Standard Operating Procedure Rigaku SmartLab XRD

II. Grazing Incidence XRD (GIXRD)



Yale West Campus Materials Characterization Core *ywcmatsci.yale.edu* ESC II, Room A119C 810 West Campus Drive West Haven, CT 06516

- 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 Room A119C 810 West Campus Drive West Haven, CT 06516

b) Primary Staff Contact

Dr. Min Li Tel: 203-737-8270 Email: min.li@yale.edu Office: ESC II, Room E119D

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 System Status Check

- 1) Please check to make sure the instrument sliding door is closed and there is no beeping sound once getting into the XRD room.
- 2) If there is a beeping sound from the instrument and meanwhile the OPERATE light is

flashing as show below. Hit the white OFF button on the front panel above and contact manager immediately.



3) Meanwhile, please check the floor at the back corner of the machine and make sure no water leaking on the floor. If no water leaking and the manager can't be reached, before leaving, put a machine down not on the machine sliding door.



- 4) If there is water leaking, it must be taken care before leaving. Please go to the service corridor A117 outside the Core in the next door and shut down the **XRD chiller** at the end of the corridor. The key is hang on the shelf right across the manager's office.
- 5) Come back to the XRD room and clean the water leak with paper tower.
- 6) Leave a machine down note on the machine sliding door.

3 Sample Preparation

- 1) Wear gloves and clean the sample holder with provided IPO.
- 2) The film sample can be put on the back of the glass powder sample holder. Make sure the sample is placed at the center of the square as shown below.



4 XRD Computer Login

1) Login FOM system: click on Click here to login with NetID to unlock the screen lock.

Clic	k here	to login	with Net	1D		
Click he	ere if yo	Or Du do no	ot have a	NetID		
			Or	Or 🖵	Click here to login with NetID Or Click here if you do not have a NetID	Or 🖵

- 2) Check system status:
 - a) Open the SmartLab Guildance 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.



- 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,
 - If the machine was used within one day, choose Use everyday and click Execute button. It takes 15 minutes for the system to reach the operation power of 40 kV and 44 mA.
 - > If the instrument has not been used for more than one days, choose Not used for 2days-1 week. It takes 30 minutes to warm up the X-ray tube.

Startup	x
Timer	
11/11/2016 🗐 🔻 07:00:03 🚔	
Estimated BE: 2016/11/11 07:14:36	
Generator usage Use everyday	-
XG set: Set	-
Voltage(kV): 40 Current(mA): 44	
Execute OK Can	cel

5 Optics Alignment

- 1) Open the GIXRD package file in User defined tab on the right side of the software window.
- 2) Insert the **PB** slit into the **CBO** adapter and click on **1** Optics Alignment (PB/PSA) in the **Package/Macro Measurement** window below:

Startup Shutdown Execute	
1 Optics Alignment (PB/PSA)	
4	
2 Sample Alignment (PB/PSA)	
<u>. </u>	
3 Message Box	
4 General Measurement	
₽	
5 Message Box	
₽	
6 General Measurement	
₽	
7 Message Box	
8 General Measurement	

3) Click **Execute** button on the popup **Optics Alignment (PB/PSA)** window below to start optics alignment:

Optics Alignment (PB/PS/	A)					
Change optics						
Current attribute :	Bragg-Brentano focusing					
Destination attribute :	Medium resolution parallel beam/PSA					
Optics alignment conditions Change optics without alignment						
Print out results after the second	er alignment					
	2					
Execute	Import Export OK Cancel					

4) 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, please contact manager. It could indicate the software communication issue.



5) 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.





- 6) Click OK on the Optics Alignment (PB/PSA) window in Step 2) above after finish.
- 6 Sample Alignment
 - 1) Remove the Center slit from the height reference sample plate.
 - 2) 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.



3) Click on 2 Sample Alignment (PB/PSA) as highlighted in the Package/Macro Measurement

window below. For samples with similar heights, only the first sample alignment is required.

Startup Ke Execute
1 Optics Alignment (PB/PSA)
2 Sample Alignment (PB/PSA)
3 Message Box
<u>.</u>
4 General Measurement
<u>.</u>
5 Message Box
6 General Measurement
7 Message Box
Ū.
8 General Measurement

4) 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.

