Pole Figures - Texture

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

Hardware Setup

2. ***X-Ray Tube***
   The system must be setup with the x-ray tube using the point source. **Contact the SMIF staff person responsible for XDIF1 system to assist with converting the X-ray tube to a point source.** Follow the procedure in Section 1, Page 14 of the equipment manual to change the X-Ray tube from the line source to the point source configuration

3. Incident Beam Optics

- Mount the cross slit on the incident beam PREFIX. Open the cross slits to a reasonable size depending on the sample size (e.g., 2mm x 2mm).

4. Diffracted Beam Optics

- Mount the parallel plate collimator (PPC) PREFIX
  - Remove the 0.04 rads soller slits
  - Verify that detector 1 is present.

5. Close the enclosure doors

The doors must be closed when starting the software

User Setup

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

7. Enter your user name and password.

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

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

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

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

Optics Setup

12. 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**: Crossed Slits(knob adjustable)
    - **Divergence slit**: Enter the cross slit height setting (e.g., 2mm)
    - **Anti Scatter slit**: None
    - **Mask**: Enter the cross slit width setting (e.g., 2mm)
    - **Mirror**: None
    - **Monochromator**: None
    - **Beam Attenuator**: None
    - **Filter:** None
    - **Soller Slit:** None

    Click OK to apply these settings and close the window

13. 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**: 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:** None
    - **Monochromator:** None

    Click OK to apply these settings and close the window

14. Select the Instrument Settings tab. Double click on the Generator settings. Set the generator to:
    - **Tension**: 45 kV
    - **Current**: 40 mA
    - Verify that the “Point Focus” option is selected

    Click OK to apply these settings and close the window

Sample Mounting and Alignment

(Using the Dial Gauge Method)

15. Open the enclosure doors and mount the sample as flat as possible on the stage

16. Close the enclosure doors (the doors must be closed for the stage to move)

17. Select the **Instrument Settings** tab and double click on an item related to the **Positions**.

18. Enter 0 in all of the coordinate fields except for Z.

19. Enter an approximate starting position for the Z height. (e.g., 7.5mm for a Si wafer) Click Apply to move the sample stage and leave the positions window open

Optimize Sample Height (Z)

Note: You must use the dial gauge method when using the point source configuration

20. Open the enclosure doors and mount the dial gauge on the stage. Then close the enclosure doors.

21. Adjust the Z position of the sample stage (enter a Z value and click Apply) until the dial gauge 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 dial gauge.) Press the *OK* button to close the Positions window.

22. Open the enclosure doors and remove the dial gauge. Then close the enclosure doors.

Peak Find

23. Select the **Instrument Settings** tab and double click on an item related to the **Positions**
    - Enter the expected 2theta value for the peak of interest and 0 in the *offset* field.
    - ClicK OK to apply these settings and close the window

24. If the sample to be analyzed is a multiple phase polycrystalline sample it may be beneficial to do a scan in the area of the angle where the pole figure will be collected.
    - Select Measure/ Manual Scan from the main menu.
    - Enter the following parameters in the Manual Scan window:
      - Scan Axis: 2theta/omega
      - Range: 10.0

- Step Size: 0.04
- Time per step: 0.1
- Click the start button to begin the scan

