Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

XRD – PB Instructions

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These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

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1. Package measurement flow

This manual describes how to set the measurement conditions and how to execute the Package measurement when the General (medium resolution PB) Package measurement is selected. Also, the conditions can be set and the measurement can be executed in the same way when one of the other General Package measurements is selected.

Figure 1.1 shows the procedural flow for a General (medium resolution PB) Package measurement.



Fig. 1.1 General (medium resolution PB) Package measurement procedural flow



2. Measurement procedures

1. Startup

Before measurement, set the output of the x-ray generator (XG) as described below.

(1) Click the Startup button on the flow bar to open the Startup dialog box.



(2) Uncheck the Timer box.

Startup		X
📕 Timer		
Start	C End	
9/13/2006	• 01:01	:01 🕂
Estimated BE:	2006/09/13	02:16:43
Generator usa	ge: Use every	day 💌
XG set:	Set	•
Voltage(kV) :	40 Curre	nt(mA): 30
Execute	ОК	Cancel

Fig. 2.1.1 Startup dialog box

- (3) Select an appropriate setting in the Generator usage box, based on the frequency of XG usage.
- (4) Select Set in the XG set box.
- (5) Enter the following values in the Voltage (kV) and Current (mA) boxes.

When using 3-kW sealed tube	40 kV, 30 mA
When using 9-kW rotating anode	45 kV, 200 mA

- Click the Execute button. (6)
- (7) The aging operation begins. The XG output will be set to the values entered in step (5) within 30 to 60 minutes.





2. Hardware setup

Before measurement, the equipment configuration must be set up for a General Package measurement.

The hardware setup method is described below.

(1) Select the Hardware Configuration command under the Options menu to open the Hardware Configuration dialog box.



(2) The Hardware Configuration dialog box shows the current hardware configuration.



Fig. 2.2.1 Hardware Configuration dialog box

(3) Table 2.2.1 shows the hardware configuration that enables the General Package measurement. If the configuration units shown in the Hardware Configuration dialog box differ from those indicated in Table 2.2.1, install the units specified in Table 2.2.1 at the designated locations, referring to the Horizontal Sample Mount X-Ray Diffractometer for Thin Film Analysis Instruction Manual (ME11550A).

Hardware configuration	Configuration units
X-ray generator	Cu target
Incident optics	CBO unit
	Standard incident optical unit
	Standard incident slit box
Goniometer	SmartLab or SmartLab(in-plane)
Base attachment configuration	Standard chi cradle
Attachment	Any
Receiving optics	Standard receiving slit box # 1
	Standard receiving optical unit # 1
	Standard receiving optical unit # 2
	Standard receiving slit box # 2
	Standard attenuator
Detector	Scintillation counter SC-70

Table 2.2.1 Hardware configuration for the General Package measurement

For example, if the target is set to Mo in the X-Ray Generator dialog box, you must change the target setting to Cu, since the General Package measurement will not be made unless the Cu target is used. For sealed tubes, you must replace the Mo tube with the Cu tube.

(4) Click the X-Ray Generator button to open the X-Ray Generator dialog box.

X-Ray Generator			
Name:	3kW	Hermetic	Ψ.
Target:	Mo	•	
Ka1 Ka2	Ka	Kb Filter	Default
0.70931 0.71361	0.7107	48 0.63230 Zr	
Focus:	1mm	x 10mm 👤	
Focus type:	Norm	al 💌	
Max. load:	3.00	3.000kW(0.002kW	
Max. tube voltage:	60	20-60kV(1kV Step)	
Max. filament current	60	2-60mA(1mA Step)	
			1
		OK	Cancel

Fig. 2.2.2 X-Ray Generator dialog box

- (5) Select Cu in the Target box. Change other applicable parameters, such as wavelength.
- (6) Click the OK button to close the X-Ray Generator dialog box and register the changes made in the Hardware Configuration dialog box.
- (7) If necessary, change configuration units using the Incident Optics, Receiving Optics, and Detector buttons. Modify the conditions in the Hardware Configuration dialog box in the same way.
- (8) Click the Update button in the Hardware Configuration dialog box.

Tip: Mounted configuration units are automatically detected for Base Attachment Configuration and Attachment.

(9) Confirm that each configuration unit displayed in the Hardware Configuration dialog box corresponds to the configuration unit indicated in Table 2.2.1, then click the Close button to close the dialog box.