Sample Alignment (PB/PSA)	3
Sample alignment conditions	
🔿 No height alignment	
Ourved sample (Z scan only)	
Flat sample	
Sample thickness (mm): 0.5	
Run recommended sequence Customize conditions Customize	
✓ Put a sample when the sample alignment starts.	
Put a sample every time the sample alignment starts in a repeated measurement.	
Print alignment result.	
	?
Execute Import Export OK Cancel	

5) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment** (**PB**) window will be back active in ~ 2 minutes after finish.



- 6) Click OK on the Sample Alignment (PB/PSA) window in Step 4) above to quit the window.
- 7 Sample Measurement
 - 1) **Skip Steps 3-5** inside the window below if the interested film peak position is already known from previous measurements. Otherwise proceed from the steps below.

Startup	Execute
1 Optics A	lignment (PB/PSA)
	Ŷ
2 Sample A	Alignment (PB/PSA)
3 M	lessage Box
	♦
4 Gener	al Measurement
	₽
5 M	lessage Box
	Û
6 Gener	al Measurement
	₽.
7 M	lessage Box
	Û
8 Gener	al Measurement
Click on	3 Message Box

2) Click on <u>3Message Box</u> button as highlighted in the **Package/Macro Measurement** window below. Read the message and click OK to close.

Message Box	×
Icon : Sequence Japanese message :	•
English message : Run general theta/2theta	scan to find interested pe
ОК	? Cancel

3) Click on

4 General Measurement

button as highlighted in the Package/Macro

Measurement window below. This measurement is to have a full range scan of the thin film to find peak of interest.

Startup 🔀 Execute
1 Optics Alignment (PB/PSA)
$\overline{\Omega}$
2 Sample Alignment (PB/PSA)
3 Message Box
4 General Measurement
V
5 Message Box
6 General Measurement
7 Message Box
<u></u>
8 General Measurement

- 4) 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.
- <mark>> Step (deg): 0.0500</mark>
- > 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

l	File pa	ime : 💽	XRD LIs	ers\Min Li\GIXRD	\SOP1.ras)					
		e name :			por mas										
	Memo	_							_						
	Menio	·													
	Manual	l exchange slit o	ondition	IS						Det	tector sett	ting		Data acquisition	mode
	s	oller/PSC		ISL	PSA		Soller	N		D	etector #	2 (HyPix 3	3000(H)) 🔻		•
		(deg)		(mm)	(deg)		(deg)	c)						b)	
	5.0	•	10.0		/pen	▼ None		Rea	d current slits						
ſ	Measur	rement conditio	IS												
	Exec.	Scan ax	s	Mode	Range	Start	Stop	Step	Speed	IS	RS1	RS2	Attenuator	Comment	Optior
						(deg)	(deg)	(deg) [Ouration time	mm 🔻	mm 🔻	mm			
) 💽) 📃 💽		_	-			
	1 🔽	Theta/2-Theta	-	Continuous 🔻	Absolute	▼ 5.0000	90.0000	0.0400	10.0000	1.000	20.000	20.1			Set
	2	z-meta/omeg	a v	conunuous •	ADSOIUTE	- 3.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Jer
	3 🔳	2-Theta/Omeg		Continuous 🔻	Absolute	→ 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Set
		2-Theta/Omeg		Continuous 🔻	Absolute	→ 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Set
	4		a 🔻	Continuous 🔻	Absolute	- 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Set
	5	2-Theta/Omeg			1					1.000	1.000	1.000			Set
	5	2-Theta/Ome		Continuous 🔻	Absolute	▼ 5.0000	90.0000	0.0100	3.0000						
	5 📄 6 📄 7 📄	2-Theta/Omeg	ja –	Continuous 🔻	Absolute	▼ 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			
	5 🖸 6 🖸 7 🗖 8	2-Theta/Omeg 2-Theta/Omeg 2-Theta/Omeg	ja v	Continuous V Continuous V	Absolute Absolute	▼ 5.0000▼ 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Set
	5 🗍 6 💭 7 💭 8 💭 9	2-Theta/Omeg 2-Theta/Omeg 2-Theta/Omeg 2-Theta/Omeg	ja v ja v ja v	Continuous V Continuous V Continuous V	Absolute Absolute Absolute	 ▼ 5.0000 ▼ 5.0000 ▼ 5.0000 	90.0000 90.0000 90.0000	0.0100 0.0100 0.0100	3.0000 3.0000 3.0000	1.000	1.000	1.000 1.000	· · · · · · · · · · · · · · · · · · ·		Set Set Set
	5 🗍 6 💭 7 💭 8 💭 9	2-Theta/Omeg 2-Theta/Omeg 2-Theta/Omeg	ja v ja v ja v	Continuous V Continuous V	Absolute Absolute	▼ 5.0000▼ 5.0000	90.0000	0.0100	3.0000	1.000	1.000	1.000			Set

