Standard Operating Procedure
Rigaku SmartLab XRD

II. Grazing Incidence XRD (GIXRD)
> FOLLOW the SOP strictly to keep the instrument in good condition. No explorations allowed on software unless permitted by lab manager

> NEVER use your own USB drive on the XPS computer. Data can be either uploaded to Yale Box, or copied to the Jump Drive provided by the Core.

> NEVER surf the web on the XPS computer to minimize the risk of the computer being hacked

> Users should acknowledge MCC in their publications. Please check the following link for details:

http://ywcmatsci.yale.edu/publications

> The core reserves the right to use the data for core promotion
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Rigaku SmartLab XRD Standard Operating Procedure

1 Introduction

a) Instrument features:
   > Full automated alignment under computer control
   > A high-efficiency 2D detector (Hypix 3000) with high-count rate
   > Cross Beam Optics (CBO) permits easy switching between focusing (BB) and parallel beam (PB) geometries without reconfiguration
   > In-plane diffraction arm for in-plane measurements without reconfiguration
   > High temperature (~1500 C) measurements in air, vacuum and helium
   > Air-Tight Sample Container for contamination free measurement

2) Location

   Materials Characterization Core
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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please follow strictly the SOP to keep the facility under good condition. No explorations on program allowed unless approved by core manager.
2 Sample Preparation

1) **Wear gloves** and clean the sample holder with provided IPO.
2) Powder samples should be milled to below 5 µm to minimize measurement deviations due to particle size effects.
3) Two types of powder sample holders, the zero-background holder and glass holder are provided as shown below. An amorphous hump signal could appear in the spectra on glass holder, which can be avoided if use the zero-background holder.

![Zero-background and Glass holder images]

4) Put powder samples at the center of the circle on the zero-background holder or square on the glass holder as shown below; cover the powder with a weighing paper and press and rotate sample with a glass slide to flat the sample surface.

![Sample placement images]

3 XRD Computer Login

1) Login FOM system: click on **Click here to login with NetID** to unlock the screen lock.
2) Check system status:
   a) Open the SmartLab Guidance software if it was closed (login: administrator, password: rigaku).
   b) Check the highlighted boxes on the bottom left of the window as shown below. Make sure that the “CBO selection slit” is set at BB, Bragg-Brentano focusing mode, and if the X-ray is at shutdown status, no number display for Tube voltage and Tube current.

![Hardware Status](image)

   c) If the Tube voltage and Tube current read 40 kV and 44 mA, skip Step d) and e) below.
   d) If the system is at shutdown status, click **Startup** button in the Package/Macro Measurement window below:
e) In the popup **Startup** window below, choose **Use everyday** if the machine was used within 24 hours and click **Execute** button. It takes **15 minutes** for the system to reach the operation power of **40 kV** and **44 mA**.

![Startup window](image)

4 User Package Definition

1) On the **Package** window at the top right of software window, find **Preinstalled > General Measurement > General (Bragg-Brentano Focusing)** package as shown below:
2) Double Click on **General (Bragg-Brentano Focusing)** to open the package in the **Package/Macro Measurement** window below which composes of three parts as shown below:

![Image of Package/Macro Measurement window]

3) Click on **Save As** button on above window to save the package as user defined package which will appear in the **User defined** tab on the top right side of the software window. On the bottom of **User defined** tab click 📁 to create a user folder and move saved package file into the folder by clicking 📁.

5 **Sample Loading**

1) Press the **Door Lock** button on the instrument cabinet door, wait till the button flashes, and then grab the handle (highlighted below) to slide the door open.

![Image of handle]

2) Push the glass or zero background holder all the way into the **height reference sample plate** as shown below. Align the holder to the center of the **plate** as highlighted by a dashed yellow line.
Note: If no enough sample to cover the entire holder, try to extend the sample area along the longer side of holder (dashed yellow line)

3) Insert the right slit size IS L (length limiting incident slit) as shown in the left picture below. Choose typical sizes from 2, 5, or 10 mm. The x-ray beam size is doubled when reaching sample surface as shown on the illustration below. For instance, if choosing 5 mm slit size, the exposed sample surface area/length will become ~ 10 mm across the X-ray beam.

4) Close the cabinet door and hit Door Lock button

6 Optics Alignment

1) Insert the PB slit into the CBO adapter and click on 1 Optics Alignment (PB/PSA) in the Package/Macro Measurement window below:
2) Click **Execute** button on the popup **Optics Alignment (PB/PSA)** window below to start optics alignment:

3) Follow the instructions on the popup **SmartMessage** window below to replace required parts and click **OK** to continue. The **Optics Alignment (PB/PSA)** window will be active after finish in ~10 **minutes**.  
**Note**: Keep an eye on alignment process, if the calibration curves become stuck for more than 5 minutes, it could indicate the software communication issue. Computer or program restart may be required.
4) If the following window appears, follow the instruction on the window to proceed and click OK after finish. **Make sure** the units are tightened. **Remember** also remove the PSA open in order to place PSA_0.114 unit.
5) Click OK on the Optics Alignment (PB/PSA) window in Step 2) above after finish.

