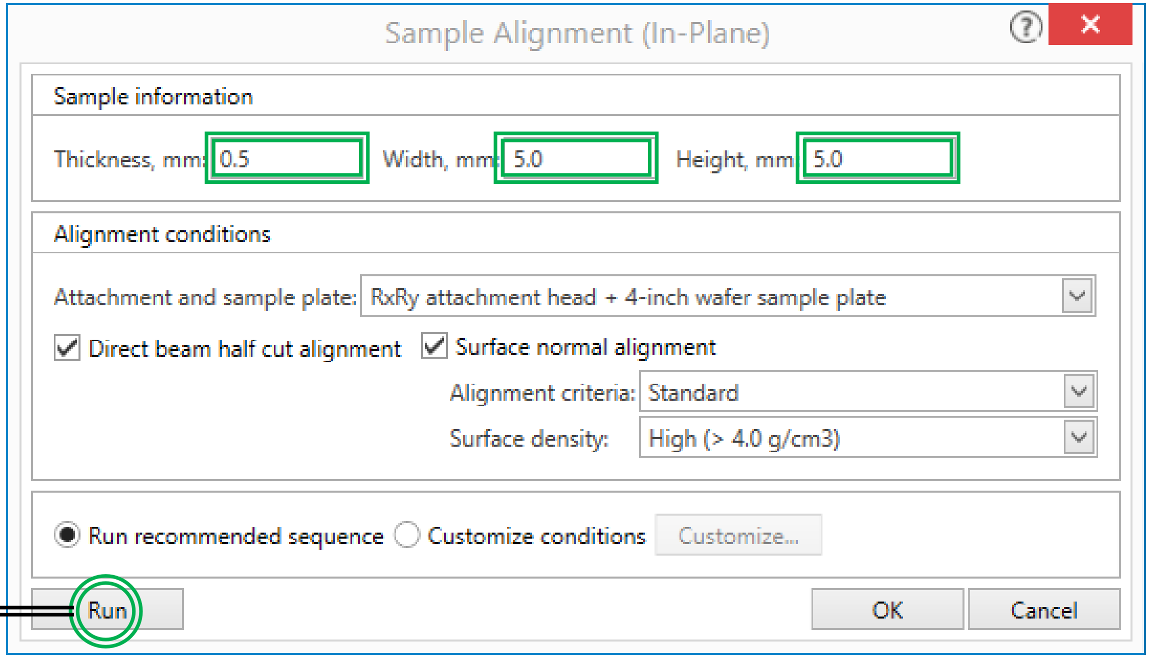


8. Left-click on ***Sample Alignment (In-Plane)*** to set ***Sample Info***

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



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

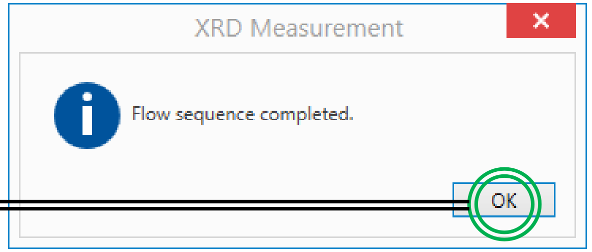


10. Confirm the following are checked:
    - Direct beam half cut alignment
    - Surface normal alignment
    - Put a sample when the sample alignment starts

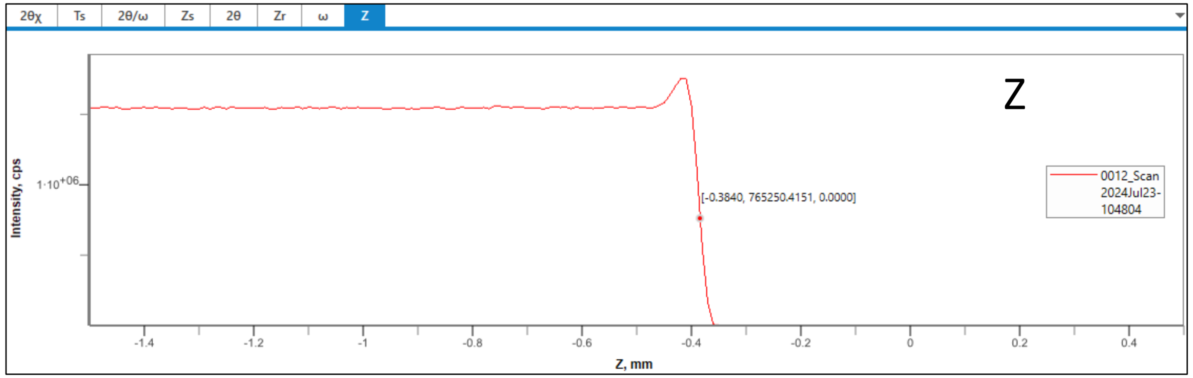
11. Click ***Run***

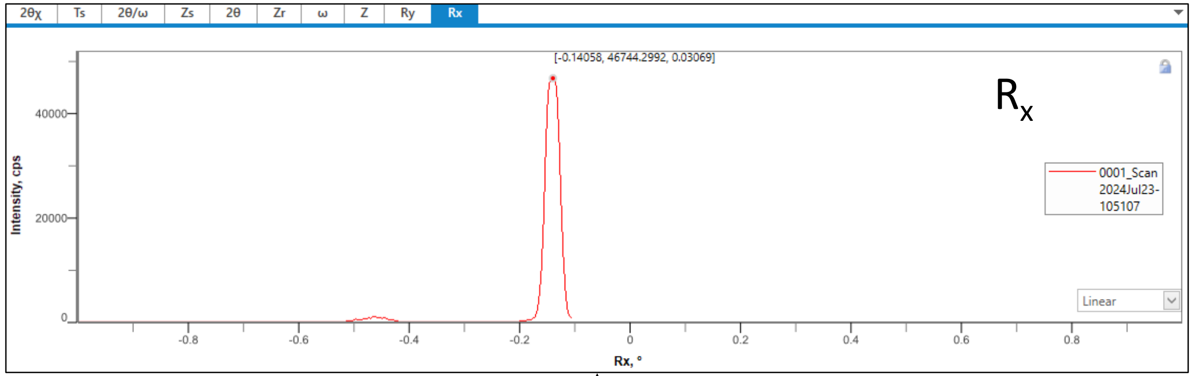
12. If ***Sample Alignment (In-Plane)*** fails, try changing surface density (e.g. High)

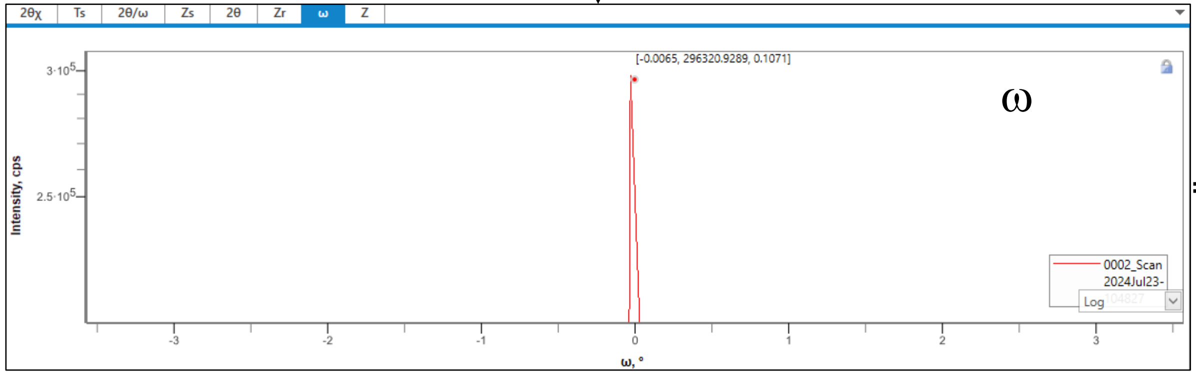
13. Wait for the ***Flow sequence completed*** prompt to continue by clicking ***OK***

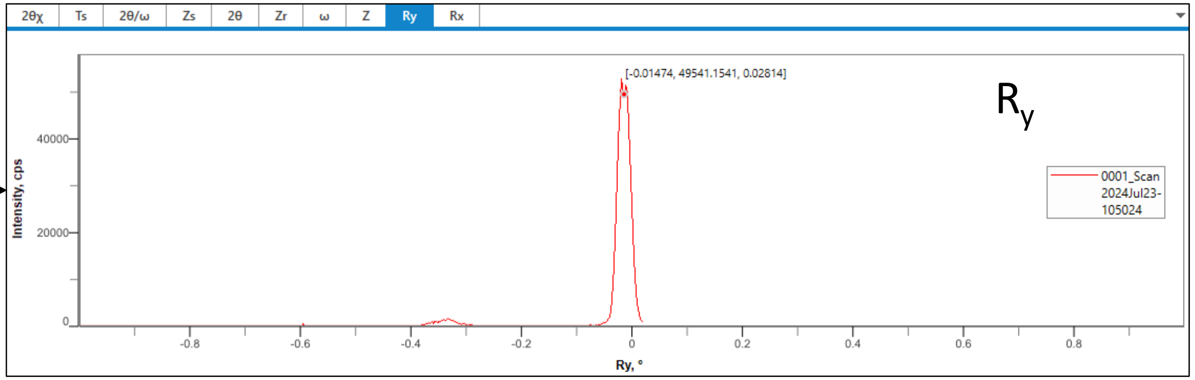


XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 4/9

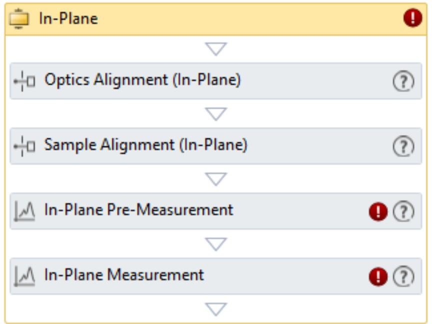






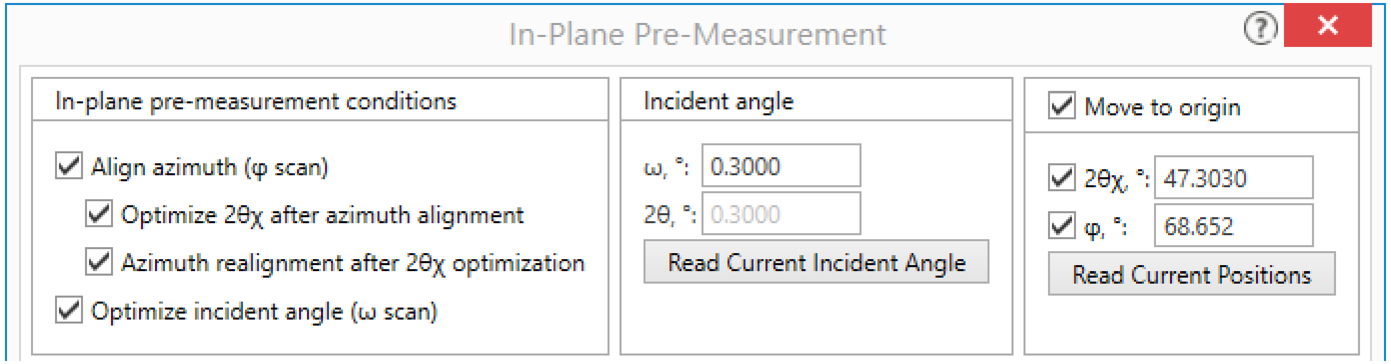


14. Left-click on ***In-Plane Pre-Measurement***



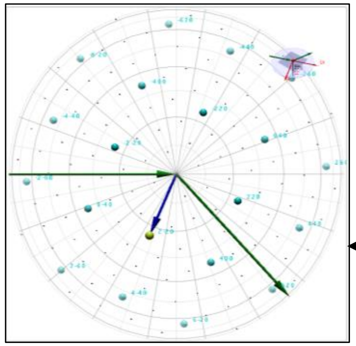
XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 5/9

15. Recommend the following is checked:
    - Align azimuth (φ scan)
    - Optimize $2\theta_\chi$ after azimuth alignment
    - Azimuth realignment after $2\theta_\chi$ optimization
    - Optimize incident angle (ω scan)



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

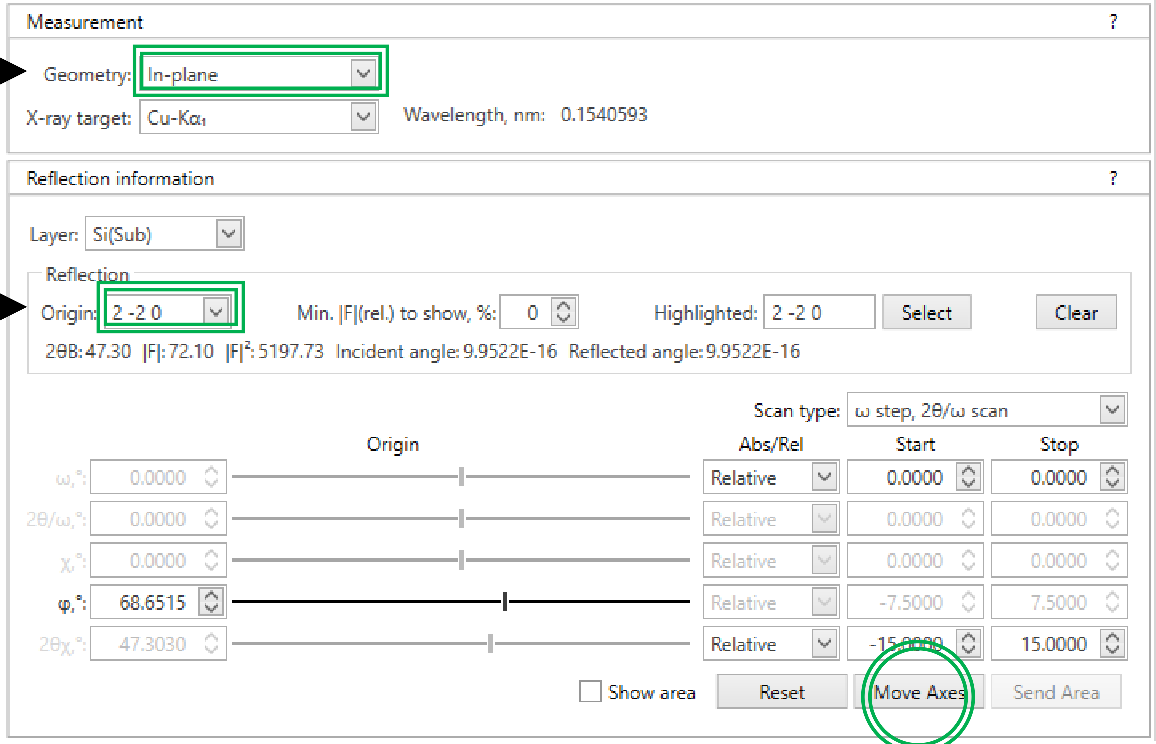
16. Open the ***RS Viewer*** and find the Si **(2 -2 0)** plane



