UltimaⅣ operation procedure
～Thin film attachment～
～Reflectivity measurement～

Start up

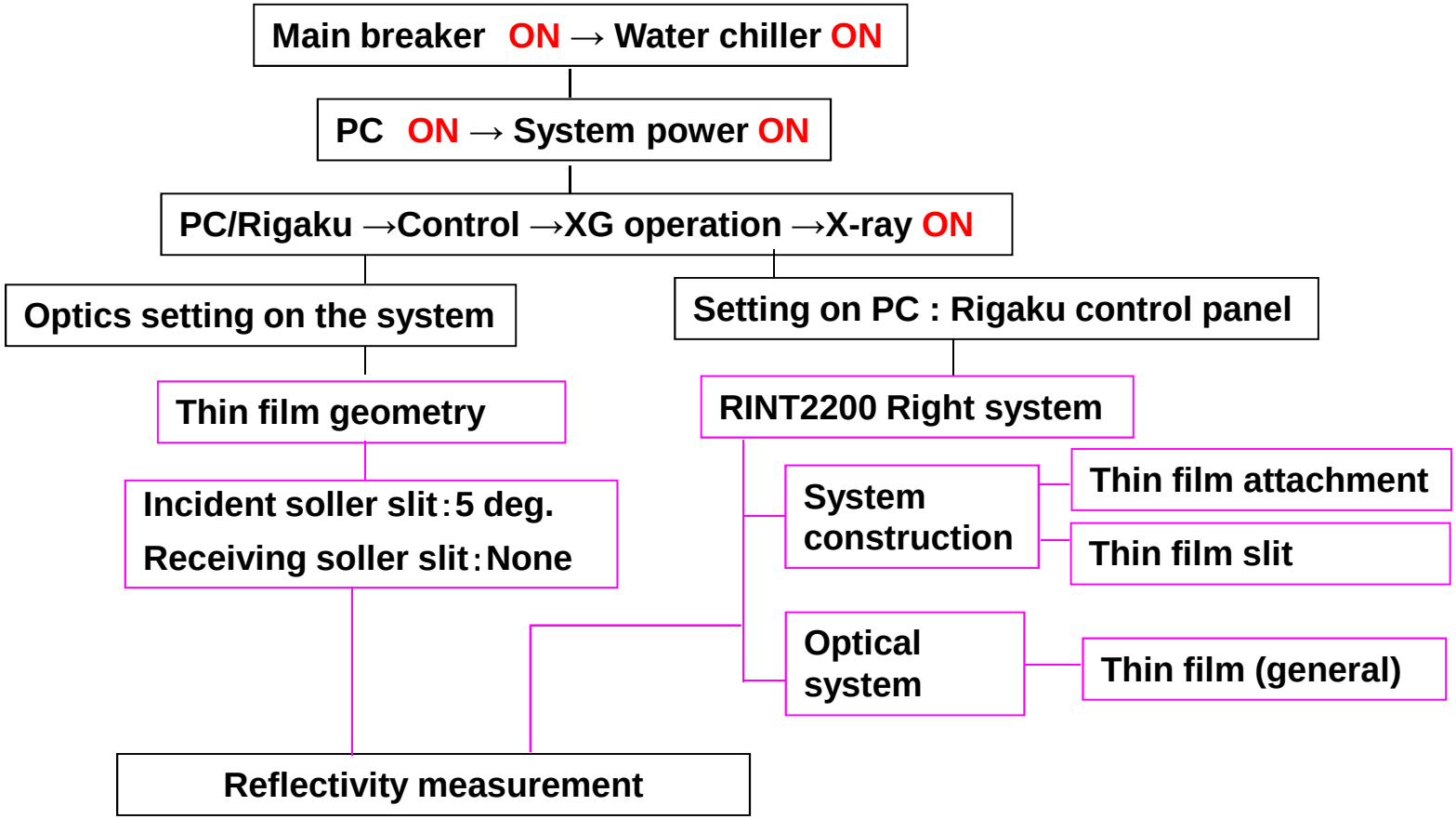
Setting of the optical system

Sample setting

Reflectivity measurement

Shut down

Flow chart of Reflectivity measurement



Note: This flow chart started after finishing the alignment of the thin film attachment.

Start up

1. System power ON

① Switch main breaker ON.

② Turn water chiller ON.

③ Turn the computer power ON.

④ Log on dialog box is displayed.

⑤ Turn on the main breaker on the system in the front side.

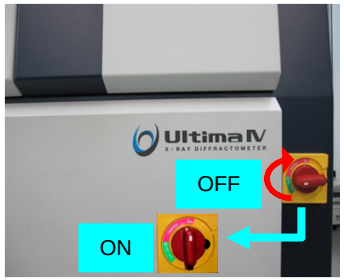
①

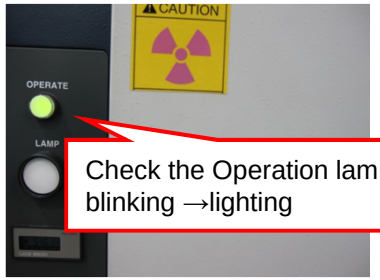
②

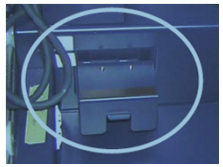
③

④

⑤







Note: The cover of the attachment connector must be kept close when turning on main breaker on the system.

Start up

2. PC screen operation

① User name：administrator

Password：None

② Check the measurement server icon at the bottom menu bar of the main screen.

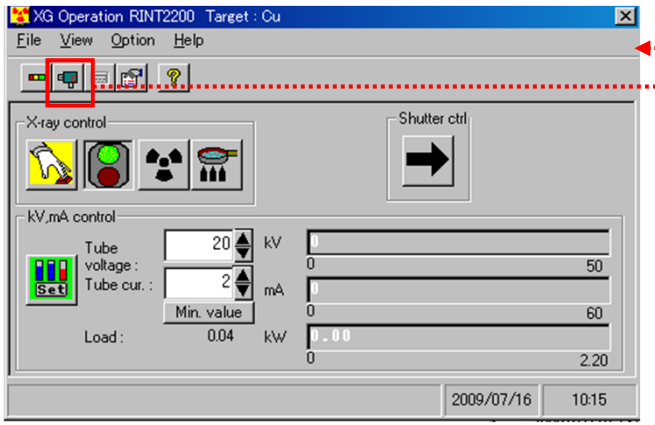
Blinking server icon turns to blue and becomes stable.

