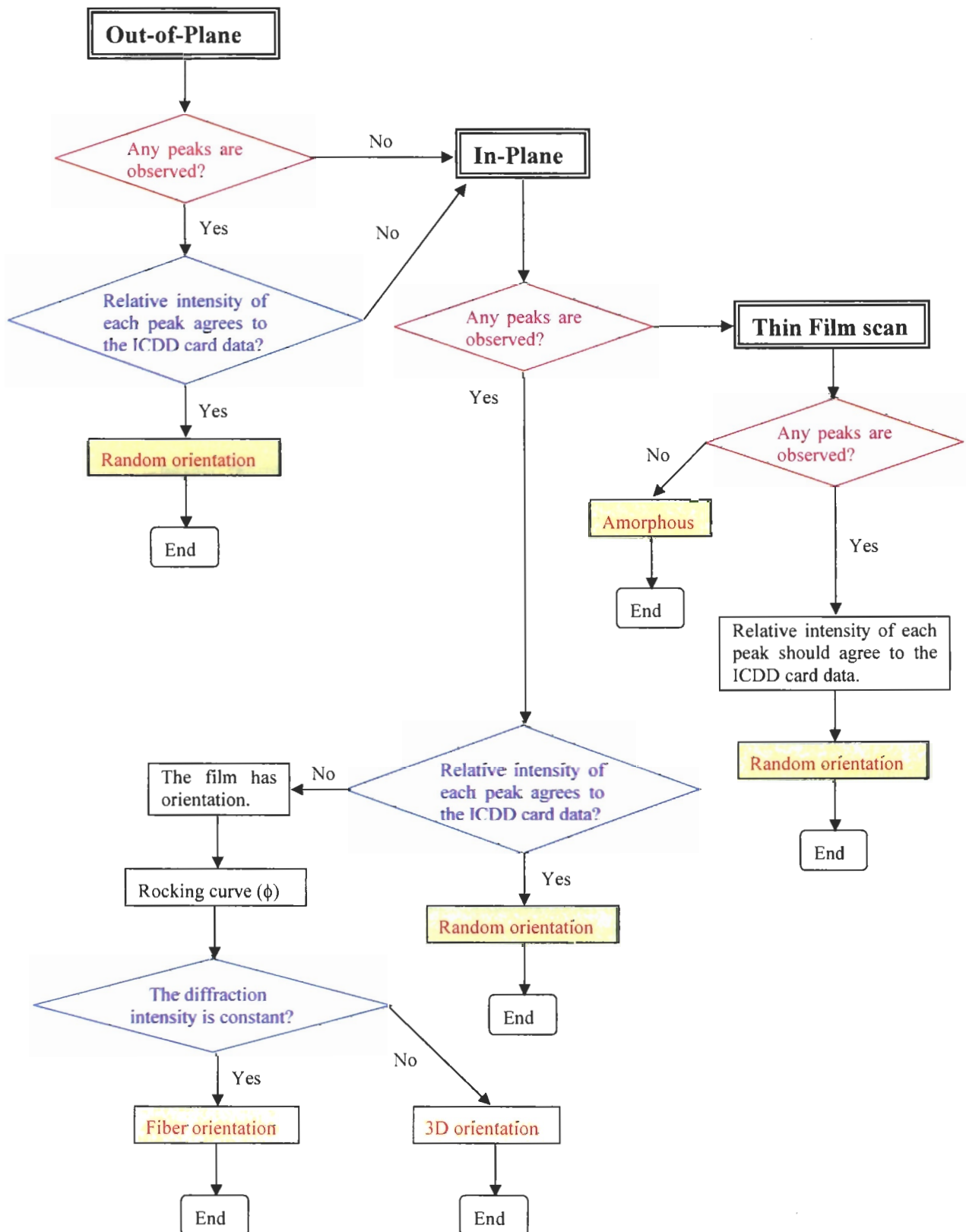


1. X-ray diffraction

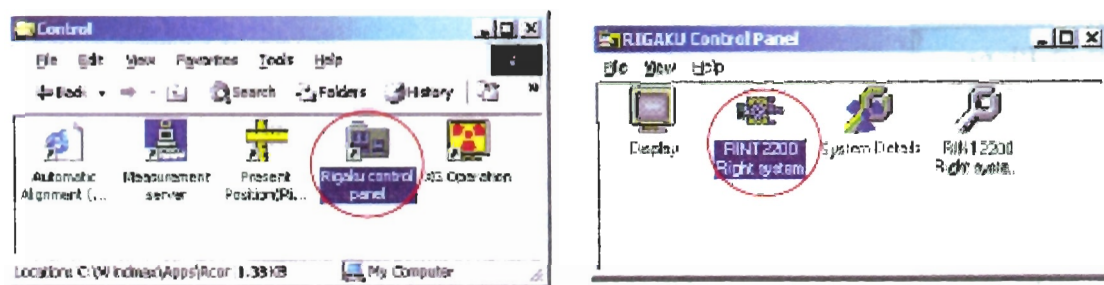
1-1 Flow chart



1-2 X-ray diffraction Measurement

1-2-1 Setting up the geometry system

Open "Rigaku control panel" from the Rigaku folder. Double click "RINT 2200 Rigaku system."



Select following items;

Attachment :

Attachment for thin film/ UltimaIII

Incident monochro:

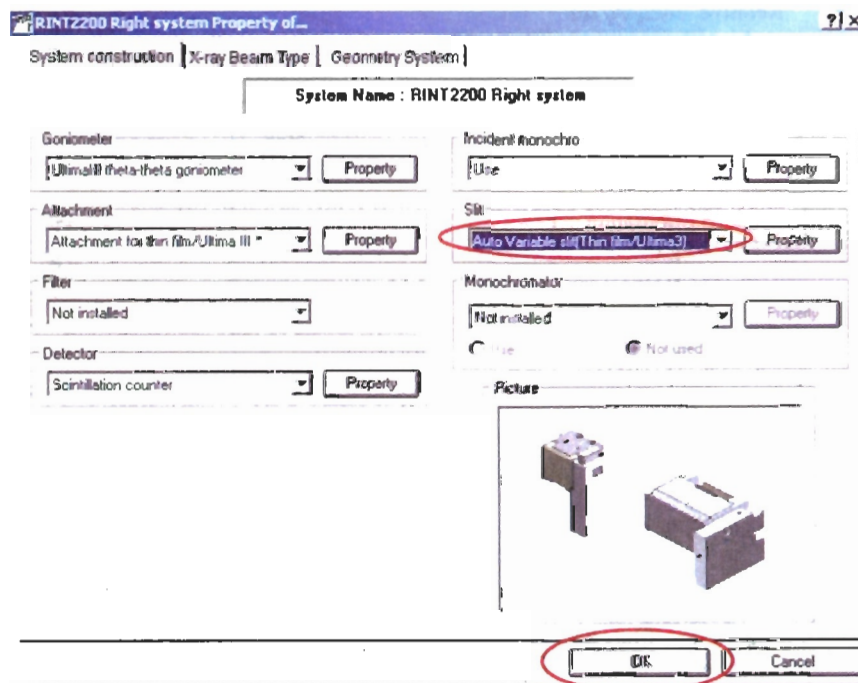
Use

Slit:

Auto Variable slit(Thin film/
UltimaIII)

Monochrometer:

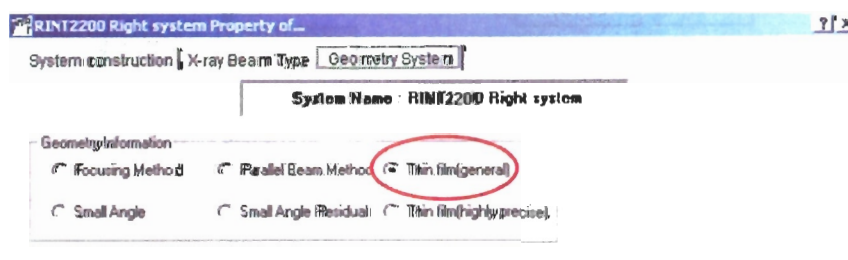
Not installed



Geometry Information:

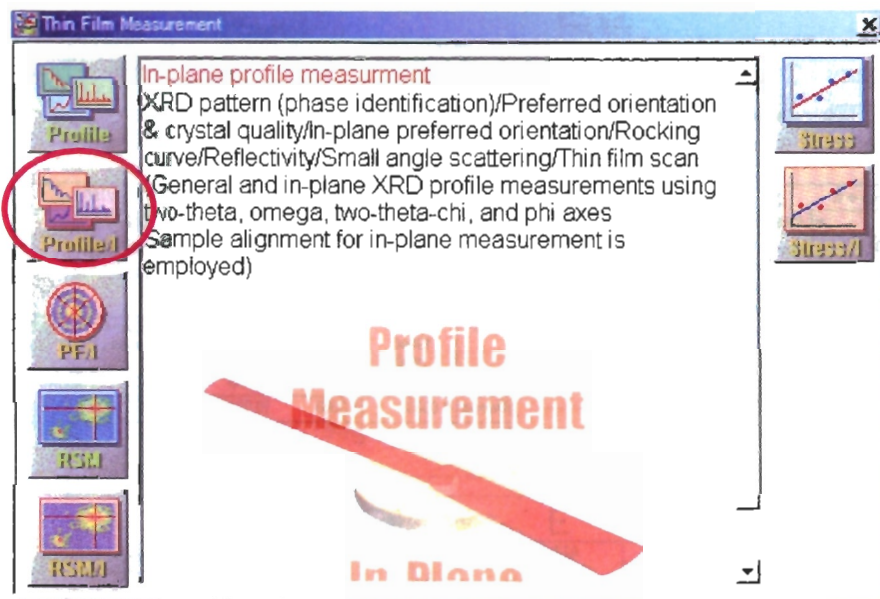
Thin film (general) or

Thin film (highly precise)

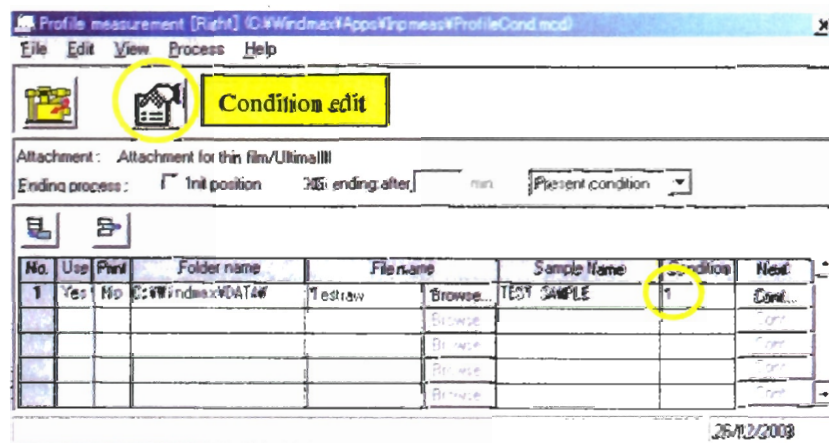


1-2-2 Out-of-Plane Measurement

Open [Start] – [Programs] - [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu.]



Select [Profile/In-Plane] measurement.



Input the folder name, the file name and the sample name. Switch “Use” to “Yes” (otherwise the measurement will not be performed!) To print the raw data (measured profile and the scanning condition) after the measurement, switch “Print” to “Yes.”

If you want to perform a series of scans, click “No. ” and “copy” button; the measurement condition will be copied. To remove conditions, use “Remove” button.

To edit measurement conditions, click on the “condition edit” button or double-click on the number input in [Condition.]

An example of scanning condition for Out-of-Plane measurements in as follows;

The screenshot displays the 'In-Plane Profile Measurement' software window. It includes a menu bar (File, Edit, Help), a sample selection bar (No.1 to No.12), and several configuration sections.

Operator: Administrator
Unit: counts
Default scale: 1000 counts
☒ Auto scale

☐ Delete previous setting results
☒ Set 'Omega = Two-Theta/Z'

Specimen Alignment

Scan Sequence: Omega → Z
 ATT: 1/100
 Repeat: 2 Cycles
 DS: 0.10mm
 Threshold: 1000 counts
 SS: 0.2mm
 Peak Search Method: Peak-top
 RS: 0.20mm

Scan condition setting
 Specimen Thickness: 1.00 mm **Set**

Axis	Start [mm]	End [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-2.000	0.000	0.010	4.0000	0.500
Omega	-0.5200	0.5200	0.0100	4.0000	0.5000

Precise Alignment

Scan Sequence: Omega → Z (Fine)
 ATT: 1/100
 Repeat: 2 Cycles
 DS: 0.10mm
 Threshold: 1000 counts
 SS: 0.2mm
 Peak Search Method: Peak-top
 RS: 0.20mm

Two-Theta/Omega: 69.1300 deg

Axis	-Delta (Start) [mm]	+Delta (End) [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-0.500	0.500	0.005	2.0000	0.500
Omega	-1.0000	1.0000	0.0040	4.0000	0.5000
Rx/Ry	-3.000	3.000	0.050	15.000	3.000

Profile Measurement

Scan axis: 2Theta/Omega
 Scan method: Continuous
 Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	40.0000	69.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input checked="" type="checkbox"/>	69.0000	69.6000	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input checked="" type="checkbox"/>	69.6000	100.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	298.5710	296.7910	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	317.1390	322.4990	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	336.7810	337.7810	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta: 0.0000 deg
 Omega: 0.0000 deg
 2ThetaChi: 0.000 deg
 Phi: 0.000 deg

Read Current Position

Range which can be inputted: -2.9800 to 139.9800
 Measurement time: 00:12
 16:18

☆ Specimen alignment

“Specimen alignment” has to be performed before Out-of-Plane measurement. Input “Specimen Thickness” and click “Set.” The recommended scanning conditions of Z and Omega axes are input automatically.

“Precise Alignment” is used when users need to be sure the direction of lattice plane observed with $2\theta/\omega$ scanning. When the critical angle for total reflection is input to “Two Theta/Omega,” the lattice planes parallel to the sample surface are observed; when the diffraction angle of the substrate is input, the lattice planes parallel to the substrate’s lattice planes are observed.

TTRAXIII

☆ Out-of-Plane measurement (2Theta/Omega scan)

“2theta/omega” is selected as “Scan axis.” The scanning range is determined by referring the ICDD card data.

The sampling step should be 1/5 ~ 1/10 of a diffraction peak. Switch the attenuator (ATT) at the middle of the scanning range according to the peak intensity, if necessary.

Profile Measurement

Scan axis: 2Theta/Omega Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	40.0000	69.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input checked="" type="checkbox"/>	69.0000	69.6000	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input checked="" type="checkbox"/>	69.6000	100.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	206.5710	206.7810	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	317.1380	322.4880	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	386.7810	387.7810	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta 0.0000 deg

Omega 0.0000 deg

2ThetaChi 0.0000 deg

Phi 0.0000 deg

Read Current Position

Range which can be inputted: -2.9800 to 139.9800 Measurement time: 00:12 18:19

☆ Rocking curve (Omega)

“Omega” is selected as “Scan axis.” Input the 2theta angle of the diffraction peak and the half value into

“2Theta” and “Omega” in “Origin”, respectively.