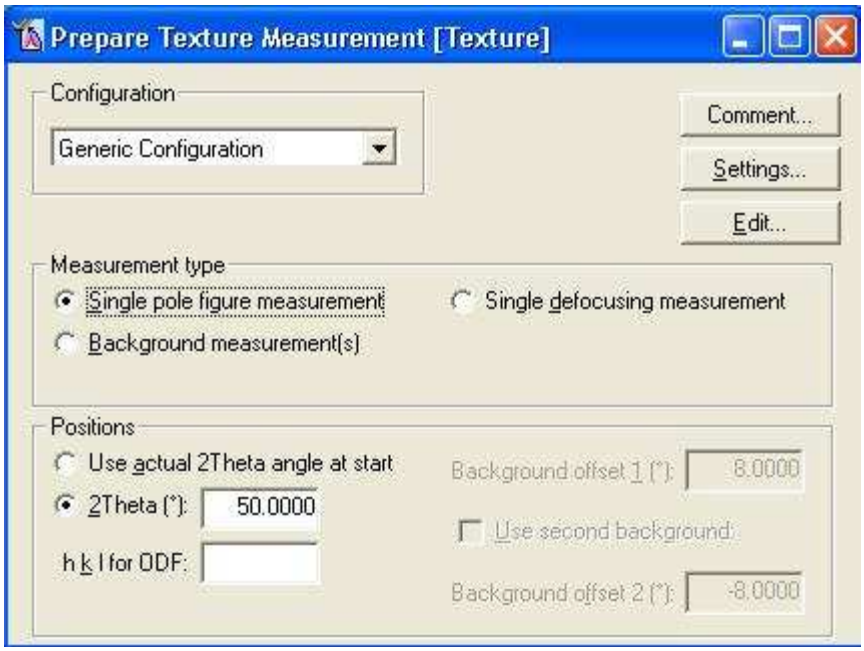
25. Note where any peaks are found, what phases they come from.

26. Close the scan windows.

Measurement Programs

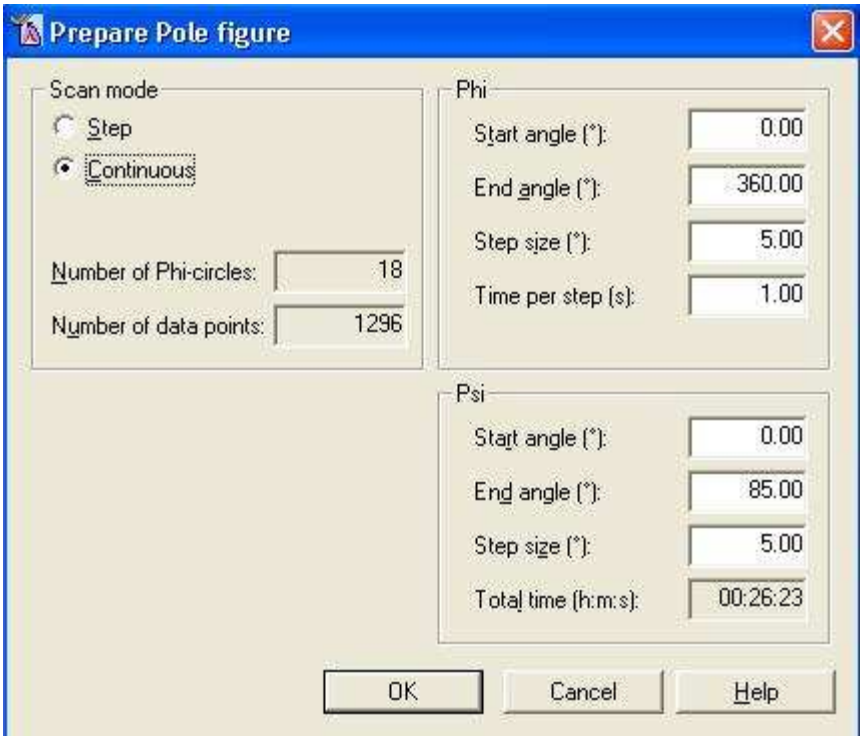
27. Create a new measurement program or edit an existing program if needed.
    - To create a new measurement program:
        - Select **File/New Program**
        - Select Program Type: **Texture Measurements**
        - Click OK
    - To load and edit an existing program:
        - Select **File/Open Program**
        - Click "Browse" to search for your measurement program
        - Select desired program from the list and click OK

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



- **Configuration:** Generic Configuration
- **Measurement type:** Single pole figure measurement
- **2Theta:** Use selected 2Theta angle found in step 25 for the peak of interest