Fig. 2.2.3 Hardware Configuration dialog box

2.3 Setting Package measurement conditions

Package measurement	Configuration Parts
General	• Optics Alignment (PB)
(medium resolution PB)	Sample Alignment
	General Measurement
General	• Optics Alignment (PB-Ge(220)x2)
(high resolution PB-Ge(220)x2)	Sample Alignment
	General Measurement
General	• Optics Alignment (PB-Ge(400)x2)
(high resolution PB-Ge(400)x2)	Sample Alignment
	General Measurement
General	• Optics Alignment (PB-Ge(220)x4)
(ultra-high resolution PB-(220)x4)	Sample Alignment
	General Measurement
General	• Optics Alignment (PB-Ge(440)x4)
(ultra-high resolution PB-(440)x4)	Sample Alignment
	General Measurement

The Parts included in the General Package measurements are shown in the table below.

To perform the Package measurements, you must set the conditions of each of the three Parts individually. Described below is how to set the conditions of each Part.

CAUTION: If another Package measurement is selected or another task such as the Manual Control task is chosen, discard the set Part conditions. To save the set conditions to a file, click the Export button in each dialog box or save Package measurement conditions as described in the "Tip" section in Subsection 2.3.3 (6).

2.3.1 Setting Optics Alignment Part conditions

Described below is how to set the conditions of the Optics Alignment (PB) Part. Conditions can be set for the other Optics Alignment Parts in the same way.

(1) Click the Optics Alignment (PB) button on the flow bar to open the Optics Alignment (PB) dialog box.

Optics Alignment	(PB)						
Change optics							
Current attribute	Bragg-Brentano focusing						
Destination attribute	Medium resolution parallel beam/RS						
Optics alignment conditions							
Change optics (quick alignment only)							
Optics alignment nam	Optics alignment name @Medium resolution PB						
F Alignment for in-plane measurement							
Print out results a	after alignment	?					
Execute Im	nport Export DK	Cancel					

Fig. 2.3.1 Optics Alignment (PB) dialog box

(2) Uncheck the Change optics (quick alignment only) box.



- (3) In the Optics alignment name box, select a location for storing optics alignment results.
 - To store optics alignment results under a new optics alignment name, click the OK or Cancel button to close the Optics Alignment (PB) dialog box. Then, select the Optics Management command from the Options menu to open the Optics Management dialog box and add a new optics alignment name. After adding a new optics alignment name, return to step (1) in this subsection. For more information on creating an optics alignment name, refer to Chapter 17 of the SmartLab Guidance Reference Manual (ME13365A).
- (4) To print the optics alignment results, check the Print out results after alignment box.

Tip: If both the Change optics (quick alignment only) and Print out results after alignment boxes are checked, the alignment results stored under the selected optics alignment name will be printed.

(5) Click the OK button to close the dialog box.

Tip:

- 2.3.2 Setting Sample Alignment Part conditions
 - (1) Click the Sample Alignment button on the flow bar to open the Sample Alignment dialog box.

Sample information		🛛 🗹 Direct beam half	cut alignment conditio
Sample thickness(mm) Sample width(mm) Sample height(mm)	1.0 10.0 10.0	Surface normal Alignment criteria	slignment conditions Standard Medium
Clear omega offset		Set chi, phi = 0	Customize
Put a sample when the Put a sample every tin	sample align the sample	ment starts. e alignment starts in a rep	peated measurement.

Fig. 2.3.2 Sample Alignment dialog box

(2) Enter the sample thickness, sample width and sample height in the Sample thickness (mm), Sample width (mm) and Sample height (mm) boxes.



- (3) Check the Direct beam half cut alignment conditions box.
- (4) If the incident x-ray angle to the sample surface has to be precisely determined to make a measurement such as a reflectivity measurement, check the Surface normal alignment conditions box.
- (5) If the Surface normal alignment conditions box is checked, select Standard in the Alignment criteria box. And select the nominal density of the surface layer from Very low, Low, Medium, or High in the Surface density box.



(6) Check the Clear omega offset and Set chi, phi = 0 boxes.

Tip: If the results of the previous sample alignment are kept, the next sample alignment uses this position as a reference and may not work correctly. In ordinary cases, check the Clear omega offset and Set chi, phi = 0 boxes.

