In-Plane Diffraction Measurements

The following instructions are meant to be a guide, but many of the scan parameters may change based on your samples and experience. In-plane measurements are an advanced technique, so we will only train people who are very comfortable with the machine from running other measurements. You MUST schedule with Linda at least 1-2 days in advance and finish your run during normal business hours because Linda has to change the x-ray tube from line focus to point focus before the measurement and back to line focus after the measurement. The x-ray tube may need to be realigned after changing back to line focus. Please report any problems immediately to Linda Sauer (Room 14, 625-0776, lksauer@tc.umn.edu). If you have any questions, please ask. The optics are delicate, please do not bump. If the x-rays are off, NEVER attempt to turn them back on.

Hardware Setup

1. The system must be setup with the x-ray tube using the point source. The system is usually in line focus, so you must schedule in advance to have Linda change the system. The x-ray lens must be mounted on the incident beam side. If these conditions are not met, STOP and contact Linda.

2. The cross-slit collimator on the front of the x-ray lens is used with the point focus to generate a line focus in the plane of the tilted sample. The top micrometer should be adjusted to 0.5 and the side micrometer should be adjusted to 3. For alignment purposes, the Cu 0.1 manual attenuator should be inserted in the x-ray lens optic.
3. The parallel plate collimator must be attached on the diffracted beam side with Detector 1. The parallel plate receiving slit should be inserted for alignment purposes and then removed when measuring samples.

Note: The following are the definitions of the angles in the software –
\( \omega \) – angle between incident x-rays and sample surface
\( 2\theta \) – angle between incident x-rays and detector
\( \psi \) – sample tilt
\( \phi \) – in-plane sample rotation
\( x,y \) – in-plane displacement of sample
\( z \) – vertical displacement of sample
User Setup

4. Log onto the computer:  *User*: Charfac code # (for example 2310); *Password*: Domain: CharFac.

5. Open the **Data Collector** program.

6. Enter your user name (created during training, please write down) and password.


8. Select **Point Focus** as the **Configuration** and press the **OK** button.

9. A message dialog box will appear. Any line with a yellow triangle tells what the software is assuming. Press the **OK** button. If a red stop sign appears, you cannot continue, please find an x-ray staff member for assistance.

10. If you previously had set sample offsets, a dialog may appear asking if you want to apply these offsets. **Yes** keeps the sample offsets, **No** clears them. Unless you were the last person to use the machine, it is a very good idea to clear the offsets.

Optics Setup

11. Selected the **Incident Beam Optics** tab and double click on one of the items. The **PreFIX Module** should be selected to be the **X-ray Lens**. The **Divergence Slit** should be set to **3 mm** and the **Mask** should be set to **0.5 mm** (determined by openings of the cross-slit collimator). The **Beam Attenuator** should be **none** (the attenuator will be removed after alignment).

12. Select the **Diffracted Beam Optics** tab and double click on one of the items. The **PreFix Module** should be the Parallel Plate Collimator. Select **none** for the **Receiving Slit** (the slit will be removed after alignment). Make sure the **Detector** is **mini prop large window 1** and the **Wavelength** is **K alpha 1**.

13. Select the **Instrument Settings** tab, double click an item, select the **X-ray** tab, and set the **Generator** to **45 kV** and **40 mA**.

Sample Mounting

14. Select the **Instrument Settings** tab, double click an item, select the **Position** tab, set **Psi** to **90 degrees** (**VERY IMPORTANT**)!, and set **Z** to **0 mm**. Press the **Apply** button to move the stage.

15. Remove the sample holder by rotating until it releases.

16. Mount the sample as flat as possible on the center of the stage with double sided tape or spring clips for large wafer samples.
17. Replace the sample holder.

18. Place the manual Cu 0.2 attenuator in the incident beam optics.

Positioning the sample and checking the zero position of the detector

19. Select **Customize/Option** from the main menu. Make sure the *Single Crystal Mode* box is checked (this allows a negative value of omega to be achieved). Close this window.

20. **Caution:** Only do this step if the sample holder is tilted down so *Psi* is 90 degrees as in the above picture. Select the *Instrument Settings* tab, double click an item, select the *Position* tab, set 2Theta to 0 degrees, Omega to -90 degrees, and press OK.

