Rocking Curves with the Hybrid

Hardware Setup

1. The system must be setup with the x-ray tube using the line source. The hybrid monochromator must be mounted on the incident beam side. Make sure the beam attenuator cable is plugged into the hybrid.

2. The ½ degree divergence slit must be in the hybrid and the manual attenuators must NOT be in the hybrid. If the sample is less than 25 mm an appropriate beam mask should be selected.

3. The rocking curve (RC) + triple axis must be mounted on the diffracted beam side. Make sure no slit is in the rocking curve optic.

User Setup

4. Open the **Organiser** program.

5. Enter your user name and password.

6. Select the **Users & Projects** menu.

7. Select **Select Projects** to select an existing project or **Edit Projects** to create a new project. (See page 3-10 in the X'Pert Organiser User's Guide for more information on creating new projects.)

8. Select **Modules/X'Pert Data Collector** or the Data Collector button on the tool bar to open the Data Collector Program.

9. Select **Instrument/Connect** in the Data Collector. The Go On Line box will appear.

10. Select HR as the configuration and rocking curve as the beam path. Then press the OK button.

Optics Setup

11. Selected the incident beam optics tab and double click on one of the item. The PREFIX module will be the hybrid. The ½ degree slit should be selected. The beam attenuator should have a usage of do not switch and the activated box should be checked. The correct beam mask should be selected.

12. Select the diffracted beam optics tab and double click on one of the items. Set the receiving slit to 1.0 mm Rocking Curve. Make sure the detector is mini prop large window 2 and the wavelength is K alpha 1.

13. Select the Goniometer tab and set the generator to 45 kV and 40 mA.

Sample Mounting

14. Press the *Change position* button and enter 90 degrees in the psi field so the sample can be easily mounted on the stage. Press the *Apply* button to move the stage.

15. Mount the sample as flat as possible on the stage.

16. Mount the micrometer on the stage and close the enclosure doors.

17. Move the z position of the sample stage until the micrometer reads 1.0. (The small inner dial will read 1 and the large outer dial should point to the 0 at the top of the micrometer.)

18. Remove the micrometer and close the doors.

19. Return the stage to its upright position by setting psi to 0 degrees and pressing the *OK* button.

Peak Optimization

20. If the lattice parameters and angles for this material are know and it will be studied often this information can be stored in the system. Once this information is stored the reciprocal space interface can be used to drive the goniometer to specific reflections. To input this information continue with step 21. If it is unknown go to step 25.

21. Select **Customise/Options** and make sure the radio button in front of Single crystal mode is checked. Close this window.

22. Select **Customise/Unit cells** and then press the insert button.

23. Enter all the requested information for the unit cell. The primary and secondary hkl are the growth vector and a wafer flat vector respectively. (If no wafer flat is present any vector perpendicular to the growth vector is sufficient.) The hkl values must be entered as integers with spaces between them.

24. After entering the all the information press the *OK* button and notice the new cell added to the list. Then press the *OK* button.

25. Before a rocking curve is collected for an extended measuring time preliminary scans are usually done to optimize the peak(s) of interest. This can be done either manually using manual measurements, or automatically by creating an optimization program. The manual method will be outlined here.

26. Select **Measure/Manual Scan** from the main menu.

27. Select omega in the *scan axis* field, enter 0.01 in the *step size* field, 6 in the *range* field, and 0.1 in the *time per step* field.

28. Enter the substrate material in the *unit cell* field. (If unit cell information was not entered, enter the 2theta value for the peak of interest in the 2theta field and 0 in the offset field and press the start button. Then, continue with step 30.)

29. Enter the plane of interest in the *HKL* field. This will set all the angles to the theoretical values for this reflection. Then press the start button.

30. If a peak is not found execute the following scan: phi as the *scan axis* field, enter 0.1 in the *step size* field, 200 in the *range* field, and 0.1 in the *time per step* field. Once a peak is found continue with step 31.

31. After the measurement is completed press the right mouse button and select **Move mode**.

32. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. If more than one peak is present choose the narrowest, most intense peak. This will move the goniometer to the selected position.

33. Back in the *manual scan* window, enter the follow parameters: psi in the *scan axis* field, enter 0.05 in the *step size* field, 8 in the *range* field, and 0.1 in the *time per step* field. Then, press the start button. (This is in the case of a symmetric reflection. If an asymmetric reflection is being studied enter phi for the *scan axis* and leave all the other scan parameters as suggested here.)