Profile Measurement

Scan axis: Omega Scan method: Continuous Range Setting: Relative

Exec.	-Delta [Start] [deg.]	+Delta [End] [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	-10.0000	10.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	69.0000	69.6000	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	69.6000	100.0000	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	206.5710	206.7810	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	317.1380	322.4880	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	386.7810	387.7810	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta 43.3800 deg

Omega 21.6900 deg

2ThetaChi 0.0000 deg

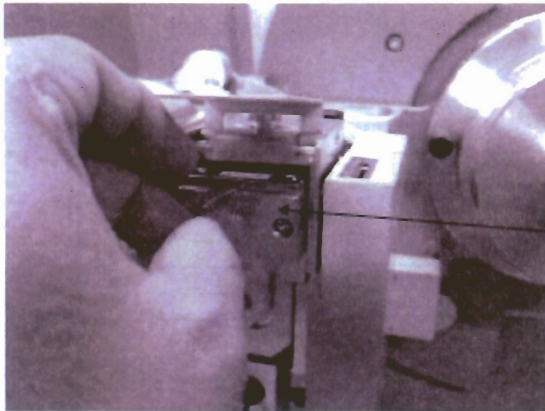
Phi 0.0000 deg

Read Current Position

Range which can be inputted: -2.0000 to 120.0000 Measurement time: 00:04 18:21

1-2-3 In-Plane Measurement

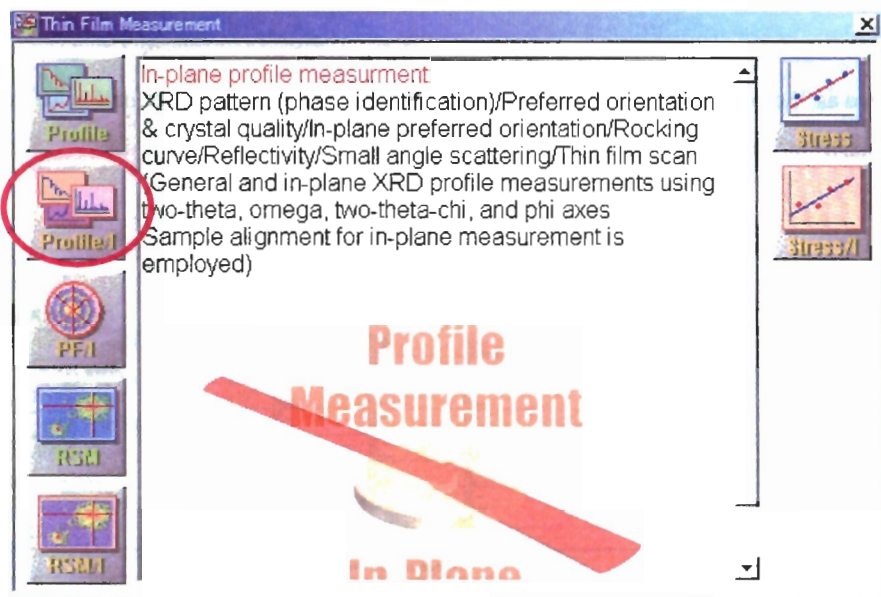
Before starting the measurements, vertical divergent Soller slits are installed.



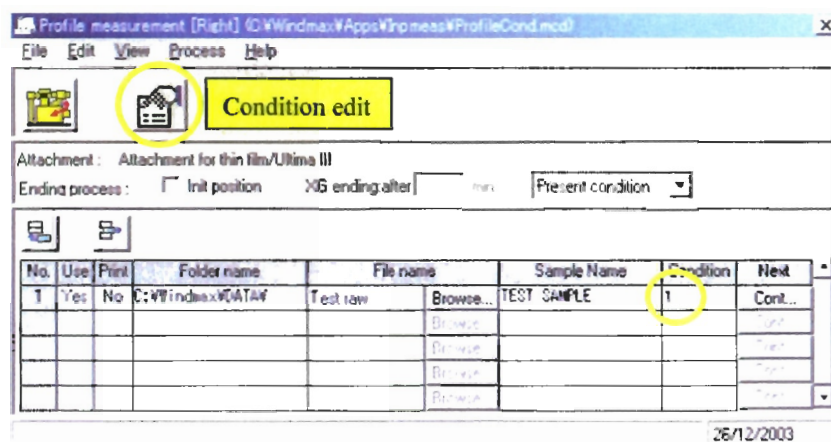
0.5° vertical divergence
Soller slit



Open [Start] – [Programs] - [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu.]



Select [Profile/In-Plane] measurement.



This is an example of scanning condition for preliminary scan of In-Plane measurement.

In-Plane Profile Measurement

File Edit Help

No.1 | No.2 | No.3 | No.4 | No.5 | No.6 | No.7 | No.8 | No.9 | No.10 | No.11 | No.12 |

Operator: Administrator Memo: _____

Unit: counts Default scale: 1000 counts ☒ Auto scale

☐ Delete previous setting results ☒ Set 'Omega = Two-Theta/2'

☒ Specimen Alignment ☒ Precise Alignment

Scan Sequence: Omega -> Z ATT: 1/100 DS: 0.10mm SS: 0.2mm RS: 0.20mm

Repeat: 2 Cycles Threshold: 1000 counts Peak Search Method: Peak-top

Scan condition setting Specimen Thickness: 1.00 mm Set

Axis	Start [mm]	End [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-2.000	0.000	0.010	4.0000	0.500
Omega	-0.5200	0.5200	0.0100	4.0000	0.5000

Two-Theta/Omega: 0.4000 deg

Axis	-Delta (Start) [mm]	+Delta (End) [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-0.500	0.500	0.005	2.0000	0.500
Omega	-1.0000	1.0000	0.0040	4.0000	0.5000
Phi, Psi				15.000	0.000

☒ Profile Measurement

Scan axis: 2Theta/Omega Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	0.0000	3.0000	0.0100	3.0000	1/800	0.10mm	0.2mm	0.20mm
<input type="checkbox"/>	112.3800	112.3800	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.3800	143.3800	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	231.9510	240.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.5130	365.8760	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.1610	331.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin: 2theta: 0.000 deg Omega: 0.000 deg 2Theta/Omega: 100 deg Phi: 0.000 deg

Read Current Position

Range which can be inputted: -12.000 to 12.000 Measurement time: 00:01 16.23

☆ Specimen alignment

For In-Plane measurement, both "Specimen alignment" and "Precise Alignment" has to be performed before measurement. The critical angle for total reflection is input to "Two-theta/Omega"; this procedure makes the incident angle (Omega) constant for any phi angle.

☆ Preliminary measurement

① Examination of critical angle

To observe the diffraction from the samples, incident x-rays have to penetrate to the sample. When the incident angle is smaller than the critical angle for total reflection, the penetration depth is just few nanometers and sometimes diffracted intensity becomes extremely low. To estimate the critical angle, perform 2Theta/Omega scan around critical angle (0~3 deg.) Read critical angle θ_c and input the value into "2Theta" and "Omega" in "Origin".

② Finding peaks

Check on the diffraction angles of (hkl)s which expected to be observed in In-Plane measurements from the results of Out-of-Plane measurement. Perform 2ThetaChi/Phi scanning around the angles.

Profile Measurement

Scan axis: 2ThetaChi/Phi Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	70.000	80.000	0.040	5.000	Open	0.10mm	Open	Open
<input type="checkbox"/>	112.380	112.980	0.040	5.000	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.380	143.380	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	221.951	240.161	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.512	365.973	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.143	331.143	0.040	5.000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta 0.4500 deg

Omega 0.4500 deg

2ThetaChi 0.000 deg

Phi 0.000 deg

Read Current Position

Range which can be inputted: -10.000 to 2.000 Measurement time: 00:02 16:27

If any peaks are not observed, it is supposed that the film is three-dimensionally oriented. In this case, try Phi scan with fixing 2ThetaChi angle at a diffraction angle estimated above.

Profile Measurement

Scan axis: Phi Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	0.000	360.000	0.100	90.00	Open	0.10mm	Open	Open
<input type="checkbox"/>	112.380	112.980	0.040	5.00	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.380	143.380	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	221.951	240.161	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.512	365.973	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.143	331.143	0.040	5.00	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta 0.4500 deg

Omega 0.4500 deg

2ThetaChi 74.130 deg

Phi 0.000 deg

Read Current Position

Range which can be inputted: 0.0000 to 77.0000 Measurement time: 00:04 16:28

③ Estimation of incident angle

Input the peak position into "2ThetaChi" and "Phi" at "Origin", respectively. Perform a Omega scan with the condition above. Find the incident angle that gives the highest intensity. Input the value to "2Theta" and "Omega".

Profile Measurement

Scan axis: Omega Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	0.0000	1.0000	0.0050	0.5000	Open	0.10mm	Open	Open
<input type="checkbox"/>	112.3800	112.9800	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.3800	143.3800	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	221.9510	240.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.5130	365.9730	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.1430	331.1430	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

Origin

2theta 0.4500 deg

Omega 0.4500 deg

2ThetaChi 74.130 deg

Phi 37.064 deg

Read Current Position

Range which can be inputted: -12.000 to 12.000 Measurement time: 00:02 16:28

TTRAXIII

☆In-Plane measurement (2ThetaChi/Phi scan)

“2ThetaChi/Phi” is selected as “Scan axis”. The scanning range is determined by referring to the ICDD card data. The sampling step should be 1/5 ~ 1/10 of a diffraction peak.

HINT : Any diffraction peaks can not be narrower than the resolution of the Soller slits (0.4deg. with standard Soller slit, and 0.12deg. with thin film PSA).

Profile Measurement

Scan axis: 2ThetaChi/Phi Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	20.000	80.000	0.040	2.000	Open	0.10mm	Open	Open
<input type="checkbox"/>	112.380	122.380	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.980	122.980	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	331.951	340.161	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.518	365.878	0.040	5.000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.161	337.161	0.040	5.000	Open	1.00mm	1mm	1.00mm

Connect All Ranges

☒ Origin

2theta: 0.4500 deg
 Omega: 0.4500 deg
 2ThetaChi: 0.000 deg
 Phi: 0.000 deg

Read Current Position

Range which can be inputted: 0.0001 to 10.0000 Measurement time: 00:30 16:40

☆Rocking curve (Phi)

“Phi” is selected as “Scan axis”. Input the 2Theta angle of the diffraction peak into “2ThetaChi”.

Profile Measurement

Scan axis: Phi Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	0.000	360.000	2.000	60.00	Open	0.10mm	Open	Open
<input type="checkbox"/>	112.380	112.980	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.980	143.380	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	331.951	340.161	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.518	365.878	0.040	5.00	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.161	337.161	0.040	5.00	Open	1.00mm	1mm	1.00mm

Connect All Ranges

☒ Origin

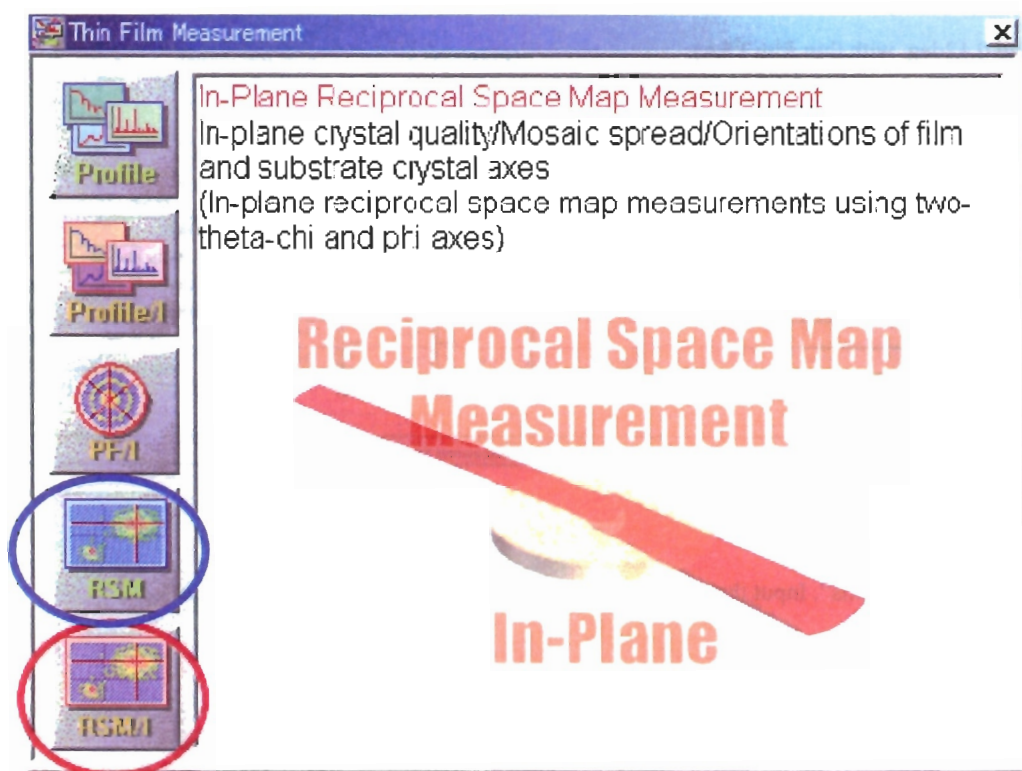
2theta: 0.4500 deg
 Omega: 0.4500 deg
 2ThetaChi: 74.000 deg
 Phi: 0.000 deg

Read Current Position

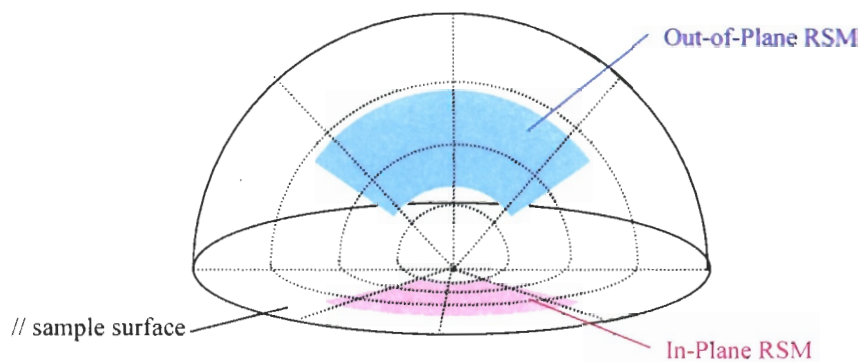
Range which can be inputted: -10.000 to 2.000 Measurement time: 00:06 16:37

1-2-4 Reciprocal Lattice Map

Open [Start] – [Programs] – [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu].



Select [RSM] for Out-of-Plane reciprocal space mapping, or [RSM/I] for In-Plane reciprocal space mapping, respectively.



☆ Specimen alignment

Perform the same procedures with Out-of-Plane or In-Plane measurements, respectively.

TTRAXIII

☆ Preliminary measurement

Refer to the results of 2Theta/Omeg (2ThetaChi/Phi) and Omega (Phi) scans to select the proper scanning ranges, sampling steps and scan speed.

☆ Main measurement

A reciprocal space map is made of two dimensional data of d-value (calculated from 2Theta/Omega (2ThetaChi/Phi) scans) and crystal direction (obtained from Omega (Phi) scans). There are two ways to collect data;

- Omega step -- 2Theta/Omega scan
- 2Theta/Omega step - Omega scan

The mode which contains fewer number of scan will be finished faster than the other ; the positioning motion between each scan takes several minutes.