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

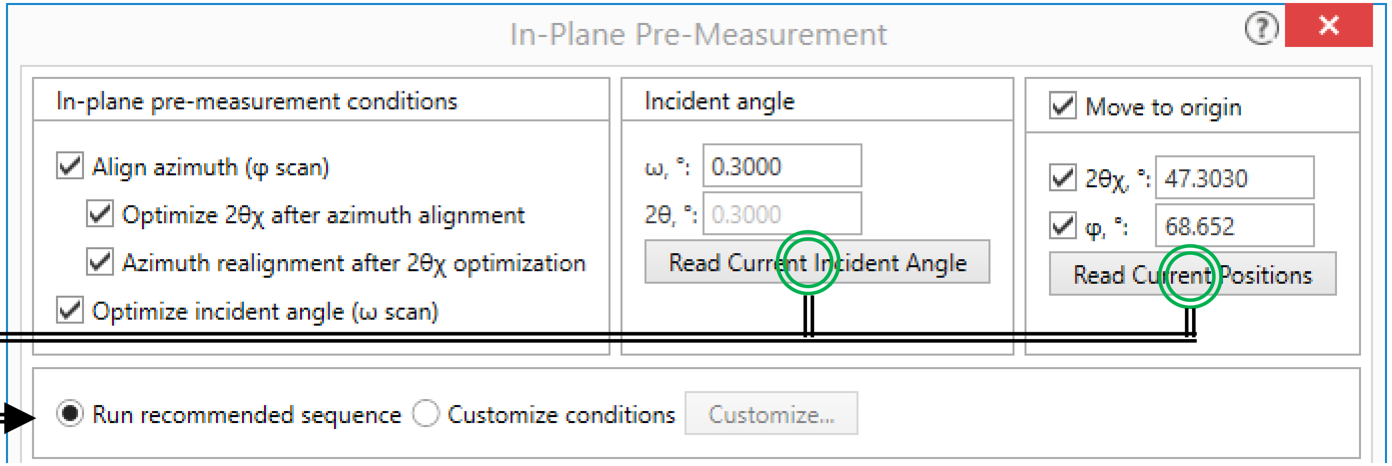
18. Click on ***Move Axes***

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



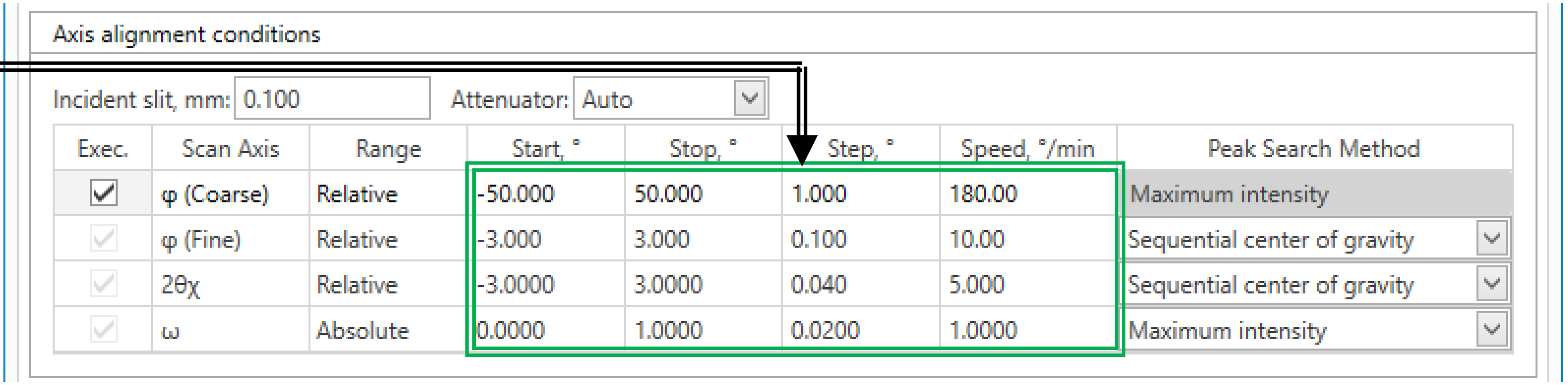
XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi$ – 6/9

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

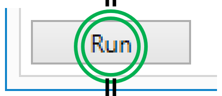


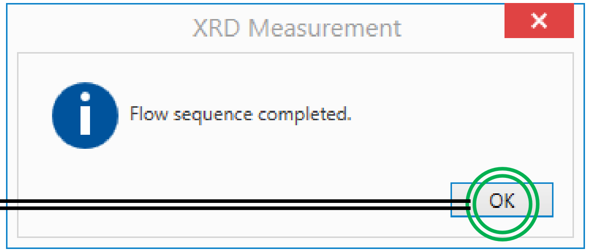
21. Use the ***Run recommended sequence*** or click ***Customize conditions***

22. Adjust the following parameters if desired:
   - ***Start***
   - ***Stop***
   - ***Step***
   - ***Speed***



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



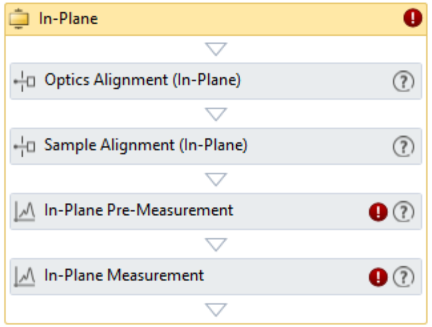


XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 7/9

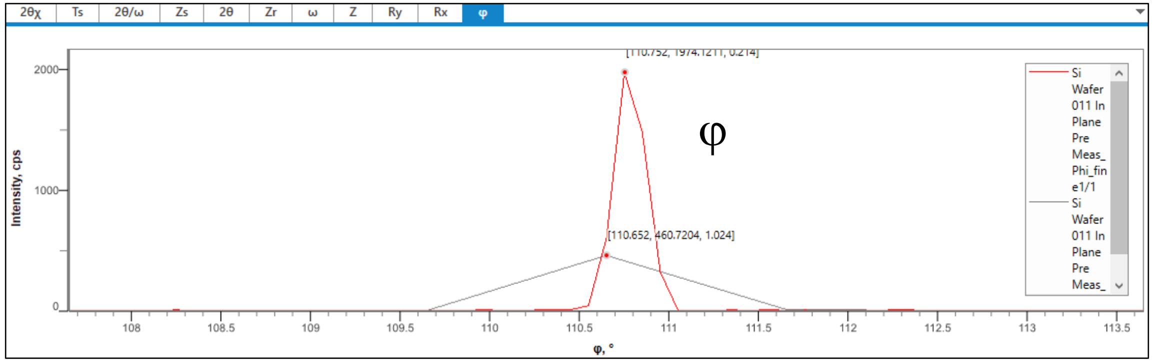
20. Once completed, the optimal φ and $\mathbf{2\theta_\chi}$ parameters should be determined

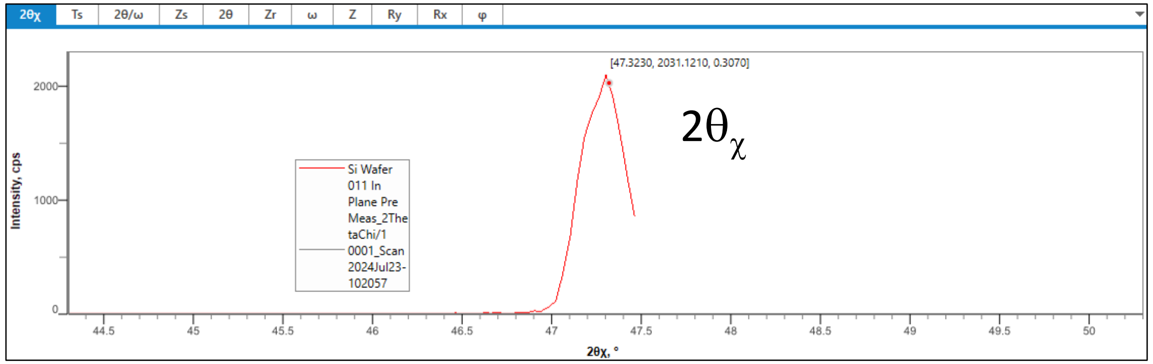
21. If the parameters are not clearly optimized, check if the search is wide enough or if the initial parameters were appropriate

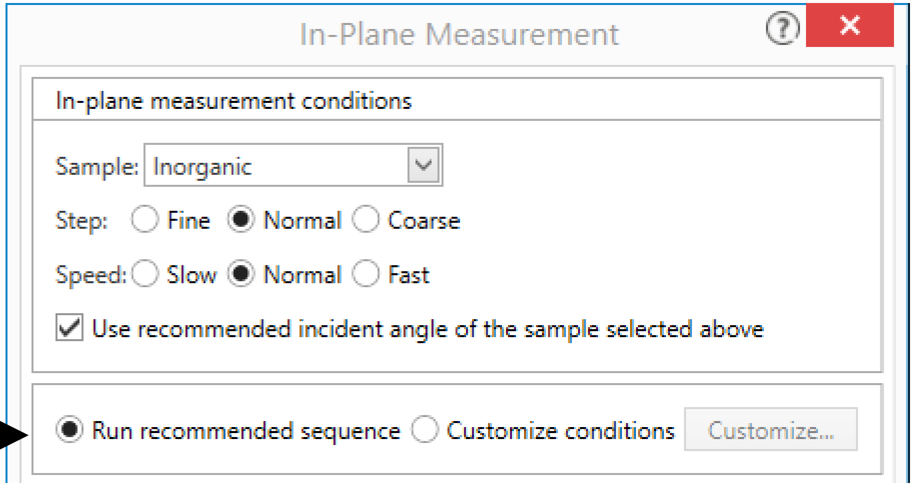
22. Left-click on ***In-Plane Measurement***



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

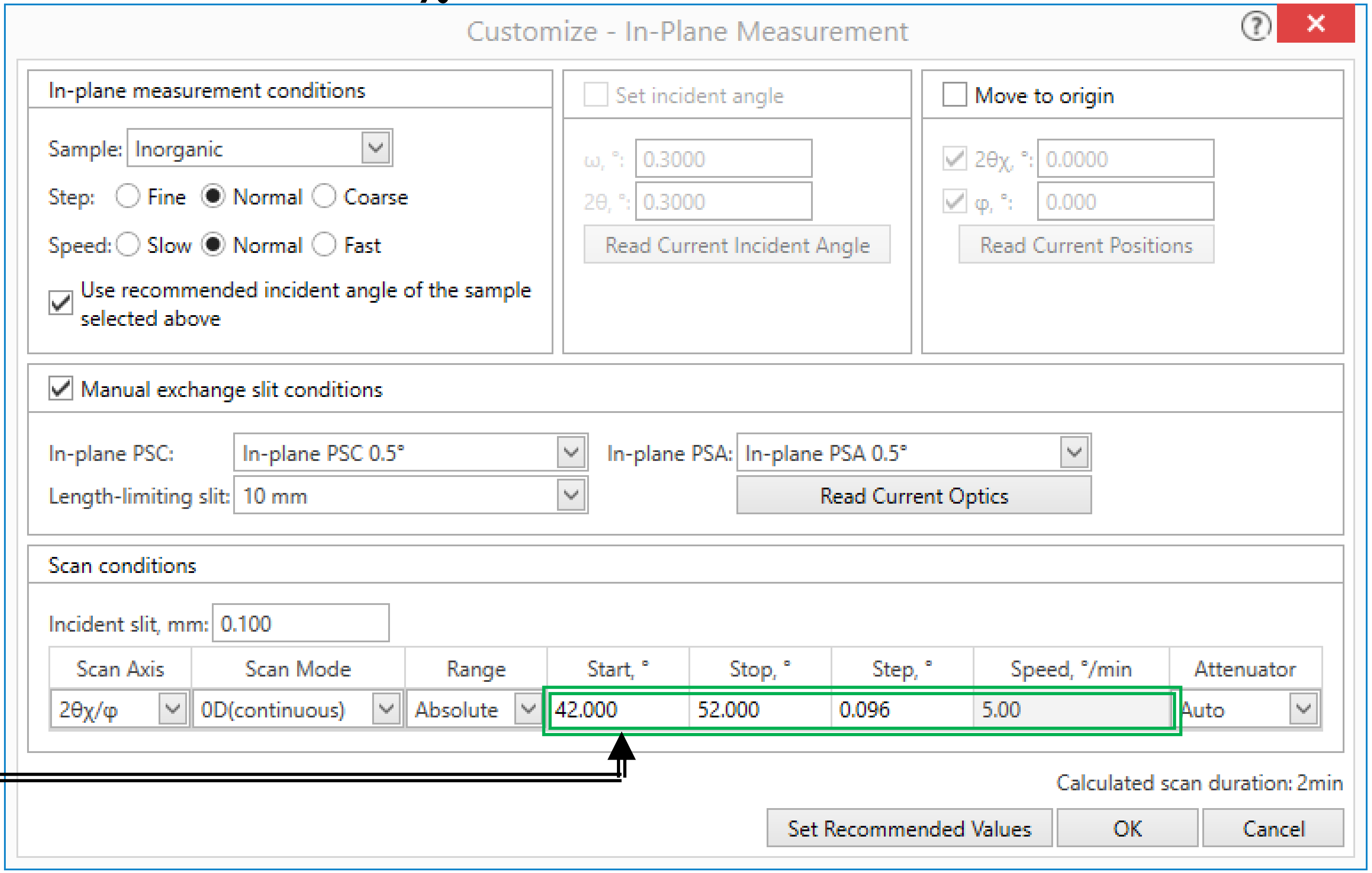






XIII. In-Plane Measurement or $2\theta_{\chi}/\varphi$ – 8/9

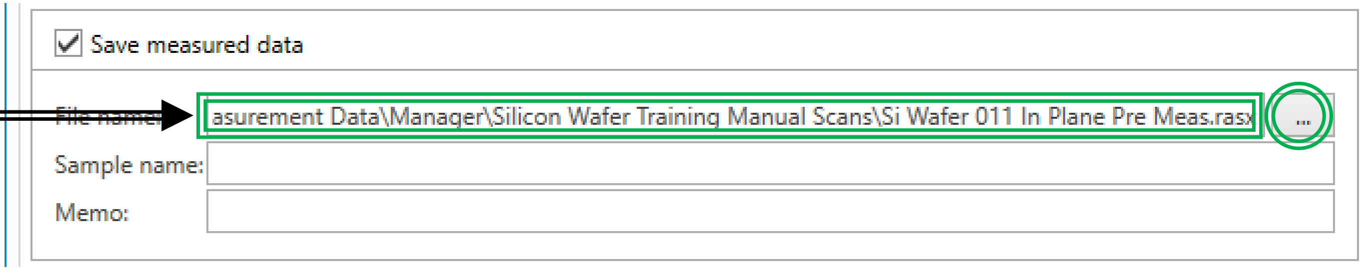
24. Click on ***Read Current Positions*** so that both the **2θχ** and **φ** are updated here if ***Move to origin*** is checked