34. After the measurement is completed press the right mouse button and select **Move mode**.

35. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. If two peaks occur select the lowest intensity point between the two peaks.

36. Back in the *manual scan* window, enter the following parameters: omega in the *scan axis* field, enter 0.002 in the *step size* field, 1 in the *range* field, and 0.1 in the *time per step* field. Press the start button.

37. If the omega peak position has not changed since the first omega scan executed in step 29 continue with step 38. (The peak is in the same position if the center of gravity of the current peak on the screen is located at the omega position of the instrument at the completion of the measurement.) If the omega peak position has changed return to step 33.

38. Insert the 1.0 mm slit in the receiving slit holder.

39. Back in the *manual scan* window, enter the follow parameters: 2theta in the *scan axis* field, enter 0.01 in the *step size* field, 3 in the *range* field, and 0.1 in the *time per step* field. Press the start button.

40. After the measurement is completed press the right mouse button and select **Move mode**.

41. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. If more than one peak is present choose the narrowest, most intense peak. This will move the goniometer to the selected position.

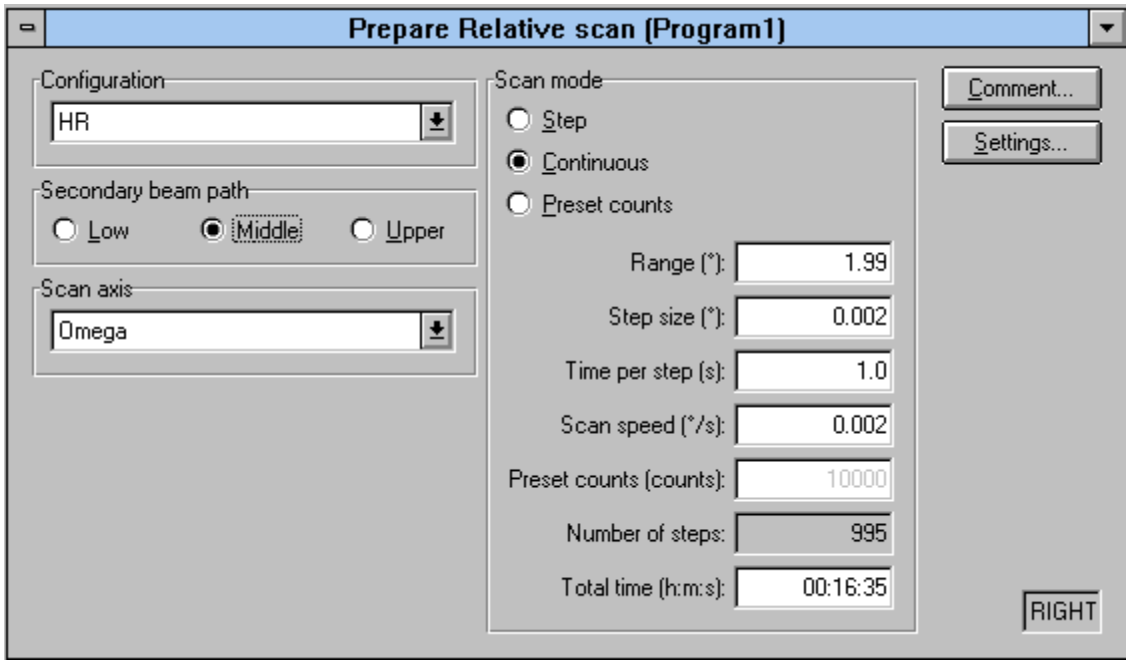
42. Close the manual scan windows.

43. Back in the control window select the incident beam optics tab. Double click on an item related to the beam attenuator. Change the usage field to 'switch at preset intensity', set the activate level to 450,000, and the deactivate level to 400,000. Make sure the attenuation factor is correctly set. If it is not change the value using the menu item **Customize/Beam Attenuators**.

Measurement Program

44. Select **File/New Program/Relative scan.**

45. The window show on the next page will appear. Enter the information explained below the figure.



Make sure that rocking curve is selected as the secondary beam path.

The scan axis should be omega-2theta.

The scan axis is always continuous.

The step size should be 0.002 degrees.

The time per step should be in the range of 0.5 – 5.0.

46. Select **File/Save as** and enter a name for the program and then press the *OK* button. Close this window.

Measuring

47. Select **Measure/Program/Relative scan**.

48. Enter the program name chosen in step 46 if it does not already appear. Enter a data set name and sample identification. **Do not modify any of the angular fields, the HKL field or the Unit cell field.** Press the start button.