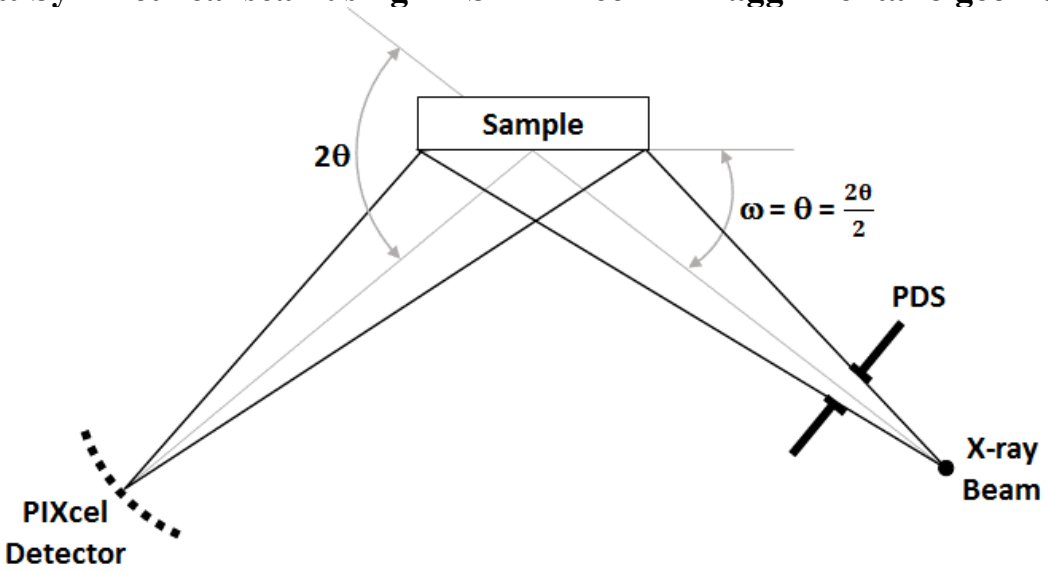
2θ-ω Symmetrical scan using PDS + PIXcel in Bragg-Brentano geometry



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 **PDS** + **PIXcel**.
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 – **PDS (Programmable Divergence Slit)**.
   a. Instead of **Ni Filter** insert **Cu 0.1mm attenuator**.
   b. If sample's vertical dimension is smaller than 25 mm, insert correct size **Mask**.
4. Diffracted Beam Optics – **PIXcel**.
   a. Make sure **Soller Slit** is inserted.

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* – **Programmable Divergence Slit**.
      - *Divergence Slit* – select *Usage* = "Fixed" and set to 1/32°.
      - *Anti-Scatter slit* – select **None**.
      - *Mask* – select appropriate **Mask**.
      - *Beam attenuator* – **None**.
      - *Filter* – select **Beta Filter**.
      - *Soller Slit* – select 0.04°.
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 **PIXcel**.
      - *Anti-Scatter slit* – select **None**.
      - *Receiving Slit* – **None**.
      - *Filter* – select **None**.
      - *Soller Slit* – select 0.04°.
      - *Detector* – select *Usage* = "Receiving Slit (0D)" and set active length to 0.16 mm.

- *Beam Attenuator* – **None**.

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 5 mm move it back to at least 5 mm.
2. Move all other motors to zero positions.
3. Make sure **Cu 0.1mm attenuator** is inserted.
4. From Menu Select *Measure* → *Manual Scan*.
5. From the *Scan Axis* drop down menu select **2Theta**.
6. Enter *Range* = 1º, *Step Size* = 0.005º, and *Time per Step* = 0.2sec. Then press *Start*.
7. 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.
8. In *User Settings – Sample Offsets* set current **2Theta** position to zero.

V. Sample Mounting

1. Mount 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).

VI. Moving Sample into the Beam Position

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.

VII. Direct beam alignments are complete

1. Close shutter.
2. Select **Incident Beam Optics** tab.
   a. The *Divergence Slit* can be used in "Fixed" and "Automatic" modes:
      - "Fixed" mode. Select slit divergence to desired value ranging from 1/32º to 4º. *Note: Make sure that at the lowest 2Theta angle the horizontal beam irradiated length matches the horizontal sample length.*
      - "Automatic" mode. Select proper irradiated length of the sample.
   b. Remove **Cu 0.1mm attenuator** and insert **Ni Beta Filter**.
3. Select **Diffracted Beam Optics** tab.
   a. *Detector* – select *Usage* = "Scanning (1D)" and set *Active Length* = 2.511º.

VIII. Measurement

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 perform *2Theta-Omega* scan first move **2Theta** and **Omega** to middle positions of the scan range. This is a symmetrical scan which means **Omega** = (**2Theta**)/2.

3. In **Manual Scan** window select *Scan Axis* **2Theta-Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start*.
4. When scan is completed, save it through *File* → *Save As* menu. Manual Scan will be lost if it is not saved.

IX. Logging out

1. Close the shutter.
2. Move all angles to zero positions and **Z** to 5 mm.
3. Lower the power of the x-ray tube to 40 kV and 20 mA.
4. Close **Data Collector**.
5. *Disable* instrument in **Badger**.