REFLECTIVITY
(Film thickness between 1 and 200 nm)

1. Log into the User Log System on the SMIF web site

Hardware Setup

X-Ray Tube

The line focus configuration of the x-ray tube is used. This is the default configuration of the system and no changes are needed in the X-ray source for this procedure.

2. ***Incident Beam Optics***

- Mount the x-ray mirror on the incident beam PREFIX. Make sure that the attenuator cable is plugged into the mirror.
- Insert the 1/32 degree divergence slit in the mirror. (The 1/16 degree slit can be used if greater intensity is needed)
- If the sample height is less than 25 mm insert an appropriate beam mask.

3. ***Diffracted Beam Optics***

- Mount the parallel plate collimator (PPC) or the Programmable Receiving Slit (PRS) on the diffracted beam PREFIX
    a. The Parallel Plate Collimator (PPC) works well with films less than 100nm thick.
    b. The Programmable Receiving Slit (PRS) has the flexibility of adjusting the slit size from 10 um up to 2 mm. Therefore it works well with films thicker than 100 nm, which usually require a higher resolution and thus a smaller receiving slit width.

- If the parallel plate collimator (PPC) is used:
  Make sure its collimator slit (0.27) is placed in front of detector and that the 0.04 rads soller slits are behind the collimator and that detector 1 is present.
- If the programmable receiving slit (PRS) is used:
  Make sure that the 0.04 rads soller slits are behind the collimator and that detector 1 is present.

4. Close the enclosure doors

- The doors must be closed when starting the software

User Setup

5. Open the **X'Pert Data Collector** program.

6. Enter your user name and password.

7. Select **Instrument/Connect** from the pull down menu. The Connect box will appear.

8. Select the "Generic" configuration. Click the OK button

9. Click the OK button to close the status messages that appear

10. If it appears, click Yes on the message box that asks if you want to apply the sample offsets

Optics Setup

11. Select the **Incident Beam Optics** tab. Double click on any item in the list to open the incident beam optics settings window.

    Select or verify the following settings by clicking on the appropriate tab:

    - **Prefix module**: Mirror Cu W/Si (parabolic MRD)
    - **Divergence slit**: Fixed Slit 1/32°
    - **Anti Scatter slit**: None
    - **Mask**: Select the beam mask size you are using (or None if not using a mask)
    - **Mirror**: Inc. Beam Cu W/Si (parabolic MRD)
    - **Monochromator**: None
    - **Beam Attenuator**:
        - **Type:** Progr. beam attenuator Ni 0.125mm
        - **Attenuation Factor:** 181.00 (Click Select to change)
        - **Usage:** Do Not Switch (for sample alignment)
        - **Activated Box:** selected (check mark appears)
    - **Filter:** None
    - **Soller Slit:** None

    Click OK to apply these settings and close the window

12. Select the **Diffracted Beam Optics** tab. Double click on any item in the list to open the diffracted beam optics settings window.
    Select or verify the following settings by clicking on the appropriate tab:
    - **Prefix module**: Select the incident beam optics you are using:
        - Parallel Plate Collimator 0.27° or
        - Programmable receiving slit with FASS
    - **Anti Scatter slit**: None
    - **Receiving slit**: Select depending on the incident beam optics you are using:
        - PPC: Parallel Plate Collimator Slit
        - PRS: Set to desired value (in mm)
    - **Collimator**: Select depending on the incident beam optics you are using
        - PPC: Parallel Plate Collimator 0.27°
        - PRS: None
    - **Detector**:
        - **Type:** Proportion detector Xe[1] (Make sure detector [1] is selected)
        - **Used wavelength:** K-alpha
    - **Mirror:** None
    - **Beam attenuator:** None
    - **Filter:** None
    - **Mask:** None
    - **Soller Slit:** Soller 0.04 rad.
    - **Monochromator:** Select depending on the incident beam optics you are using
        - PPC: None
        - PRS: Diffracted beam monochr. For Cu (curved crystal)