(7) Select the Run recommended sequence radio button.



(8) Click the OK button to close the dialog box.

2.3.3 Setting General Measurement Part conditions

(1) Click the General Measurement button on the flow bar to open the General Measurement dialog box.

Gen	eral	Measure	ement																$\mathbf{\times}$
58	iyê m	easuremen	t data																
Fi	e nan	ne	C:\Doc	umen	ts and Set	tings	administrat	or\M	y Docume	nts\Rigaku	Test								
Sa	mple	name																	
M	emo							_											
M	anual	exchange	slit cond	ditions	5	_		_			_			Detector	setting				
Soll (de	er/PS	c	ISL (mm)			PSA (den	,		Soller (dea)					Detect	or #1 (SC-	70) 🔻			
No	ne		10.0)	•	Non	e	-	None		Read	current slits		1.0					_
1127	safaur	ement con	litions			-			3		-			_					
Đ	kec.	Sca	an axis		Mode	6	Range	4	Start	Stop	Step	Speed	IS	RS1	R52	Attenuator	Comment	Measureme	ent
				l.				-	(deg)	(deg)	(deg)	Duration time	mm 💌	mm 🔻	mm			origin	
		D There is		V	Carling	V	Abaabaa	V -	2 0000	V	0.0100	V V 2.0000	1.000	1.000	<u></u>	V		V V Sohip	
1	-	2-metalo	neya	-	Continuot	15 -	Absolute		3.0000	00.0000	0.0100	3.0000	1.000	1.000				Setup	
4	F	2. Thetal	amega		Continuo	R <u></u>	Absolute		2.0000	80.0000	0.0100	3,0000	1.000	1.000				Sehin	-
4	F	C. Thatale	mega		Continued		Abcolute	-	3 0000	an 0000	n atos	3 0000	1,000	1.000				Sehip	
5	F	2. Theball	mega	-	Continuos		Absolute		3.0000	20 0000	0.0100	3.0000	1,000	1.000				Setur	
6	Г	2-Theta/C	imeda	-	Continuos		Absolute	+	3.0000	80.0000	0.0100	3.0000	1,000	1.000	<u> </u>			Setup	
7	Г	2-Theta/C	mega	+	Continuóu	15 -	Absolute	+	3.0000	80.0000	0.0100	3,8080	1.000	1.000				Setup	
8	Г	2-Theta/C	mega	-	Continuou	15 9	Absolute	-	3.0000	80.0000	0.0100	3,8000	1.000	1.000		~		Setup	
9	Г	2-Theta/C	Imega	Ŧ	Continuou	is 🐨	Absolute	Ψ.	3.0000	80.0000	0.0100	3,0000	1.000	1.000		Ψ.		Setup	
10	Г	2-Theta/C	mega	Ŧ	Continuol	15 -	Absolute	Ŧ	3.0000	0000:08	0.0100	3.0000	1.080	1.080		· ·		Setup	-
Г	Drive	the 4 axes	to the	curre	nt zero po:	sitions	after the r	neas	urement	completed.									
Cal	culate	ed scan dur	ation :	00:25	:40													_	?
	Exec	ute													Import	Export	ок	Cancel	

Fig. 2.3.3 General Measurement dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the data measurement, the measurement data will be saved with the specified file name.
 - 1. Click the [...] button to open the Save As dialog box.

Save As			? 🔀
Save in: [My Documents	• + 1	🕂 💷-
My Music	S		
File <u>n</u> ame:	RigakuTest.ras		Save
			1



- 2. Enter the settings in the Save in and File name boxes.
- 3. Click the Save button.

(3) Enter any required information in the Sample name and Memo boxes (optional).



- (4) Set the manual exchange slit conditions.
- (5) Select "SC-70" from Detector setting.
- (6) Set the measurement conditions.



To set the manual exchange slit conditions and measurement conditions, refer to "General Measurement Part" Help Topic of the online help section of the SmartLab Guidance software.

- (7) If the four axes (2-theta, omega, chi, phi) are driven to the current zero positions after the data measurement, check the Drive the 4 axes to the current zero positions after the measurement completed box.
- (8) Click the OK button to close the dialog box.