25. Adjust the following parameters if desired:
    - ***Start***
    - ***Stop***
    - ***Step***
    - ***Speed***

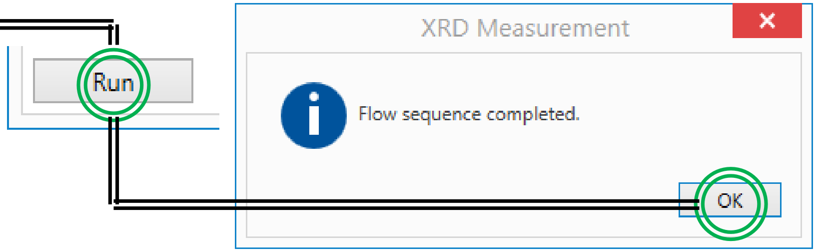
For training with Silicon: See example

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

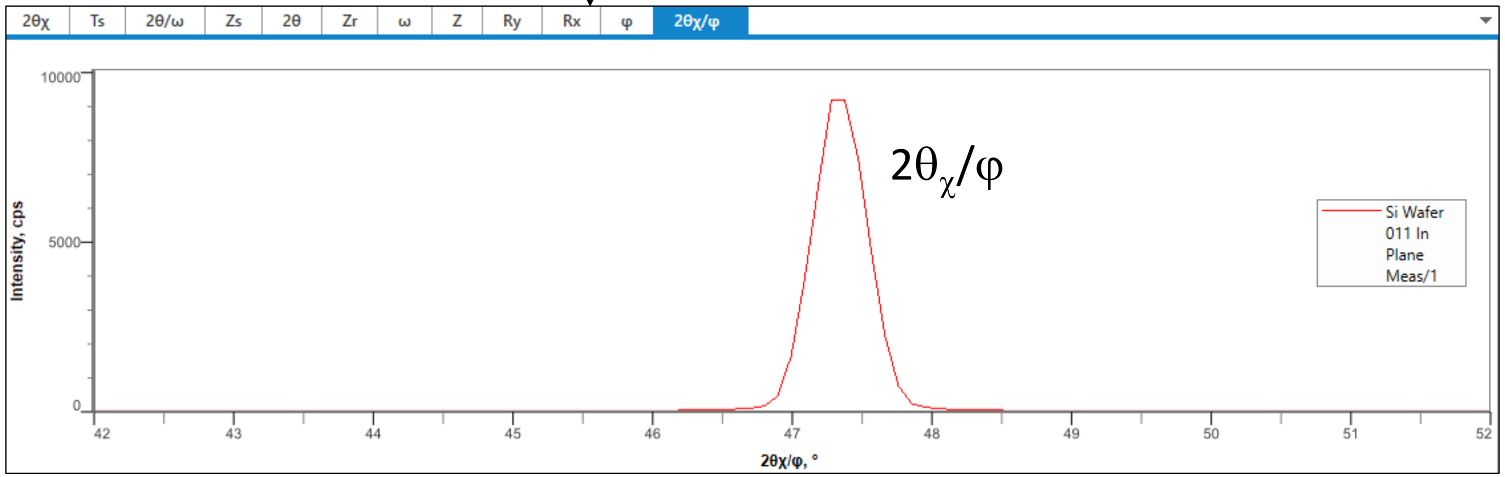


XIII. In-Plane Measurement or $2\theta_\chi/\varphi$ – 9/9

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



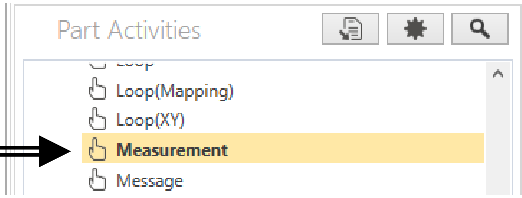
28. The $\mathbf{2\theta_\chi/\varphi}$ measurement should look like this

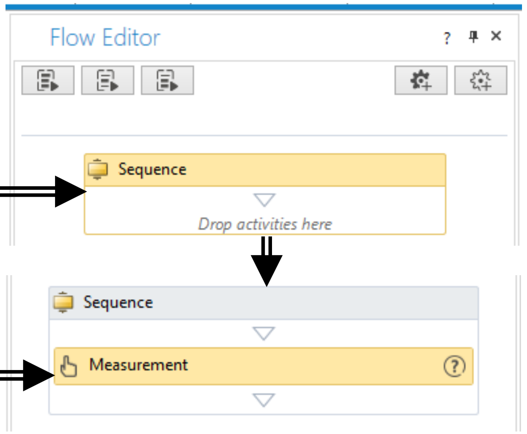


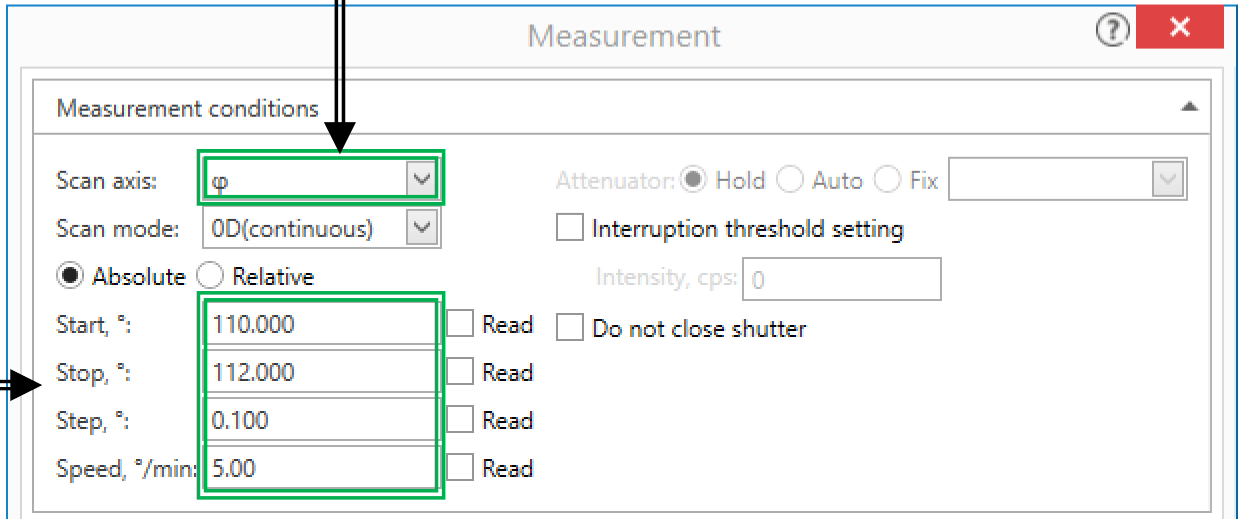
XIV. In-Plane Azimuth or Phi(φ) Scan – 1/2

- This sequence will perform an In-Plane Azimuth or Phi(φ) 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 φ for ***Scan axis***
6. Configure the following:
   - ***Start***
   - ***Stop***
   - ***Step***
   - ***Speed***

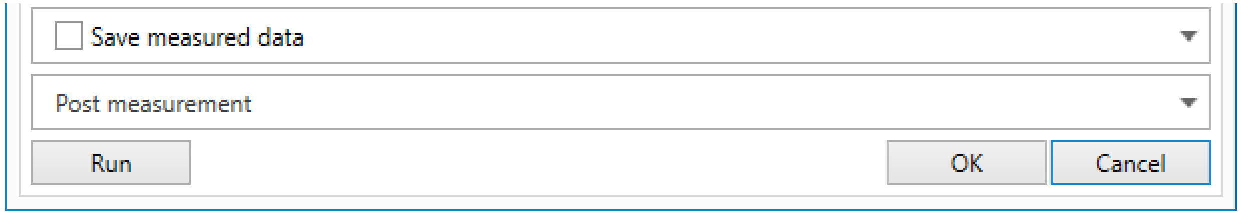




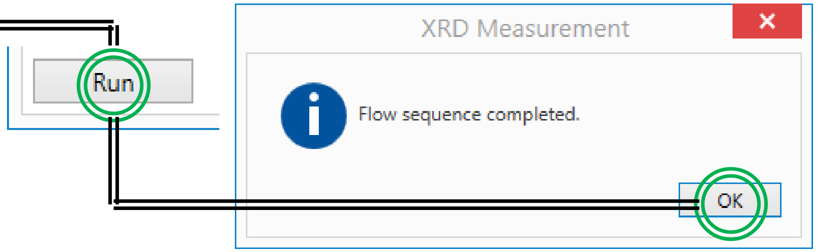


XIV. In-Plane Azimuth or Phi(φ) Scan – 2/2

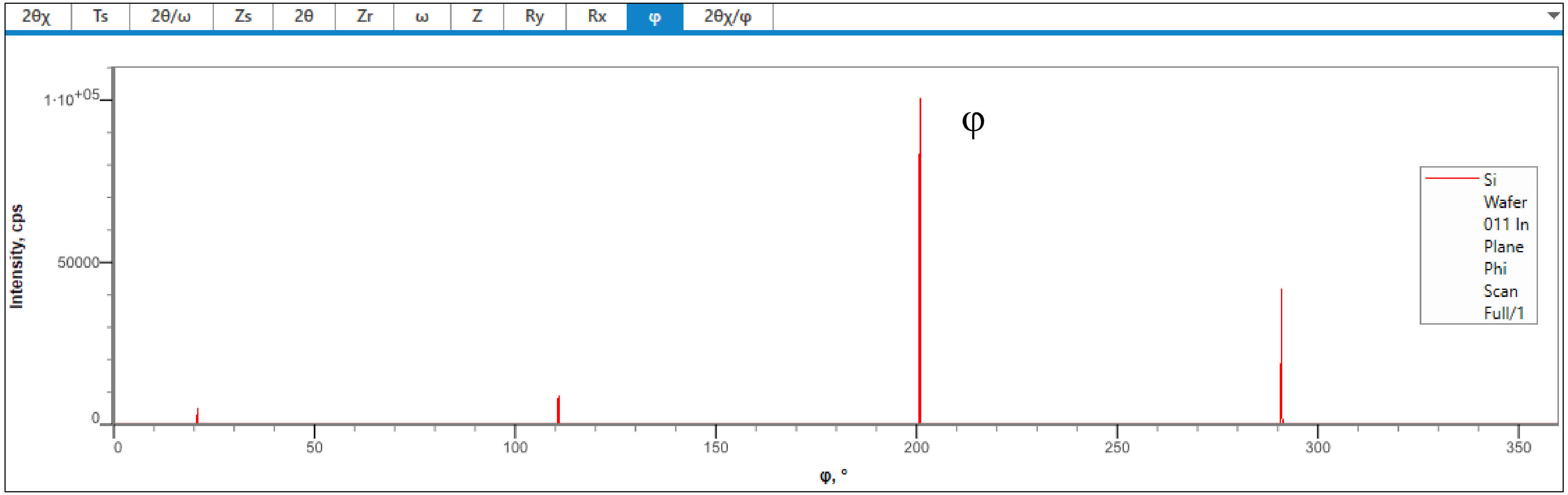
7. Input your desired ***File name*** and ***File location*** here



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



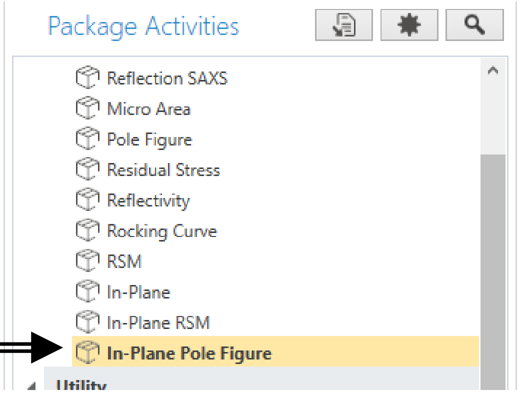
9. If the parameters were chosen properly, you should eventually see a series of peaks appear at the appropriate φ positions

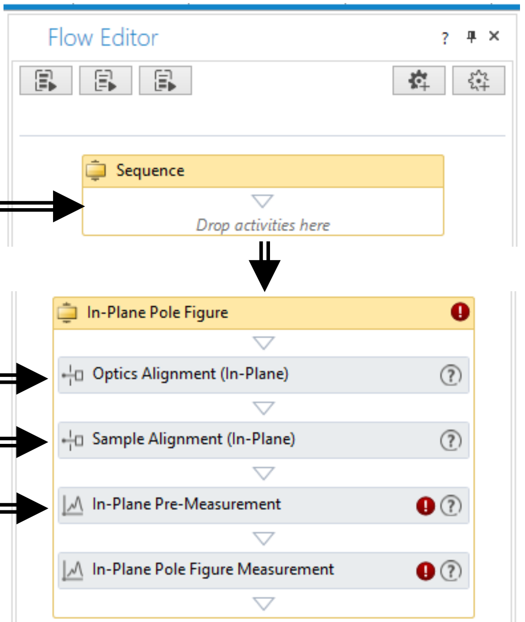


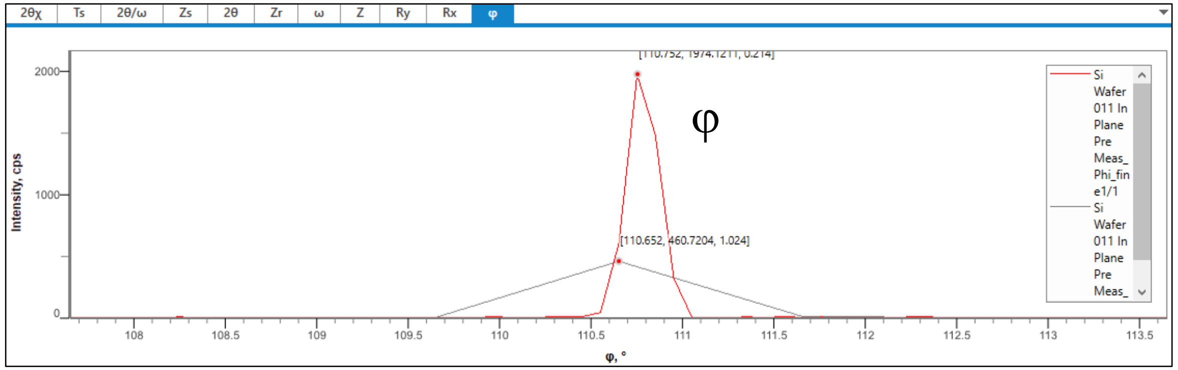
XV. In-Plane Pole Figure – 1/4

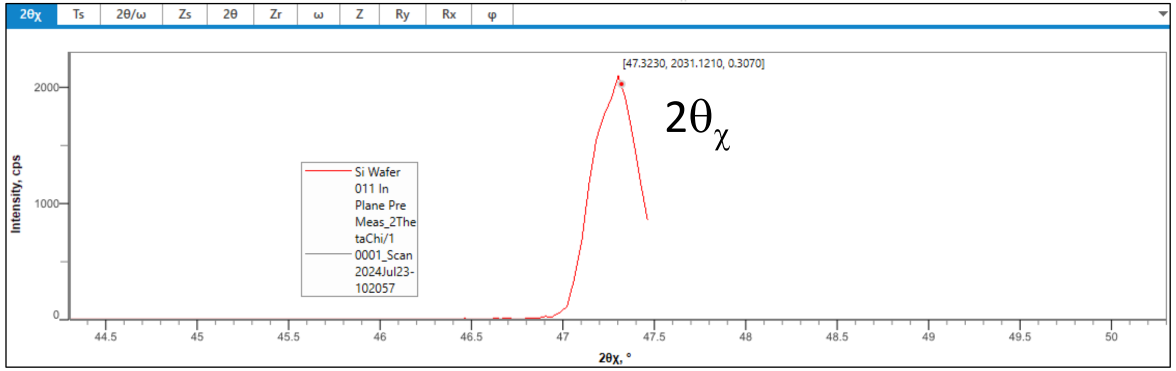
- This sequence will perform an In-Plane Pole Figure using Parallel Beam optics