    Click OK to apply these settings and close the window

13. Select the Instrument Settings tab. Double click on the Generator settings. Set the generator to:
    - **Tension**: 45 kV
    - **Current**: 40 mA

    Click OK to apply these settings and close the window

Sample Mounting and Alignment

(Using the Beam Bisection Method)

14. Select the **Instrument Settings** tab and double click on an item related to the **Positions**.
    For the first sample:
    - Enter 0 in all of the coordinate fields, except set Z = 0.05
    - Click the OK button to move the stage

    For subsequent samples
    - Enter 0 in all of the coordinate fields except for Z (leave Z at the same value as the previous sample)
    - Click the OK button to move the stage

15. ***For the first sample:***
    - Open the enclosure doors and mount the sample as flat as possible on the stage. After mounting the sample, close the enclosure doors. Skip to step 16.

    For subsequent samples:
    - Open the enclosure doors and remove the previous sample, then mount the next sample as flat as possible on the stage.
    - Close the enclosure doors
    - Select the incident beam optics tab and double click on any item. Make the following changes in the incident beam optics settings window:
        - **Beam Attenuator**:
            - **Usage:** Do Not Switch (for sample alignment)
            - **Activated Box:** selected (check mark appears)

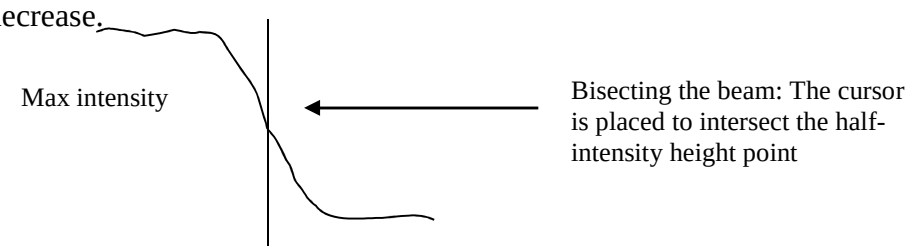
        Click OK to apply these settings and close the window
    - Skip to Step 18

16. <u>Detector (2theta) Alignment (</u>***Only Performed for the First Sample)***
    - Select **Measure/ Manual** Scan from the main menu.
    - Enter the following parameters in the Manual Scan window:
        - **Scan Axis**: 2theta
        - **Range**: 2.0
        - **Step Size**: 0.01
        - **Time per step**: 0.1
    - Click the start button to begin the scan
    - After the measurement is completed, you will find the highest peak in the scan data and move the goniometer to this position. If two main peaks are present, choose the midpoint between the two peaks**.**
        - Press the right mouse button and select **Move mode**
        - Press and hold the left mouse button and move the cursor to place the green line at the maximum of the peak (or the midpoint between the two main peaks). This will move the goniometer to the selected position.
    - Select the User Settings/ Sample Offsets from the main menu. Enter 0 in the *2theta* field. Press *OK***.**

17. <u>Move Sample Height (Z) to an Initial Value</u> **(*Only Performed for the First Sample)***
    - Select the **Instrument** Settings tab and double click on an item related to the **Positions.** Enter an approximate starting position for the Z height. (e.g., 7.5mm for a Si wafer) Click OK to move the sample stage.

18. <u>Sample Height (Z) Alignment</u>
    - Enter the following parameters in the Manual Scan window:
        - **Scan Axis**: Z
        - **Range**: 2.0
        - **Step Size**: 0.01
        - **Time per step**: 0.1
    - Click the start button to begin the scan

- The resulting scan should look like two plateau regions connected through a gradual decrease.



