REFLECTIVITY
(film thickness between 10 and 1,500 Angstroms)

Hardware Setup

1. The system must be setup with the x-ray tube using the line source, the Slit on the incident beam side (make sure the attenuator is plugged in), set to 1/32 degree. Mount the 0.27 degree parallel plate collimator with its collimator slit in place on the diffracted beam side.

User Setup

2. Open the **Organiser** program.
3. Enter your user name and password.
4. Select the **Users & Projects** menu.
5. Choose **Select Projects** to select an existing project or **Edit Projects** to create a new project, then choose **Select Projects** to choose this newly created project.
6. Select **Modules/X'Pert Data Collector** or the Data Collector button on the tool bar to open the Data Collector Program.
7. Select **Instrument/connect** in the Data Collector. The Go On Line box will appear.
8. Select the *Mirror + PPC* for your optics. Then press the OK button.

Direct Beam Intensity Determination

9. Press the *Instrument setting* tab in the tree view. Double click any item in the tree view to prompt another window. Enter 0 in the *2theta* field, 0 in the *offset* field (this will set omega to 0), 0 in the *phi* field, and 0 in the *psi*, x, y, and z fields. Press the OK button to move to these positions.
10. Press the *X-ray* tab. Set the generator power to 45 kV and 40 mA.
11. Select the incident beam optics tab. Double click any item to prompt another window. Select the proper optic components in the new window. **Set the beam attenuator to 'do not switch' and 'activated'.** Press the OK button.
12. Select the diffracted beam optics tab. Double click any item to prompt another window. Select the proper optic components for the secondary beam side.
13. Select **Measure/ Manual Scan** from the main menu.
14. Enter the following parameters: 2theta in the *scan axis* field, enter 0.01 in the *step size* field, 2 in the *range* field, and 0.1 in the *time per step* field, then press the start button.
15. After the measurement is completed, press the right mouse button and select **Move mode.**
16. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. This will move the goniometer to the selected position. Note the intensity value (write it down).
17. Select the **Utilities/ Sample Offsets** from the main menu. Enter 0 in the *2theta* field. Close this window.

Bisecting the Beam with the Sample

18. Back in the Control window to move Psi to 90 degree. This step makes sample mounting easier. Note: if you are comfortable mounting your sample in the upright position, you can leave psi at 0, and move on to step 19.
19. Mount the sample on the stage. The long direction should be horizontal.
20. Check the *open shutter* box. Press the *change position* button and enter 0 for psi and adjust the z position of the sample stage until the counts read between ten and twenty percent of the value noted in step 15. Then press OK.
21. Back in the **Manual Scan** window, enter the following parameters: omega in the *scan axis* field, enter 0.005 in the *step size* field, 2 in the *range* field, and 0.1 in the *time per step* field, then press the start button.
22. After the measurement is completed press the right mouse button and select **Move mode.**
23. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. This will move the goniometer to the selected position.
24. If the current intensity is now **half** (within 1 percent) of the intensity noted in step 15 continue with step 24. If it is more or less than half, then return to the Control window. Increase z to decrease the intensity or decrease z to increase the intensity until it is within 1 percent of half of the value noted in step 15.
25. Select **Customize/Option** from the main menu. Make sure the *Single Crystal Mode* box is **not** checked. Close this window.

Optimizing Reflected Intensity

26. Back in the **Manual Scan** window, enter the following parameters: 2theta/omega in the *scan axis* field, 1.6 in the *2theta* field, 0.005 in the *step size* field, 3 in the *range* field, and 0.2 in the *time per step* field. Then press the start button.
27. Select **Axes** and *logarithmic* scale.
28. After the measurement is completed press the right mouse button and select **Move mode**.
29. Press and hold the left mouse button until the cursor 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.)
30. Back in the **Manual Scan** window, enter the following parameters: omega in the *scan axis* field, 0.005 in the *step size* field, 1 in the *range* field, and 0.2 in the *time per step* field. Then, press the start button.
31. After the measurement is completed select **Axes** and *linear* scale, then press the right mouse button and select **Move mode**.
32. Press and hold down the left mouse button until the cursor is placed over the center of gravity of the peak. 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. This will move the goniometer to the selected position.
33. Back in the **Manual Scan** window, enter the following parameters: psi in the *scan axis* field, 0.05 in the *step size* field, 10 in the *range* field, and 0.2 in the *time per step* field. Then press the start button.

34. After the measurement is completed press the right mouse button and select the Move mode.
35. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. This will move the goniometer to the selected position.
36. Back in the **Manual Scan** window, enter the following parameters: omega in the *scan axis* field, 0.005 in the *step size* field, 1 in the *range* field, and 0.2 in the *time per step* field. Then, press the start button.
37. After the measurement is completed press the right mouse button and select the *Move mode*.
38. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. **Before releasing the left mouse button compare the position displayed in the top center of the screen with the one displayed for omega on the status bar at the bottom of the screen. If these numbers are within 0.1 degrees of each other continue with step 38.** If the difference is greater than 0.1 then go back to step 32 after the goniometer moves.
39. Back in the **Manual scan** window, enter the following parameters: 2theta/omega in the *scan axis* field, 3.6 in the *2theta* field, 0.005 in the *step size* field, 7 in the *range* field, and 0.1 in *the time per step* field. Then, press the start button.
40. Select **Axes** and *logarithmic* scale.
41. After the measurement is completed, make a note of where the critical angle is. You will select a start angle that is at least 0.1 degree lower. Close both the **Scan display** and **prepare Manual Scan** windows.
42. Select **Utilities/sample offset** from the main menu. Enter exactly half of the value in the *2theta* field into the *omega* field. Close the window.

Setting the Attenuator

43. Select the *Incident Beam* optics tab. Press the *Change optics* button. Set the attenuator to 'preset intensity'. The in level should be 500,000 and the out level should be 450,000. Press the okay button.

Measurement Program

44. Select **File/New Program/Absolute Scan.**
45. Enter the following information: Gonio in the *scan axis* field, set the start angle to whatever you determined appropriate in step 40, set the end angle to 5, enter 0.005 in the *step size* field, enter 1 in the *time per step* field.
46. **Select File/Save** as and enter a filename for this scan program and then push the OK button. Close this window.

Measuring

47. Select **Measure/Program/Absolute scan**. Choose the reflectivity program you just created.
48. Enter a dataset name and a description. Press the start button.