Reciprocal Space Map Measurement

File Edit Help

No.1 No.2 No.3 No.4 No.5 No.6 No.7 No.8 No.9 No.10 No.11 No.12

Operator Administrator Memo

Unit cps Default scale 1000 cps ☒ Auto scale ☒ Multi line profile

☐ Use the previous setting results ☒ Set Omega = Two-Theta/2

☐ Specimen Alignment

Scan Sequence Omega Chi Z ATT 1/500

Repeat 1 Cycles DS 1.00mm

Threshold 1000 cps SS 0.05mm

Peak Search Method Peak loc RS 0.05mm

Scan condition setting

Specimen Thickness 1.00 mm Set

☐ Precise Alignment

Scan Sequence Omega Chi Z ATT 1/500

Repeat 2 Cycles DS 1.00mm

Threshold 1000 cps SS 0.05mm

Peak Search Method Peak loc RS 0.05mm

Two-Theta/Omega 2.4000 deg

Axis	-Delta (Start) (mm)	+Delta (End) (mm)	Sampling Step (mm)	Speed (mm/min)	Delta Back (mm)
Z	-0.500	0.500	0.100	0.0000	0.500
Omega	-0.5000	0.5000	0.1000	0.0000	0.5000
Phi	0.0000	0.0000	0.0000	0.0000	0.0000

Set Phi = 0 for Phi scan Alignment

☒ Reciprocal Space Map Measurement

Method Omega step - 2Theta/Omega continuous scan

Measure

Range Setting -2.0000 to 2.0000 Calculate Origin

Mesh Condition	-Delta (Start) (1/ang.)	+Delta (End) (1/ang.)	Delta (1/ang.)	Origin (1/ang.)	Fixed Time (sec)
02	0.00000000	0.00000000	0.00000000	0.00000000	1.0000
03	0.00000000	0.00000000	0.00000000	0.00000000	1.0000

SIR

ATT Open

DS 1.00mm

SS 1mm

RS 1.00mm

☒ Origin

2theta 69.2400 deg

Omega 34.6201 deg

2ThetaChi 1.000 deg

Phi 0.000 deg

Read Current Position

Radial Measurement

Range Setting Relative

Range Setting Absolute

Step Move Condition	-Delta (Start) (deg.)	+Delta (End) (deg.)	Sampling Step (deg.)	Scan Condition	Start (deg.)	End (deg.)	Sampling Step (deg.)	Speed (deg./min.)
Omega	-5.0000	5.0000	0.2500	2Theta/Omega	42.0000	72.0000	0.0800	20.0000

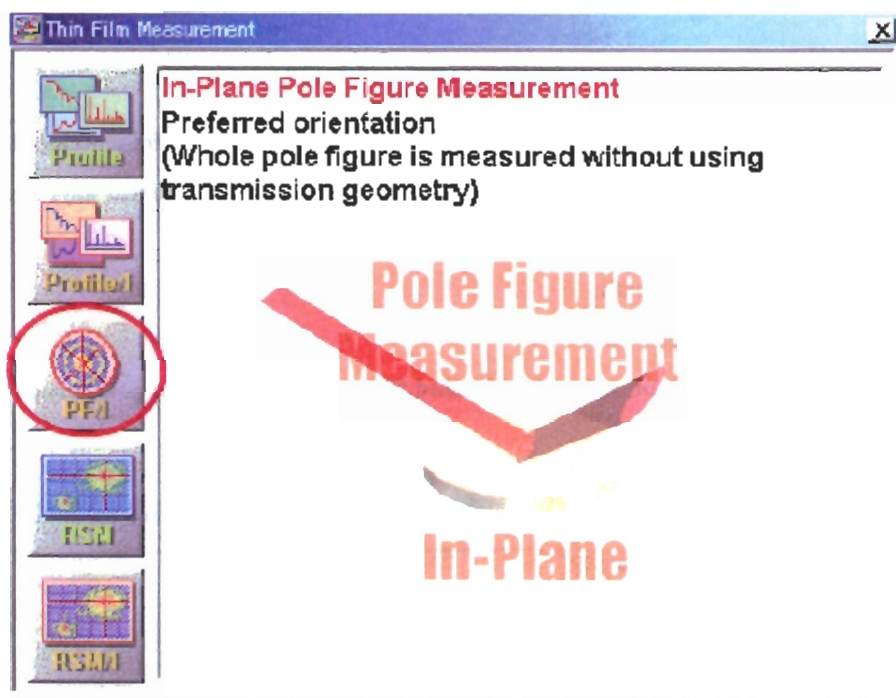
Range which can be inputted: -3.0000 to 140.0000

Measurement point: 41 x 376 = 15416 Measurement time: 01:03

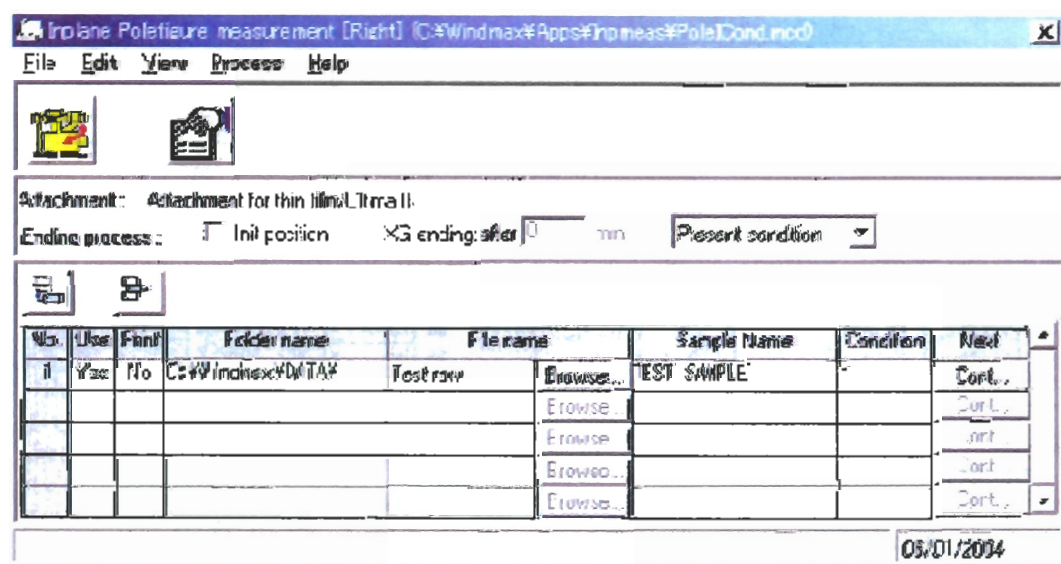
16.49

1-2-5 Pole Figure

Open [Start] – [Programs] – [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu].



Select [PF1] for pole figure measurement.



To edit scanning conditions, click on the "Condition edit" button or double click on the number input in [Condition].

The following is an example of specimen alignment / scanning conditions for pole figure measurement

In-Plane Pole Figure Measurement

File Edit Help

No.1 | No.2 | No.3 | No.4 | No.5 | No.6 | No.7 | No.8 | No.9 | No.10 | No.11 | No.12

Operator: Administrator Memo: _____

Unit: counts Default scale: 1000 counts ☒ Auto scale ☒ Multi line profile

☐ Delete previous setting results ☒ Set 'Omega = Two-Theta/2'

☒ Specimen Alignment

Scan Sequence: Omega → Z ATT: 1/800

Repeat: 2 Cycles DS: 0.10mm

Threshold: 1000 counts SS: 0.2mm

Peak Search Method: Peak-top RS: 0.20mm

Scan condition setting
Specimen Thickness: 1.00 mm

Axis	Start [mm]	End [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-2.000	0.000	0.010	4.0000	0.500
Omega	-0.5200	0.5200	0.0100	4.0000	0.5000

☒ Precise Alignment

Scan Sequence: Omega (Fine) ATT: 1/800

Repeat: 2 Cycles DS: 0.10mm

Threshold: 1000 counts SS: 0.2mm

Peak Search Method: Peak-top RS: 0.20mm

Two-Theta/Omega: 69.1300 deg.

Axis	-Delta (Start) [deg.]	+Delta (End) [deg.]	Sampling Step [deg.]	Speed [deg./min.]	Delta(Back) [deg.]
Omega	-0.5000	0.5000	0.0100	2.0000	0.5000
Phi	---	---	---	---	0.500

☒ Pole Figure Measurement

Omega Min: 0.5000 deg. Whole Area

alpha

Range Setting	Start [deg.]	End [deg.]	Sampling Step [deg.]
Absolute	0.00	90.00	2.00

beta

Scan method	Range Setting	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]
Continuous	Absolute	0.000	60.000	2.000	90.00

Pole Orientation

2ThetaB: 43.3000 deg. <=>

alpha: 0.00 deg.

beta: 0.000 deg. <=>

SR

ATT: Open

DS: 0.50mm

SS: 1mm

RS: 1.00mm

Origin

2theta: 0.000 deg.

Omega: 0.000 deg.

2thetaCh: 0.000 deg.

Phi: 0.000 deg.

Range which can be inputted: -3.0000 to 140.0000 Measurement time: 00:31 19:02

☆ Specimen alignment

“Specimen alignment” has to be performed before pole figure measurement. Input “Specimen Thickness” and click “Set.” The recommended scanning conditions of Z and Omega axes are input automatically.

“Precise Alignment” is used when users need to be sure the direction of lattice plane observed at the center of a pole figure ($\alpha=90\text{deg.}$). When the critical angle for total reflection is input to “Two Theta/Omega,” the lattice planes parallel to the sample surface are positioned at the center; when the diffraction angle of the substrate is input, the lattice planes parallel to the substrate’s lattice planes are positioned at the center.

2. High- resolution x-ray diffraction

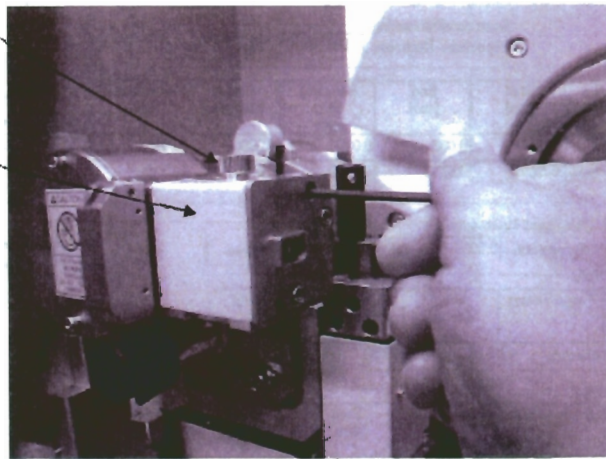
2-1 Measurement

2-1-1 Setting up the geometry system

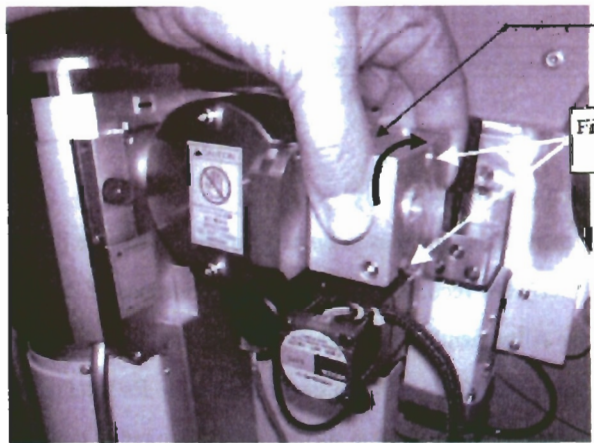
In this section, high- resolution optical system with Ge(220) incident monochromator is used to observe diffraction peaks from high-quality epitaxial films and single crystals. Adjust each optic by following the procedure mentioned in the manual.

① Move θ -s to 45deg.

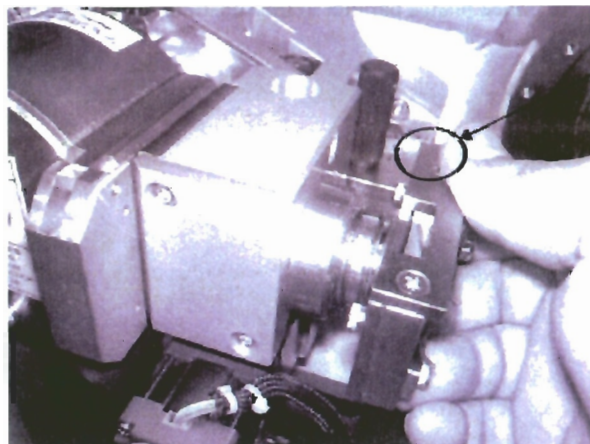
Remove the incident-side slit and the incident Soller slit.



Attaching the incident Ge(220) channel cut Monochromator.



Attaching the dedicated incident- side slit.



TTRAXIII

Open "Rigaku control panel" from the Rigaku folder. Double click "RINT 2200 Rigaku system."

Select following items;

Attachment:

Attachment for thin film/

UltimaIII

Incident monochro:

Use

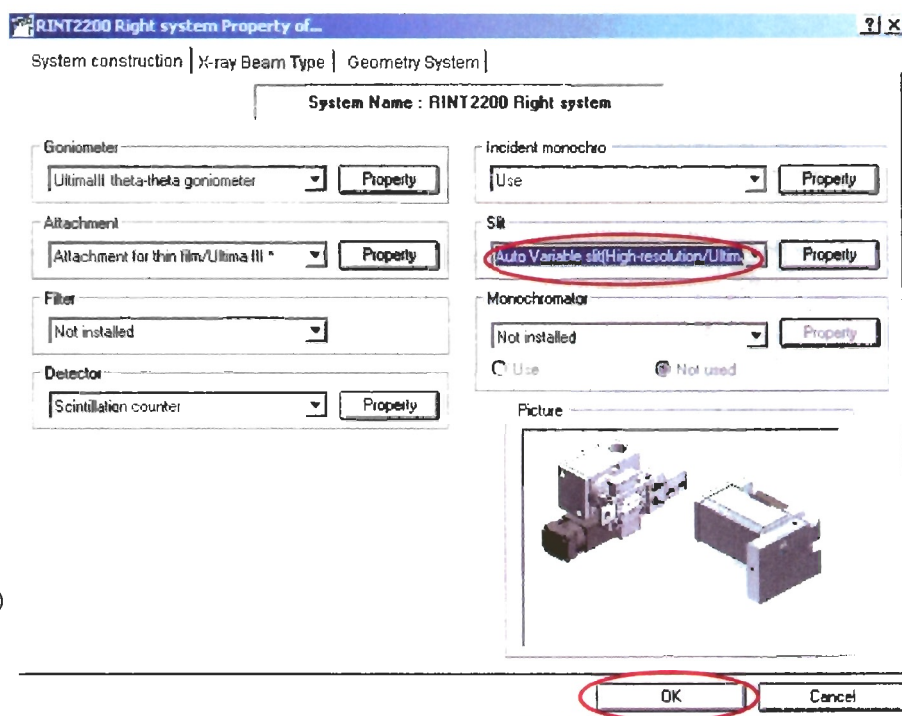
Slit:

Auto variable slit

(High- resolution/UltimaIII)

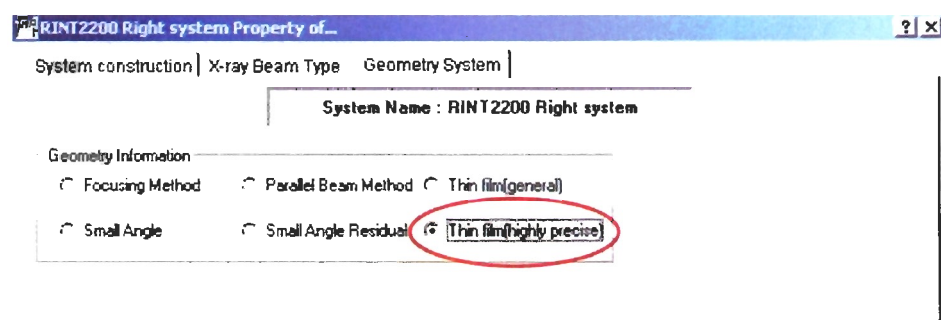
Monochromator:

Not installed



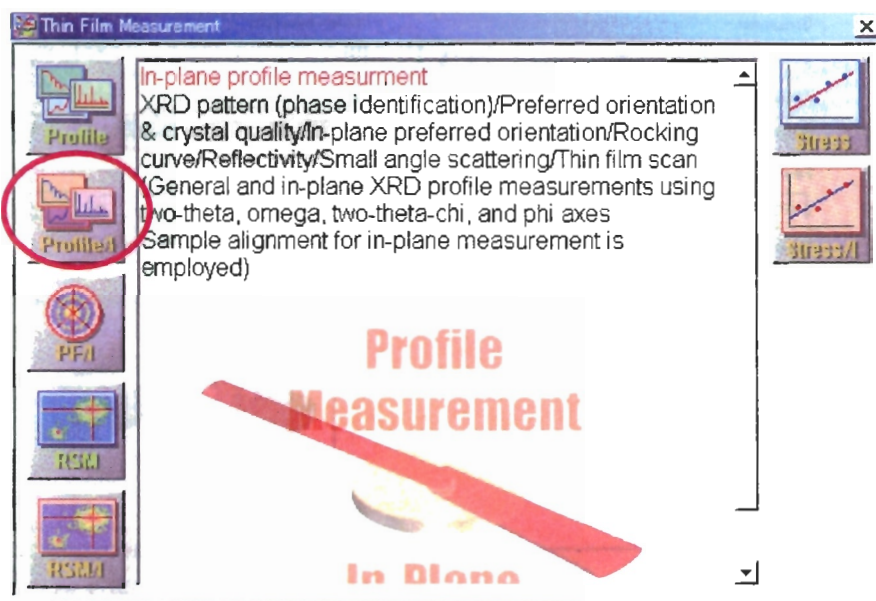
Geometry Information:

Thin film (highly precise)

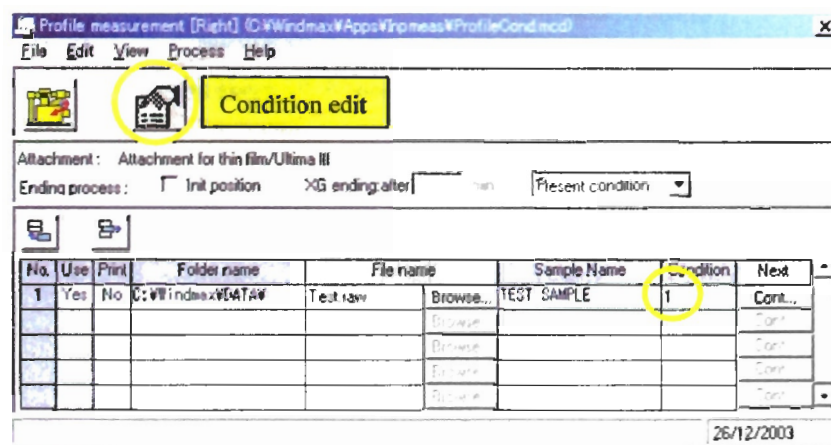


2-1-2 Rocking curve Measurement

Open [Start] – [Programs] - [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu.]