- Press the right mouse button and select Move mode
- Press and hold the left mouse button and move the cursor to place the green line in the middle of the plateau regions where the intensity is half of the full intensity. This will move the sample to bisect the main beam**.**

19. <u>Sample Tilt (Omega) Alignment</u>
    - Enter the following parameters in the Manual Scan window:
      - **Scan Axis**: Omega
      - **Range**: 4.0
      - **Step Size**: 0.01
      - **Time per step**: 0.1
    - Click the start button to begin the scan
    - After the measurement is completed find the peak in the scan data and move the goniometer to this position
      - Press the right mouse button and select **Peak Mode**
      - The software will automatically determine the peak position.
      - Press the "*move to*" button to move the goniometer to the selected position, then click close.
    - Select User Settings/Sample Offsets from the main menu. Enter zero into the *omega* field. Close the window.
    - Repeat another Z scan as before and move the sample to bisect the main beam. You do not have to repeat another omega scan after the second Z scan**.**

Optimizing Reflected Intensity

20. Select the **Instrument Settings** tab and double click on any item related to the **Positions**
    - Enter 1.6 for the 2theta value
    - Click *OK.*

21. Back in the **Manual Scan** window, enter the following parameters:
    - **Scan Axis**: 2theta/omega
    - **Range**: 3.0
    - **Step Size**: 0.005
    - **Time per step**: 0.2
    - Click the start button to begin the scan

22. Select **Axes** and *logarithmic* scale.

23. After the measurement is completed:

- Right click on the plot, select **Axes** and **Logarithmic** scale
- Press the right mouse button and select **Move mode**
- Press and hold the left mouse button and move the cursor until the green line is placed over the maximum intensity of the first (lowest angle) clearly visible fringe. This will move the goniometer to the selected position. (If no fringes are visible just pick a spot to the right of the critical angle.)

24. Back in the **Manual Scan** window, enter the following parameters to optimize omega:

- **Scan Axis**: omega
- **Range**: 1.0
- **Step Size**: 0.005
- **Time per step**: 0.2
- Click the start button to begin the scan

25. After the measurement is completed:

- Right click on the plot, select **Axes** and **Linear** scale
- Press the right mouse button and select **Move mode**
- Press and hold the left mouse button and move the cursor until the green line is placed over the peak in the scan. If two broad low intensity peaks are present, reflectivity on this sample will not be possible due to high interface roughness. If three peaks are present, with a sharp peak in the middle, select the position of the sharp peak in the middle.

26. Back in the **Manual Scan** window, enter the following parameters to optimize Chi:

- **Scan Axis**: chi
- **Range**: 10.0
- **Step Size**: 0.05
- **Time per step**: 0.2
- Click the start button to begin the scan

27. After the measurement is completed:

- Right click on the plot, select **Axes** and **Linear** scale
- Press the right mouse button and select **Move mode**
- Press and hold the left mouse button and move the cursor until the green line is placed over the peak in the scan

28. Back in the **Manual Scan** window, enter the following parameters to re-optimize omega now that chi has been optimized:

- **Scan Axis**: omega
- **Range**: 1.0
- **Step Size**: 0.005
- **Time per step**: 0.2
- Click the start button to begin the scan

29. After the measurement is completed:

- Press the right mouse button and select **Move mode**
- Press and hold the left mouse button and move the cursor until the green line is placed over the peak in the scan
- If the new omega value is within 0.1 degrees of the previous omega value, continue with step 30. If the difference is greater than 0.1 degrees, go back to step 26-29 to reoptimize chi and omega.

30. Select **User Settings/sample offsets** from the main menu. Enter exactly half of the value in the *2theta* field into the *omega* field. Close the window.

31. Set the optics for sample measurements**:**

- Select the incident beam optics tab and double click on any item. Make the following changes in the incident beam optics settings window:
    - **Beam Attenuator**:
        - **Usage:** At pre-set intensity
        - **Activate at level: 550000**
        - **De-activate at level: 400000**