γ.	Chiek the		
	(i)Tip:	To save all the	e Part conditions set in Subsections 2.3.1 through 2.3.3 to a single
	\checkmark	file, click the S	Save as button on the flow bar to open the Save As dialog box,
		then enter the	settings in the Save in and File name boxes. Click the Save
		button.	
		Select-alsaved 1	Package measurement conditions file (*.sqp) in the box showing
		the flow bar to	load-he conditions stored in that file. 🌾 🗈 📸 🏢 -
		Chan Music	
			s
			-
		F1	
		rile <u>n</u> ame:	BBopticsDefault.sdp
		Save as type:	Package files(*.sqp) Cancel
			Constal (modium resolution DD /DCA)
			General (medium resolution PD/FSA)
		BBo	pticsDefault.sgp <c:\documents -<="" and="" set="" th=""></c:\documents>
			pave as Set default conditions

2.4 Customizing Part conditions

i

The user is free to set scan conditions and slit conditions of the Sample Alignment Part. Use the Customize dialog box to set scan conditions and slit conditions.

Tip: To confirm the conditions set for Run recommended sequence, click the Set recommended values button in the Customize dialog box.

2.4.1 Customizing Sample Alignment Part conditions

- (1) Select the Customize conditions radio button in the Sample Alignment dialog box.
- (2) Click the Customize button to open the Customize dialog box.

Custor	nize - Samp	le Alignment	0				
Sample	information			Direct	beam half cut	alignment conditic	ins
Samp	le thickness(mr	n) 1.0		💌 Surfac	ce normal align	ment conditions	
Samp	le width(mm)	10.0	_	Alignme	ent criteria	Standard	•
Samp	le height(mm)	10.0		Surface	e density	Medium	•
				2			
🔽 Clea	ar omega offset	I.	Set chi, phi = 0).			
Slit cor	nditions			Scatterin	g angle for alig	nment	
IS(m	m) IS L(m	m) RS1(m	m) RS2(mm)				
0.050	10.0	• 0.050	20.000	Alignn	nent 2-Theta(d	eg) 0.5000	
🔽 Dire	ct beam half cu	ut alignment mea	surement condit	lions			
At	tenuator 1/10	0000 💌	Intensity	threshold(cps)	5000		
	Scan axis	Range	Start	Stop	Step	Speed	Delta
7			(mm)	(mm)	(mm)	(mm/min)	(mm)
1	Z	Absolute	-3.0000	1.0000	0.0100	8.0000	0.5000
	Scan axis	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Delta (deg)
[Omega	Absolute	-3.0000	3.0000	0.0100	8.0000	1.0000
🔽 Surt	ace normal alig	nment measure	ment conditions				
At	tenuator 1/10	100 💌	Intensity	threshold(cps)	3000	Repeat	3 🔹
	Scan axis	Range	Start (mm)	Stop (mm)	Step (mm)	Speed (mm/min)	Delta (mm)
[Z	Relative	-0.3000	0.3000	0.0100	4.0000	0.3000
Exec.	Scan axis	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Delta (deg)
I	Omega	Relative	-0.2000	0.2000	0.0020	1.0000	0.1500
	Chi	Relative	-3.000	3.000	0.050	15.000	3.000
						Set recomm	nended values
							Close

Fig. 2.4.1 Customize dialog box

The conditions set in the Sample Alignment dialog box are indicated in the Sample information, Direct beam half cut alignment conditions, Surface normal alignment conditions sections, and the Clear omega offset, Set chi, phi = 0 boxes.

- (3) If necessary, set conditions in the Slit conditions, Scattering angle for alignment, Direct beam half cut alignment measurement conditions and Surface normal alignment measurement conditions sections.
 - CAUTION: Clicking the Set recommended values button changes settings in the Slit conditions, Scattering angle for half alignment, Direct beam cut alignment measurement conditions and Surface normal alignment measurement conditions sections the to values recommended based on the settings specified in the Sample information and Surface normal alignment conditions sections. For more information, refer to "Sample Alignment Part" Help Topic of the online help section of the SmartLab Guidance software.
- (4) After setting the conditions, click the Close button to close the Customize dialog box.

2.5 Executing a Package measurement

Described below is the procedure for executing the complete series of measurement operations from the optics alignment to the general measurement.



(2) Click the Run button on the flow bar.