Select [Profile/In-Plane] measurement.



To edit scanning conditions, click on the “condition edit” button or double-click on the number input in [Condition.]

☆ Specimen alignment

XRD measurement for epitaxial films and single crystal substrates requires precise azimuth alignment because the distribution width of reciprocal lattice points are narrow so that the each goniometer axis should be arranged precisely.

In-Plane Profile Measurement

File Edit Help

No.1 | No.2 | No.3 | No.4 | No.5 | No.6 | No.7 | No.8 | No.9 | No.10 | No.11 | No.12

Operator: Administrator Memo: _____

Unit: counts Default scale: 1000 counts ☒ Auto scale

☐ Delete previous setting results ☒ Set $\Omega = 2\theta/Z$

☒ Specimen Alignment

Scan Sequence: $\Omega \rightarrow Z$ ATT: 1/100

Repeat: 2 Cycles DS: 0.10mm

Threshold: 1000 counts SS: 0.2mm

Peak Search Method: Peak-top RS: 0.20mm

Scan condition setting
Specimen Thickness: 1.00 mm Set

Axis	Start [mm]	End [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-2.000	0.000	0.010	4.0000	0.500
Ω	-0.5200	0.5200	0.0100	4.0000	0.5000

☒ Precise Alignment

Scan Sequence: $\Omega \rightarrow Z$ (Fine) ATT: 1/100

Repeat: 2 Cycles DS: 0.10mm

Threshold: 1000 counts SS: 0.2mm

Peak Search Method: Peak-top RS: 0.20mm

Two-Theta/ Ω : 69.1300 deg.

Axis	-Delta (Start) [mm]	+Delta (End) [mm]	Sampling Step [mm]	Speed [mm/min.]	Delta(Back) [mm]
Z	-0.500	0.500	0.005	2.0000	0.500
Ω	-1.0000	1.0000	0.0040	4.0000	0.5000
Rotary	-0.000	0.000	0.000	5.000	0.000

☒ Profile Measurement

Scan axis: 2Theta/ Ω Scan method: Continuous Range Setting: Relative

Exac.	-Delta (Start) [deg.]	+Delta (End) [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	-1.0000	1.0000	0.0040	0.2000	1/800	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.3800	112.9800	0.0400	5.0000	1/100	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.9800	142.3800	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	231.9510	240.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.5180	365.8780	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	390.1610	391.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges ☒ Origin

2theta: 69.1300 deg

Ω : 34.5650 deg

2ThetaCh: 0.000 deg

Φ : 0.000 deg

Read Current Position

Range which can be inputted: 0.001 to 2.000 Measurement time: 00:10 17:32

☆ High resolution rocking curve (symmetric scan)

The symmetric rocking curve observes the crystal planes almost parallel to the sample surface. Since the "Precise Alignment" procedure makes those crystal planes being observed, perform 2theta.omega scan right after the alignment. Input the diffraction angle of the substrate into "2Theta" and the half value into "Omega" in "Origin", respectively.

TTRAXIII

☆High- resolution rocking curve (asymmetric scan)

To observe crystal planes not parallel to the sample surface, the difference between incident and exit angles should be equal to the angle between the observing crystal planes and the symmetric planes. The angle between $(h_1k_1l_1)$ and $(h_2k_2l_2)$ is calculated with the following equation (for cubic system),

$$\text{Cubic system : } \cos \phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)}}$$

Also phi angle (in-plane sample rotation) should be at proper position to observe asymmetric planes. Perform phi scan before starting main measurements and find the proper position.

☒ Profile Measurement

Scan axis: 2Theta/Omega Scan method: Continuous Range Setting: Relative

Exec.	-Delta (Start) [deg]	+Delta (End) [deg]	Sampling Step [deg]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	-1.5000	1.5000	0.0040	0.2000	1/800	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.3800	112.9800	0.0400	5.0000	1/1.00	1.00mm	1mm	1.00mm
<input type="checkbox"/>	112.3800	143.3800	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	331.9510	340.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	360.5180	365.8780	0.0400	5.0000	Open	1.00mm	1mm	1.00mm
<input type="checkbox"/>	330.1610	331.1610	0.0400	5.0000	Open	1.00mm	1mm	1.00mm

Connect All Ranges ☒

Origin

2theta 88.0283 deg

Omega 8.7500 deg

2ThetaChi 0.000 deg

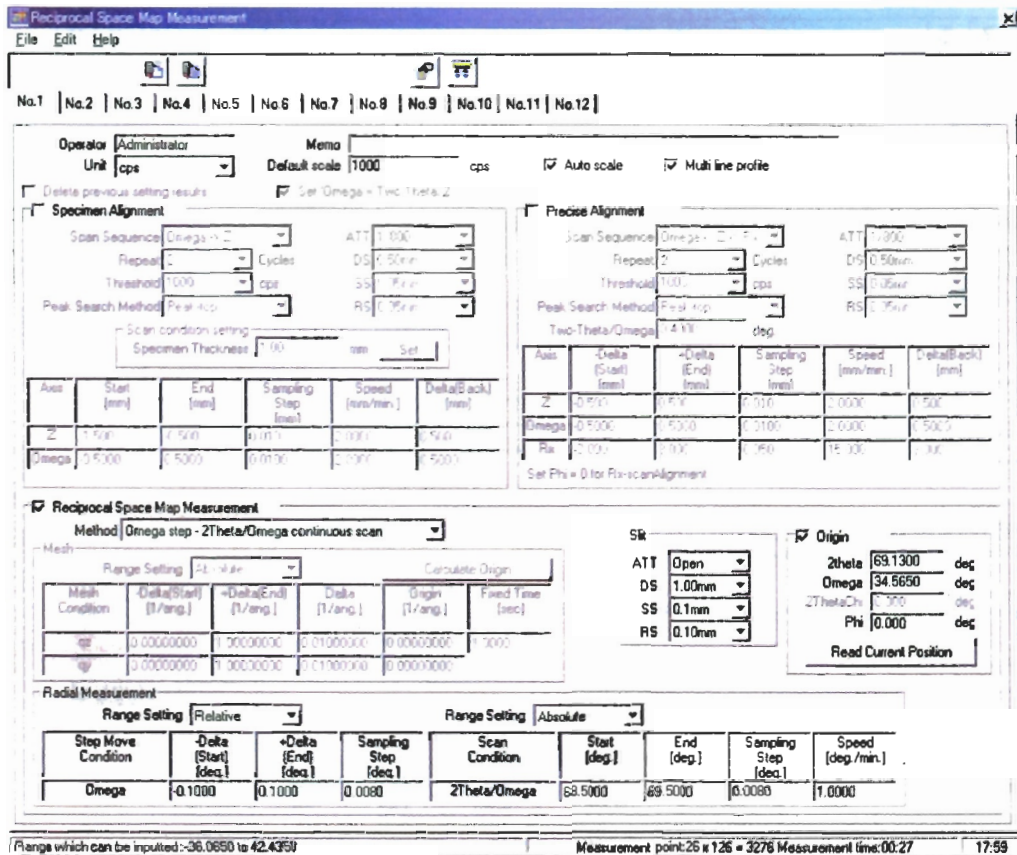
Phi -2.500 deg

Read Current Position

Range which can be inputted: -20.4980 to 51.9697 Measurement time: 00:15 17:50

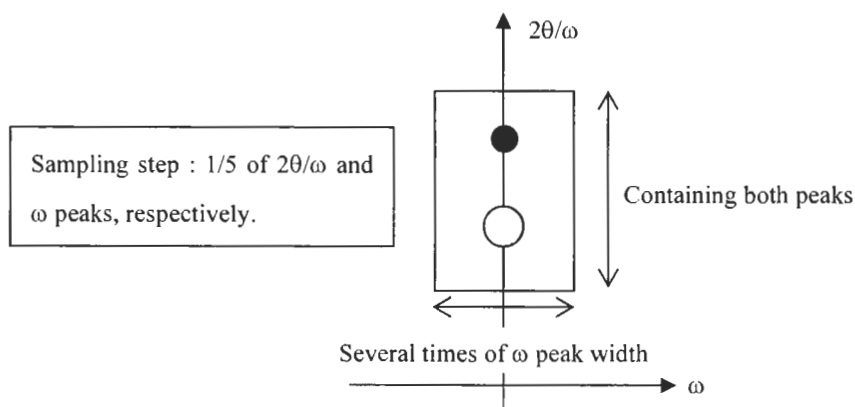
2-1-3 Reciprocal Lattice Mapping

Reciprocal lattice mapping is used to obtain the coordinates of reciprocal lattice points and the degree of relaxation for epitaxial films. An example of scanning condition for SiGe (004) / Si (004) samples are as follows;



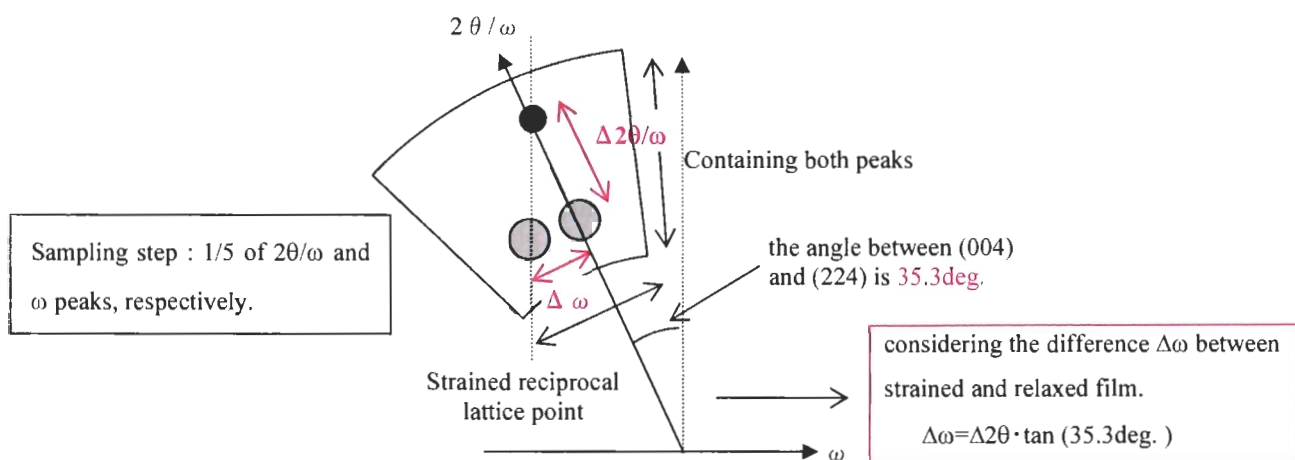
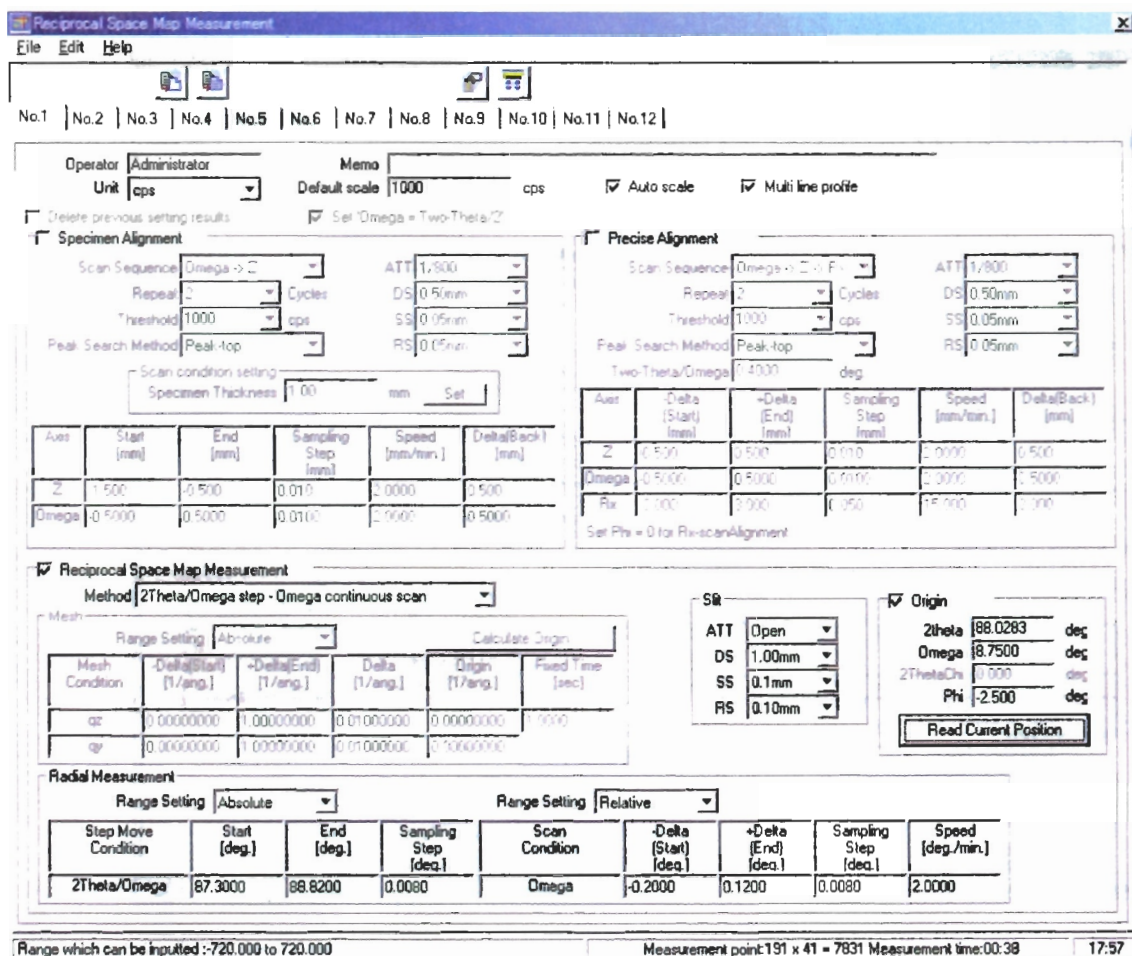
Measurement mode (Omega step – 2Theta/Omega scan or 2Theta/Omega step – Omega scan) is selected as the total number of scan becomes fewer. Because reciprocal space map consists of successive 1D ($2\theta/\omega$ or ω) scans and it takes several ten seconds to position scan axes (axis) to the start angle in every intervals.

To observe the spread of reciprocal lattice point, the range of measurement should be wide enough. The scan range of $2\theta/\omega$ is determined by referring to rocking curve profile as the intensity decreases to the background level at the both end. The range of Omega should be several times larger than the ω peak width.




The following is a example of the scanning condition for SiGe (224) / Si (224) samples.

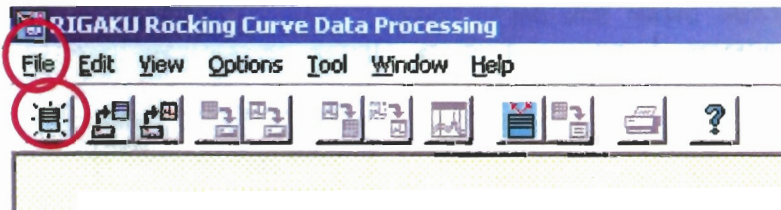
Considering the epitaxial films can be totally strained, the range of Ω should be wider than the case of symmetric RSM measurement.