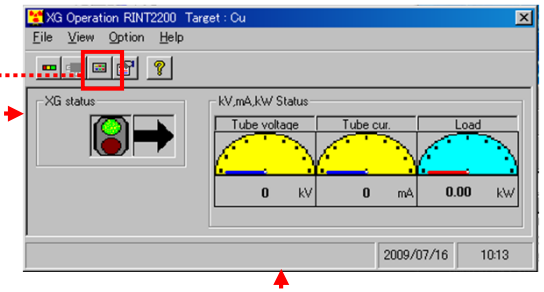
③ Open ‘Rigaku’ folder from main screen→Open ‘Control’ →Open ‘XG operation’

Start up

3. X-ray ON

① XG operation screen display





♦Default screen of XG operation

♦Open the XG operation screen by clicking XG operation button.

② Check the Ready lamp

③ Click the X-ray button →X-ray ON

Note: Water flow starts automatically after the X-ray is turned ON.

Start up

4. Aging operation

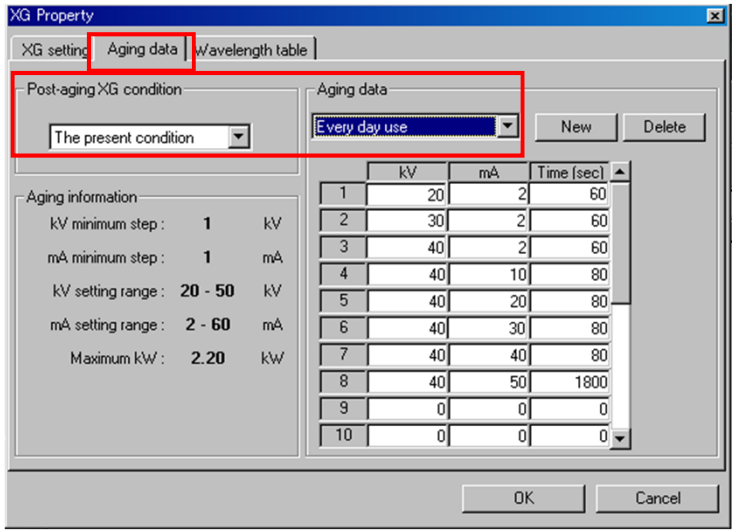
① Click the Aging button , then aging starts

Note: Perform aging operation as the parallel beam method needs to hold the tube voltage and current for 30 minutes at the working load

② When aging is finished, the aging button becomes

Note: Aging table setting is necessary.
Choose ‘every day use data’ for aging data and choose ‘The present condition’ for post XG aging condition.

Click for XG property.



Note: Type of aging data
1. When using new target
2. Long time (more than 15 days) not in use
3. Every day use

Other aging table is created using ‘New’ button.

Setting of Thin film optical system

5. Setting of thin film optical system on the instrument

①X-ray target（Ts axis）

②Multilayer mirror

③Selection slit：PB（Parallel beam method)

④Incident soller slit：5 deg.