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 **φ** and **2θχ** should be found



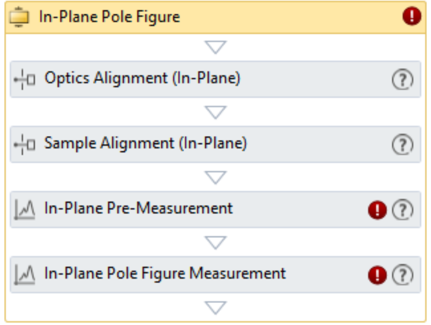




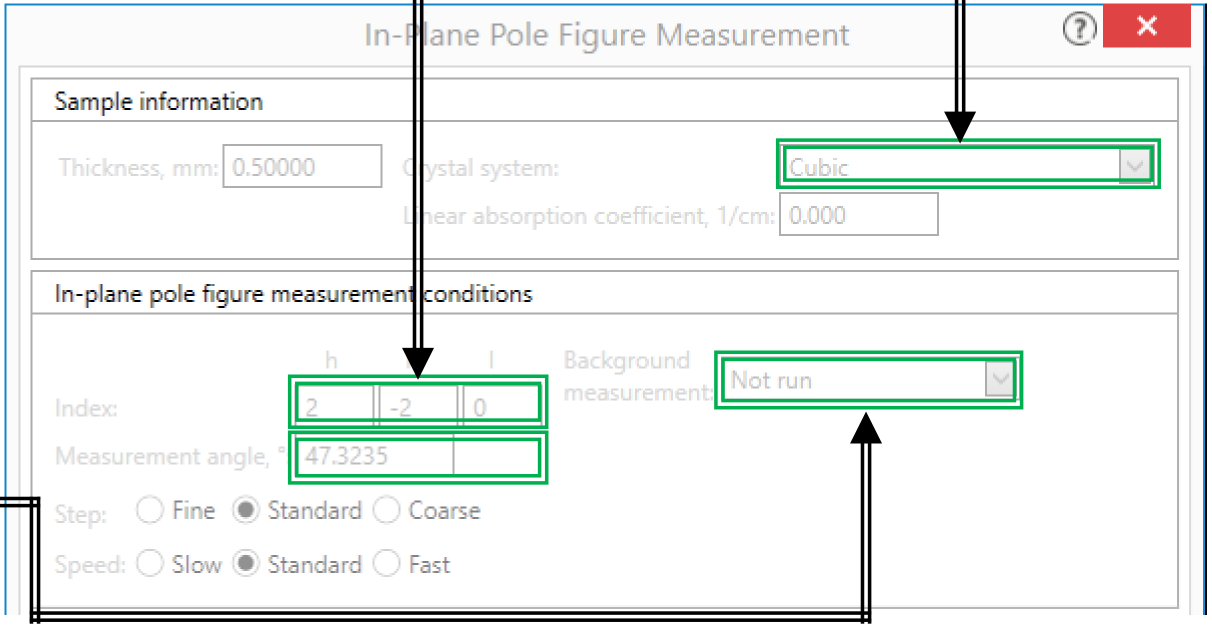


XV. In-Plane Pole Figure – 2/4

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

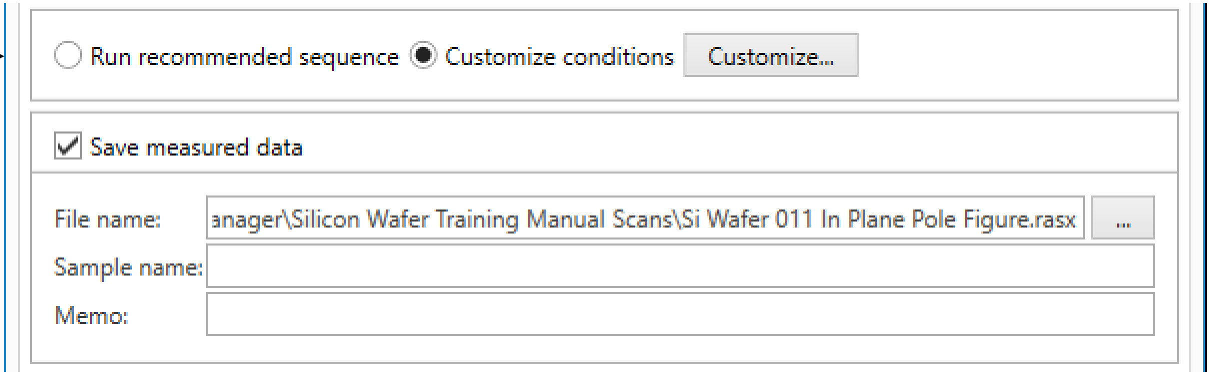


7. Remember to input the following parameters!
   - ***Crystal system*** (i.e. Cubic)
   - ***Index*** (i.e. 2 -2 0)
   - ***Measurement angle*** (i.e. $2\theta_\chi$)



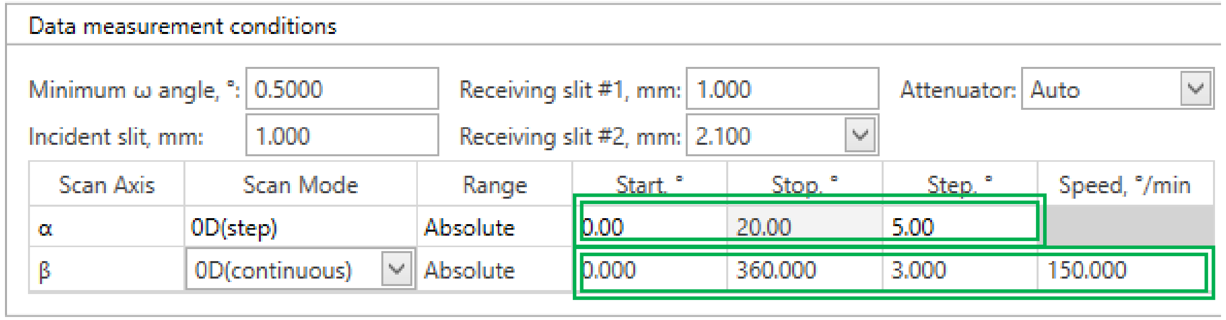
8. Choose if any ***Background measurements*** will be run if desired

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 α scan axes!
    - ***Start***
    - ***Stop***
    - ***Step***
    - ***Speed***



Data measurement conditions

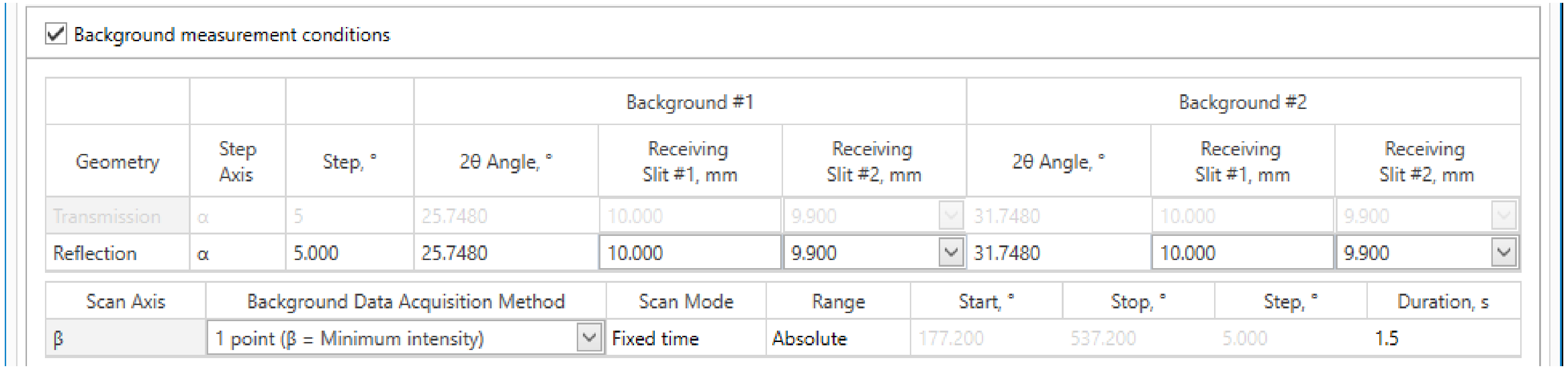
Minimum ω angle, °:	0.5000	Receiving slit #1, mm:	1.000	Attenuator:	Auto
Incident slit, mm:	1.000	Receiving slit #2, mm:	2.100		

Scan Axis	Scan Mode	Range	Start, °	Stop, °	Step, °	Speed, °/min
α	0D(step)	Absolute	0.00	20.00	5.00	
β	0D(continuous)	Absolute	0.000	360.000	3.000	150.000

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

(Note: Will the peaks appear for α and β if arbitrarily chosen?)

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



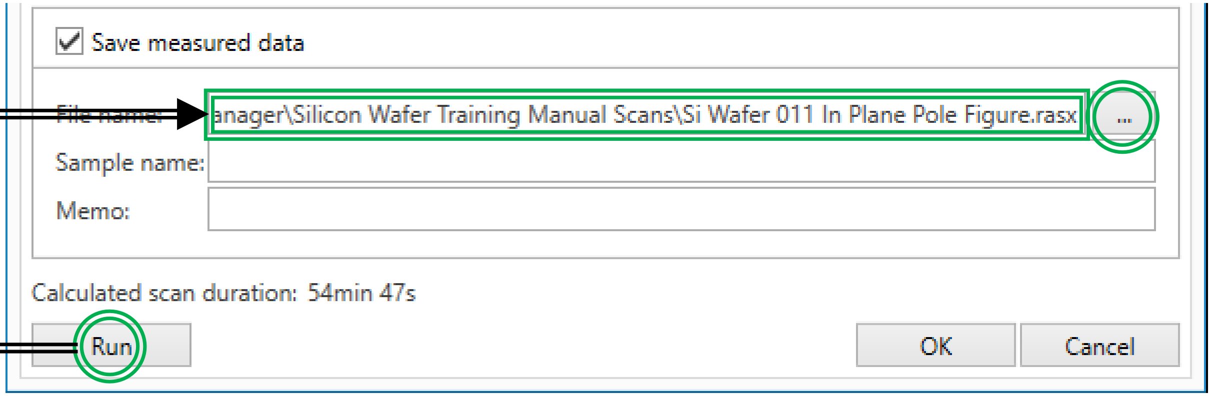
☑ Background measurement conditions

Geometry	Step Axis	Step, °	Background #1 2θ Angle, °	Background #1 Receiving Slit #1, mm	Background #1 Receiving Slit #2, mm	Background #2 2θ Angle, °	Background #2 Receiving Slit #1, mm	Background #2 Receiving Slit #2, mm
Transmission	α	5	25.7480	10.000	9.900	31.7480	10.000	9.900
Reflection	α	5.000	25.7480	10.000	9.900	31.7480	10.000	9.900

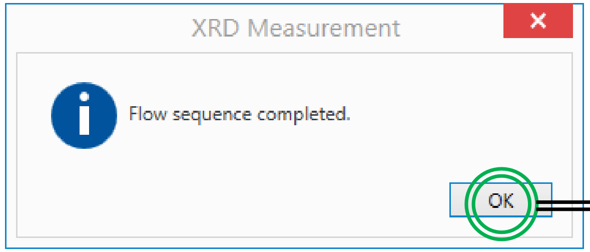
Scan Axis	Background Data Acquisition Method	Scan Mode	Range	Start, °	Stop, °	Step, °	Duration, s
β	1 point (β = Minimum intensity)	Fixed time	Absolute	177.200	537.200	5.000	1.5

XV. In-Plane Pole Figure – 4/4

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

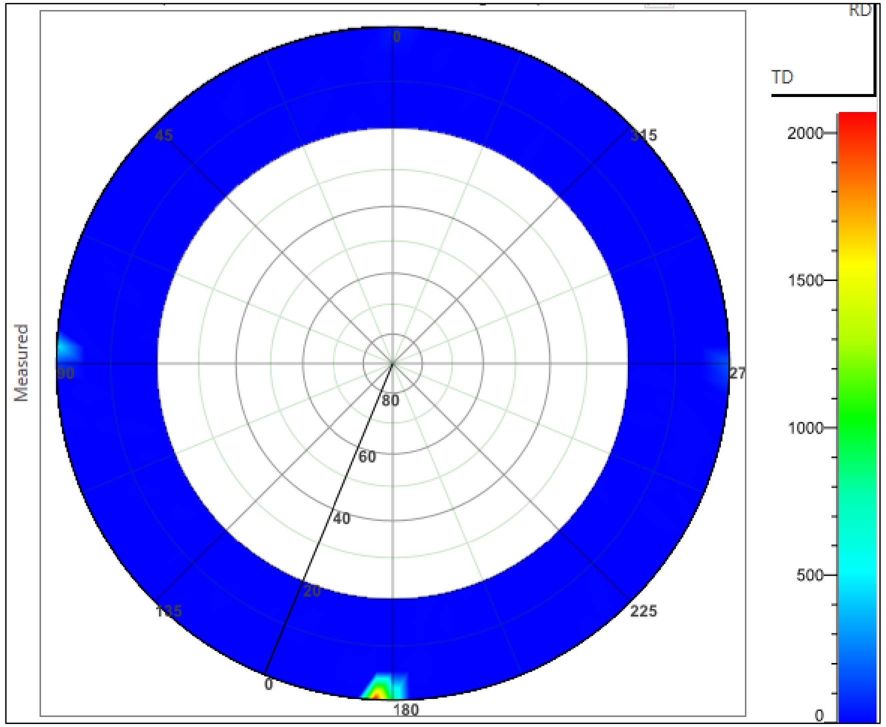


13. Click ***Run*** then ***OK*** when completed



For training with Silicon: Do not Run

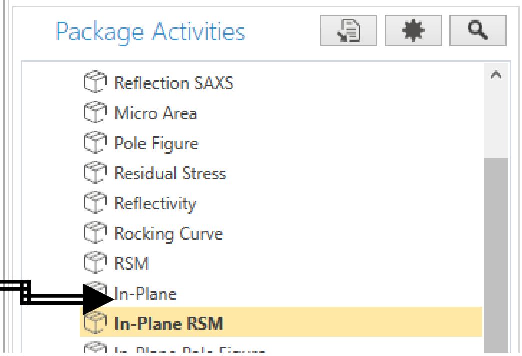
14. If the parameters were chosen properly, you should eventually see a series of peaks appear at the appropriate α and β positions



XVI. In-Plane RSM – 1/3

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

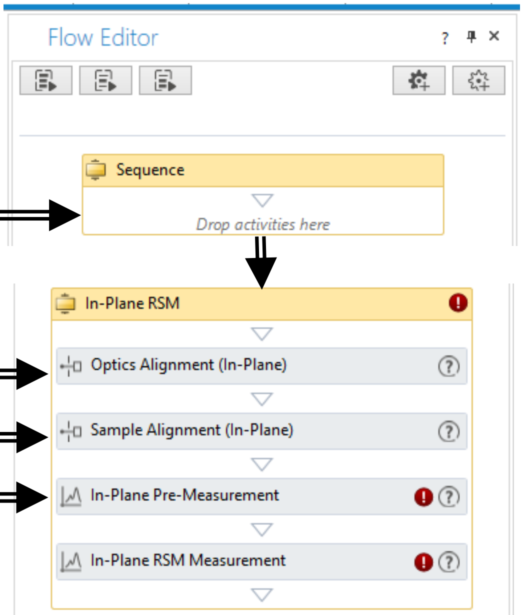
1. Select the ***In-Plane RSM*** under ***XRD Measurement > Package Activities***



