2 Theta Phase Analysis

1. Ensure you have an active XDIF1 reservation

Hardware Setup

Click HERE for a video showing the XDIF1 hardware components

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 Click HERE for Incident Beam Optics video
   - Mount the mirror on the incident beam PREFIX. Make sure that the attenuator cable is plugged into the mirror.
   - Insert the 1/32 degree slit in the mirror optics for sample alignment. (The ½ degree slit will be inserted later for the actual data collection)
   - If the sample height is less than 25 mm insert an appropriate beam mask that is smaller than the sample. (Otherwise the X-ray beam can hit the sample stage, and extra peaks at 38° and 45° will be generated)

3. Diffracted Beam Optics Click HERE for Diffracted Beam Optics video
   - Mount the parallel plate collimator (PPC) PREFIX
     - Verify that the 0.04 rads soller slits are behind the collimator and that detector 1 is present.

4. Close the enclosure doors Click HERE for closing enclosure doors video
   - The doors must be closed when starting the software
   - When the doors are open there will be one dot illuminated under “Shutter Open” in the front panel, and when the doors are closed, no dots will be illuminated.

Software Start-Up Click HERE for software start-up video

5. Open the X’Pert Data Collector program.

6. Enter your user name and password and click OK

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 Software Setup

11. Select the Incident Beam Optics tab. **Click HERE for Incident Beam Optics software set-up.**

   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. **Click HERE for Diffracted Beam Optics software set-up.**

   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:** Parallel Plate Collimator 0.27°
   - **Anti Scatter slit:** None
   - **Receiving slit:** None
   - **Collimator:** Parallel Plate Collimator 0.27°
   - **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 slits 0.04 rad.
   - **Monochromator:** None

   Click OK to apply these settings and close the window

13. Select the Instrument Settings tab. Double click on the Generator settings. **Click HERE for Setting the X-Ray generator video.** 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.
   
   Click HERE for setting the position coordinates video

   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

   Click HERE for mounting a powder sample video

15. For the first sample: Click HERE for mounting a sample to the stage video
   
   • 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.
   • Remove the 1/2 degree slit and insert the 1/32 degree slit
   • 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:
     • **Divergence slit**: slit fixed 1/32°
     • **Beam Attenuator**:
       o **Usage**: Do Not Switch (for sample alignment)
       o **Activated Box**: selected (check mark appears)
   
   Click OK to apply these settings and close the window
   • Skip to Step 18

   Click HERE for Detector alignment video

16. Detector (2theta) Alignment (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.
     o 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 **User Settings/ Sample Offsets** from the main menu. Enter 0 in the 2theta field. Press **OK**.

**Click HERE for sample height alignment video**

17. **Move Sample Height (Z) to an Initial Value (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 or 6.5mm for a glass slide) Click OK to move the sample stage.

18. **Sample Height (Z) Alignment**
- 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.

![Diagram](image.png)

- 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.

**Click HERE for sample tilt alignment video**

19. **Sample Tilt (Omega) Alignment (Optional)**
*Note: This step is optional for most samples. It is only applicable for flat, smooth samples being measured at low angles. It will not affect other types of samples whether this alignment is performed or not.*
- 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.

Click HERE for prepare for measurement video

20. Close the shutter by pressing the close shutter button on the upper menu bar

21. Close the “Prepare Manual Scan” window

22. Set the optics for sample measurements:
   • Open the enclosure doors
   • Remove the 1/32 degree slit and insert a 1/2 degree slit
   • 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:
     • Divergence slit: Fixed Slit 1/2°
     • Beam Attenuator:
       o Usage: At pre-set intensity
       o Activate at level: 550000
       o De-activate at level: 400000
     Click OK to apply these settings and close the window
Measurement Programs

Click **HERE** for measurement program video

23. 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

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

![Prepare Absolute Scan Window](image)

- **Configuration**: Generic Configuration
- **Scan Axis**: Gonio
- **Scan Mode**: Continuous
- **Start Angle and End Angle**: should be values based on where peaks of interest are expected for the material being studied. (e.g., 20 degrees to 80 degrees)
- **Step Size**: 0.05 is typical
- **Time per step**: 0.5 – 1.0 is typical

25. Select **File/Save as** and enter a name and description for this scan program and then click the Save button. Close the program window.
Measuring
Click HERE for measuring video

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

27. 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

Click HERE for Data Viewer Program and Saving Data video

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

YouTube tutorial on using HighScore for Phase Identification:
https://www.youtube.com/watch?v=dASaENbIC_4
HighScore basic steps:
   • Open data file (.xrdml) that you want to process
   • Determine Background (from Treatment menu), Accept but don’t subtract it
   • Search Peaks (from Treatment menu)
   • Perform Peak Fit (Select “Default Profile Fit” from Fitting in Analyze Menu)
   • Determine Phases (Execute Search and Match from Analysis Menu)
     • Edit Restriction Set before searching
   • Save results and/or create Report

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

Shut Down
Click HERE for shut down video

30. Unmount your sample from the stage and remove any tape left on the stage
31. 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
32. Exit the software
33. Stop the XDIF1 reservation in the CoreResearch@Duke system