	Save as	Set default conditions	
	Startup	Run	
	General (medium re	esolution PB/PSA)	?
	1 Optic:	s Alignment (PB/PSA)	
Tip:	After executing a Package flow bar. The mark indic progress.	e measurement, the mark ag cates that the Part C orrespond	ppears on a button on the ling to the button is in
	Startup	Run	
	• 1 Optics	Alignment (PB/PSA)	
		Ŷ	

(3) If the following message appears, click the No button.

(4) A message will prompt you to replace optical devices in the middle of the measurement. Confirm the message and click the OK button.

(5) When the following message appears, place the height reference sample plate on the attachment and insert the center slit into the height reference sample plate. If another message appears at the same time prompting for replacement of the optical device(s) such as the selection slit, install the specified optical device(s) as indicated by the message.

(6) Click the OK button to execute optics alignment.

The optics alignment will be performed under the recommended conditions, and it will be completed in about 10 minutes.

(7) The specific procedure in this step depends on the setting or in the flow bar. Follow the directions given below.

When 🚨 (Don't show confirmation messages) is set:

The optics alignment results will be registered in the optics management database.

When **U** (Show confirmation messages) is set:

1. The following message will appear to verify the optics alignment has been completed. Click the OK button.

2. The Optics Alignment Results dialog box will appear. Confirm the results and click the Register button. The optics alignment results will be registered in the optics management database.

)ptics details					Hardware configuration
Axis	Abs. posi	Position	Intensity	Date	XG Type: Open, Rota
	0.3633 deg	0 deg	Empty	Empty	Target: Cu Gopiometer: SmartLab
2-Thet	0.0000 deg	0 deg	Empty	Empty	Base attachment config.: Std. c
Omega	0.5013 deg	0 deg	Empty	Empty	Attachment: XY-20mm, BxBy, >
Ts	-0.20025	0 mm	Empty	Empty	Receiving slit # 2: Standard incident :
Zs	-1.56187	0 mm	Empty	Empty	Receiving slit # 1: Standard rec
) Zd	-0.09906	0 mm	Empty	Empty	Attanuator: Standard Attenuato
Z	0.0000 mm	0 mm	Empty	Empty	Delectori
£				>	
Register					Do not register Cancel

value for each axis, but does not register the optics alignment results in the optics management database.

(8) The following message will appear. Based on this message, place the sample spacer on the attachment, place the wafer sample plate on the sample spacer, and place the sample on the wafer sample plate. If another message appears at the same time prompting for replacement of the manual exchange slit(s) such as the length limiting slit, install the specified manual exchange slit(s) as indicated by the message.

SmartMessage	
	Hide figures
Place the sample spacer (0-3 mm) and the wafer sample plate (standard) on the	attachment. 📥
Place the sample on the wafer sample plate.	
(*** <>> <	
sample spacer wafer sample plate wafer sample	
Insert length limiting slit 5 (mm) in incident slit box.	
-> Length limiting slit 10 (mm) is currently on incident slit box.	
	~
ок	Stop

(9) Click the OK button to execute sample alignment.

Direct beam half cut alignment and surface normal alignment will be performed under the specified conditions. The sample alignment will be completed in about 10 minutes.

Only when **(Show confirmation messages)** is set on the flow bar, the message will appear to verify the sample alignment has been completed. Click the OK button.

Message	
Sample alignment has been completed.	~
	3
ОК	Stop

(10) If the manual exchange slit(s) differs from those specified in the General Measurement dialog box, a message will appear like below. If instructed by the message, install the specified manual exchange slit(s) as prompted.

(11) Click the OK button to execute a data measurement.

The data measurement will be made under the specified conditions.

(12) After completion of the measurement, the measurement data will be saved under the file name set in Subsection 2.3.3 (2).

Only when $\[Med]$ (Show confirmation messages) is set on the flow bar, the message will appear to verify the data measurement has been completed. Click the OK button.

This is the end of the General Package measurement.

2.6 Changing configuration units

After a Package measurement (or Part) is executed, and the hardware configuration is not set for the Package measurement or the Part, the Hardware Configuration dialog box will open. A message appears below the dialog box prompting you to change the configuration units preventing the use of the Package measurement or the Part.

For example, if the target has been set to Mo in the X-Ray Generator dialog box, the displayed message will show "Target: Change Mo to Cu.", since the target must be Cu for the General Package measurements.