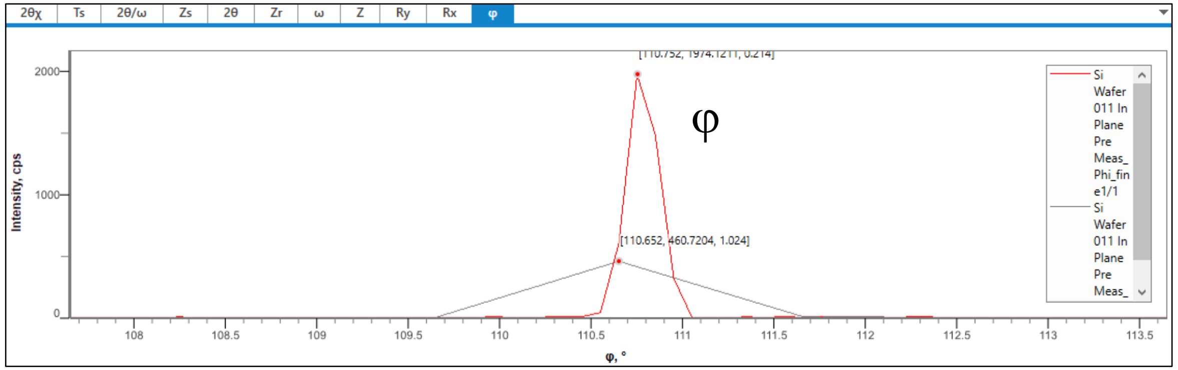
2. Drag the ***In-Plane RSM Figure*** into the ***Flow Editor*** in ***Sequence***

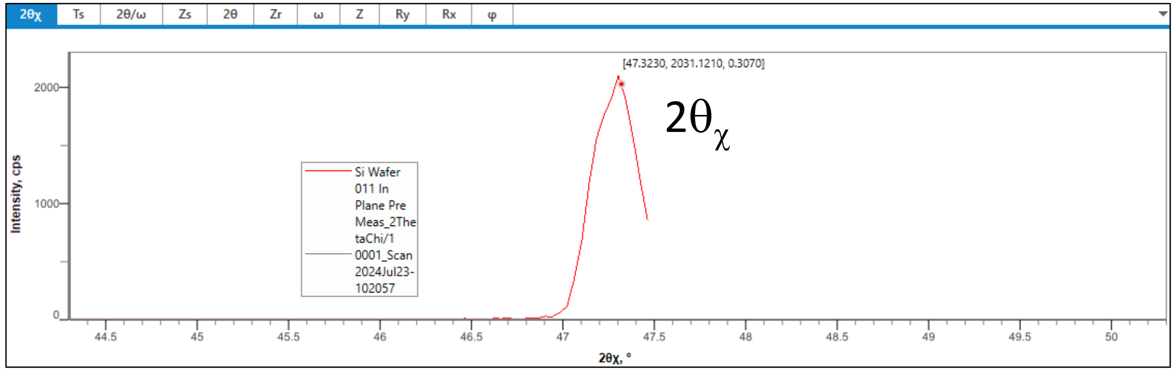
3. If ***Optics Alignment (In-Plane)*** and ***Sample Alignment (In-Plane)*** were previously performed, then skip to ***Step 4***
(see ***XIII. In-Plane Measurement*** for review)

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

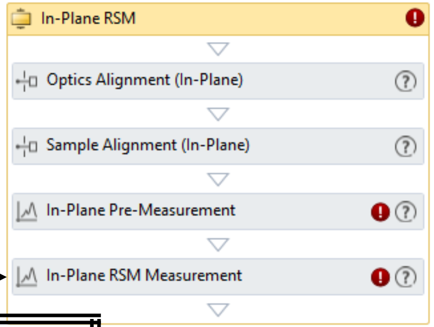


5. Perform the ***In-Plane Pre-Measurement***
(see ***XIII. In-Plane Measurement*** for review) – optimized **φ** and **2θχ** should be found





XVI. In-Plane RSM – 2/3

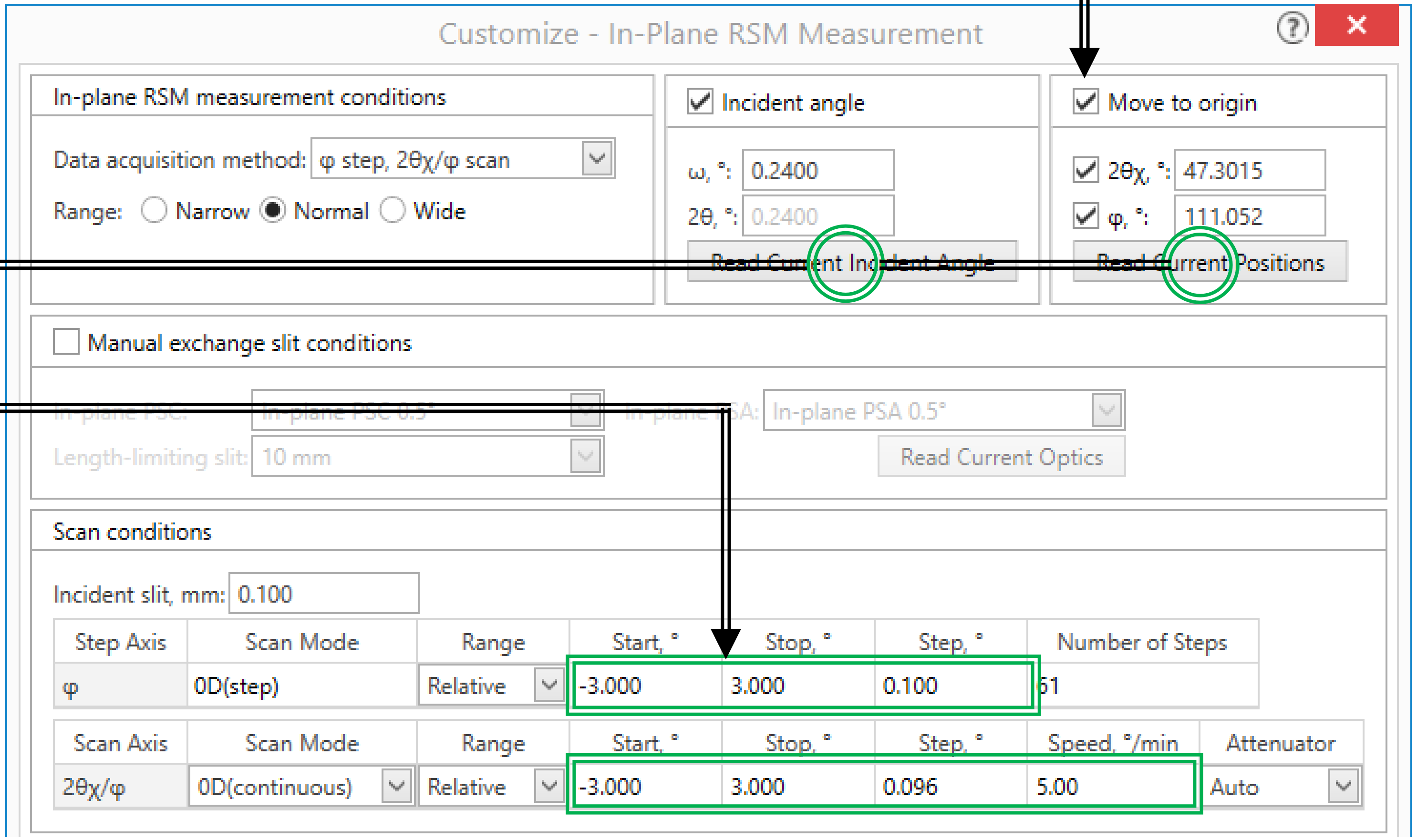


6. Left click ***In-Plane RSM Measurement***

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

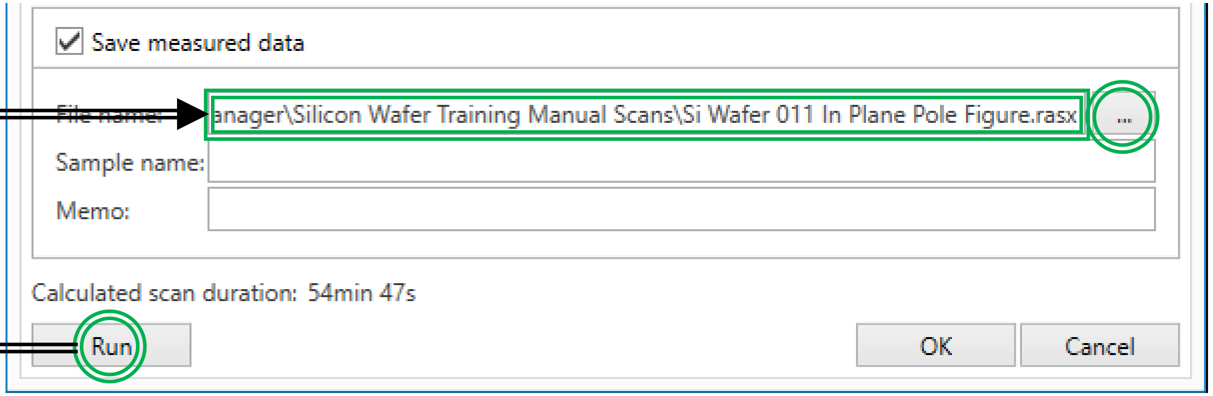
8. Click ***Read Current Positions*** and ***Read Current Incident Angle*** to set the proper ***Origin***

9. Configure the following:
   - ***Start***
   - ***Stop***
   - ***Step***
   - ***Speed***

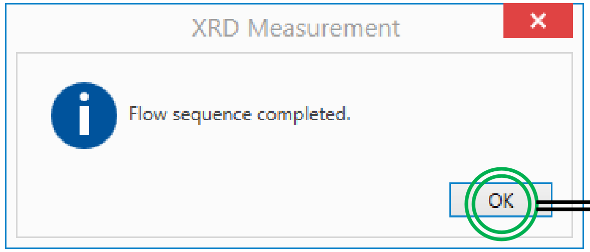


XVI. In-Plane RSM – 3/3

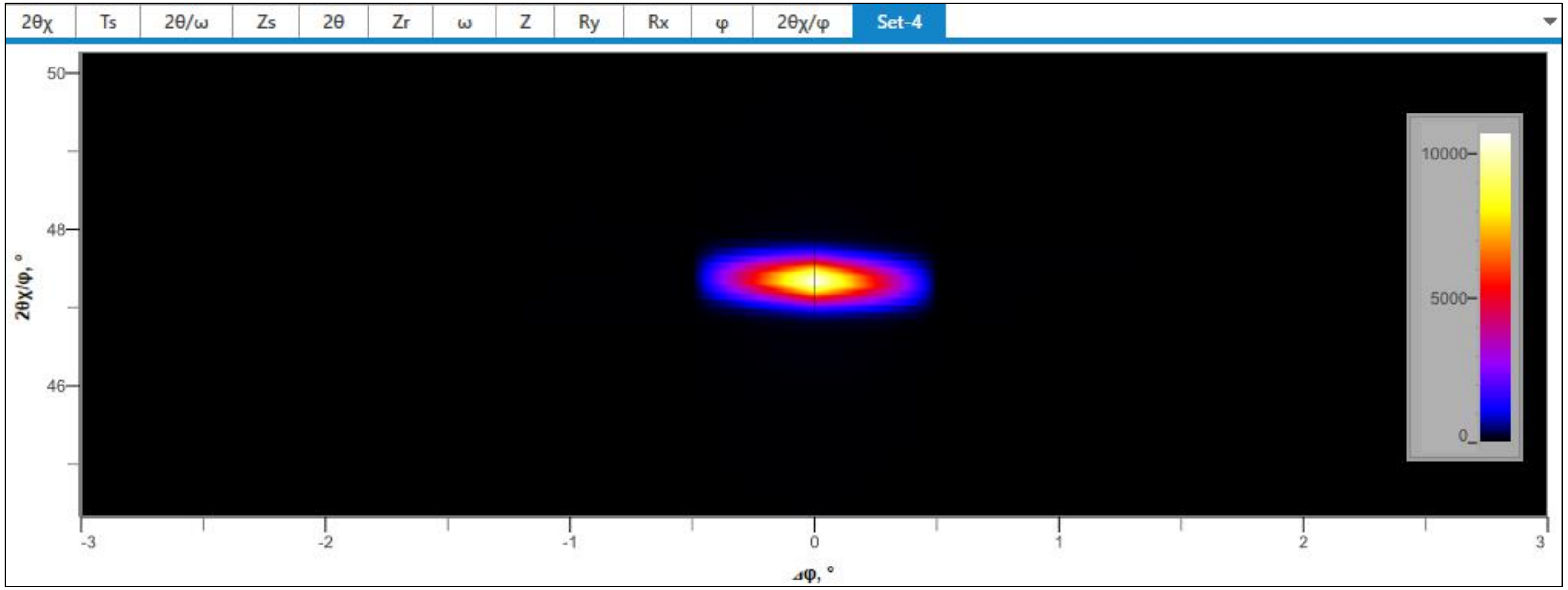
10. Input your desired ***File name*** and ***File location*** here



