Rocking Curve and 2θ − ω scans using Hybrid Monochromator (X'Pert_1 only)

I. Login

1. Start **Data Collector**
2. Type your *UserName* and *Password.*
3. Select *Instrument – Connect.*
4. Choose Configuration **Hybrid + RC/TA**.
5. Below Configuration window select *Diffracted Beam Path* as **RC.**
6. Click *OK.*

II. Hardware Setup

1. X-ray Tube is in *Line Focus*.
2. Goniometer Resolution set to "High 0.0001 deg".
3. Incident Beam Optics – **Hybrid Monochromator**
   a. *Divergence Slit* – $1/2^{o}$.
   b. If sample is smaller than 25 mm, insert correct size *Mask.*
      Note: if you have to change incident beam optics please first turn Automatic attenuator to "Activate" status and then unplug attenuator cable.
4. Diffracted Beam Optics – **RC/TA**
   a. **RC** beam path can be used with or without receiving slits. Without slits detector will have widest angular acceptance in 2θ.
   b. **TA** beam path does not use any slits.

III. Data Collector Software

1. Select the *Incident Beam* optics tab
   a. Double click any item. Incident beam optics window will appear.
   b. Go through all tabs and select proper optic components.
   c. Set *Automatic Attenuator Usage* to "Do not switch" and "Activated".
2. Select the *Diffracted Beam* optics tab
   a. Double click any item. Diffracted beam optics window will appear.
   b. Go through all tabs and select proper optic components.
3. Select *Instrument Settings* tab
   a. Double click any item in the tree view to prompt another window.
   b. Press *X-ray* tab. Set generator power to 45 kV and 40 mA.

IV. Sample Mounting

1. Mount sample. If the sample is large, supplied clips can be used to hold the sample.
2. If in the *Instrument Settings* tab X = 0.0 and Y = 0.0, beam is positioned at the center of a sample stage (aluminum disk).

V. Moving Sample into the Beam Position

1. Using supplied Micrometer
   a. Mount micrometer onto the MRD cradle. Close the doors.
   b. In the *Instruments Settings* window, double click any item in the tree view to prompt another window.
   c. Move Z until micrometer reads (1.00±0.02) mm. This is the correct sample height.
2. Using direct beam
   a. Place $1/16^{o}$ slit into RC path slit holder.
   b. Place $1/32^{o}$ slit into **Hybrid Monochromator**.
   c. In *Instruments Settings* check Z position. Sample should not interfere with the direct beam. If not sure, remove sample and move Z back to at least 5mm.

d. Move all other motors to zero positions.
e. From Menu Select *Measure – Manual Scan.*
f. From the *Scan Axis* drop down menu select 2Theta.
g. Enter *Range* 1^o, *Step Size* 0.005^o, and *Time per Step* 0.1sec. Then press *Start.*
h. After scan is finished, right click on mouse and select *Move Mode*. Move Scan Axis to the center of the mass of the peak.
i. Note the direct beam intensity. If sample is removed, mount the sample as described in Part IV. In *Instruments Settings* move Z to higher values until intensity starts to drop.
j. In *Manual Scan* window from the *Scan Axis* drop down menu select Z. Enter *Range* 2mm, *Step Size* 0.01mm, and *Time per Step* 0.2sec. Then press *Start.*
k. After scan is finished, right click on mouse and select *Move Mode*. Move *Scan Axis* to the intensity value corresponding to ½ of the direct beam intensity.
l. Z is aligned. Close shutter. Remove $1/16^o$ slit from the RC optics, replace $1/32^o$ slit in the **Hybrid Monochromator** with $1/2^o$ slit.
Note: sample height alignment using direct beam will work correctly if sample size is larger than the beam size in axial direction.

VI. Aligning diffractometer on the known diffraction peak. Si(001) example

1. Select *Instrument Settings* tab
   a. Double click any item in the tree view to prompt another window.
   b. Click *Positions* tab.
   c. In *Unit Cells* select *Si_001.*
   d. In *h k l* field enter "0 0 4".
   e. Click *OK*. Diffractometer moves to Si(004) peak position.
   f. Select *Measure – Manual Scan.*
   g. Start with *Omega* Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select *Omega*. Enter *Range* 2^o, *Step Size* 0.01^o, and *Time per Step* 0.1sec. Then press *Start.*
   h. After scan is completed. Si(004) diffraction peak should be visible. Right click on mouse and select *Move Mode*. Move Scan Axis to the center of the mass of the peak.
   i. Next perform *Psi* Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select *Psi*. Enter *Range* 6^o, *Step Size* 0.03^o, and *Time per Step* 0.1sec. Then press *Start.*
   j. Right click on mouse and select *Move Mode*. Move Scan Axis to the center of the mass of the peak.
   k. Repeat *Omega* Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select *Omega*. Enter *Range* 0.2^o, *Step Size* 0.0005^o, and *Time per Step* 0.1sec. Then press *Start.*
   l. Right click on mouse and select *Move Mode*. Move Scan Axis to the center of the mass of the peak.
   m. Select *User Settings – Sample Offsets*. Enter in *Omega* and *Psi* fields theoretical Si(004) values. Click *OK.*
2. Select *Incident Beam* optics tab
   a. Double click any item. Incident beam optics window will appear.
   b. Set *Automatic Attenuator Usage* to "Preset Intensity" with *Activate* Level set to 500,000 and *Deactivate* Level to 450,000.

VII. Measurement – Symmetrical Scan

1. Simplest way to execute scan is to do a *Manual Scan*. It is a relative scan i.e. executed around current goniometer position with the range specified in *Manual Scan* window.

2. To do *2Theta – Omega* scan first move 2Theta and Omega to middle positions of the scan range.
3. In *Manual Scan* window select *Scan Axis* "2Theta – Omega" and appropriate *Range, Step Size* and *Time per Step.*
4. Click *Start*. When scan is completed, save it through *File – Save As* menu. Manual Scan will be lost if it is not saved.
5. To do *Omega* scan on the diffraction peak, first move 2Theat and Omega to the diffraction peak position.
6. In *Manual Scan* window select *Scan Axis* "Omega" and appropriate *Range, Step Size* and *Time per Step.*
7. Click *Start*. When scan is completed, save it through *File – Save As* menu. Manual Scan will be lost if it is not saved.
   Note: In symmetrical scan mode Omega axis is always ½ of the 2Theta axis.

VIII. Logging out

1. Close the shutter.
2. Switch *Automatic Attenuator* to "Do Not Switch" and "Activated".
3. Move all angles to zero positions and Z to 5mm.
4. Lower the power of the x-ray tube to 40kV and 10mA.
5. Close *Data Collector.*