Fig. 2.6.1 Hardware Configuration dialog box

If the Hardware Configuration dialog box appears, replace the configuration units as prompted by the message in the dialog box and update the contents of the Hardware Configuration dialog box.

This procedure is described below.

(1) Change the target in the x-ray generator from Mo to Cu. For sealed tubes, replace the Mo tube with the Cu tube.

(2) Click the X-Ray Generator button to open the X-Ray Generator dialog box.

Name:	BkW	Hermetic
Target:	Мо	•
Ka1 Ka2 0.70931 0.71361 (Ka 0.7107	Kb Filter Defau
Focus:	1mm	× 10mm 💌
Focus type:	Norm	nal 💌
Max. load:	3.00	3.000kW(0.002kW
Max. tube voltage:	60	20-60kV(1kV Step)
	[co	

Fig. 2.6.2 X-Ray Generator dialog box

- (3) Select Cu in the Target box. Change any other parameters as necessary.
- (4) Click the OK button to close the dialog box.
- (5) If necessary, make changes for Incident Optics, Receiving Optics, and Detector in the same way.
- (6) Click the Update button in the Hardware Configuration dialog box.
- (7) Confirm that the Hardware Configuration dialog box displays the message "Hardware check OK", then click the Close button to close the dialog box.

2~60mA(1mA Step)	Attachment RxBy	
Hardware check OK		0
<u>¢</u>		Update Close

CAUTION: To execute the measurement once again, click the Run button on the Package Measurement flow bar or the Execute button in the applicable Part dialog box.

!

7. Shutdown

After completion of all measurements, turn off the x-ray generator as described below.

(1) Click the Shutdown button on the flow bar to open the Shutdown dialog box.

(2) Uncheck the Execute box.

Shutdown).		
Execute	1.		
Shutdowr	o conditions		
XG set:	XG Off		•
Voltage	(kV); 40	- Current(r	nA): 30
Execute		ОК	Cancel

Fig. 2.7.1 Shutdown dialog box

- (3) Select XG Off in the XG set box.
- (4) Click the Execute button.
- (5) The shutdown operation is executed. The x-ray generator will be turned off in about 10 minutes.

For information on other functions available from the Shutdown dialog box, refer to Chapter 21 of the SmartLab Guidance Reference Manual (ME13365A).

Shoulder of $K \alpha_1$ radiation?

A 2-theta/omega scan of Si(004) collected with multilayer mirror and slit collimation optics is shown in the figure. A shoulder as indicated by the arrow in the figure can be observed on the lower side of the Cu K_{α 1} diffraction peak when a single crystal that gives high diffraction intensity is used as the sample. The radiation whose wavelength corresponds to this shoulder is called a "K satellite" or a "nondiagram line." The radiation is known to come from an electronic level created by a doubly ionized atom and has slightly higher energy than that of the ordinary K α_1 created by a singly ionized atom. Since the excitation probability of this radiation is extremely low, the K satellite (nondiagram line) is observed only when the diffraction intensity of the Cu K α radiation is high.

Reference:

"X-Ray Spectroscopy: An Introduction (Springer Series in Optical Sciences)", by B. K. Agarwal, Springer

3. Executing a Part individually

In this chapter, how to execute the following Parts individually is described.

Optics Alignment (PB) Part Sample Alignment Part General Measurement Part

1. Executing the Optics Alignment Part

Described below is how to execute the Optics Alignment (PB) Part. The other Optics Alignment Parts can be executed in the same way.

(1) Click the Optics Alignment (PB) button on the flow bar to open the Optics Alignment (PB) dialog box.

Optics Alignment	(PB)							
Change optics								
Current attribute	Current attribute Bragg-Brentano focusing							
Destination attribute	Destination attribute Medium resolution parallel beam/RS							
Optics alignment conc	litions							
Change optics (c	juick alignr	nent only)						
Optics alignment nam	ne @Mediu	um resolution F	°В	•				
Alignment for in-p	lane meas	urement						
F Print out results a	after alignm	ent		?				
Execute In	nport	Export	ОК	Cancel				
1								

Fig. 3.1.1 Optics Alignment (PB) dialog box

(2) Uncheck the Change optics (quick alignment only) box.

Tip:

Tip:

: Check the Change optics (quick alignment only) box to switch optics for the data measurement using the alignment results stored under the selected optics alignment name by performing a quick alignment.