⑤Incident slit box DS

+Height limit slit 10mm

⑥Thin film attachment

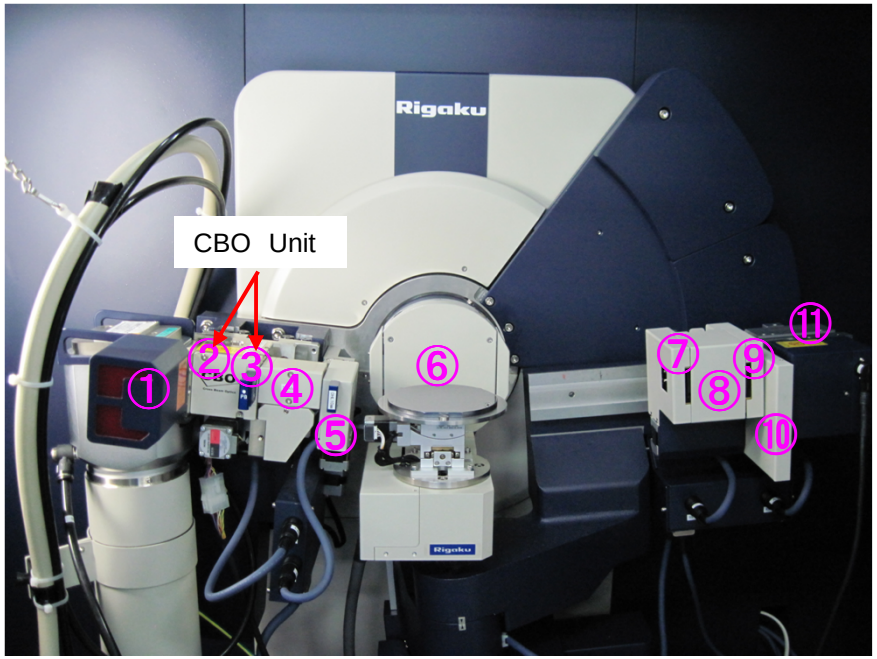
⑦Receiving slit box SS

⑧Receiving soller slit：None

⑨Receiving slit box RS

⑩Attenuator

⑪Scintillation counter SC



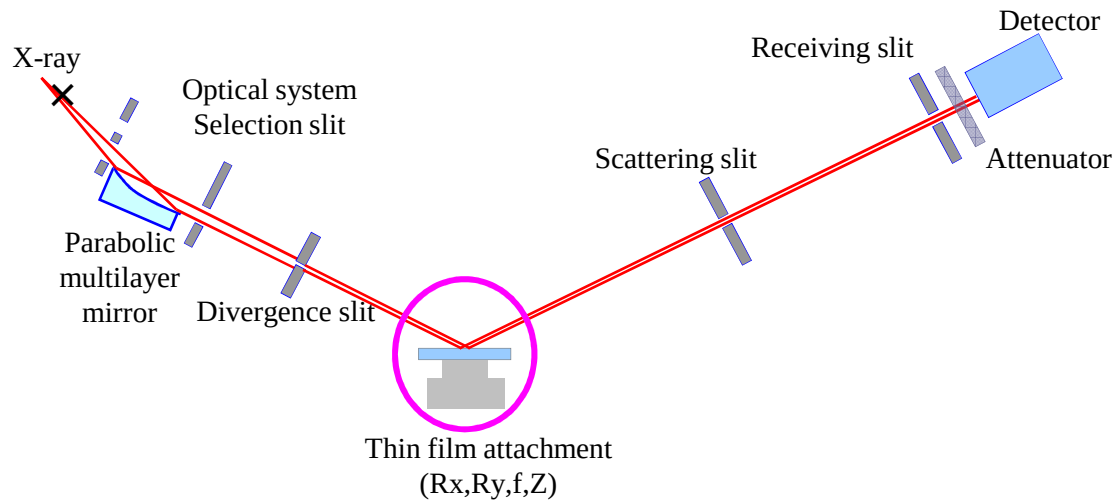
Note: Basically, soller slits are not required for reflectivity measurement. The incident side soller slit has been kept to avoid any vacant space.

Thin film optical system

6. Outline of the thin film optical system

Incident monochromator crystal	Resolution (X-ray Divergence angle)	Total thickness of thin films
None (Multilayer mirror)	0.05 deg.	1 to 100 nm
Ge(220)2-bounce monochromator	0.010 to 0.015 deg.	100 to 300 nm

Slit collimation

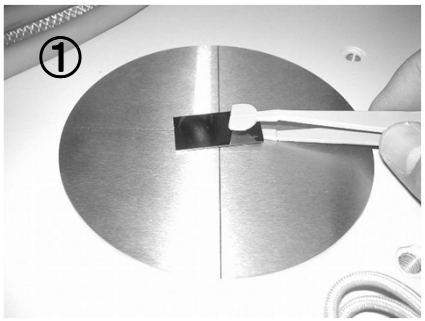


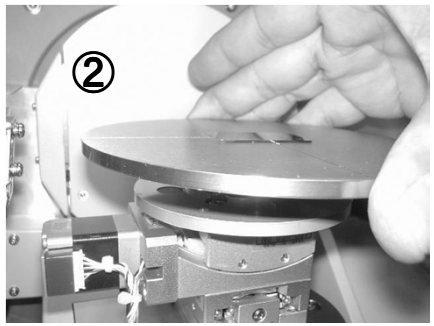
Note: In this measurement, slit collimation optics was chosen. Slit collimation means collimated X-ray by slits.

Sample setting

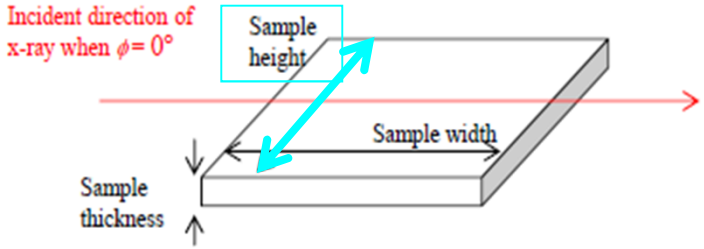
7. Sample setting

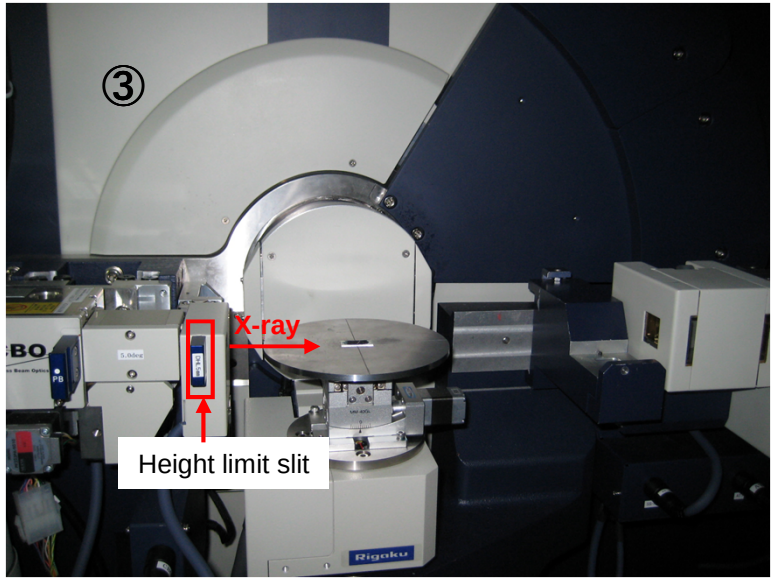
① Set the sample on the center of the sample plate.
② Replace the sample plate on the Thin film attachment.