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



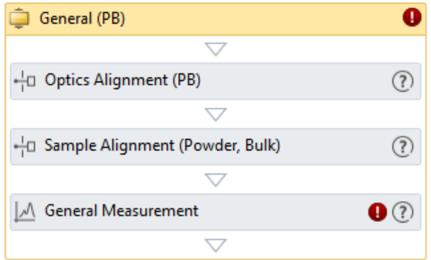
For training with Silicon: Do not Run



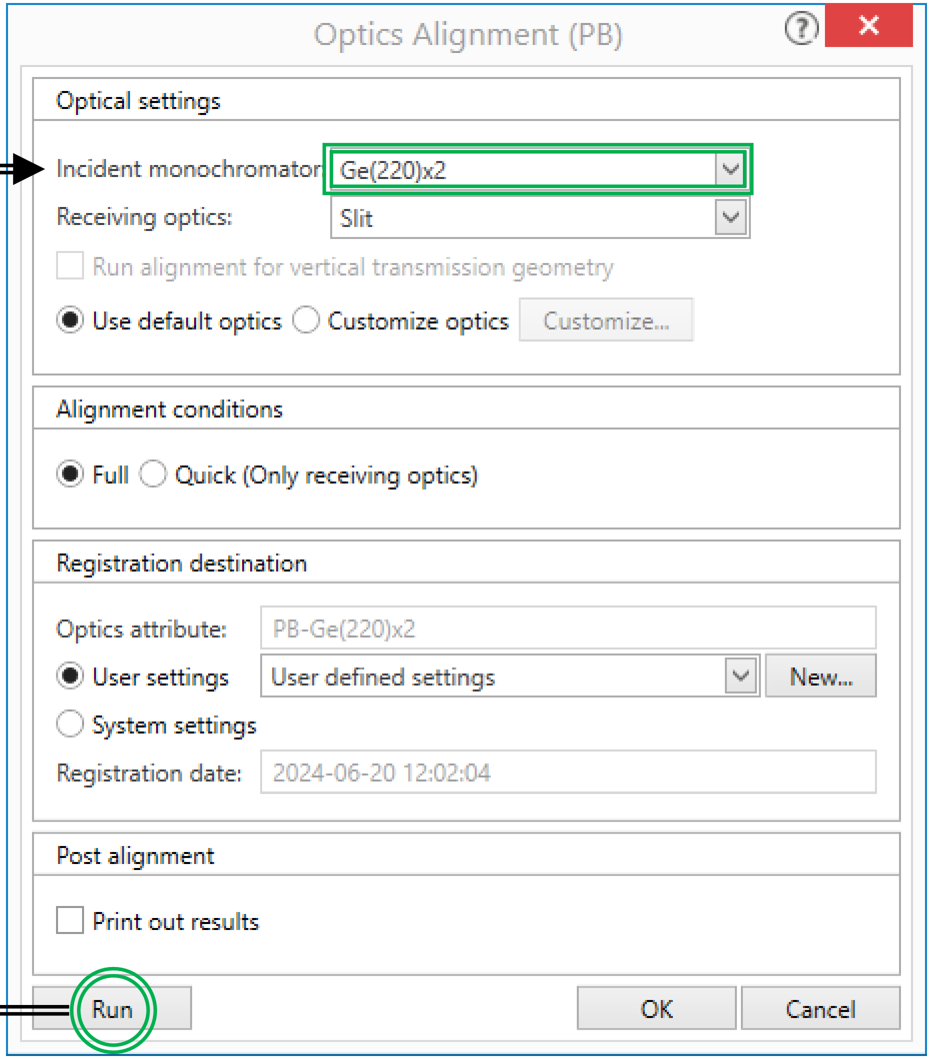
XVII. Monochromator Ge(220)x2 – 1/2

- 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

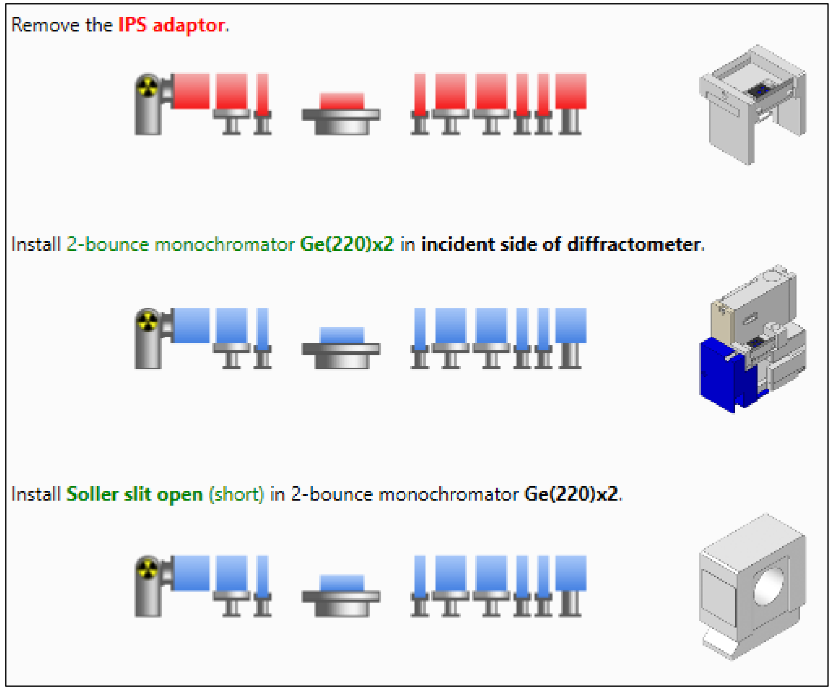


2. Select ***Ge(220)x2*** for the ***Incident monochromator***
3. Confirm ***Use default optics*** is selected under ***Optical settings***
4. Confirm ***Full*** is selected under ***Alignment conditions***
5. Confirm ***User settings*** is selected, then click ***Run***
6. A ***Smart Message*** will 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**)



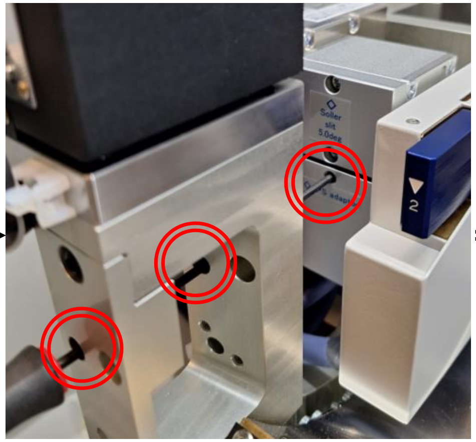
XVII. Monochromator Ge(220)x2 – 2/2

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

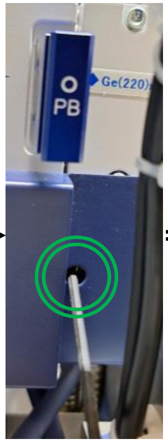


Identify right ***Connector Cables*** first

Loosen set screw to remove ***IPS***



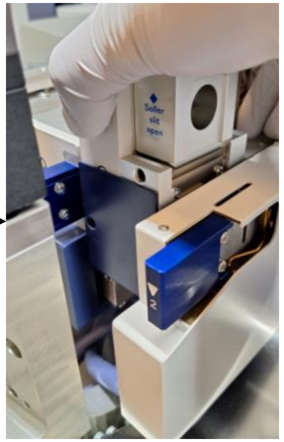
Loosen set screw ***IPS Adapter***



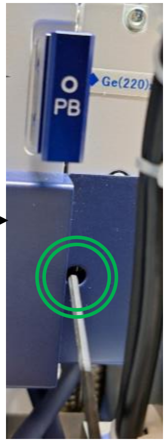
Detach the ***Connector Cables*** for ***IPS adaptor***

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

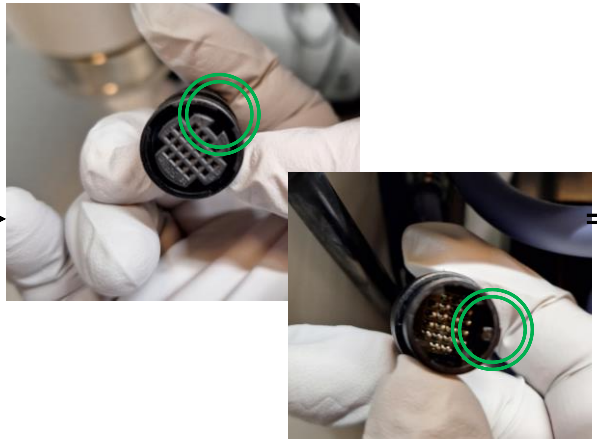
Carefully install ***Ge(220)x2*** – align along the slot



Gently tighten set screws – just ***Snug***



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

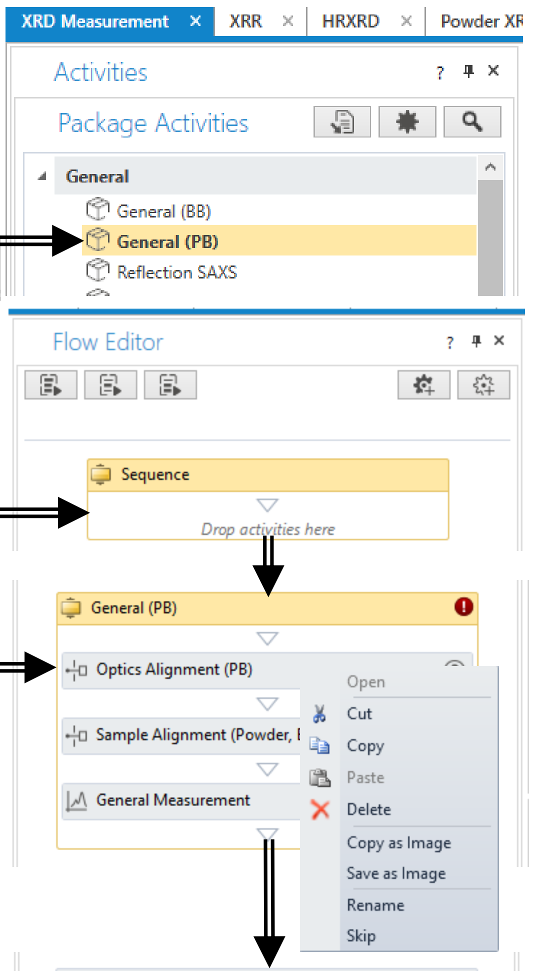


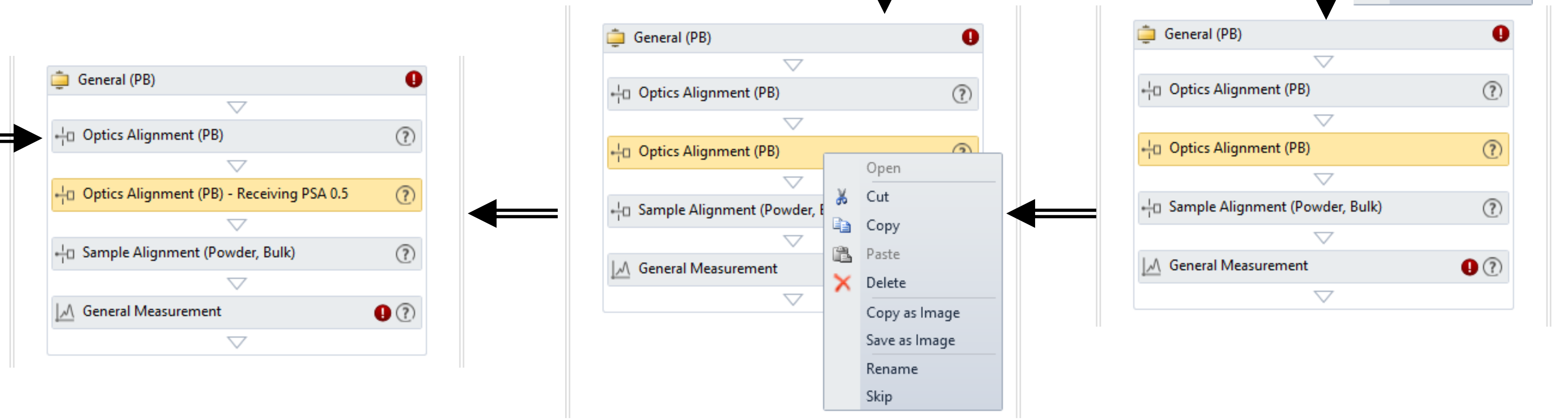
Connect the ***Connector Cables*** for ***Ge(220)x2***

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 ***2nd 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

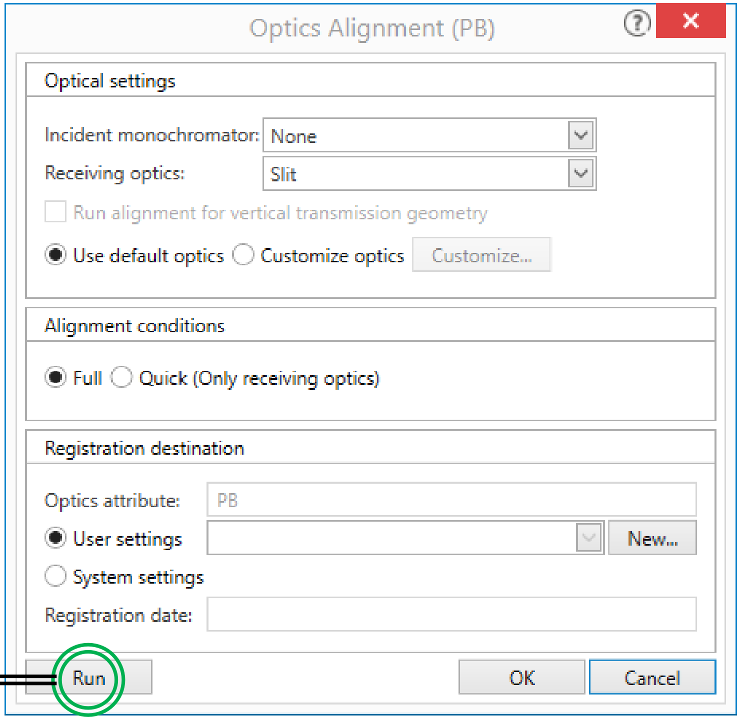
6. Confirm the following are selected:
   - ***Optical settings*** → ***Use default optics***
   - ***Alignment conditions*** → ***Full***
   - ***Registration destination*** → ***User settings***

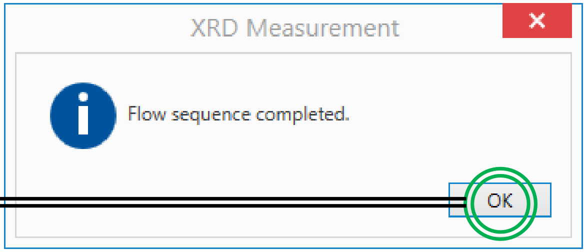
7. Click ***Run***
   and then ***OK*** when completed

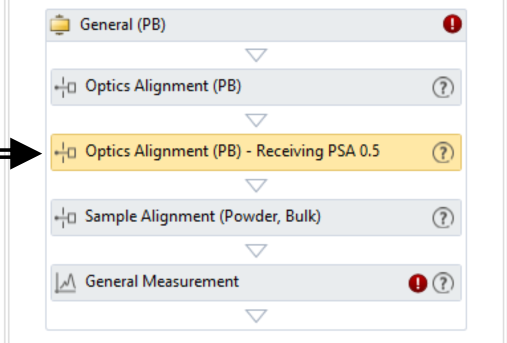
8. Left-click on ***Optics Alignment (PB) – Receiving PSA 0.5*** tab

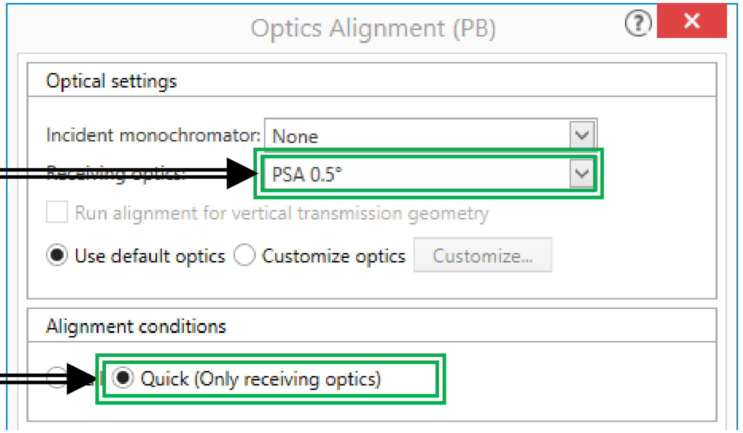
9. Change the ***Receiving optics*** to ***PSA 0.5°***
   and check ***Quick (Only receiving optics)***