2-2 Outline of rocking curve simulation software

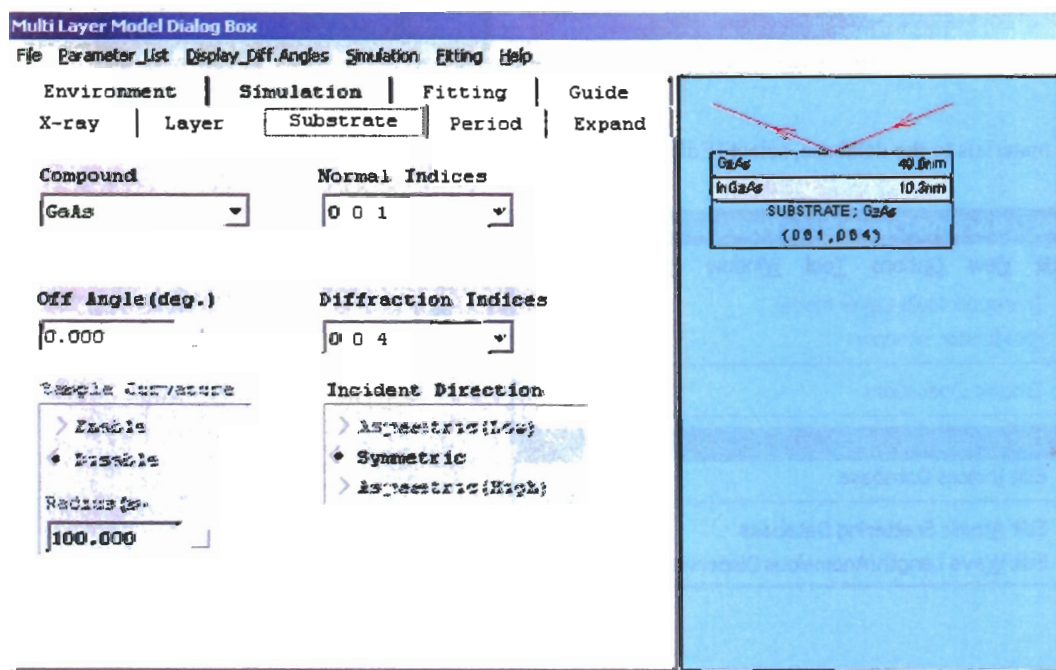
2-2-1 Sample structure and x-ray divergence

Click  or select [File] - [New].



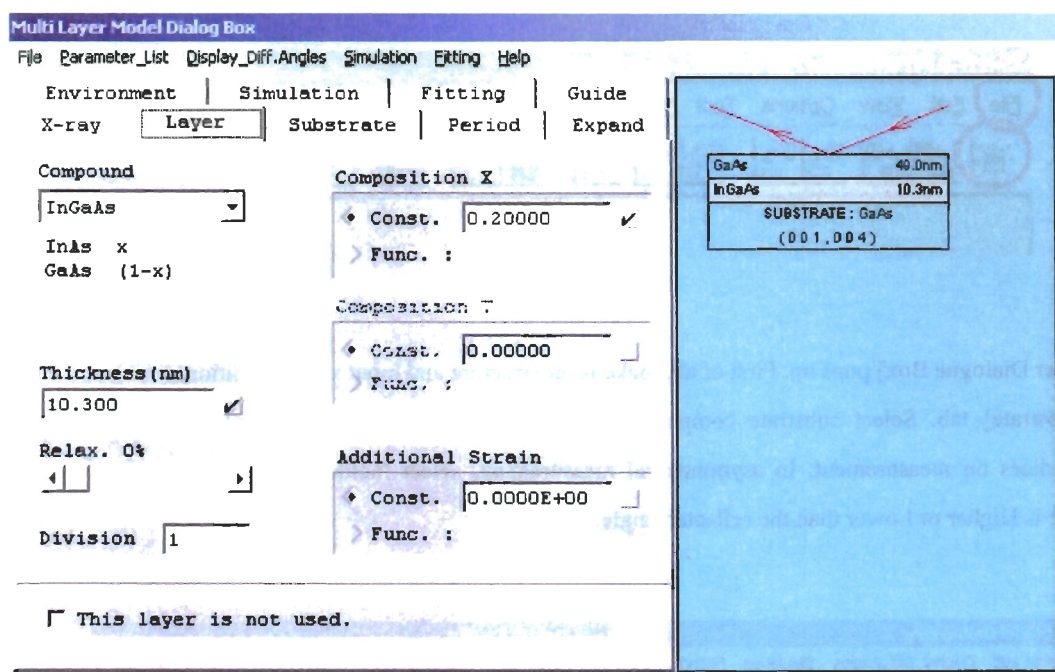
[Multi Layer Dialogue Box] pops up. First of all, make layer structure and input x-ray condition.

Click [Substrate] tab. Select substrate compound, surface normal indices, offset angle if it has, and diffraction indices on measurement. In asymmetrical measurement, select Incident Direction based on the incident angle is Higher or Lower than the reflected angle.

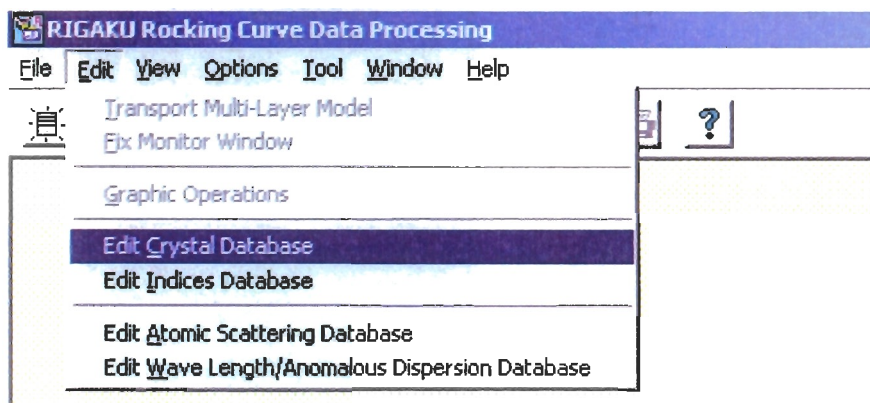


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To add films, double-click SUBSTRATE in the illustration. Select compound, input layer thickness and percentage of the composition X in the case of solid solution. Layers are copied/ deleted by double-clicking on the film in the illustration with left/ right mouse button, respectively.



To add new materials to the database, select [Edit] - [Edit Crystal Database] .



Elements (e.g. Si and Ge) and binary compounds (e.g. GaAs) are included in Basic Crystal list. Crystallographical coefficients are necessary to add new Basic Crystal to the database. Solid solution consists of two basic crystals. New solid solution model is registered if the basic crystals have common crystal system. The values of crystallographical coefficients become the linear combination of each basic crystal.

Editing Crystal Database

Crystal

☒ Basic Crystal ☐ Solid Solution

Open

AlAs
AlN
AlP
AlSb
GaAs
GaN

Exit

Help

Editing Solid Solution Database

Solid Solution Name : AlGaAs

N of Basic Crystals : 2

Structure of the Solid Solution

Crystal 1 AlAs x

Crystal 2 GaAs (1-x)

Crystal 3

Crystal 4

Delete from Database

Basic Crystal List

AlAs	Cubic
AlN	Hexag.
AlP	Cubic
AlSb	Cubic
GaAs	Cubic
GaN	Hexag.
GaP	Cubic
GaSb	Cubic

Save Cancel Help

Editing Basic Crystal Database

Crystal Parameters

Name (ID) AlAs

Crystal System Cubic

Cell Param. a 5.66000 (Å)

c 5.66000

Stiffness C11 12.50000 (1.0E+10 N/m²)

C12 5.34000

C44 5.42000

Temp. Factor B 0.00000

N of Atoms 8 /unit

Delete from Database

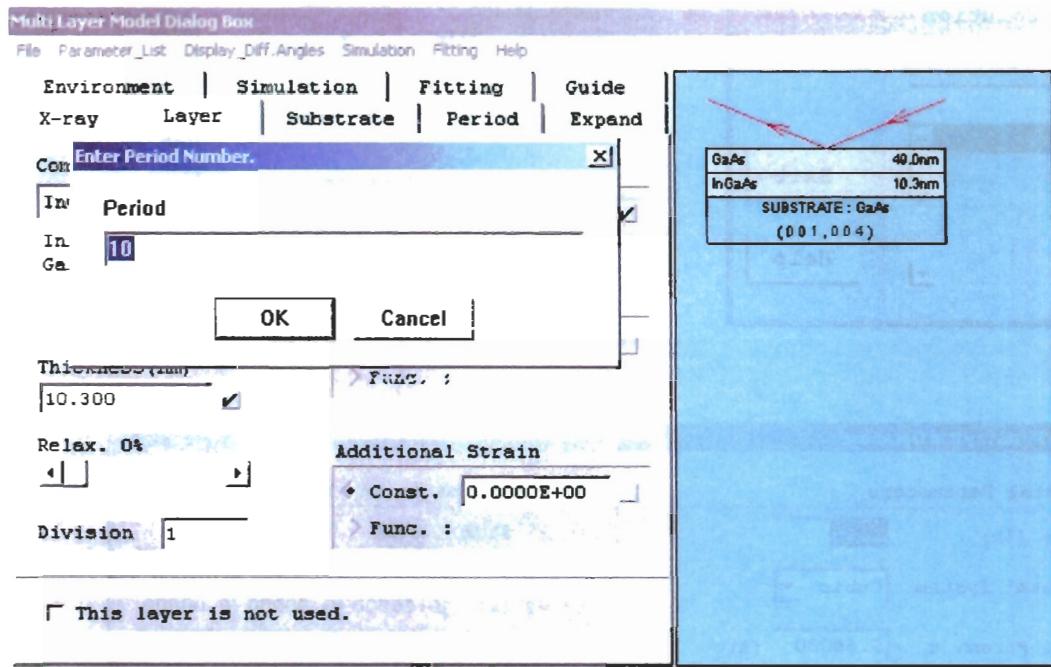
Save Cancel Help

Atom Positions in the Unit Cell

Atom	a(X)	b(Y)	c(Z)
1 Al	0.00000	0.00000	0.00000
2 Al	0.50000	0.50000	0.00000
3 Al	0.50000	0.00000	0.50000
4 Al	0.00000	0.50000	0.50000
5 As	0.25000	0.25000	0.25000
6 As	0.75000	0.75000	0.25000
7 As	0.75000	0.25000	0.75000
8 As	0.25000	0.75000	0.75000
9	0.00000	0.00000	0.00000
10	0.00000	0.00000	0.00000
11	0.00000	0.00000	0.00000
12	0.00000	0.00000	0.00000

TTRAXIII

If the sample has periodical structures, select the layers composing superlattice then [Enter Period Number] window pops up. Input the number of time.



The diffraction angle of those films and substrate are displayed by selecting [Display_Diff.Angles].

Diffraction Angle Display [?] [X]

Material	Bragg(deg.)	Incident(deg.)	A.U.(sec.)	a(Å)	c(Å)	Delta/d
GaAs	33.0259	33.0259	0.0	5.65325	5.65325	0.00%
InGaAs	32.0167	32.0167	-3633.0	5.65325	5.81161	2.80%
GaAs	33.0259	33.0259	0.0	5.65325	5.65325	0.00%

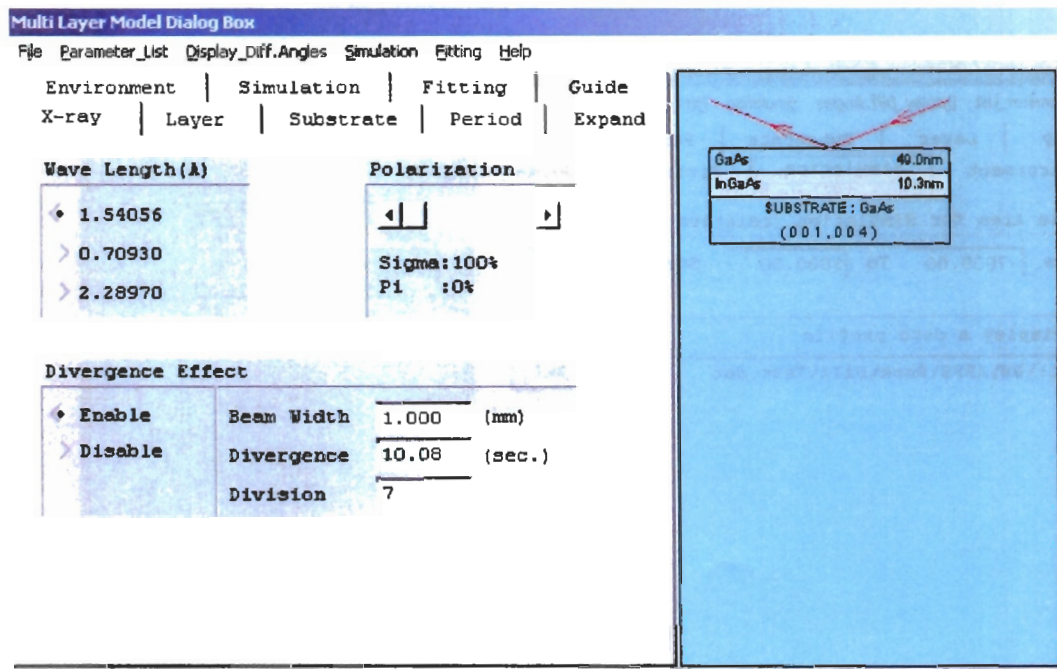
Surface Indices : 0 0 1

Diffraction Indices : 0 0 4

Renew

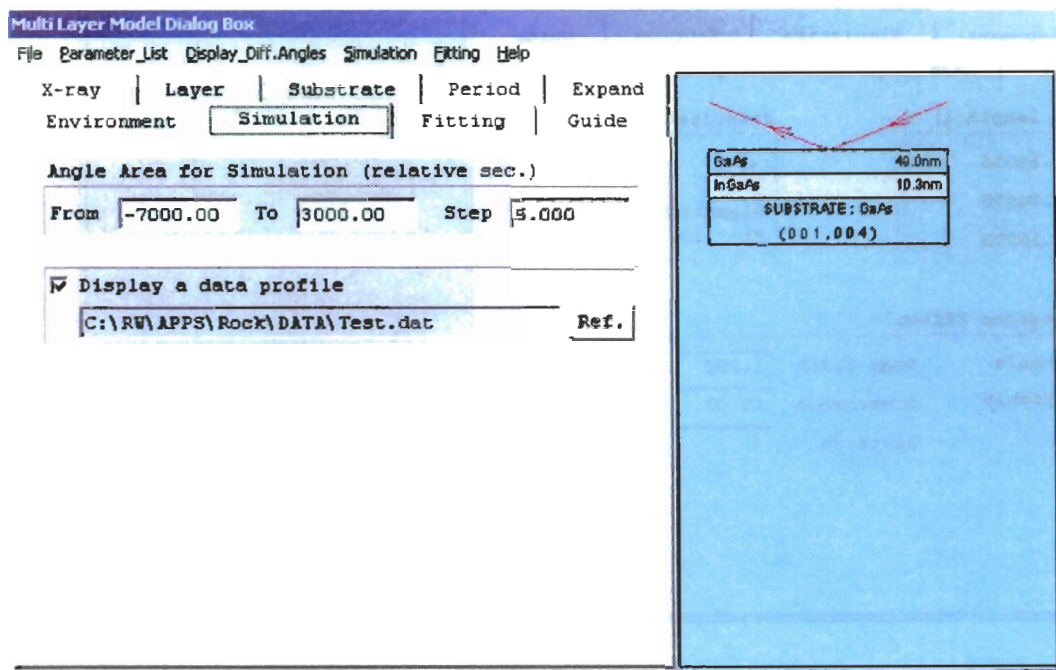
TTRAXIII

Input x-ray wavelength, polarization and divergence. The divergence angle is selected by incident optical system. For [Polarization], σ polarization should normally be selected.