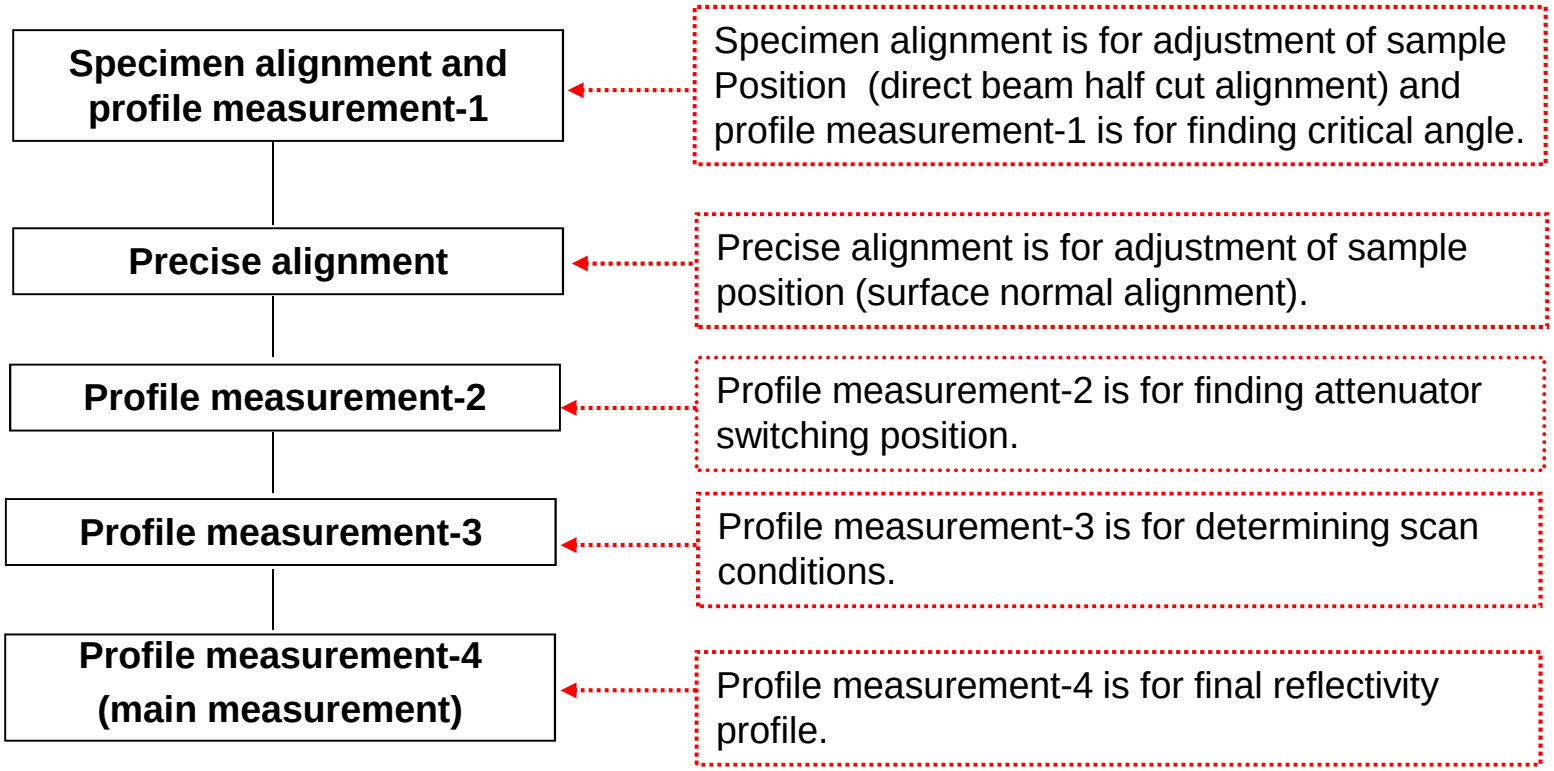


③ Use proper height limit slit so that X-ray doesn't exceed the height of the sample.





Flow chart for detailed Reflectivity measurement



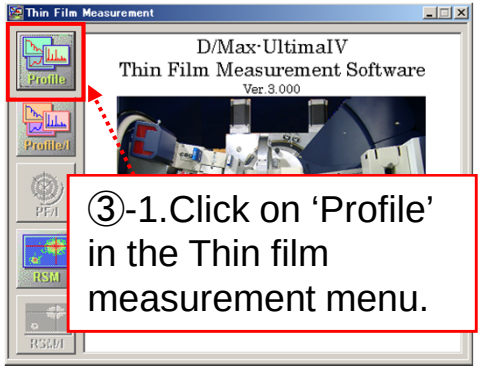
Reflectivity measurement

8. Reflectivity measurement for thin film

① Click ‘Rigaku’ folder

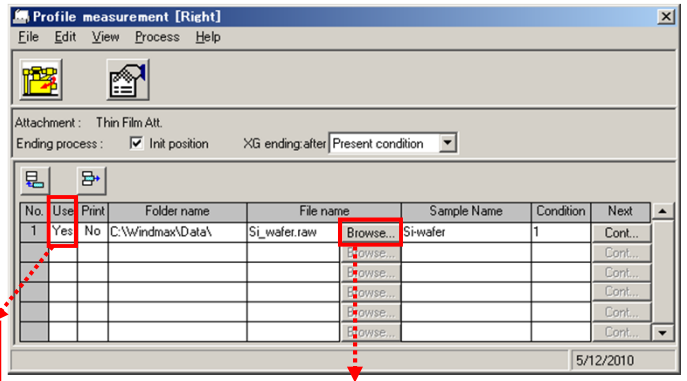
② Click ‘Right Measurement’ Right Measurement

③ Click ‘Thin Film Meas. menu’



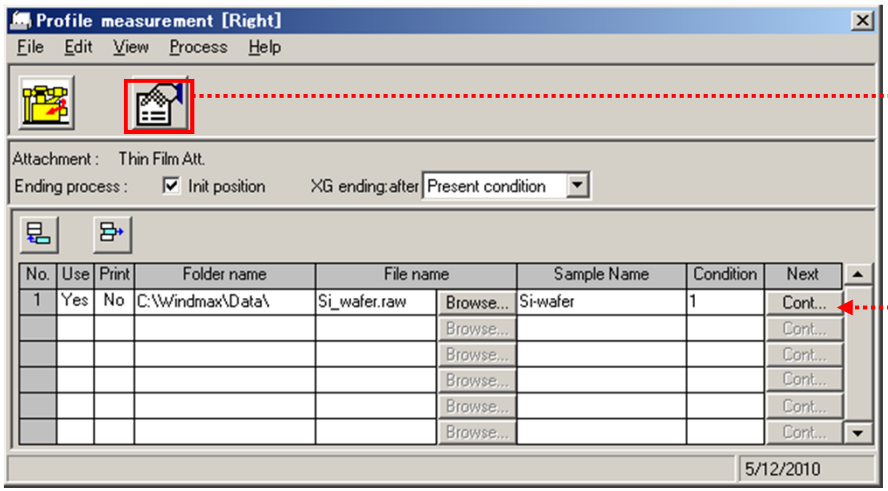
③-1.Click on ‘Profile’ in the Thin film measurement menu.

