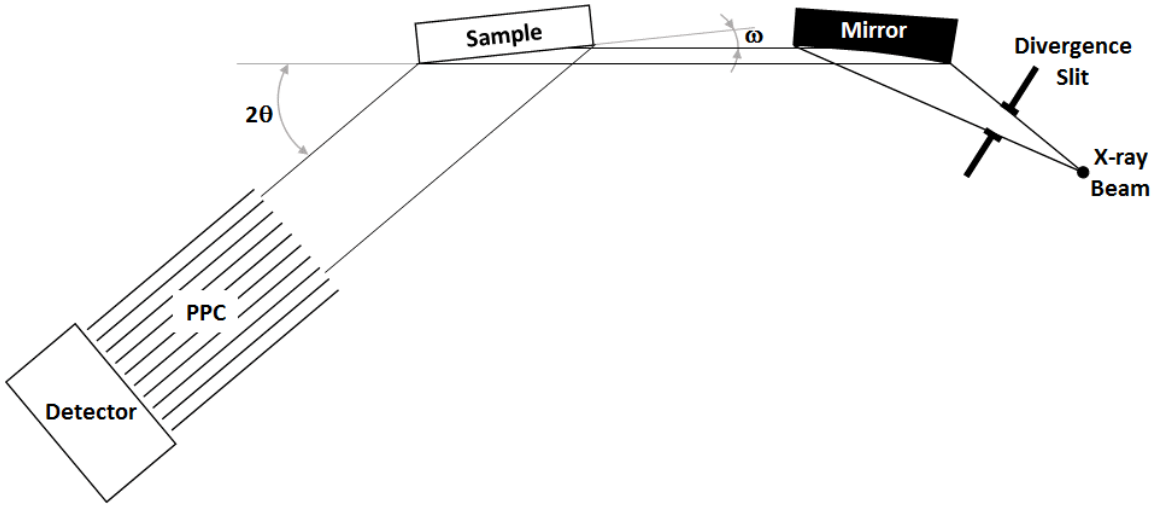
Grazing Incidence Diffraction using Mirror + PPC



I. Login

1. *Enable* instrument in **Badger**.
2. Start **Data Collector**.
3. Login by typing your "User Name" and "Password".
4. From the menu select *Instrument* → *Connect*.
5. Choose Configuration **Mirror + PPC**.
6. Click *OK*.

II. Hardware Setup for Initial Alignment

1. X-ray Tube is in "Line Focus".
2. Goniometer Resolution set to "Normal 0.001 deg".
3. Incident Beam Optics – **Mirror**
   Note: if you have to change incident beam optics please first turn Automatic attenuator to "Activate" status and then unplug attenuator cable.
   a. Insert 1/32° **Divergence Slit** into **Mirror** optics.
   b. If sample's vertical dimension is smaller than 25 mm, insert correct size **Mask**.
4. Diffracted Beam Optics – **PPC**
   a. Insert **PPC Receiving Slit** into **Parallel Plate Collimator** optics.

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:
      - *PreFIX Module* – select **Mirror**.
      - *Divergence Slit* – select 1/32° **Divergence Slit**.
      - *Anti-Scatter Slit* – select **None**.
      - *Mask* – select appropriate **Mask**.
      - *Beam Attenuator* – **Progr. Beam Attenuator**. For initial alignment set *Usage* = "Do not switch" and *Status* = "Activated". Make sure the *Description* = "Mirror". If not, click *Select* and select Mirror attenuator.
      - *Filter* – select **None** or **Beta filter** if you will be using one.
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:
      - *PreFIX Module* – select **Parallel Plate Collimator 0.27°**.
      - *Anti-Scatter Slit* – select **None**.
      - *Receiving Slit* – select **PPC Receiving Slit**.
      - *Filter* – select **None** or **Beta filter** if you will be using one.

- *Monochromator* – select **None** or **Flat Graphite Monochromator** if you use one. By default there is no monochromator mounted.