29. Click on **Edit** and the window shown below will appear. Enter the information as explained below



Texture measurements are 2-axis scans with Phi as the continuous scan axis and Chi as the step scan axis

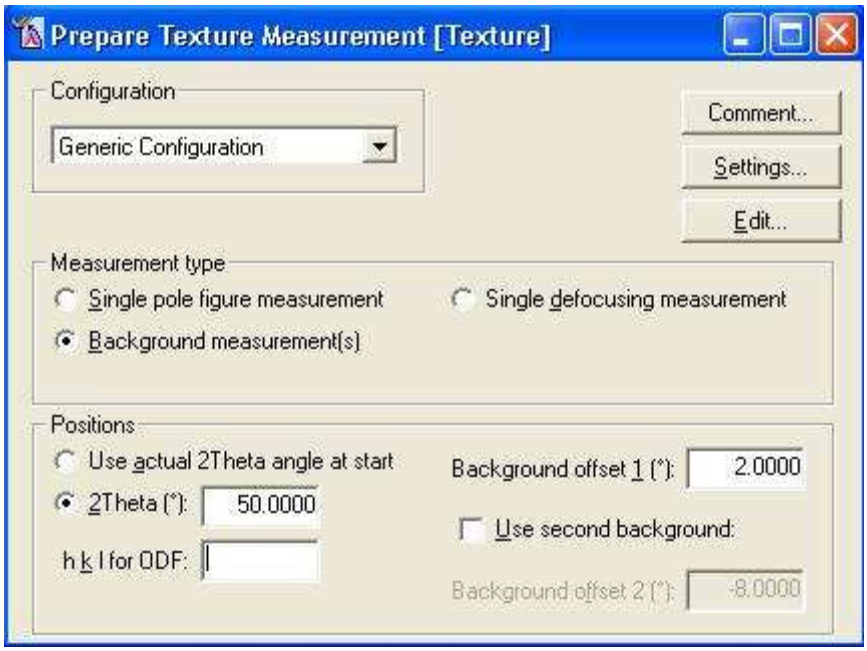
- **Scan Mode:** Continuous
- **Phi:**
  - **Start Angle:** 0 is typical
  - **End Angle:** 360 is typical
  - **Step Size:** depends on the degree of preferred orientation. Strong texture needs smaller step sizes
- **Chi**
  - **Start Angle:** 0 is typical
  - **End Angle:** 85 is typical
  - **Step Size:** depends on the degree of preferred orientation. Strong texture needs smaller step sizes

30. Select **File/Save as** and enter a name for the program and then press the OK button.

31. If a background correction is desired for the pole figure continue with step 32. If not skip to step 40.

32. Create a new background measurement program or edit an existing program if needed.
    - To create a new background measurement program:
        - Select **File/New Program**
        - Select Program Type: **Texture Measurements**
        - 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