③-2.Click on “NO” to change to “YES”.



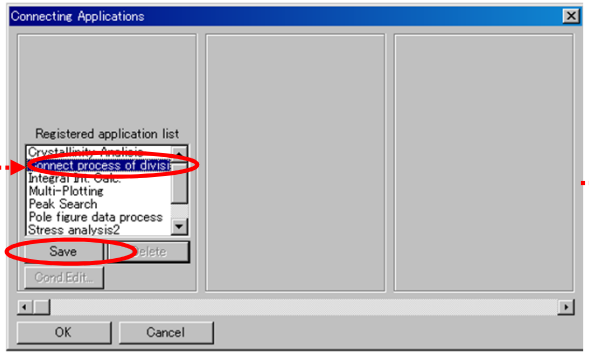
③-3. Click ‘browse’ to create folder and file name.

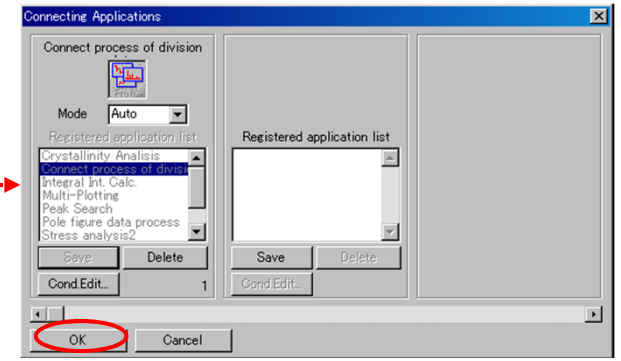
Reflectivity measurement



③-4 Click ‘cont..’ button.

③-5 Select ‘connect process of division’ and click ‘save’, then ‘OK’.





③-6 Click on ‘Measurement condition’ icon.

Note: ‘Connect process of division’ is done only for one time.

Rigaku

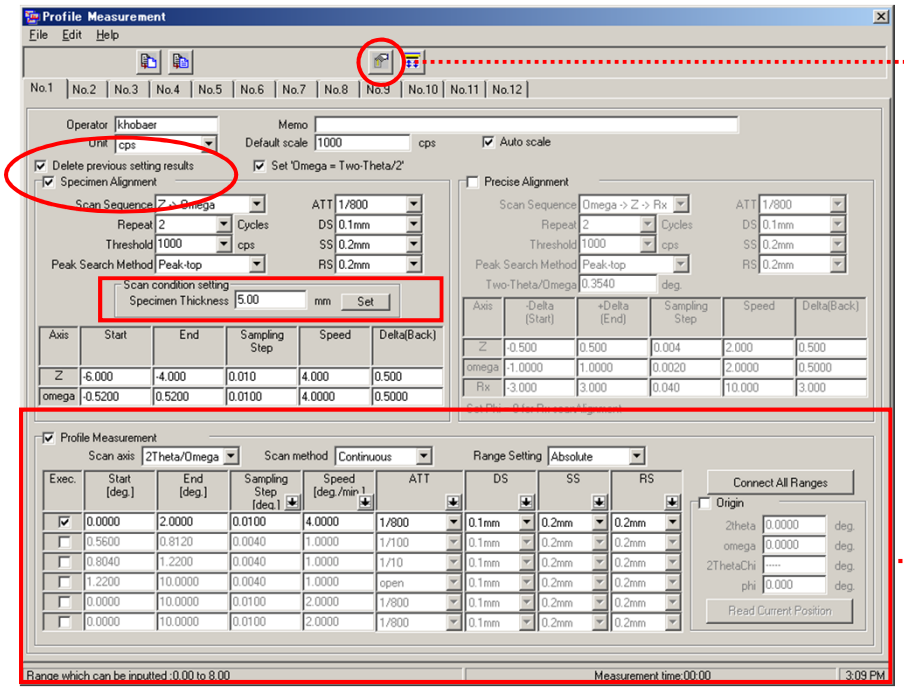
Reflectivity measurement step-1

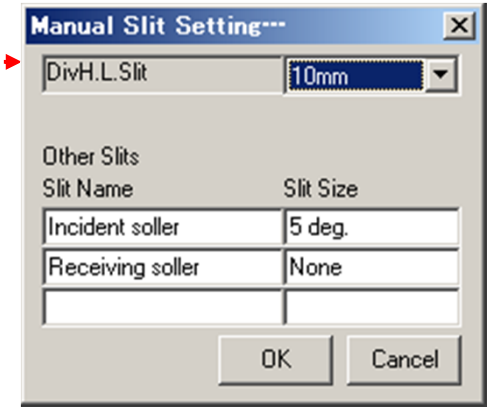
9. Specimen alignment and profile measurement-1

① Click icon and input soller slit information.

② Check ‘Specimen Alignment’ and ‘Delete previous setting results’

②－1 Input the actual thickness of the sample.