(3) In the Optics alignment name box, select a location for storing optics alignment results.

To store optics alignment results under a new optics alignment name, click the OK or Cancel button to close the Optics Alignment (PB) dialog box. Then, select the Optics Management command from the Options menu to open the Optics Management dialog box and add a new optics alignment name. After adding a new optics alignment name, return to step (1) in this section. For more information on creating an optics alignment name, refer to Chapter 17 of the SmartLab Guidance Reference Manual (ME13365A).

(4) To print the optics alignment results, check the Print out results after alignment box.

If both the Change optics (quick alignment only) and Print out results after alignment boxes are checked, the alignment results stored under the selected optics alignment name will be printed.

- (5) Click the Execute button in the Optics Alignment (PB) dialog box.
- (6) Optics alignment is executed followed by steps (5) through (7) in Section 2.5.

2. Executing the Sample Alignment Part

(1) Click the Sample Alignment button on the flow bar to open the Sample Alignment dialog box.

Sample Alignmen	t		
Sample information		🔽 Direct beam half c	ut alignment conditions
Sample thickness(mi	m) 1.0	Surface normal ali	gnment conditions
Sample width(mm)	10.0	Alignment criteria	Standard 💌
Sample height(mm)	10.0	Surface density	Medium
Run recommended Put a sample where	I sequence) the sample alig	C Customize conditions	Customize
F Put a sample ever	y time the sampl	e alignment starts in a repe	ated measurement.

Fig. 3.2.1 Sample Alignment dialog box

(2) Enter the sample thickness, sample width and sample height in the Sample thickness (mm), Sample width (mm) and Sample height (mm) boxes.

- (3) Check the Direct beam half cut alignment conditions box.
- (4) If the incident x-ray angle to the sample surface has to be precisely determined to make a measurement such as a reflectivity measurement, check the Surface normal alignment conditions box.
- (5) If the Surface normal alignment conditions box is checked, select Standard in the Alignment criteria box. And select the nominal density of the surface layer from Very low, Low, Medium, or High in the Surface density box.

(6) Check the Clear omega offset and Set chi, phi = 0 boxes.

If the results of the previous sample alignment are kept, the next sample alignment uses this position as a reference and may not work correctly. In ordinary cases, check the Clear omega offset and Set chi, phi = 0 boxes.

(7) Select the Run recommended sequence radio button.

To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

2.4.1 Customizing Sample Alignment Part conditions

- (8) Click the Execute button in the Sample Alignment dialog box.
- (9) Sample alignment is executed followed by steps (8) and (9) in Section 2.5.

CAUTION: If the current hardware configuration setting is different from that for the operation of the Sample Alignment Part, the sample alignment will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

2.6 Changing configuration units

3. Executing the General Measurement Part

(1) Click the General Measurement button on the flow bar to open the General Measurement dialog box.