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. Diffractometer Zero Alignment

1. In **Instruments Settings** check **Z** position. If it is larger than 5mm move it back to at least 5mm.
2. Move all other motors to zero positions.
3. From Menu select *Measure* → *Manual Scan*.
4. From the *Scan Axis* drop down menu select **2Theta**.
5. Enter *Range* = 1°, *Step Size* = 0.005°, and *Time per Step* = 0.1sec. Then press *Start*.
6. After scan is finished, move **2Theta** axis to a peak position using one of the two ways:
   a. Peak Mode. Right click on mouse and select *Peak Mode*. New window will appear showing the **2Theta** position of the peak. Click *Move To*. Close the window.
   b. Move Mode. Right click on mouse and select *Move Mode*. Move **2Theta** to the center of the mass of the peak.
7. Select *User Settings* → *Sample Offsets* and set current **2Theta** position to zero.
8. Note the direct beam intensity.

V. Sample Mounting

1. Mount sample using scotch tape. Usually for GID long direction should be horizontal. If the sample is large, supplied clips can be used instead of scotch tape.
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).

VI. Moving Sample into the Beam Position and Aligning Sample Parallel to the Beam

1. Note the direct beam intensity. In **Instruments Settings** move **Z** to higher values until intensity starts to drop.
2. **Z** alignment can be performed using either optimization program or manually:
   a. Using optimization program.
      - Select *Measure* → *Program*. New window with user written programs will appear.
      - From the *Measurement Type* select *Optimize Program*.
      - Find proper program that says "Opt Z" and select it.
      - *Click OK* and start the scan.
   b. Manually.
      - *Select Measure* → *Manual Scan*.
      - 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. Press *Start*.
      - *After* scan is finished, right click on mouse and select *Move Mode*.
      - *Move* **Z** to the intensity value corresponding to ½ of the direct beam intensity.
3. Check if the sample surface is parallel to the beam.
   a. In **Manual Scan** window from the *Scan Axis* drop down menu select **Omega**.
   b. Enter *Range* = 2°, *Step Size* = 0.01°, and *Time per Step* = 0.1sec. Press *Start*.
   c. If **Omega** position (hairline) is not at the peak position, right click on mouse and select *Move Mode*. Move **Omega** to the center of the peak.
4. Repeat step 2 and then 3.
5. If after steps 3a and 3b hairline is positioned at the peak center, alignment is complete. Otherwise, repeat steps 2 and 3 again.
6. In *User Settings* → *Sample Offsets* set current **Omega** position to zero.
7. Close the shutter.

VII. Direct beam alignments are complete

1. Close shutter.
2. Determine the length of the sample.

3. Use the “Mirror Irradiation Length.pdf” graph to determine proper **Divergence Slit** at chosen incident beam angle **Omega** position. Choose **Divergence Slit** and **Omega** combination such that horizontal beam irradiation length matches horizontal sample size. *Note: The 1/2^o slit will provide highest intensity.*
4. Select **Incident Beam Optics** tab. Insert the proper **Divergence Slit**.
5. Select **Diffracted Beam Optics** tab. Remove PPC slit for highest intensity. Use PPC slit to improve resolution.

VIII. Measurement

1. In the **Incident Beam Optics** tab set *Beam Attenuator Usage* – “Preset Intensity” with *Activate Level* = 500,000 and *Deactivate Level* = 450,000.
2. 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 scan range specified in **Manual Scan** window.
3. To perform *2Theta* scan, first move **2Theta** to the middle position of the scan range.
4. Move **Omega** to position determined from “Mirror Irradiation Length.pdf” graph.
5. In **Manual Scan** window select *Scan Axis* **2Theta** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start*.
6. When scan is completed, save it through *File* $\rightarrow$ *Save As* menu. Manual Scan will be lost if it is not saved.

IX. Logging out

1. Close the shutter.
2. *Beam Attenuator – Usage* = “Do Not Switch” and *Status* = “Activated”.
3. Move all angles to zero positions and **Z** to 5mm.
4. Lower the power of the x-ray tube to 40kV and 20mA.
5. Close **Data Collector**.
6. *Disable* instrument in **Badger**.