5) Follow the instruction on SmartMessage window below to replace the **PSA_0.114deg** unit with **PSA_open** and click **OK**:





- 6) Click **OK** button on **General Measurement** window and popup **File Save** window and find the peak position of the interest in saved spectrum.
- 7) Skip the **steps 5-7** inside the window below if the X-ray grazing angle is known from previous measurements on similar samples. Otherwise proceed with steps below.

Startup Ke Execute	
1 Optics Alignment (PB/PSA)	
3 Message Box	
4 General Measurement	
6 General Measurement	
7 Message Box	
8 General Measurement	
8) Click on 5 Message Box	button above and read the message in the Message

Box below and click **OK** to close.

Message Box
Icon : Sequence Japanese message :
English message : Run Omega scan at fixed 2-theta peak position to oj
? OK Cancel

9) Click the ^{6 General Measurement} in the window below decide the X-ray grazing angle (**Omega** scan) with the detector fixed at the 2-theta peak position found in

U (0 /
4 Ge	eneral Measurement
10	otics Alignment (PB/PSA)
	Ŷ
2 Sa	mple Alignment (PB/PSA)
	Ŷ
	3 Message Box
-	Ŷ
4	General Measurement
	Ŷ
	5 Message Box
	Û
6	General Measurement
	Ŷ
	7 Message Box
	Ŷ
8	General Measurement
<u></u>	

- 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.

ile n	ame :	XRD Us	ers\Min Li\GIXRE)\omega scan.r	as] a)				
Jamp	le name : WT	e1												
1emo	·:													
anua	al exchange slit co	ondition	IS						Det	tector set	ing		Data acquisition r	node
5	Soller/PSC (deg)		IS L (mm)	PSA (deg)		Soller (deg)			D	etector #	2 (HyPix 3	000(H)) 🔻	b)	•
5.0	▼ [1	0.0	.).5	▼ None	•	Rea	id current slits						
leasu	rement condition	IS												
xec.	Scan axi	s	Mode	Range	Start (deg)	Stop (deg)	Step (deg) [Speed Duration time	IS mm 🔻	RS1	RS2 mm	Attenuator	Comment	Options
		T			▼		▼							
V	Omega	-	Continuous 🔻	Absolute 🔻	-2.0000	2.0000	0.0400	10.0000	1.000	20.000	20.1		D	Set
	2-Theta/Omeg	a 🔻	Continuous	Absolute 🔻	-4.9355	4.9355	0.0400	3.0000	0.200	20.000	20.000		d)	Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	10.0000	65.0000	0.0400	3.0000	0.200	20.000	20.000			Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
_	2-Theta/Omeg	a 🔻	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
	2-Theta/Omeg	a –	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
	2-Theta/Omeg	a 🔻	Continuous 🔻	Absolute 🔻	5.0000	90.0000	0.0400	3.0000	1.000	20.000	20.000			Set
				ons after the m										

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.

Axis	Action		Origin(Center) (deg)	Oscillation range (+/-) (deg)	Start (deg)	Stop (deg)	Speed (deg/min)
2-Theta :	Move to origin	-	33.0000	1.0000	0.0000	10.0000	0.0000
Omega :	None	-	0.5000	1.0000	0.0000	10.0000	5.0000
Phi :	None		0.000	1.0000	0.000	10.000	5.000
2-ThetaChi :	None	-	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	-	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	Ŧ	0.0000	1.0000	0.0000	10.0000	5.0000

- 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. Typically the angle at or slightly below dashed line is good to have enough signal intensity.