Profile Measurement

Reflectivity measurement step-1

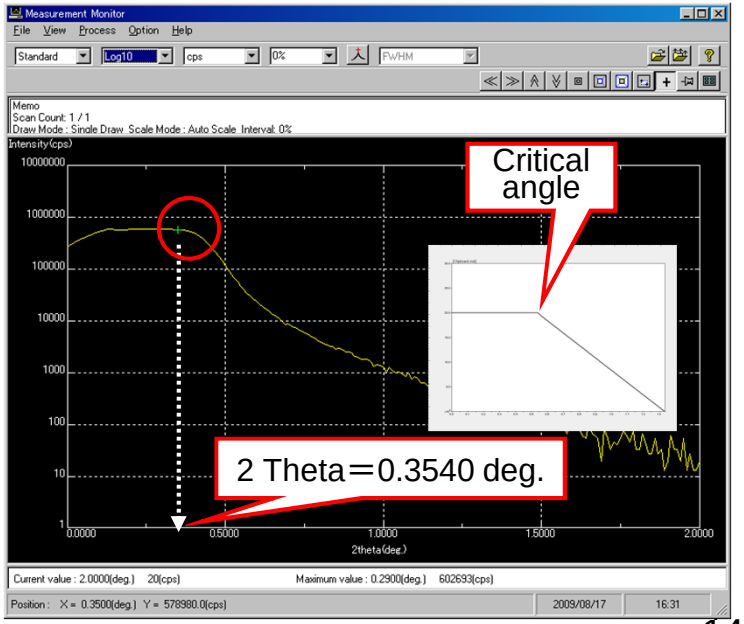
③ Check ‘Profile Measurement’ and input measurement conditions as shown below:

Scan axis	: 2 Theta/Omega	Start angle	: 0 deg.
Scan method	: Continuous	End angle	: 2 deg.
Range setting	: Absolute	Sampling step	: 0.01 deg.
		Scan rate	: 4 deg./min
		Attenuator	: 1/800
		DS	: 0.1 mm
		SS,RS	: 0.2 mm

④ Click button on the profile measurement window to execute measurement.

⑤ Take the angle just below the ‘Critical angle’.

Note: Critical angle is a position of angle after that X-rays start to penetrate thin film with increasing incident angle.

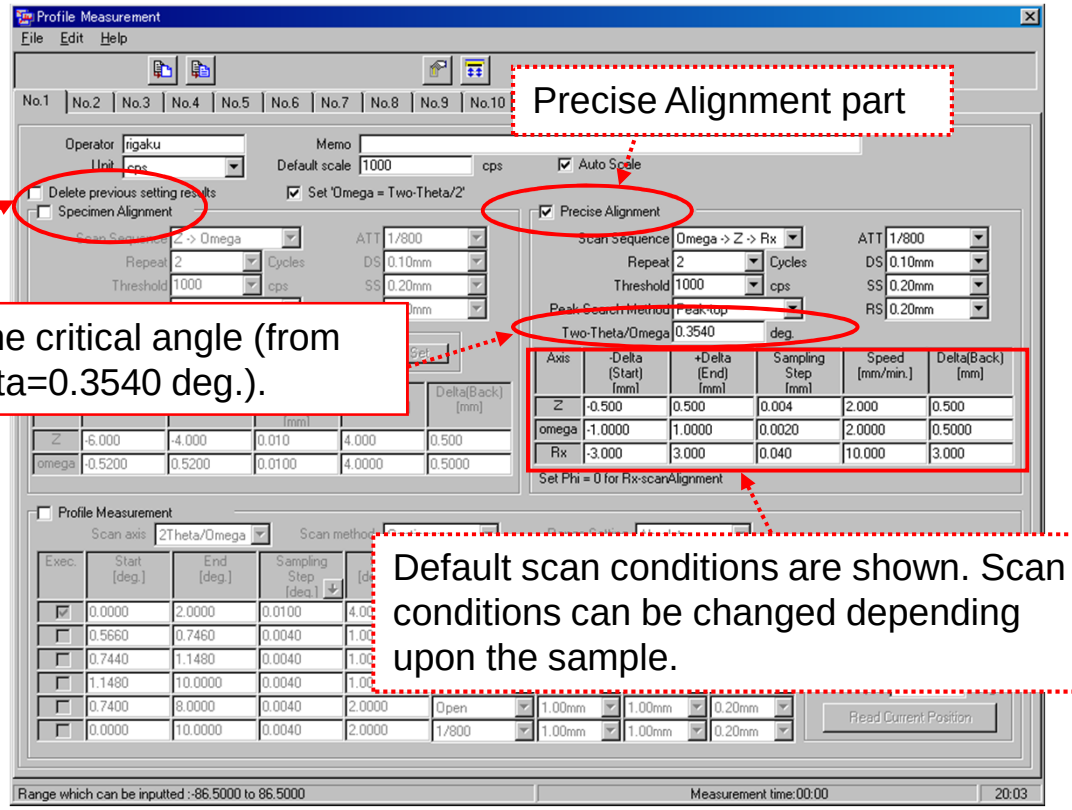


Reflectivity measurement step-2

10. Precise alignment

① Uncheck ‘Specimen Alignment’ and ‘Profile Measurement’.

② Check ‘Precise Alignment’.



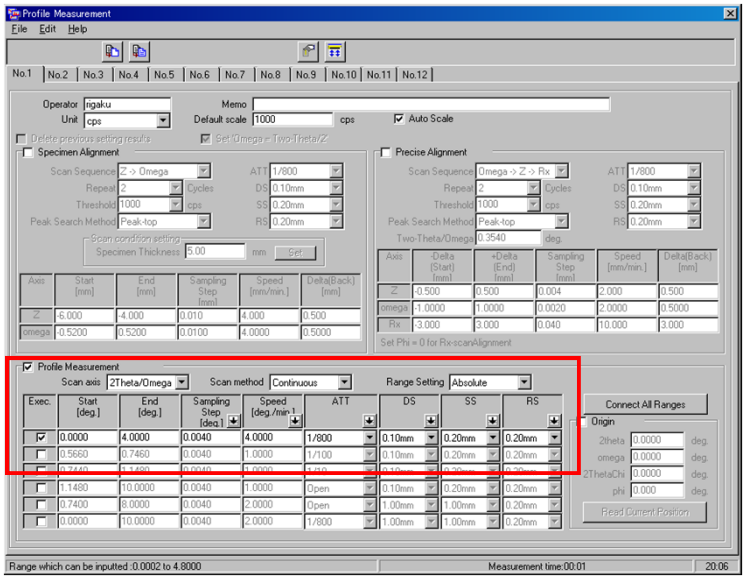
③ Click button on the profile measurement window to execute precise alignment.