Gen	eral	Measur	ement																	
54	ivê m	easuremen	nt data																	
Fil	e nar	ne	C:\Doc	umen	its and Set	tings\	administrat	:or\M	ly Docume	ents\Rigaku'	Test									
Sa	mple	name				1000		1.874	1.	1/80										1
Me	emo		<u> </u>	_				_			-									
M	aniual	excharige	slit cond	litions	i.									Detector	setting					i
Soll (de	er/PS	ic	ISL (mm)			PSA (dea)			Soller (deg)					Detecto	nr #1 (SC-	70) 💌				
No	9/ ne		10.0)	•	None	e	-	None	•	Read	current slits		1						
No.	-	event ron	ditions		_	1				_	·		_	_	_	_	_			
E>	kec.	Sc	an axis		Mode		Range		Start	Stop	Step	Speed	IS	RS1	R52	Attenuator	Comment	Measure	ment	
					10 C				(deg)	(deg)	(deg)	Duration time	mm 💌	mm 💌	nnn			origi	n	
	-			V		V		V	V	V		<u>V</u> <u>V</u>	М	V	8	V		V	<u>v</u>	1
1	N I	2-Theta/0	Omega	-	Continuou	5 *	Absolute	-	3,0000	80.0000	0.0100	3.0000	1.000	1.000		*		Setup.		-
2	-	2-Thetak	Omega	-	Continuau	S .*	Absolute	~	3.0000	80,0000	0,0100	3,8000	1.000	1.000		<u> </u>		Secup.	-	
3	-	2-Theta/(Omega	-	Continuqu	5 4	Absolute	*	3.0000	80.0000	0.0160	3.0000	1.000	1.000		· ·		Setup.		
4	1	2-Theta/0	Omega	-	Continuou	5 *	Absolute	¥.	3,0000	88,0000	8,0100	3,8886	1,000	1:000		*		Setup.	14	
5	-	2-Theta/0	Omega	*	Continuou	5 4	Absolute	7	3.0000	80.0000	0.0100	3.0000	1,800	1,000		<u>*</u>		Setup.		
6	F	2-Theta/0	Omeiga	*	Continuou	5 7	Absolute	*	3.0000	80.0000	0.0100	3.0000	1.000	1.000				Setup.		
7	Г	2-Theta/0	Omega	*	Continuou	g 👻	Absolute	4	3.0000	80,0000	0.0100	3,0000	1.000	1.000		·*		Setup.	11	
8	Г	2-Theta/(omega		Continuou	9. *	Absolute	Ŧ	3,0000	80.0000	0.8100	3.8800	1.000	1.000		Ψ.		Setup.	1	
9	Г	2-Theta/0	Omega		Continuou	5 *	Absolute	Y	3.0000	80.0000	0.0100	3,8000	1.000	1.000		7		Setup.	44	
10	Г	2-Theta/	Ómega		Continuou	5. *	Absolute	Ŧ	3,0000	80,0000	0.0100	3,0000	1,000	1,000		*		Setup.		
-	Drive	the 4 axes	s to the	curre	nt zero pos	itions	after the i	meas	urement	completed.										
Cal	culate	ed scan du	ration : I	00:25	i:40														?	
	Exec	ute													Import	Export	ок	Cano	:el	

Fig. 3.3.1 General Measurement dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the data measurement, the measurement data will be saved with the specified file name.
 - 1. Click the [...] button to open the Save As dialog box.

Save As			? 🚺
Save jn: [My Documents	- + E	r
📸 My Music 📇 My Picture:	5		
File <u>n</u> ame:	RigakuTest.ras		Save
Save as type:	RAS(*.ras)	•	Cancel

Fig. 3.3.2 Save As dialog box

- 2. Enter the settings in the Save in and File name boxes.
- 3. Click the Save button.

(3) Enter any required information in the Sample name and Memo boxes (optional).

- (4) Set the manual exchange slit conditions.
- (5) Confirm that the currently-installed detector is selected from Detector setting.

CAUTION: If another detector is selected, optics alignment and sample alignment will be required using the selected detector before executing this Part.

(6) Set the measurement conditions.

To set the manual exchange slit conditions or measurement conditions, refer to "General Measurement Part" Help Topic of the online help section of the SmartLab Guidance software.

- (7) If the four axes (2-theta, omega, chi, phi) are driven to the current zero positions after the data measurement, check the Drive the 4 axes to the current zero positions after the measurement completed box.
- (8) Click the Execute button in the General Measurement dialog box.
- (9) Data measurement is executed followed by steps (10) through (12) in Section 2.5.

CAUTION: If the current hardware configuration setting is different from that for the operation of the General Measurement Part, the data measurement will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box. 2.6 Changing configuration units

4. Troubleshooting

Problem	Response
Previously saved conditions cannot be imported through the Optics Alignment (PB) dialog box.	 Confirm that the user name displayed on the title bar of the main window is correct. Add the name of the optics alignment again in the Optics Management dialog box.
Previously saved conditions cannot be imported through the Sample Alignment dialog box.	• Confirm that the user name displayed on the title bar of the main window is correct.
Previously saved conditions cannot be imported through the General Measurement dialog box.	• Confirm that the user name displayed on the title bar of the main window is correct.
Package measurement cannot be executed.	Check to see if the XG output is as specified.Check to see if the door of the radiation enclosure is closed.
Clicking the OK button in a message box will result in the same message box reappearing.	• Confirm that the specified slit and other devices are installed correctly. Also, make sure that the necessary devices have not been removed.
Sufficient intensity cannot be obtained for data measurement.	 Check to see if an absorber is inserted in the receiving slit box. Confirm that the XG output has reached the specified levels. Measure the Si wafer reference sample using the Run recommended sequence mode and check the intensity.