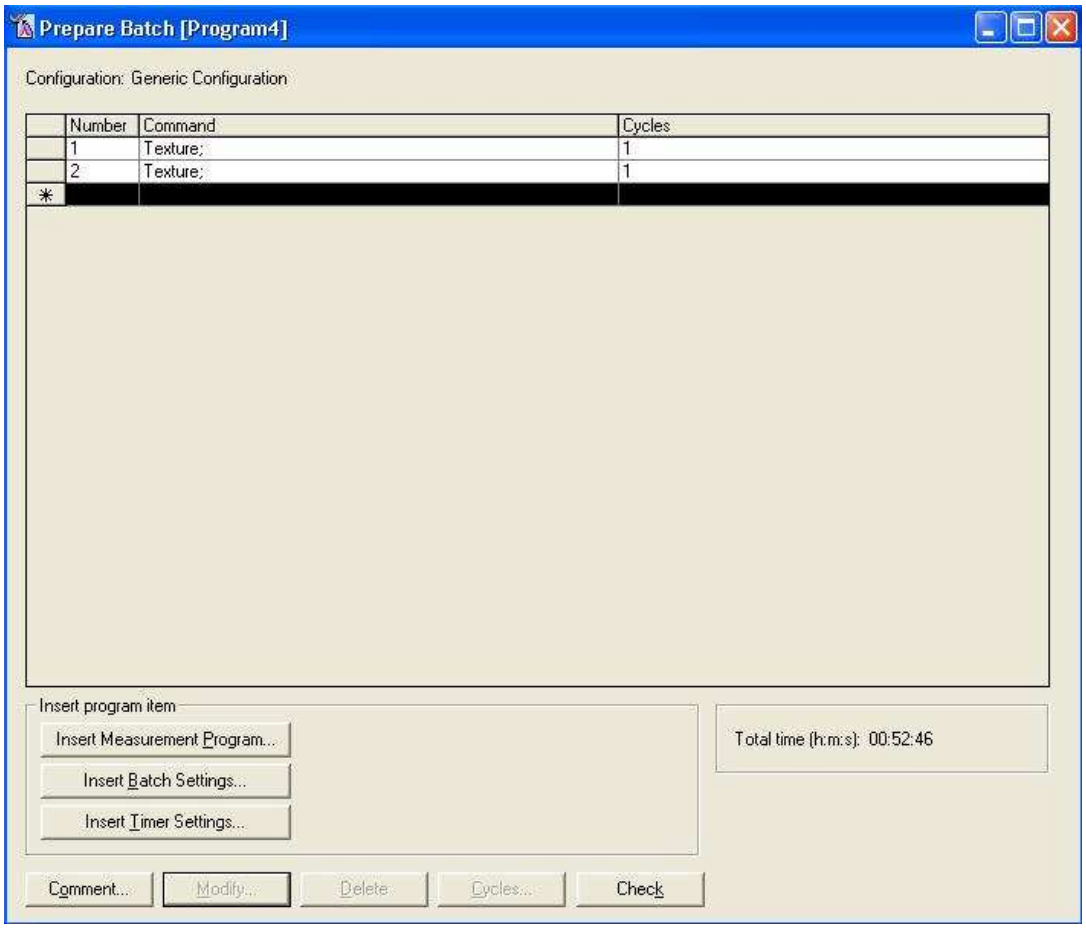
- **Configuration:** Generic Configuration
- **Measurement type:** Background measurement(s)
- **2Theta:** Use selected 2Theta angle found in step 24 for the peak of interest
- **Background offset:** 1 or 2 degrees (typical)

34. Select **File/Save as** and enter a name for the background measurement program and then press the OK button.

35. If several pole figures will be collected on the same sample and/or a background correction will be measured continue with step 36 to create a batch program. If just a single pole figure measurement is desired skip to step 40.

36. Create a new batch measurement program or edit an existing program if needed.
    - To create a new batch measurement program:
        - Select **File/New Program**
        - Select Program Type: **General Batch**
        - Select configuration: **Generic Configuration**
        - 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

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



38. Create a sequence of measurement programs that were previously created. They could be either single pole figure measurements or background measurements.
    - Click Inset Measurement Program
    - Click Browse and select desired Texture or Background program
39. Select **File/Save as** and enter a name for the batch program and then press the OK button.

Measuring

40. Select **Measure/Program**.
    - Select List of programs of type:
        - **Texture Measurement** (for a single measurement)
        - **General Batch** (for a Batch program that will run a sequence of measurements)
    - Click "Browse" to search for your measurement program
    - Select the desired measurement program from the list and click Open

41. 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)
        i. Enter a filename (sample name)
        ii. 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

42. When the measurement is completed, you can view and process the data using the X'Pert Data Viewer Program and the X'Pert Texture program

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

Shut Down

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

45. **Contact the SMIF staff person responsible for XDIF1 system to assist with converting the X-ray tube back to a line source**. Follow the procedure in Section 1, Page 14 of the equipment manual to return the X-Ray tube from the point source to the line source configuration.

46. 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

47. Exit the software

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