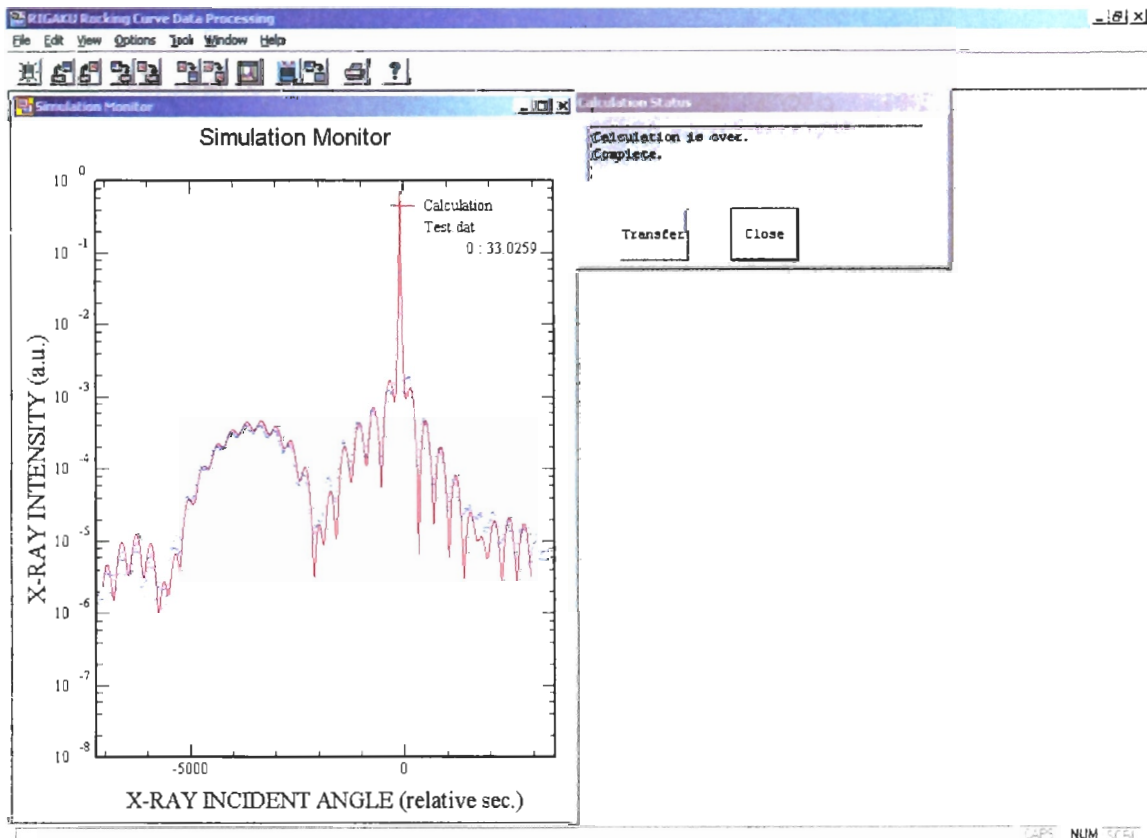


2-2-2 Comparison with measured profile


To compare calculated profile to experimental data, click [Simulation] tab, check [Display a data profile] and select experimental file. Input simulation area and step then click [Simulation] on the bar above.

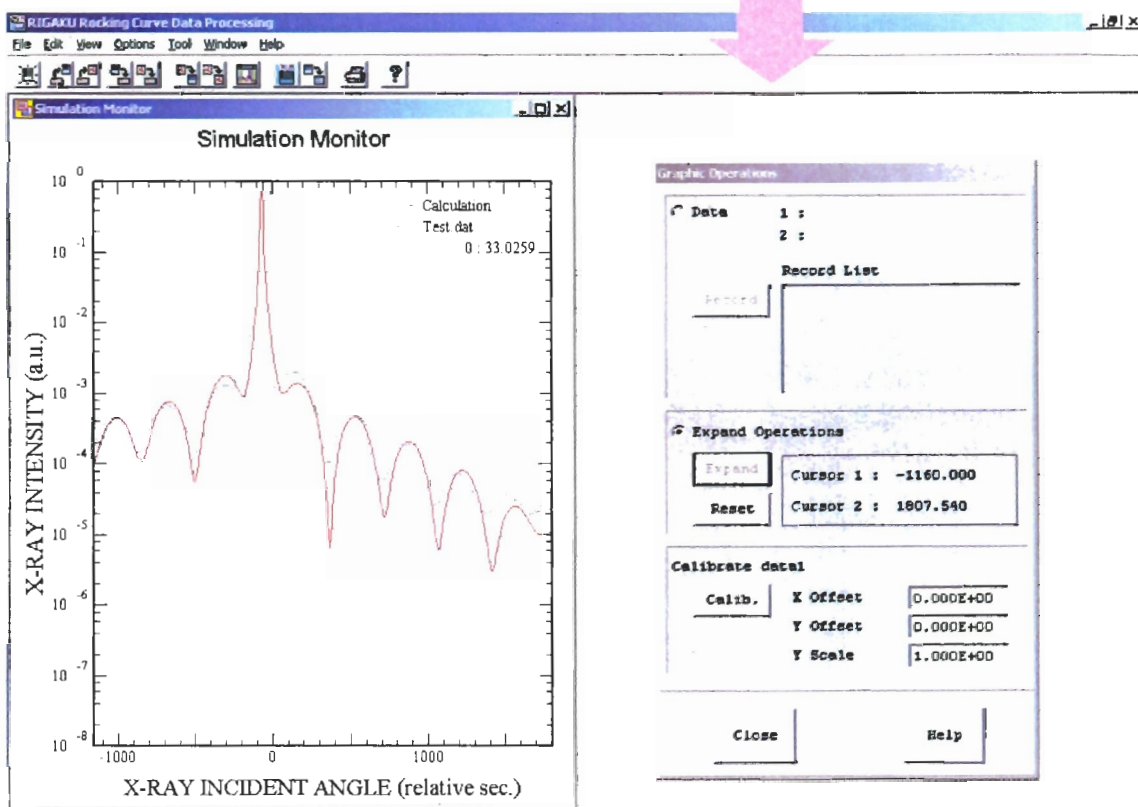
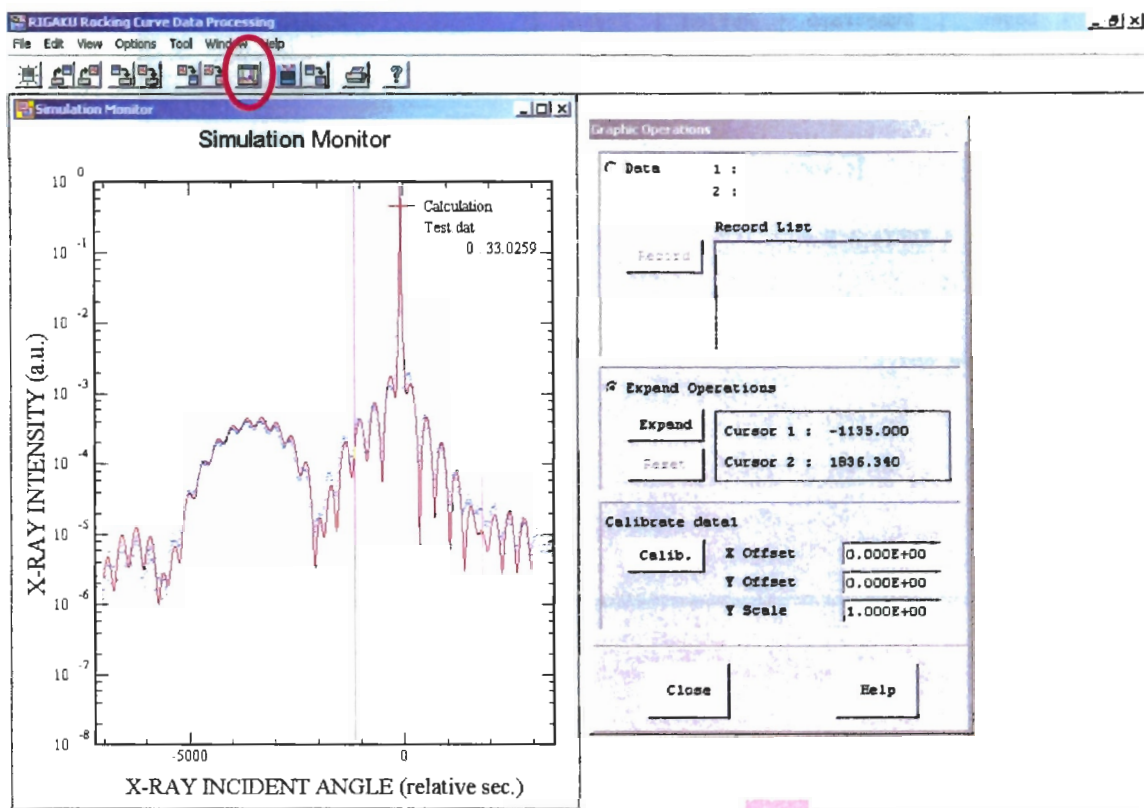


Calculated profile is shown in [Simulation Monitor] .



2-2-3 Expanding

To expand a part of a profile, click  or select [Edit] – [Graphic Operation] . Check [Expand Operations], move cursors to appropriate position, and click [Expand] .



TTRAXIII

If the peak angle differs from each other, make an offset on the calculated profile. Click [Environment] tab and input offset value. Also dynamic scale and background level are entered in this tab.

Multi Layer Model Dialog Box

File Parameter_List Display_Diff.Angles Simulation Fitting Help

X-ray | Layer | Substrate | Period | Expand

Environment | Simulation | Fitting | Guide

Theta Offset Angle(sec.) ✓

Scale Factor ✓

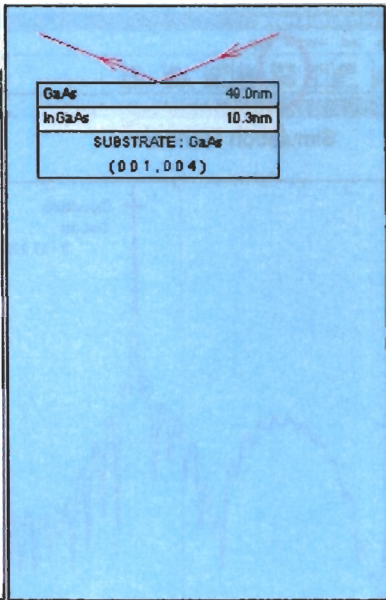
Background (= A * THETA + B)

A = ✓

B = ✓

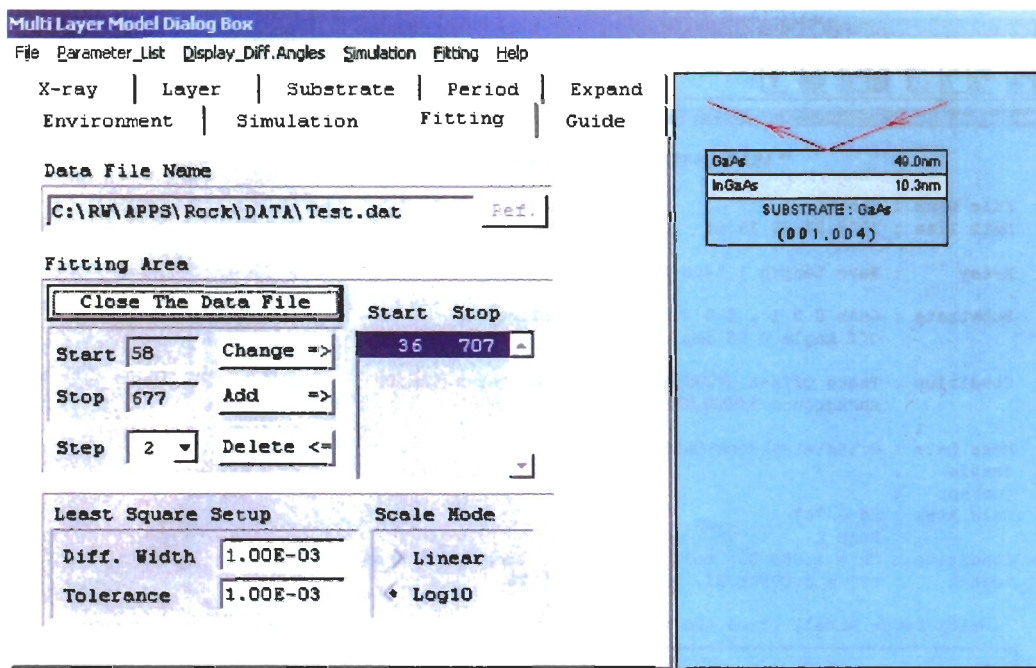
(THETA : Relative deg.)

GaAs	40.0nm
InGaAs	10.3nm
SUBSTRATE: GaAs	
(001,004)	

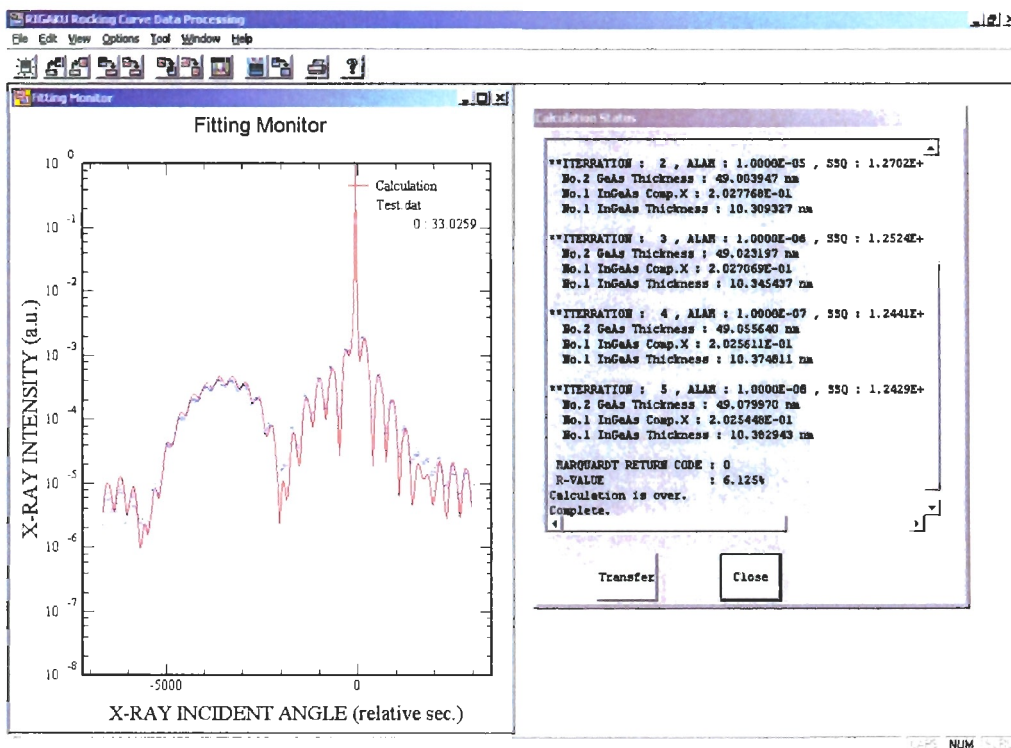


2-2-4 Profile Fitting

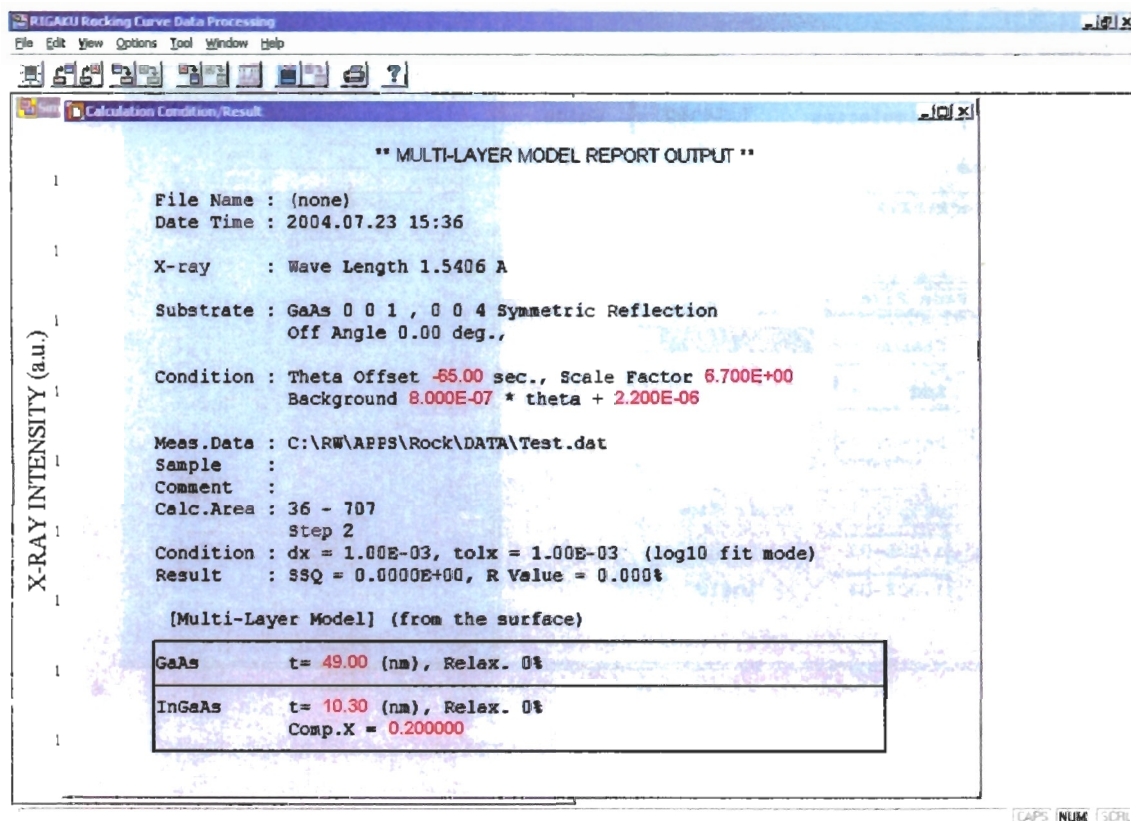
Final procedure of rocking curve analysis is profile fitting. Click [Fitting] tab, select measurement data, and input fitting area. Parameter which have checkmark in their checkbox are refined.