21. Go to the **Measure** menu and select **Manual Scan**.
22. Select 2theta for the Scan axis, Continuous for the Scan Mode, 2 deg for the Range, 0.01 deg for the Step Size, 0.2 sec for the Time Per Step, and press Start.

23. After the scan is finished, right mouse click on the graph and select Peak Mode.

24. To accept the location of the peak, press the Move To button and then select OK. To select a different position of the peak, press Cancel, right mouse click on the graph, and select Move Mode. Move the cursor until the intensity is the maximum. This will move the goniometer to this position.

25. Once the correct position of the detector is found, click on the Tools menu and select Sample Offsets. Set the current 2theta position to 0 and select OK.

Setting the correct sample height

26. To set the correct height of the sample, the incident beam must be bisected:
   a. Set Z to 8 mm, and press OK.
   b. Go to the Measure menu and select Manual Scan.
   c. Select Z for the Scan Axis, Continuous for the Scan Mode, 6 mm for the Range, 0.01 mm for the Step Size, 0.1 sec for the Time Per Step, and press Start.
   d. After the scan is finished, right mouse click on the graph and select Move Mode.
   e. Move the cursor until the intensity is ½ the maximum. This will move the goniometer to this position.
   f. Return to the Manual Scan window, select Psi for the Scan Axis, 6 degrees for the Range, 0.05 degrees for the Step Size, 0.3 sec for the Time Per Step, and press Start.
   g. Use the Move Mode again to select the maximum intensity.
   h. Repeat steps c. to g. until the values do not change. The Ranges and Step Sizes can be made smaller once the approximate correct values are determined in the first scans.

Peak Optimization

27. For the in-plane diffraction measurement to work properly, the x-rays must be incident at a small angle above the critical angle of the film. For most films, a tilt of 0.3 degrees is sufficient. If the films contain Cu, a tilt of 0.5 degrees may be needed due to the higher critical angle.

28. Select the Instrument Settings tab, double click an item, select the Position tab, set Psi to the current value plus 0.3 degrees (for example, if the Psi was optimized at 90 degrees, set Psi to 90.3 degrees), set 2theta to the correct value for the peak of interest, and press the OK button.

29. After the machine finishes moving, close the shutter by pressing the blue arrow in the toolbar, open the doors, remove the Cu attenuator from the x-ray lens, remove the receiving slit from the parallel plate collimator, and shut the doors. Only remove the attenuator and slit when the 2theta is no longer very close to 0 degrees to avoid damaging the detector.
30. Do a quick Phi scan to find your peak. Optimize the peak using Psi (this is your incident angle, which should be \( \geq .01 \)) and Omega scans (or Phi scans, omega and phi are the same movement in this orientation).

**Peak Measurement**

31. Do complete Phi scans to determine the symmetry of the crystal planes or a 2theta scan to calculate the epitaxial crystallite size.

**When Done**

32. Power the x-rays down to 40 kv and 10 ma.

33. Close the program, transfer your data (files are not backed up), and log off the computer. Please leave the work area clean.

34. Contact Linda to return the system to line focus mode. Note: If a user is scheduled to run immediately after you, you must finish \( \frac{1}{2} \) hour early allowing Linda to re-setup the system.
APPENDIX

Changing X-pert to Point Focus

1. Change incident beam to point focus optics and diffracted beam to parallel plate collimator.

2. Connect Instrument and select point focus. This will open a wizard instructing you what to do.

3. Click next to power down the X-rays (note you should be at stand-by power of 40kV and 10mA when you do this).

4. Switch the X-ray tube (open back and rotate 90degrees counter-clockwise).

5. Click next, finish and apply. (all the rest is in the instructions starting on page 1)

Changing X-pert back to Line focus

1. Change incident and diffracted optics to either thin film or rocking curve set-up.

2. Connect instrument and select line focus.

3. This will open a wizard instructing you what to do.

4. Click next to power down the X-rays (note you should be at stand-by power of 40kV and 10mA when you do this).

5. Switch the X-ray tube (open back and rotate 90degrees clockwise).

6. Click next, finish and apply.

7. Select **Customize/Option** from the main menu. Make sure the **Powder X-ray** box is checked