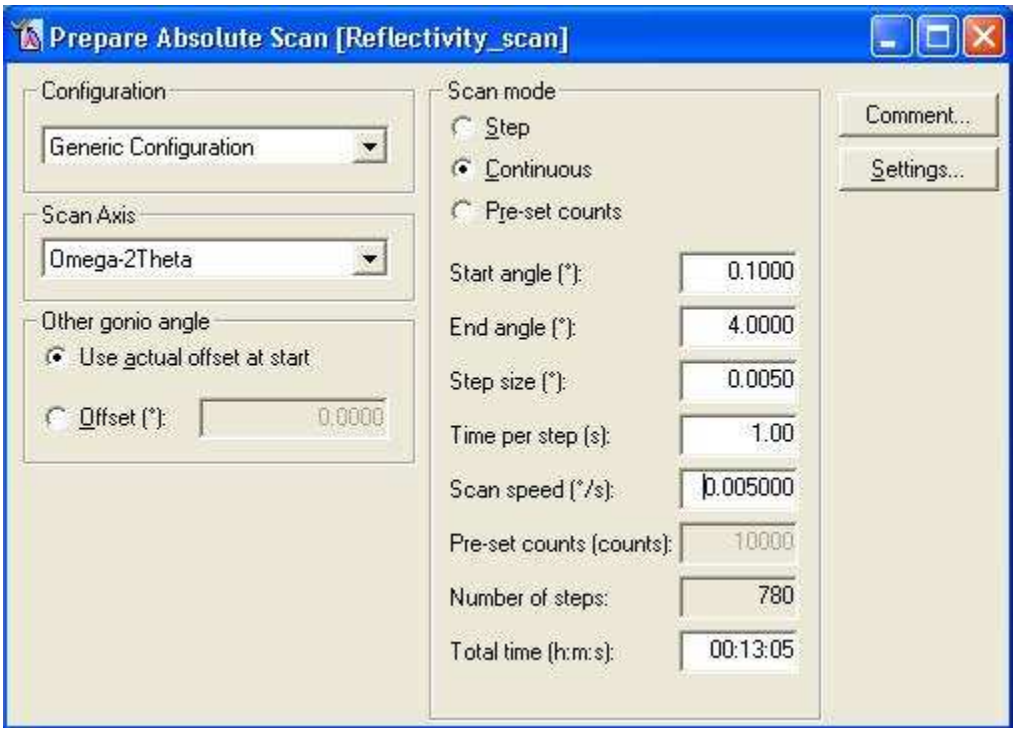
  Click OK to apply these settings and close the window

Measurement Program

32. Create a new measurement program or edit an existing program if needed.

- To create a new measurement program:
    - Select **File/New Program**
    - Select Program Type: **Absolute Scan**
    - Click OK
- To load and edit an existing program:
    - Select **File/Open Program**
    - Click “Browse” to search for your measurement program
    - Select desired program from the list and click OK

33. The window shown below will appear. Enter the information as explained below



- **Configuration:** Generic Configuration
- **Scan Axis:** Omega-2Theta
- **Scan Mode:** Continuous
- **Start Angle:** 0.1 degree less than current 2theta value (found in step 23)
- **End Angle:** 4 (or any desired range)
- **Step Size:** 0.005 is typical
- **Time per step:** 1.0 is typical

34. Select **File/Save** as and enter a name and description for this scan program and then click the OK button. Close this window.

Measuring

35. Select **Measure/Program**.
    - Click “Browse” to search for your measurement program
    - Select the desired measurement program from the list and click Open

36. A start menu will appear
    - Click on the folder icon and select or create a folder where the data will be stored. (Your data should be stored in your own folder within the XRD Data folder)
        - Enter a filename (sample name)
        - Click the Save button
    - Back in the start menu, enter comments and sample ID if desired
    - Click the OK button and the measurement will begin

37. When the measurement is completed, you can view and process the data using the X’Pert Data Viewer Program

38. For subsequent samples, return to the ***Sample Mounting and Alignment*** section

Shut Down

39. Unmount your sample from the stage and remove any tape left on the stage

40. Select the Instrument Settings tab. Double click on the Generator settings. Return the generator setting to the idle values:
    - **Tension: 45 kV**
    - **Current: 20 mA**

    Click OK to apply these settings and close the window

41. Exit the software

42. Log out of the User Log System on the SMIF web site