To start fitting procedure, click [Fitting].



The result of fitting is shown by selecting [Parameter_List] on [Multi Layer Model Dialog Box]. When clicking the menu, a message asks whether you want to display the fitting conditions. By choosing "Yes", fitted parameters are displayed by red characters.



3. X-ray reflectivity

3-1 Flow Chart

Preparation

To select the geometry system, sampling step and slit width, simulate XRR profile with GXRR software. Click  and input designed layer structure.

☞ **To select optical system**, change [Number of divisions] from 1 to 7. Input 0.04 and 0.01 into [Divergence] and examine the profiles;

If the profiles do not change clearly → Slit collimation

If 0.01 gives distinct oscillation while 0.04 gives dull shape

→ Ge(220) channel-cut monochrometer

☞ **To select step width**, change [step] value and compare the profiles.

☞ **To select the DS slit**, estimate the critical angle. To observe XRR profiles properly, it is necessary to limit the irradiation width within the sample width at critical angle. Refer to [irradiation width- incident angle] graph and select the most suitable one.


Measurement

☞ **Optics adjustment.**

☞ **Sample alignment and measurement.** Perform 2theta/omega scan with the sampling step selected above.

Analysis

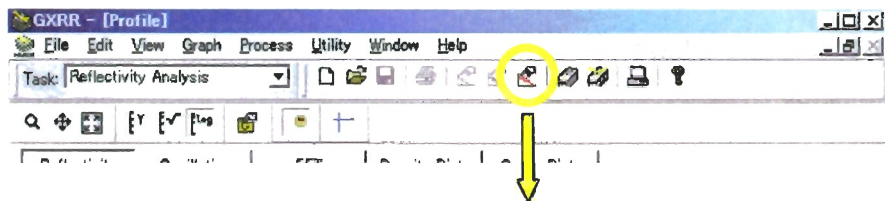
☞ **RMS fitting with GXRR software.** Click  to switch to fitting mode. Compare calculated profile to measured profile, change parameters to make it resemble measured one before execute profile fitting.

☞ If there are large differences between calculated and measured profile, the designed layer structure might be quite different from the actual structure. In such a case, Fourier Transformation  would be effective to make a proper layer model.

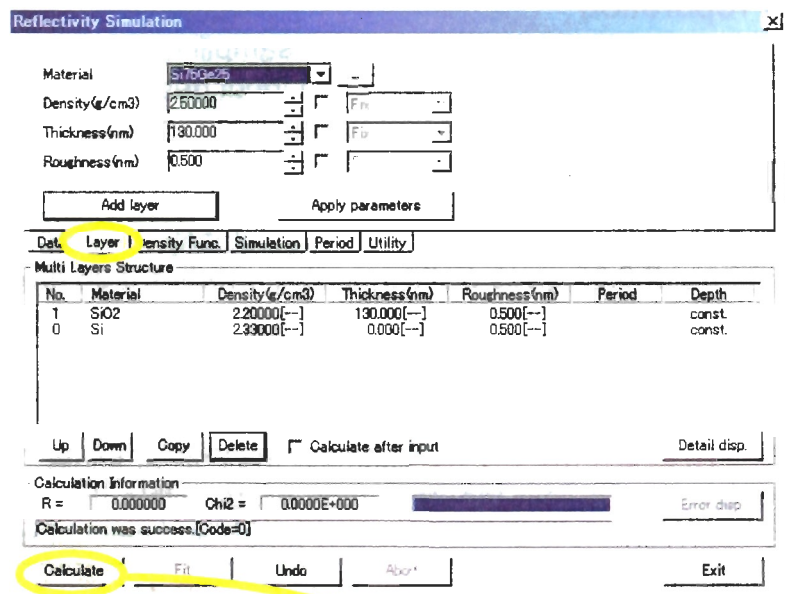
3-2 Preparation

Start GXRR software.

Click "Simulation" button.



Select "layer" tab.



Input the designed layer structure.

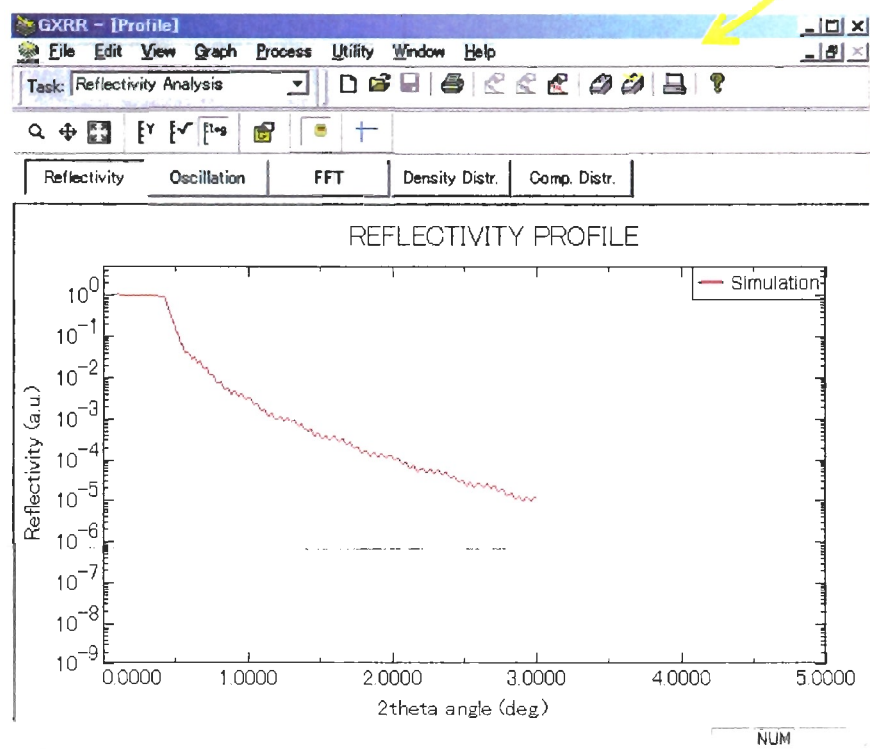
Choose the substrate/layer material from "Material" list.

Click "Add layer" button.

Change "Thickness" to the designed values.

Use "Up" and "Down" buttons to switch the order of the layers.

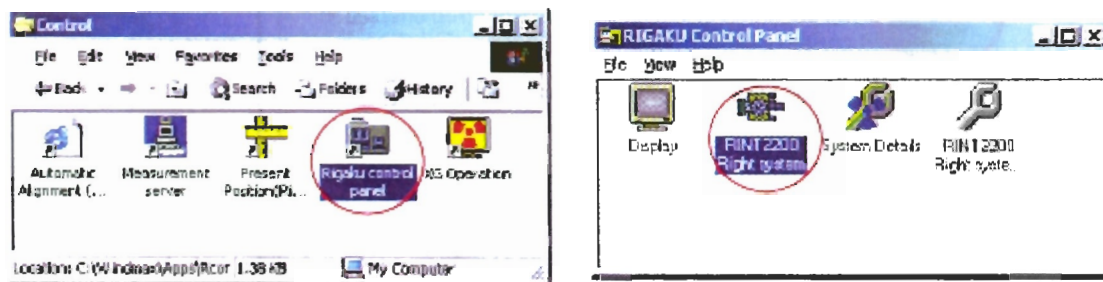
Click "Calculate" to show the simulated profile. (When "Calculate after input" is checked, the simulated profile will be calculated automatically.)



3-3 Measurement

3-3-1 Setting up the geometry system

Open "Rigaku control panel" from the Rigaku folder. Double click "RINT 2200 Rigaku system."



Select following items;

Attachment :

Attachment for thin film/ UltimaIII

Incident monochro:

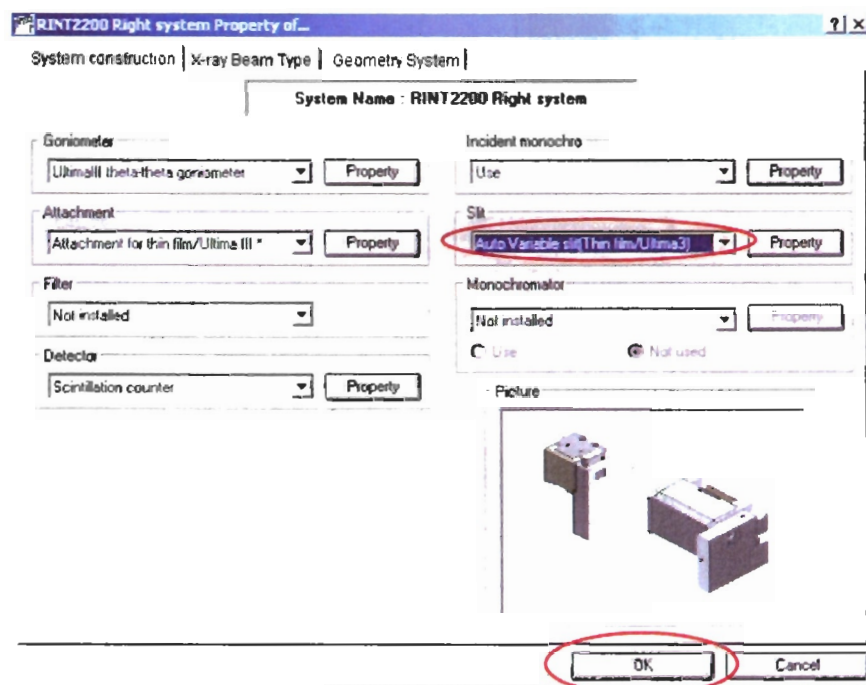
Use

Slit:

Auto Variable slit(Thin film/
UltimaIII)

Monochromator:

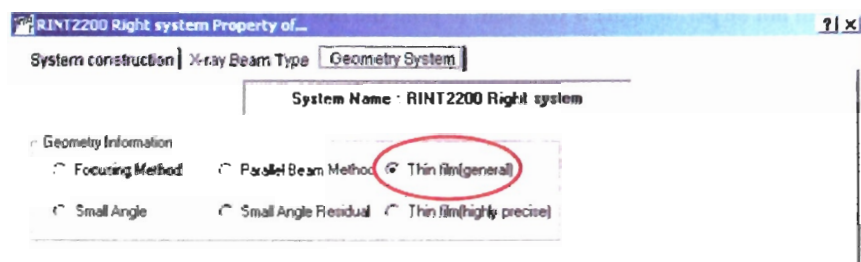
Not installed



Geometry Information:

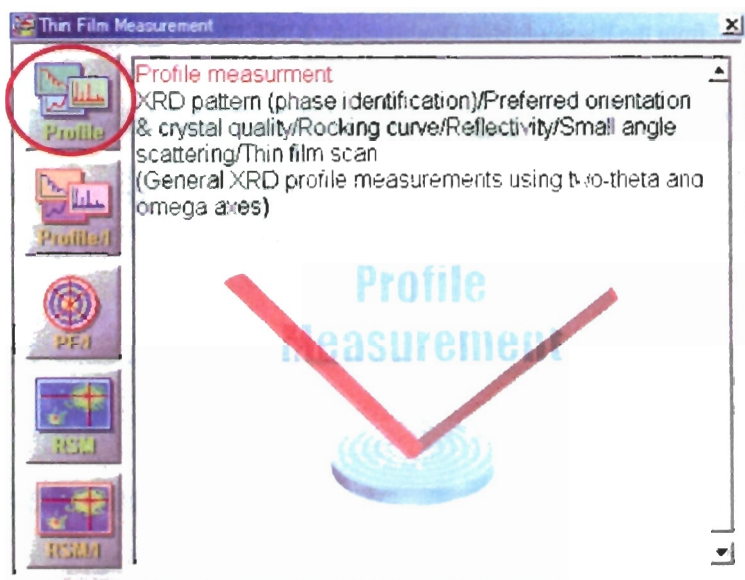
Thin film (general) or

Thin film (highly precise)

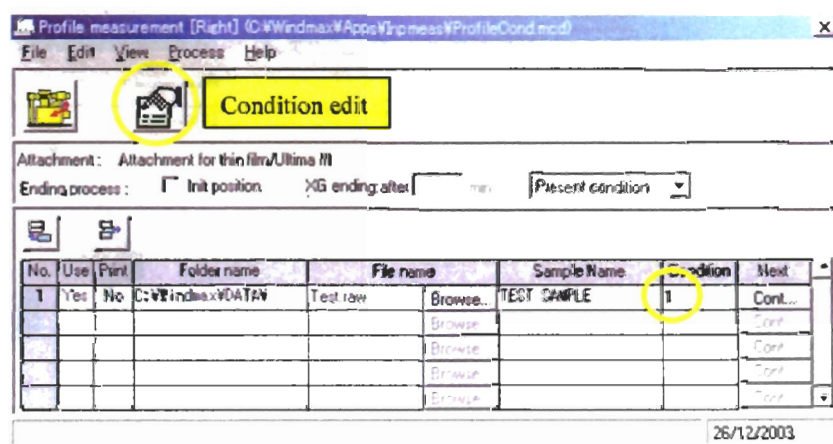


3-3-2 XRR Measurement

Open [Start] – [Programs] - [Rigaku] – [Rigaku Measurement] – [Thin Film Meas. Menu.]



Select [Profile] measurement.



Input the folder name, the file name and the sample name. Switch "Use" to "Yes" (otherwise the measurement will not be performed!) If you want to print the raw data (measured profile and the scanning condition) after the measurement, switch "Print" to "Yes."

To edit measurement conditions, click on the "condition edit" button or double-click on the number input in [Condition.]

TTRAXIII

Initialize measurement conditions

Copy measurement conditions

Manual slit setting

Offset

The screenshot shows the 'Profile Measurement' window. At the top, there are buttons for 'Initialize' and 'Copy', which are circled in red. Below these are various settings for 'Specimen Alignment' and 'Precise Alignment'. The 'Specimen Alignment' section includes fields for 'Specimen Thickness' (set to 1.00 mm) and 'Set' button. The 'Precise Alignment' section includes fields for 'Two Theta/Omega' (set to 4.000 deg) and 'Set' button. There are also tables for 'Scan condition setting' and 'Range Setting'. The 'Scan condition setting' table has columns for Axis, Start, End, Sampling Step, Speed, and Dehalback. The 'Range Setting' table has columns for Exec, Start, End, Sampling Step, Speed, ATT, DS, SS, and RS. The 'Range Setting' table is currently empty. The 'Offset' label points to the 'Offset' field in the 'Range Setting' table.

☆ Specimen alignment

For X-ray reflectivity analysis, both “Specimen Alignment” and “Precise Alignment” are needed to be done before the measurement. Check these items.

“Specimen Alignment” – Input “Specimen Thickness” and click “Set.” The recommended scanning conditions of Z and omega axes are input automatically.

“Precise Alignment” – Input the value little bit smaller than the critical angle to “Two Theta/Omega.”

☆ Preliminary measurement

To estimate the attenuator switching positions, preliminary measurement is performed. The following is the typical scanning condition.

Two theta / omega scan range : 0-3deg.