15) Click on	7 Message	Box	button as	highlighted	in the	Package/Macro
,	nent window belo	W.		88		
Startup	Execute					
1 Optio	cs Alignment (PB/PSA)					
r	Ŷ					
2 Samp	ble Alignment (PB/PSA)					
r	Ŷ					
	3 Message Box					
10						
4 Ge						
	5 Message Box					
	₽ message box					
6 Ge	eneral Measurement					
	7 Message Box					
	Q					
8 Ge	eneral Measurement					
16) Read the r	nessage and click	OK to close	the window	v below		
Message Box	_		the whiteow			
Icon : Sec	quence 🔹					

	Icon : Sequence	
	Japanese message :	
	English message :	
	Run GI 2-theta scan at optimized omega angle	
	?	
	OK	
17)	Click 8 General Measurement	in the window below to perform the final grazing
	incidence scan.	



18) 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,

eneral N	leasurement			×		\frown		~							X
Save m	easurement data														
File na	me: C:\XRD	Use	ers\Min Li\GIXRD	SOP_GIXR	D.r	as			(
Sample	e name :														
Memo	:														
Manual	exchange slit condit	tion	s							D	etector set	tina		Data acquisition r	node
	oller/PSC (deg)		IS L (mm)	PSA (deg)			Soller (deg)						3000(H)) 🔻	OD	•
5.0	▼ 10.0		• 0	.5		▼ None	•	Rea	ad current slits						
Measurement conditions															
Exec.	Scan axis		Mode	Range		Start (deg)	Stop (deg)	Step (deg)	Speed Duration time	IS mm	RS1	RS2 mm	Attenuator	Comment	Options
	(•) (T						•				
1 🔽	2-Theta	•	Continuous 🔻	Absolute	•	10.0000	80.0000	0.0400	3.0000	0.200	20.000	20.1	-		Set 🔺
2 🔳	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
3 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
4 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
5 🔳	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
6 📃	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
7 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
8 📃	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
9 🔳	2-Theta/Omega	-	Continuous 👻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000			Set
10 🔳	2-Theta/Omega	-	Continuous 🔻	Absolute	-	3.0000	80.0000	0.0100	3.0000	1.000	1.000	1.000	-		Set 👻
	ve the 4 axes to the ated scan duration :			ons after th	ie m	easuremen	t completed								?
Exec	ute										Ir	nport	Export	ок	Cancel

to origin and optimized Omega angle found in 6 General Measurement and click Close button.

19) Click Set button to open the Options window below. In the highlighted area, select Move

Axis	Action	C)rigin(Center) (deg)	Oscillation range (+/-) (deg)	Start (deg)	Stop (deg)	Speed (deg/min)
2-Theta :	None	•	0.0000	1.0000	0.0000	10.0000	0.0000
Omega :	Move to origin	•	0.5000	:.0000	0.0000	10.0000	5.0000
Phi :	None		0.000	1.0000	0.000	10.000	5.000
2-ThetaChi :	None	_	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	-	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	v	0.0000	1.0000	0.0000	10.0000	5.0000
None 🔻	None	–	0.0000	1.0000	0.0000	10.0000	5.0000

20) Go back to General Measurement window in Step 18) above and click Execute button.

8 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) Open a BB scan package file in your folder and **perform BB optical alignment**. Please check the powder XRD SOP for further instructions.
- 4) Put back the **K-Beta filter** after finish.
- 5) Remove the **center slit** from the stage.,

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6) 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:



7) 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 logging off FOM.

Shutdown		
Execute		
Shutdown conditions	🗐 PHA window # 1	926.25 mV
XG set: XG Off 🔹	🕂 Shutter	Close
	Tube voltage	40 kV
	🕜 Tube current	44 mA
Voltage(kV): 20 Current(mA): 2	🔚 X-ray ON time	19595.7 h
	😏 Theta_s	0.0000 deg
Execute OK Cancel	Detector	Second Detector
	AT1 . 1	0.0000 1

- 8) Never minimize or close SmartLab Guidance software.
- 9) Logoff FOM program: click the ¹/₂ 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.



- 10) Sign off on the logbook.
- 11) Clean the sample holders and glass slides with clean wipes IPA.
- 12) Store the sample holder and other tools back into the tool box.