7 Sample Alignment

1) Open user specific package file in User defined tab created in Section 4 User Package Definition.
2) Remove the Center slit from the height reference sample plate.
3) Flip the glass powder holder over and push the holder all the way into the height reference sample plate and place the sample on top of the square as shown below.
4) Click on as highlighted in the Package/Macro Measurement window below:

5) In the pop-up Sample Alignment (BB) window below, choose Flat sample and Input the Sample thickness (mm) above the glass holder; choose Put a sample when the sample alignment starts and click Execute button.
6) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment (PB)** window will be back active in ~ 2 minutes after finish.

![Sample Alignment (PB) window](image)

7) Click **OK** on the **Sample Alignment (BB)** window in **Step 5)** above to quit the window.
8) **Repeat** sample alignment for each sample even with the same thickness.

### 8 Sample Measurement

1) Click on **3 Message Box** button as highlighted in the **Package/Macro Measurement** window below:

![Package/Macro Measurement window](image)

2) **Skip Step 3)** - if the peak position of interest is known or for repeating similar samples.
3) Click **OK** in the popup **Message Box** below to run a **theta/theta** scan to find the peak of interest for peak intensity optimization.
4) Click on button as highlighted in the Package/Macro Measurement window below:

5) Perform following steps on the popup General Measurement window below:
   a) Specify File name and folder as highlighted in the window below.
   b) Check K beta filter method
   c) Select 1D mode
   d) Click Read current slits button
   e) Set measurement conditions:
      > Exec: click/check small box to activate line 1.
      > Mode: Continuous
      > Range: Absolute
      > Start (deg): 10.0000. Never change to below 5. The detector will be damaged.
> Stop (deg): 90.0000, the upper limit.
> Step (deg): 0.0500
> Speed Duration time (degree/min): 10.0000, choose lower speed to smooth spectra.
> IS mm: 1, recommended size.
> RS1 mm: 20.000, recommended size.
> RS2 mm: 20.1, recommended size.
> Voltage (kV): 40, maximal voltage. Never change to above 40 to damage the X-ray tube.
> Current (mA): 44, maximal current. Never change to above 44 to damage the X-ray tube.

f) Select box near Drive the 4 axes to the current zero positions after measurement completed.
g) Click Execute button to start measurement
h) Follow the instruction on SmartMessage windows if appear and hit OK

6) Replace the PSA_0.114deg unit with PSA_open as instructed in the SmartMessage window below and click OK:
7) Click OK button on **General Measurement** window and popup **File Save** window and find the peak position of the interest in saved spectrum.

8) Click on **button as highlighted in the **Package/Macro Measurement** window below:

9) Click **OK** in the popup **Message Box** below to run **Omega** scan at fixed 2-theta peak position found in **Step 5** above to optimize omega angle.
10) Perform highlighted steps on the popup **General Measurement** window below:
   a) Specify file name and folder,
   b) Make sure 0D is selected in Data acquisition mode,
   c) Select Omega in Scan axis.

11) Click **Set** button to open the Options window below. In the highlighted area, select **Move to origin** and input 2-Theta angle, the peak of interest found in **Step 5** and click **Close** button.
12) Go back to **General Measurement** window in Step 11 above and click **Execute** button.
13) Follow instruction on SmartMessage window below to install the **PSA_0.5deg** unit and click **OK** button.

14) Choose the Omega angle close to the plateau of the Omega scan curve as shown below:
15) Click on 

button as highlighted in the Package/Macro Measurement window below:

16) Click **OK** in the popup Message Box below to run GI 2-theta scan at optimized omega angle.
17) Perform highlighted steps on the popup **General Measurement** window below:
   a) Specify file name and folder,
   b) Make sure 0D is selected in Data acquisition mode,

18) Click **Set** button to open the Options window below. In the highlighted area, select **Move to origin** and optimized Omega angle found in **Step 14** and click **Close** button.
9 Checklist after Experiment

1) Back up your data to Yale Box cloud drive (box.yale.edu) or using Core USB drive. **Do not** use personal USB drive.

2) **Remove** sample from the stage. **Never leave samples inside XRD.**

3) The user who finishes near 6pm at regular time or **anytime** in off-peak hours should shutdown the instrument by clicking **Shutdown** in the window below:
4) Make sure XG Off is chosen and click Execute button in the popup Shutdown window below and watch the Tube current start to drop from 44 mA before logoff FOM.

5) Never minimize or close SmartLab Guidance software.

6) Logoff FOM program: click the icon on the taskbar below to activate the FOM program and click Logoff button in the FOM window. If any issues occurred during scan, check “Something wrong” and type message in the Comments space.

7) 

8) Clean the sample holders and glass slides with clean wipes IPA.

9) Store the sample holder and other tools back into the tool box.