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









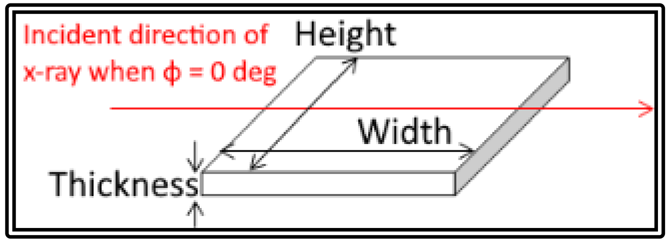
XVIII. Grazing Incidence XRD or GIXRD – 3/5

11. Left-click on ***Sample Alignment (Powder, Bulk)*** to set ***Sample Info***

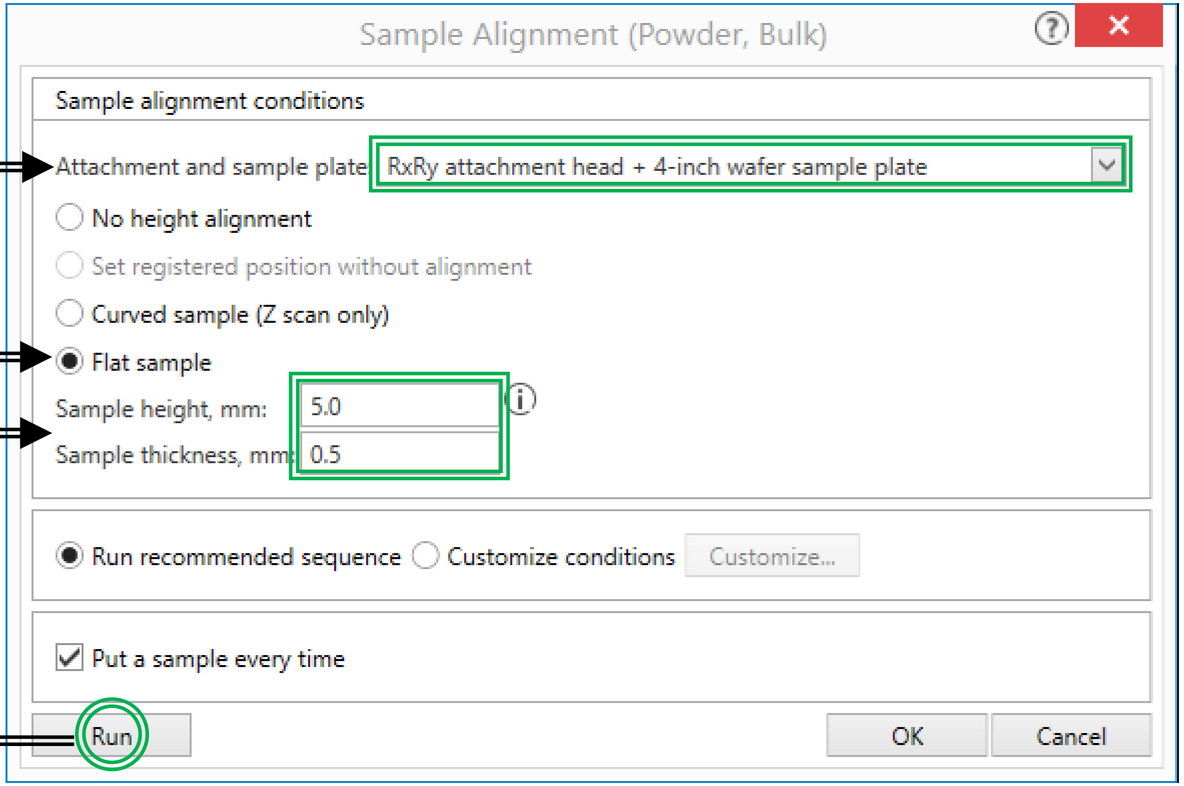
12. Set the ***Attachment and sample plate*** to ***RxRy attachment head + 4-inch wafer sample plate***

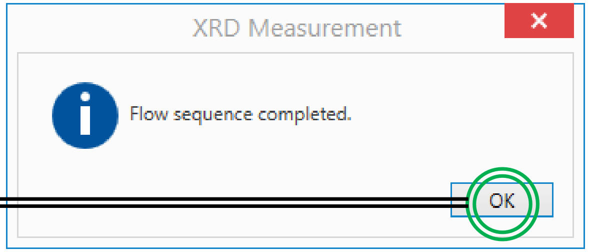
13. Select ***Flat sample***

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



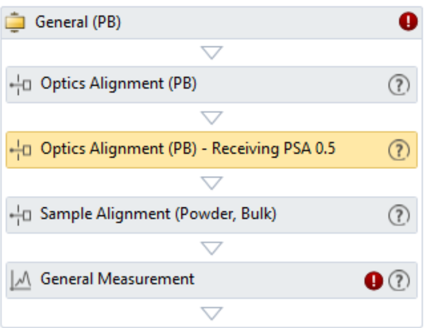
15. Click ***Run*** and then ***OK*** when completed

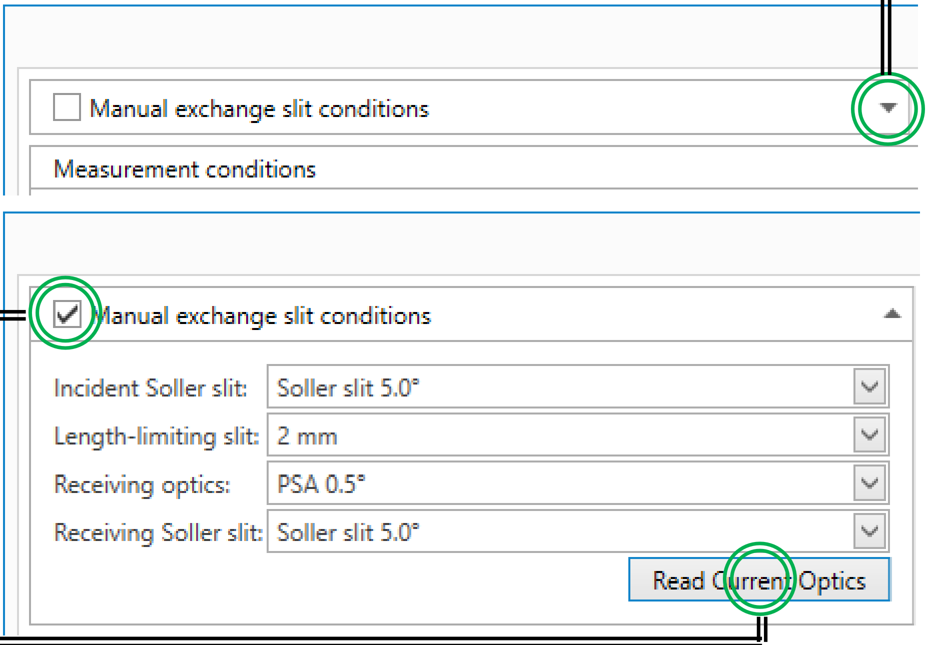


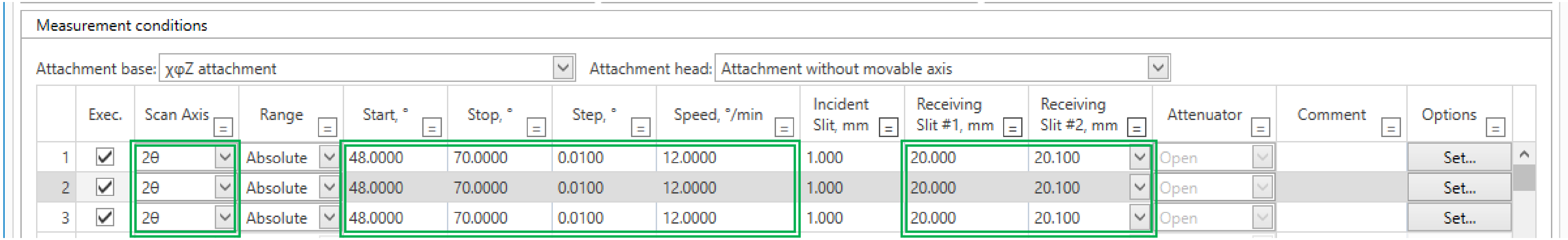


XVIII. Grazing Incidence XRD or GIXRD – 4/5

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 **2θ** 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







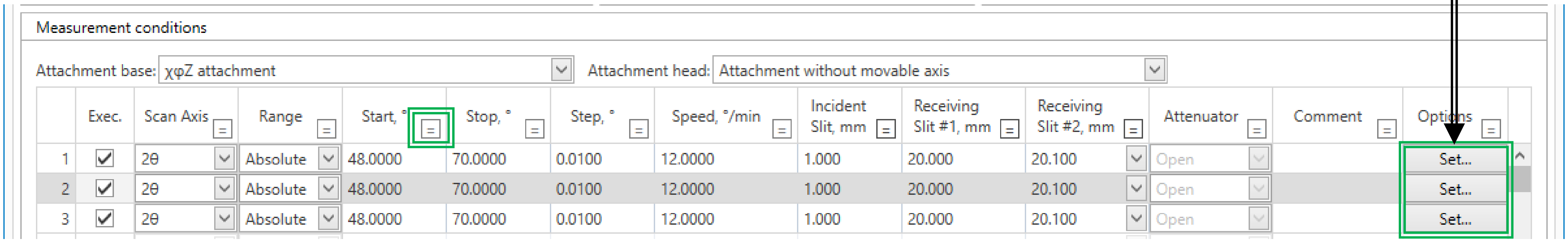
Measurement conditions

Attachment base: χφZ 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	☑	2θ	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
2	☑	2θ	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...
3	☑	2θ	Absolute	48.0000	70.0000	0.0100	12.0000	1.000	20.000	20.100	Open		Set...

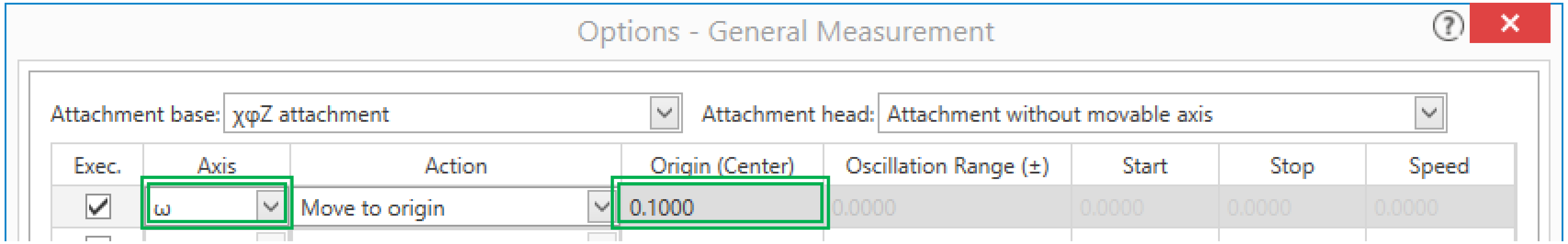
XVIII. Grazing Incidence XRD or GIXRD – 4/5