Reflectivity measurement step-3

11. Profile measurement-2

① Check on ‘Profile Measurement’ and input measurement conditions.

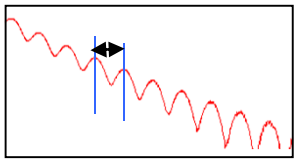
Scan axis	: 2 Theta/Omega	Sampling step	: 0.004 deg. (from simulated model)
Scan method	: Continuous	Scan rate	: 4 deg. /min
Range setting	: Absolute	Attenuator	: 1/800 for profile masurement-2
Start angle	: 0 deg.	DS	: 0.1 mm
End angle	: 4 deg.	SS, RS	: 0.2 mm



Note 1:
DS size is decided depending on the sample size ,the formula required W = DS / sin α Where, W =Irradiation width in mm, DS=Divergence slit size in mm, α= Incident angle=2 theta/2. In this measurement, 2 Theta=0.3540 deg.

Note 2:
DS size should be selected in such a way that X-ray doesn’t exceed the width of the sample.

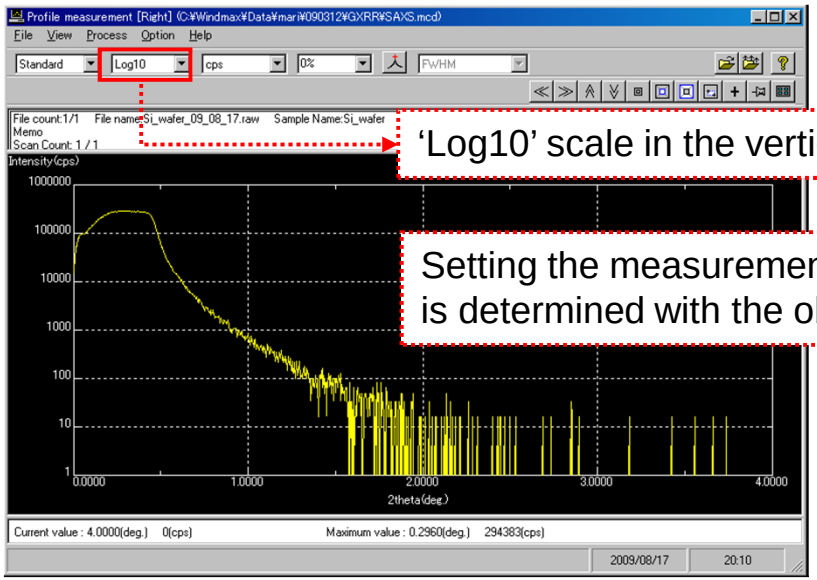
Note 3:
In this explanation, the sampling step was taken 0.004 for a sample of 100nm. However, the sampling step can be determined from simulated model using ‘GlobalFit’ software.



Sampling step=1/10 × Fringe width

② Click button on the profile measurement window to execute measurement.

Reflectivity measurement step-3



'Log10' scale in the vertical axis should be chosen.

Setting the measurement range using variable attenuator is determined with the observed intensity from this scan.

Scan range and Attenuator:

0.0 ~ 0.5620 deg. 1/800

0.5620 ~ 0.8050 deg. 1/100

0.8050 ~ 1.2200 deg. 1/10

1.2200 ~ 10.0 deg. Open

Note1:
Guideline of Attenuator change position (when using 1/800):

More than 20000cps	1/800
More than 2000cps	1/100
More than 200cps	1/10
Less than 200cps	open

Note 2: The end angle of one range must be the start angle of next range.

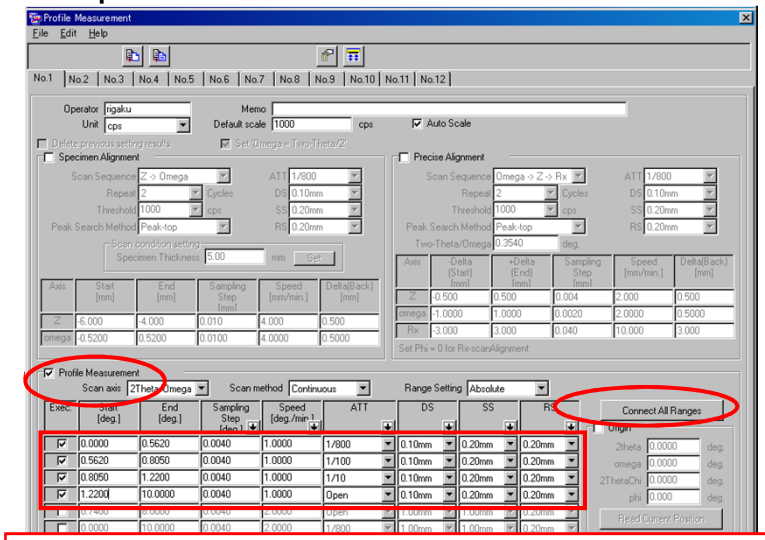
Reflectivity measurement step-4

12. Profile measurement-3

① Maintain check mark on ‘Profile Measurement’.

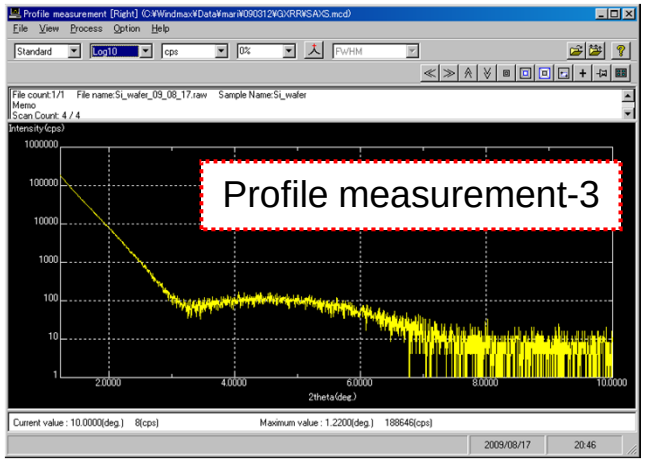
② Set the scan range and attenuator from profile measurement-2.