Sampling step : the estimated value from the simulation

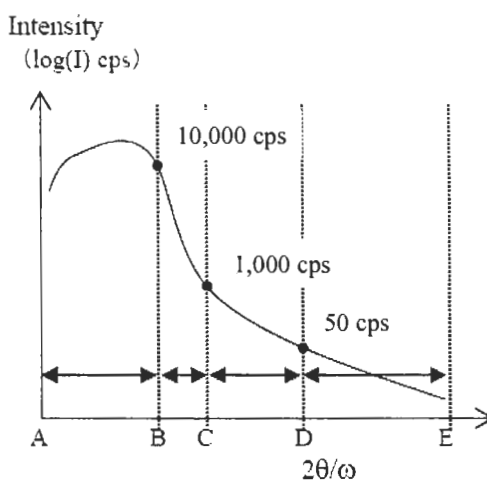
Sampling speed : 1deg./min.

Attenuator : 1/800

DS, SS, RS : the selected widths from the simulation.

The attenuator switching positions are estimated as follows.

Intensity	Attenuator is switched as
10,000 cps	1/5000 → 1/400
1,000 cps	1/400 → 1/30
50 cps	1/30 → open



☆ Main measurement

Remove the check marks at “Specimen Alignment” and “Precise Alignment” because the alignment procedures are already finished before the preliminary measurement.

Set the scan conditions and attenuator switching positions for main measurement. Click on “Connect All Ranges” to make sure that the start and end angle of each scan is continuous.

☒ Profile Measurement

Scan axis: $2\theta/\omega$ Scan method: Continuous Range Setting: Absolute

Exec.	Start [deg.]	End [deg.]	Sampling Step [deg.]	Speed [deg./min.]	ATT	DS	SS	RS
<input checked="" type="checkbox"/>	0.0000	0.3000	0.0100	2.0000	1/800	0.1mm	0.2mm	0.2mm
<input checked="" type="checkbox"/>	0.3000	0.5000	0.0100	2.0000	1/100	0.1mm	0.2mm	0.2mm
<input checked="" type="checkbox"/>	0.5000	1.2000	0.0100	2.0000	1/10	0.1mm	0.2mm	0.2mm
<input checked="" type="checkbox"/>	1.2000	5.0000	0.0100	1.0000	open	0.1mm	0.2mm	0.2mm
<input checked="" type="checkbox"/>	5.0000	10.0000	0.0100	0.5000	open	0.1mm	0.2mm	0.2mm
<input checked="" type="checkbox"/>	10.0000	10.0000	0.0100	0.0000	1/800	0.1mm	0.2mm	0.2mm

Connect All Ranges

Origin

2θ : 0.000 deg

ω : 0.000 deg

2θ Ch: 0.000 deg

ϕ : 0.000 deg

Read Current Position

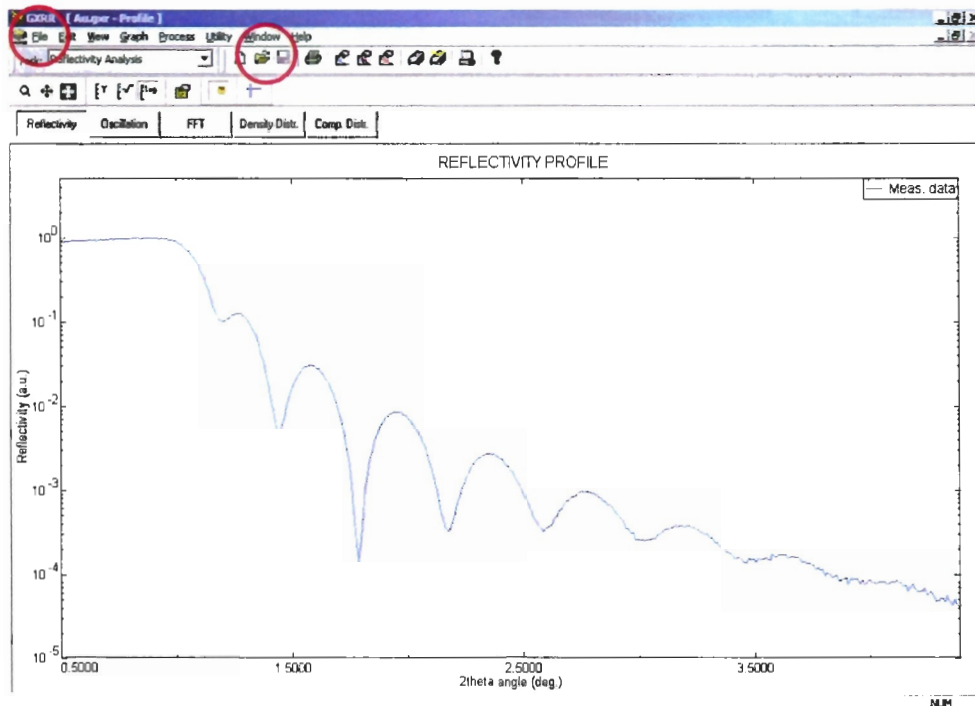
3-4 Outline of XRR analysis software (Demo data file path : C:\Program Files\Rigaku\GXRR3\Data)

3-4-1 Open experimental data


Start the GXRR software and read experimental data. Both of *.raw and *.dat are readable on GXRR.

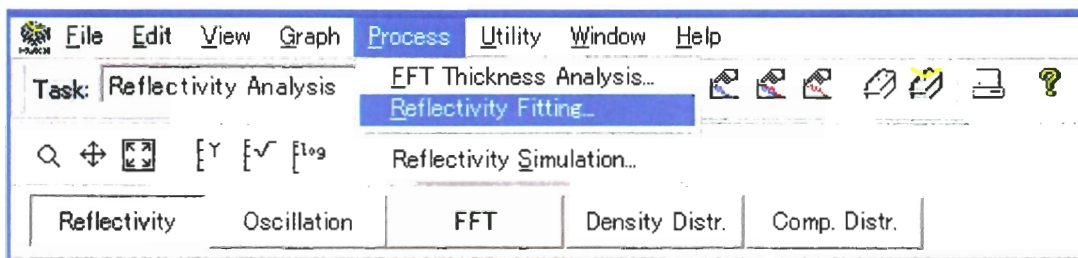
Open “Au.gxr” file saved in following path, C:\Program Files\Rigaku\GXRR3\Data.

Designed model ; Au (20nm) / Si substrate



3-4-2 Set the designed model

First, Select [Process] – [Reflectivity fitting] of click  to make a structure model of layer.



Select [Material] in [layer] tab, then select Si and click [Add layer]. Next, select Au and input [Density], [Thickness] and [roughness] as the designed values.

Remove [Thickness], [Roughness], [Density] checkbox of Au layer, [Roughness] checkbox of Si substrate and [Apply parameters] to set as fitting parameters.

Reflectivity Fitting

Material: Density(g/cm3):

Thickness(nm): ☒ Fix ☐ Surf. side: ☐ Fix ☐ Sub. side:

Roughness(nm): ☒ Fit ☐ Depth distribution:

Data **Layer** **Fitting** **Period** **Utility**

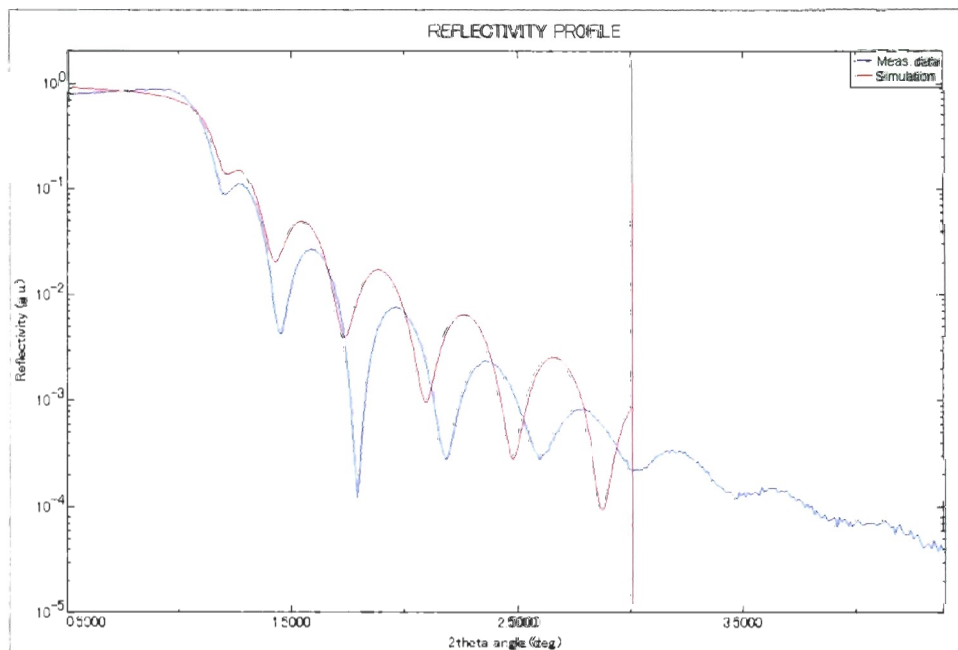
Multi Layers Structure

No.	Material	Thickness(nm)	Density(g/cm3)	Roughness(nm)	Subs. side den..	Depth	Period
1	Au	20.000	19.30000	0.500		const.	
0	Si	0.000	2.33000	0.500		const.	

☒ Calculate after input


Calculation Information
 R = 0.171732 Chi2 = 7.8463E-001
 Calculation was success.[Code=0]

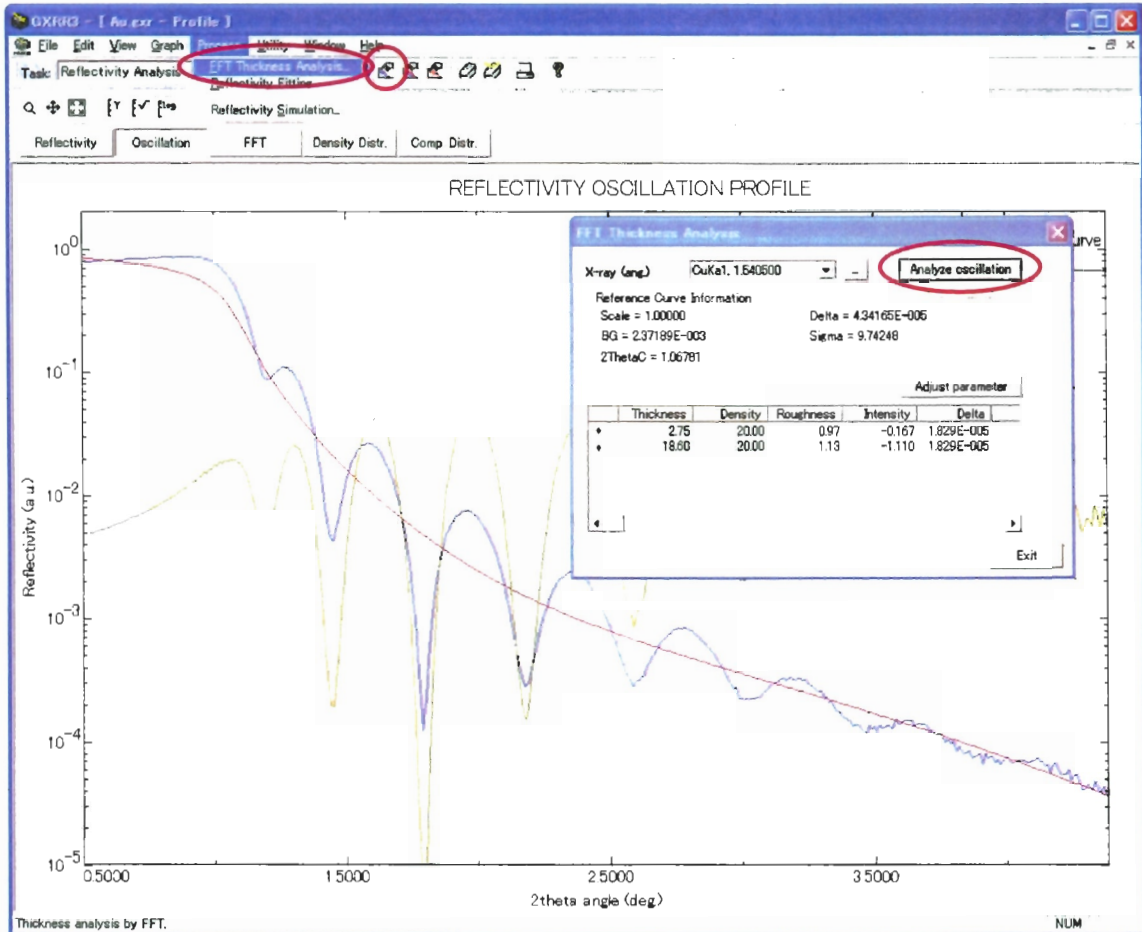
Click [Calculate] to display the calculated profile. The difference between calculated and measured profile indicates that those parameters used in the calculation differ from actual values.



3-4-3 Estimation of film thickness by Fourier transform

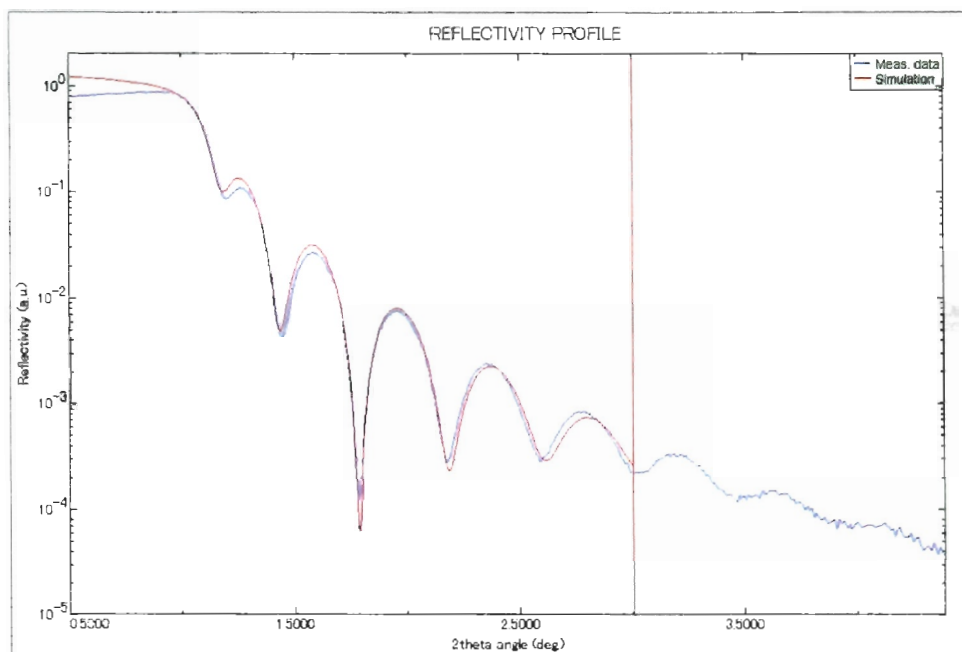
Film thickness is estimated by Fourier transform of oscillation components appearing on an XRR profile. The result is reliable in the case of single-layer film, and it is also useful for multi-layer samples because it still gives some hints for XRR analysis.

[FFT Thickness] dialogue appears by choosing [Process] – [FFT Thickness Analysis] or click . Click [Analyze oscillation]. The oscillation components are separated as follows.



This oscillation is transformed to film thickness by the period of the oscillation and the electron density of the film calculated from chemical formula and the density. The results of FFT analysis (Thickness, Density, Roughness) show on FFT Thickness Analysis dialog.

Click [Adjust parameter] to adjust the results of FFT analysis to the layer model.



3-4-4 Profile Fitting

Input [Divergence] corresponding to optical system in [Data] tab.

Profile		X-ray Beam	
2theta0 (deg.)	0.00000	<input type="checkbox"/> Fit	CuKa1 (1.540500)
Max. intensity	1.3533E+000	<input checked="" type="checkbox"/> Fit	Divergence (deg.) 0.01000
BG intensity	4.6871E-005	<input checked="" type="checkbox"/> Fit	

Data Layer **Fitting** Period Utility

Set [Fitting Region] to decide the fitting range. Ordinary, input [Start] to the critical angle and [Stop] to the angle that intensity becomes background level.

Fitting Region		Fitting Conditions	
Start angle (deg.)	0.9000	Convergence	0.01
Stop angle (deg.)	3.0000	Max. of iteration	20

Data Layer **Fitting** Period Utility

Click [Fit] to start profile fitting. Parameters which have checkmark in their checkbox are refined. Profile fitting will be considered to finish when necessary parameters converge and R-value decreases to ~0.04.