23. Left-click on ***Set…***



24. Set ω as the ***Axis***

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

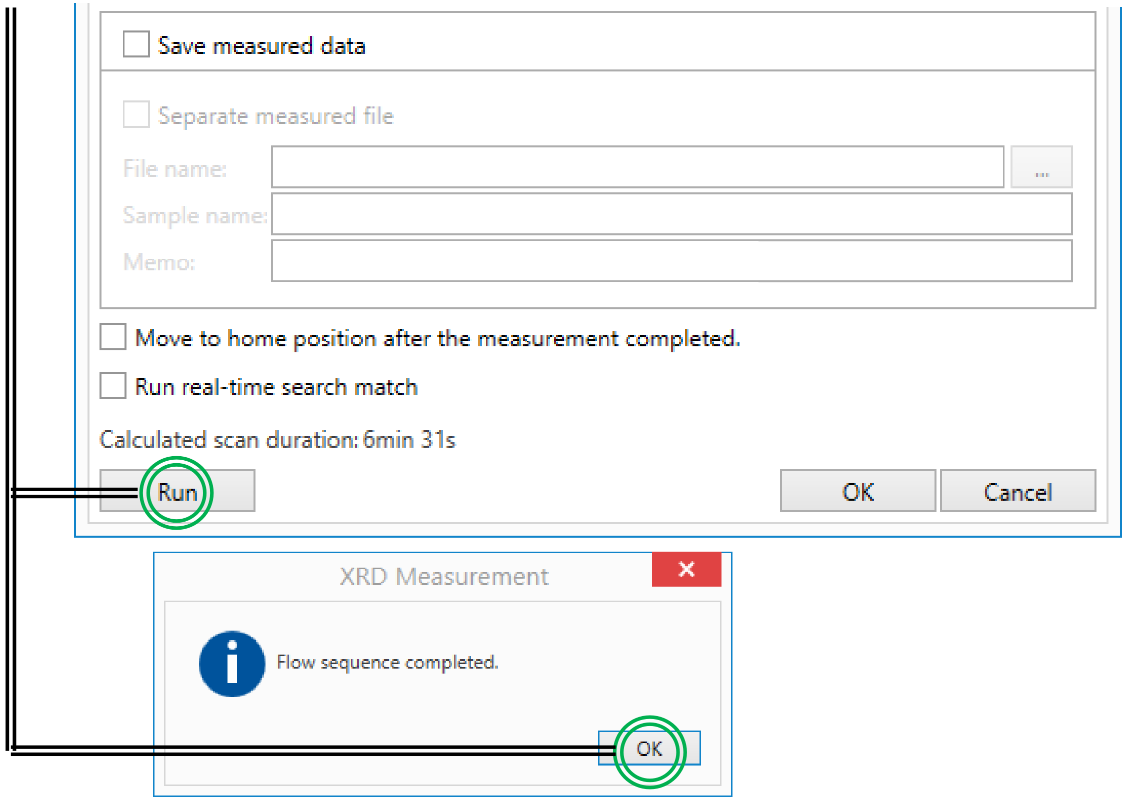


26. You may wish to vary the ω values to see the impact on the angle to your desired peak

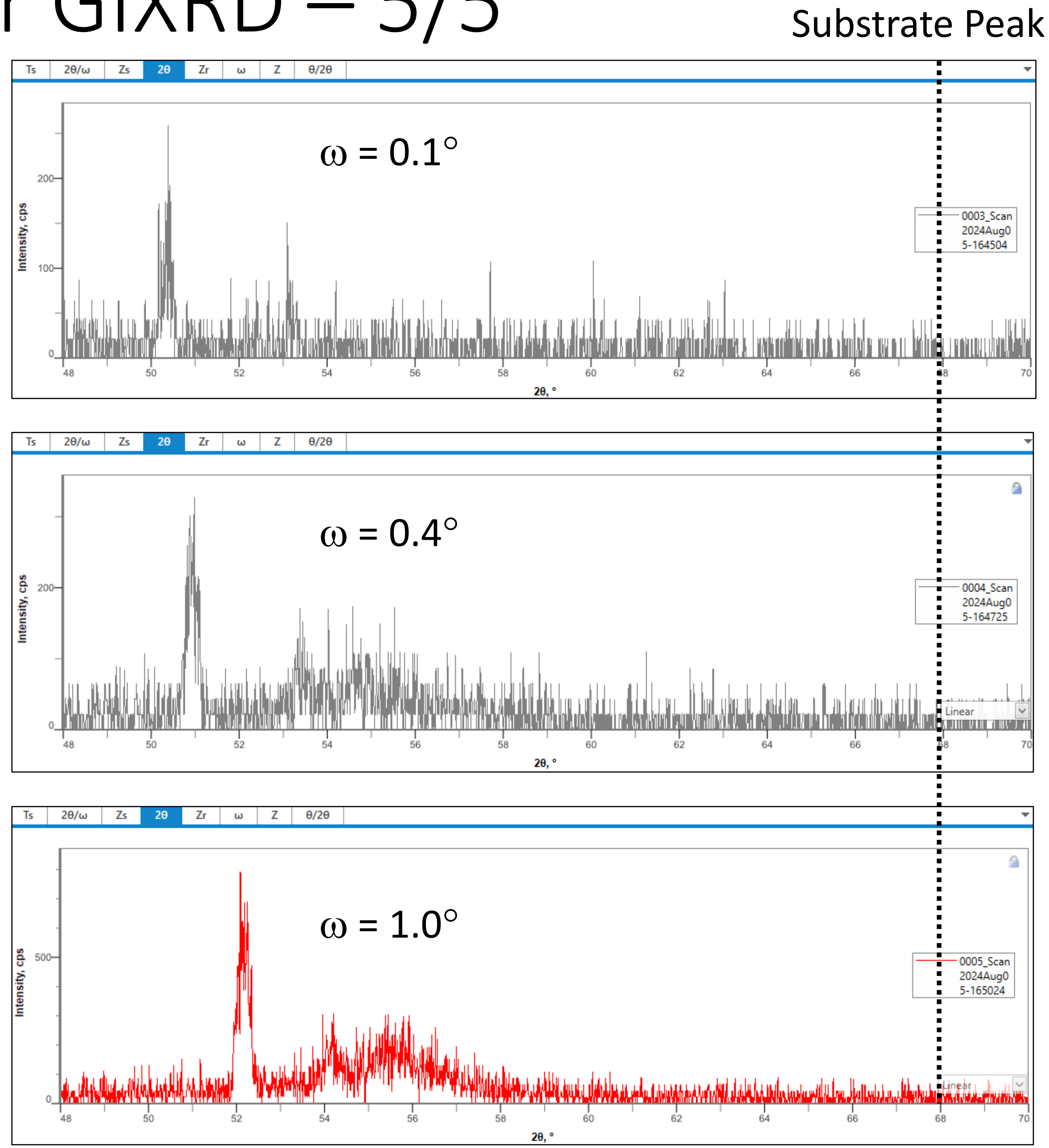
27. Clicking on the [=] box will ***Copy*** the value from the top row if you choose to keep the values the same for each row

XVIII. Grazing Incidence XRD or GIXRD – 5/5

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

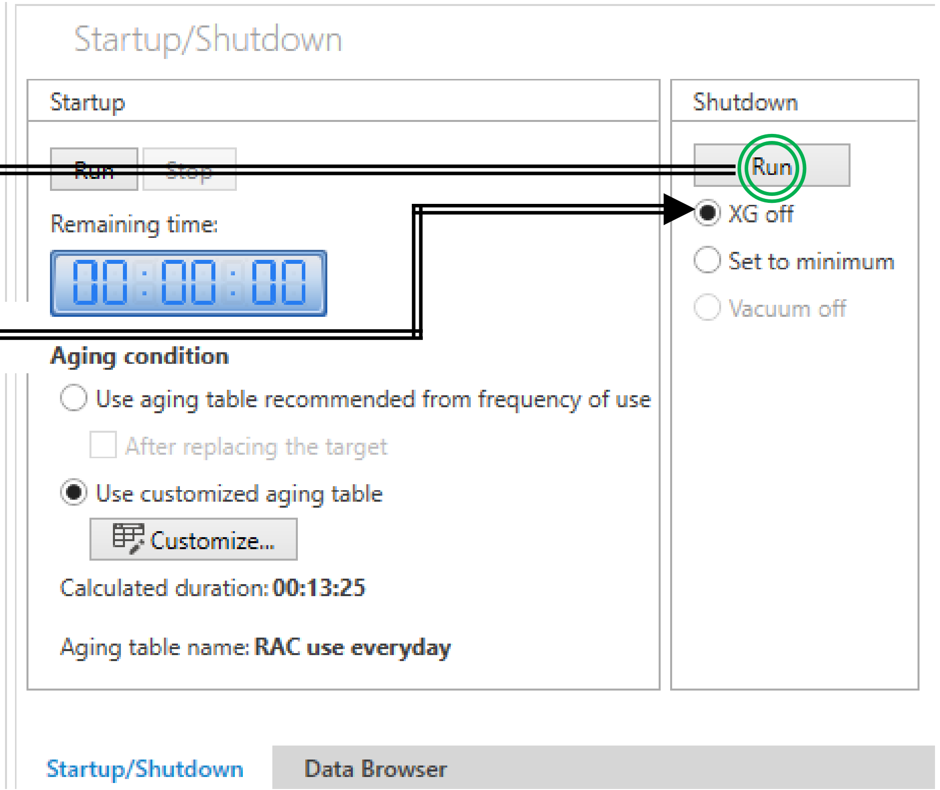


29. You may wish to run different values of ω for comparison

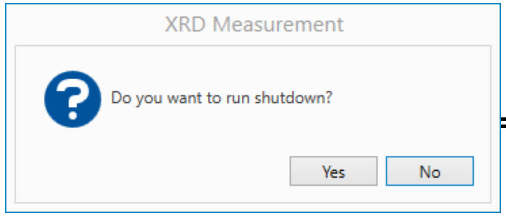


XIX. Clean-up and Shutdown – 1/1

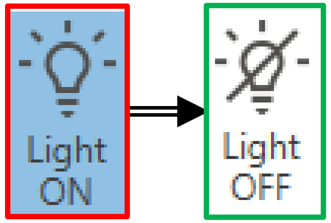
- This sequence is for cleaning up your work and shutting down equipment



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***

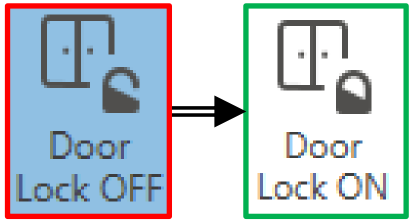


4. Turn off ***Light*** to cabinet by clicking on ***Light ON*** button



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

6. Clean up any materials, samples, or waste from work area
7. Close the ***Doors*** to cabinet, and lock doors by clicking on ***Door Lock OFF*** button

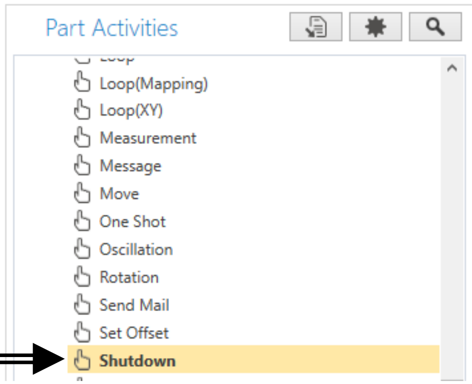


8. Close the ***Software*** by clicking on the ***X*** and record your time and make any notes in the ***Sign-In Sheet***

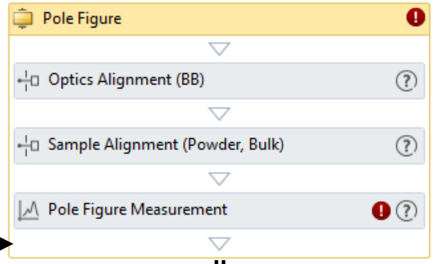
XX. Overnight Scan + Shutdown – 1/1

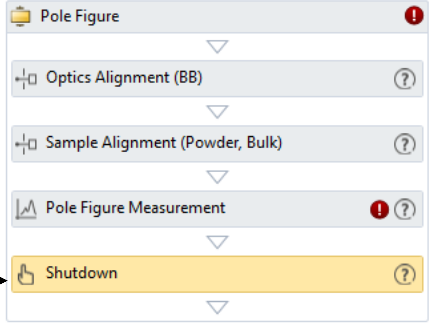
- This sequence is for planning an overnight measurement with a shutdown

1. If you plan on performing an ***Overnight Scan*** and won't return to shutdown manually, you may perform the following steps

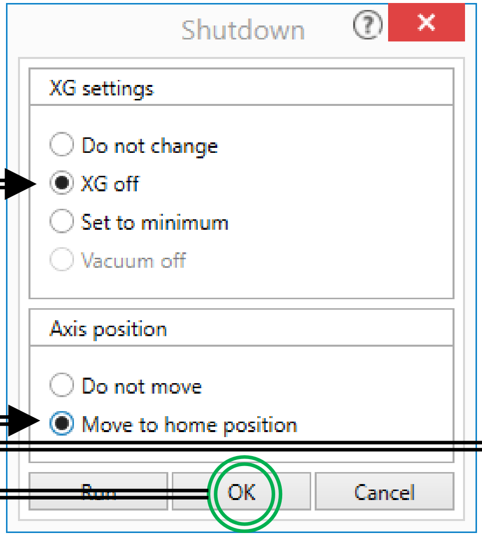


2. Find ***Shutdown*** under ***Part Activities***

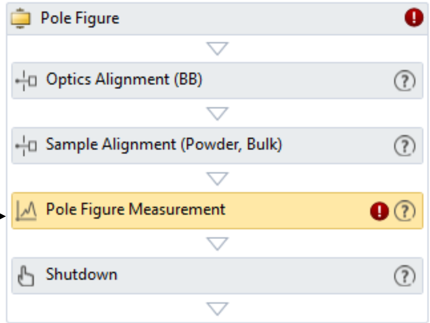




3. Drag ***Shutdown*** under the desired overnight scan (e.g. ***Pole Figure Measurement***)

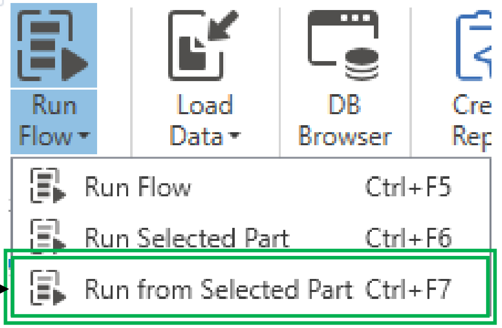


4. Left-click on ***Shutdown*** tab to confirm ***XG off*** and ***Move to home position*** are checked and click ***OK***



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!** (see ***XIX. Cleanup and Shutdown Steps 6 – 8***)

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

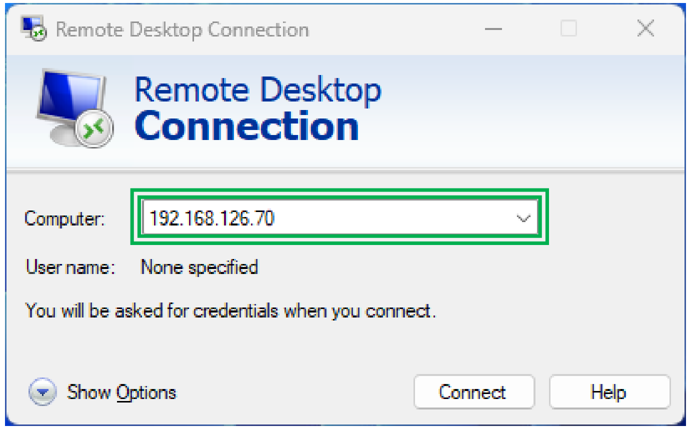
3. Cabinet will perform ***Power Up*** sequence

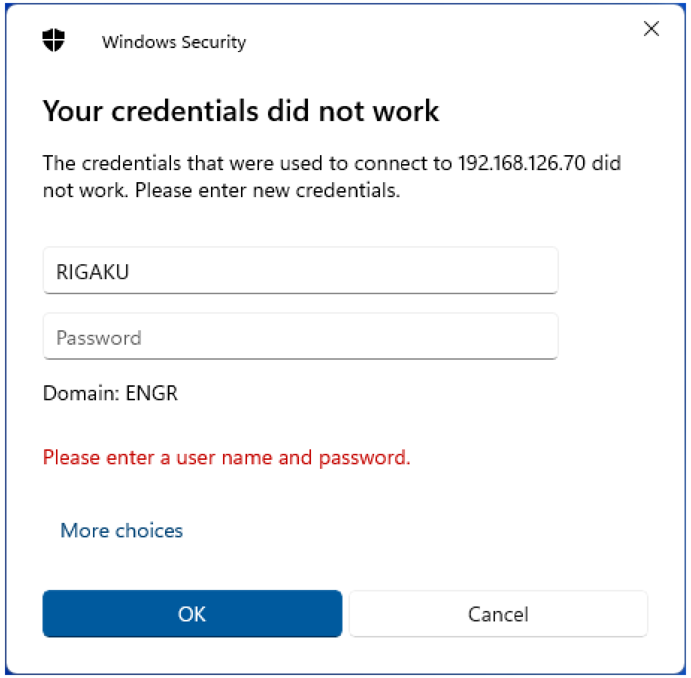
4. The ***Power ON*** lights will illuminate

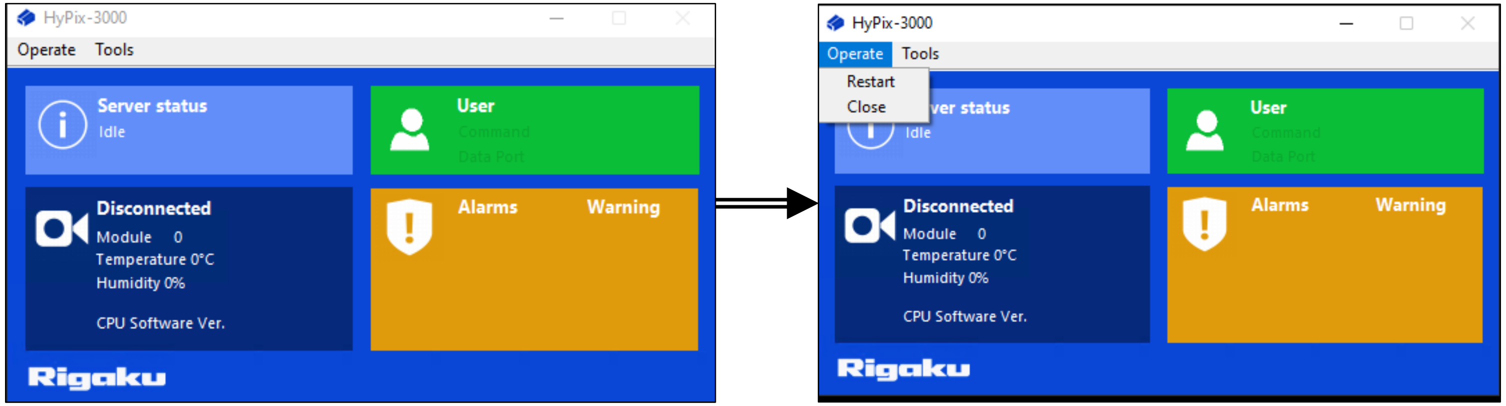
TS-B. Hypix Detector Troubleshooting

○ 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