Scan axis : 2 Theta/Omega
Scan method : Continuous
Range setting : Absolute
Scan range
and Attenuator: 0.0~0.5620 deg. 1/800
0.5620~0.8050 deg. 1/100
0.8050~1.2200 deg. 1/10
1.2200~10.0 deg. open
Sampling step : 0.004 deg. (from simulated model)
Scan rate : 1deg./min
DS : 0.1 mm
SS, RS : 0.2 mm



②－1 Click ‘Connect All Ranges’ button.

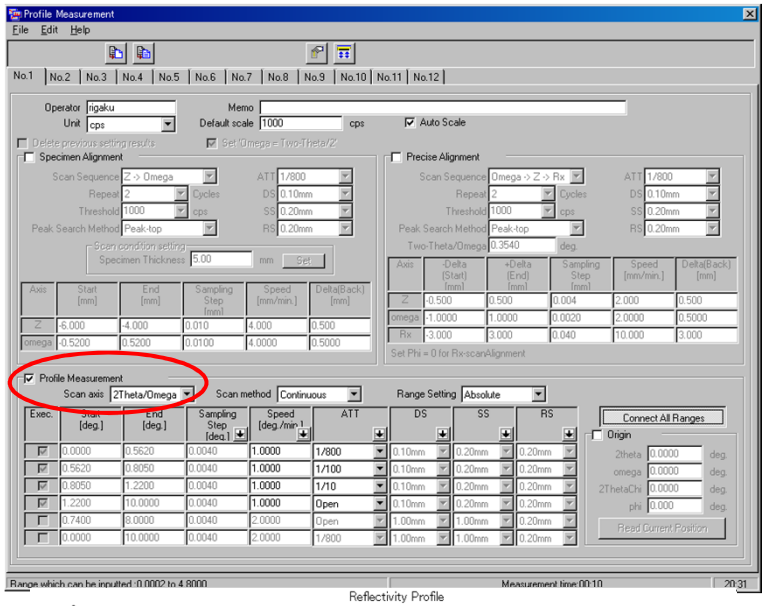
③ Click button on the profile measurement window to execute measurement.

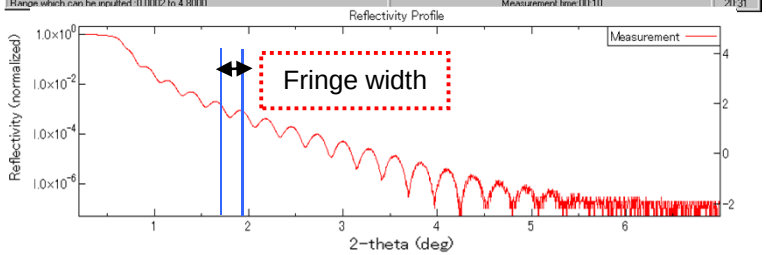


Reflectivity measurement step-5

13. Profile measurement-4 (main measurement)

① Set the measurement conditions as shown below:





Scan axis : 2 Theta/Omega
Scan method : Continuous
Range setting : Absolute
Scan range
and Attenuator: 0.0~0.5620 deg. 1/800
0.5620~0.8050 deg. 1/100
0.8050~1.2200 deg. 1/10
1.2200~10.0 deg. open
Sampling step : 0.004 deg.
Scan rate : 1 deg. /min
DS : 0.1 mm
SS, RS : 0.2 mm

Note 1: The end angle of the final measurement is determined at the background level that doesn't show any fringe.
Note 2: Sampling step is determined again checking the fringe width of the observed data.
Note 3: Generally, it takes 10-15 minutes to finish the measurement.
Note 4: Si wafer sample was measured in this experiment that has no thin film. However, if the sample contain thin film it shows the reflectivity profile as shown left.

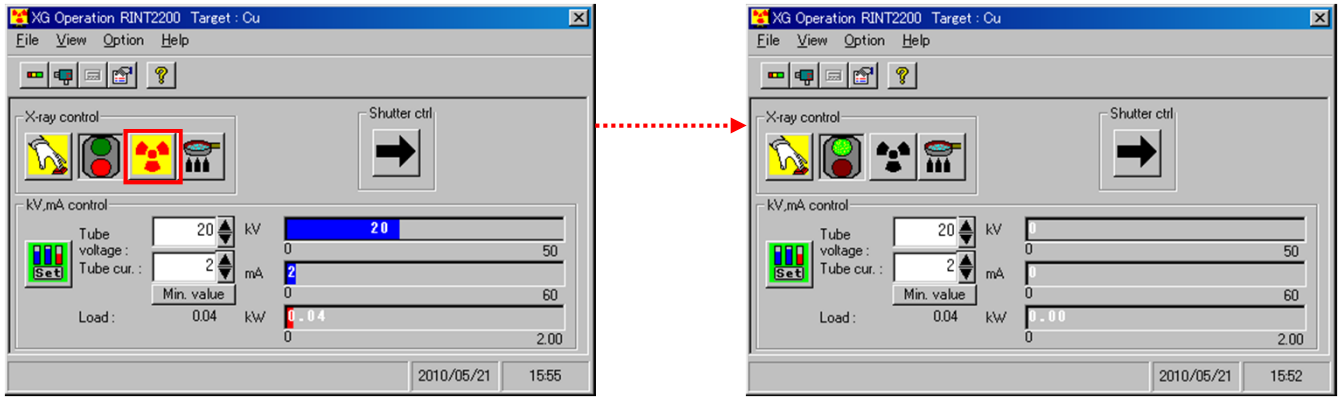
② Click button on the profile measurement window to execute measurement.

Shut down

14. From X-ray OFF to power off

① Open the XG operation screen.
② Click the XG ON/OFF button to turn XG OFF.

Note: The water supply will be automatically stopped in three minutes.



③ Turn off the main breaker on the front right side.
④ Turn off the water chiller.
⑤ Turn off the main power supply on the wall.