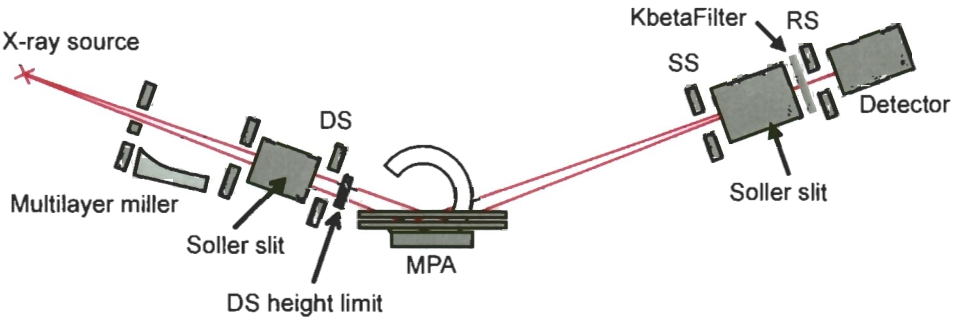
# Pole figure measurement (Schulz reflection method)

**【Optic】**




**【Measurement flow】**

**A.** Test scan

The profile is measured in wide range. Phase identification is performed and sample orientation is investigated compared to intensity ratio of database card. Then, evaluated lattice plane is decided.

**B.** Investigation of slit condition

Slit condition changes according to sample size and peak width. The peak is measured in lowest alpha angle.

**C.** Main measurement

Pole figure measurement is performed.

**【Exchange of geometry】**

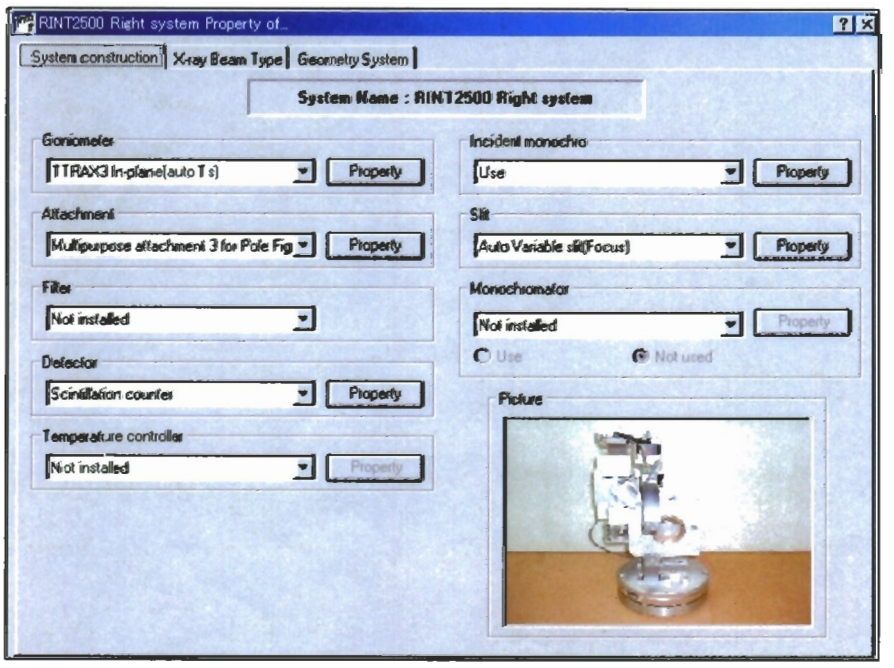
**1.** It is required to finish auto alignment of Focusing method with "Standard sample holder". In most cases, 5deg soller slit is used.

**2.** Multipurpose attachment is mounted. The power of RCD is turned off. (There is the switch on back side).

**3.** After connecting attachment cable, the power of RCD is turned on.

**4.** The sample is mounted on MPA. Rolling direction is faced to beta:0deg.

**5.** Click "RINT2500 Right system" in [Rigaku]-[Control]-[Rigaku Control Panel]. Select items as follows.



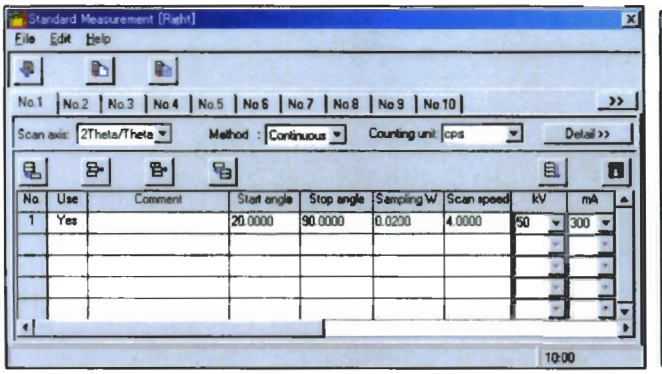

6. Click "Auto alighment" in [Rigaku]-[Control]. The optimization of theta axis and sample height is performed.

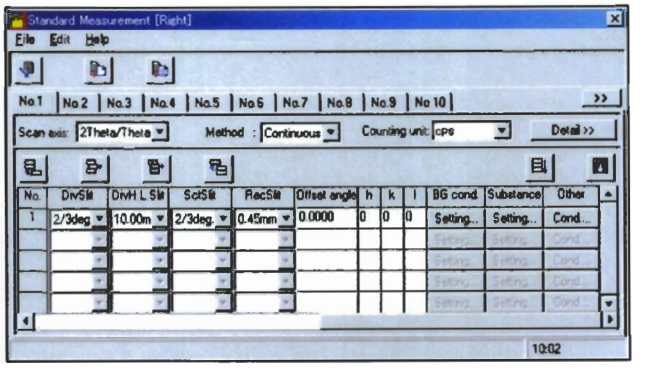


Rigaku

**【A. Test scan】**

**A-1.** Click "Standard measurement" in [Rigaku]-[Right measurement].
Enter parameters as follow.



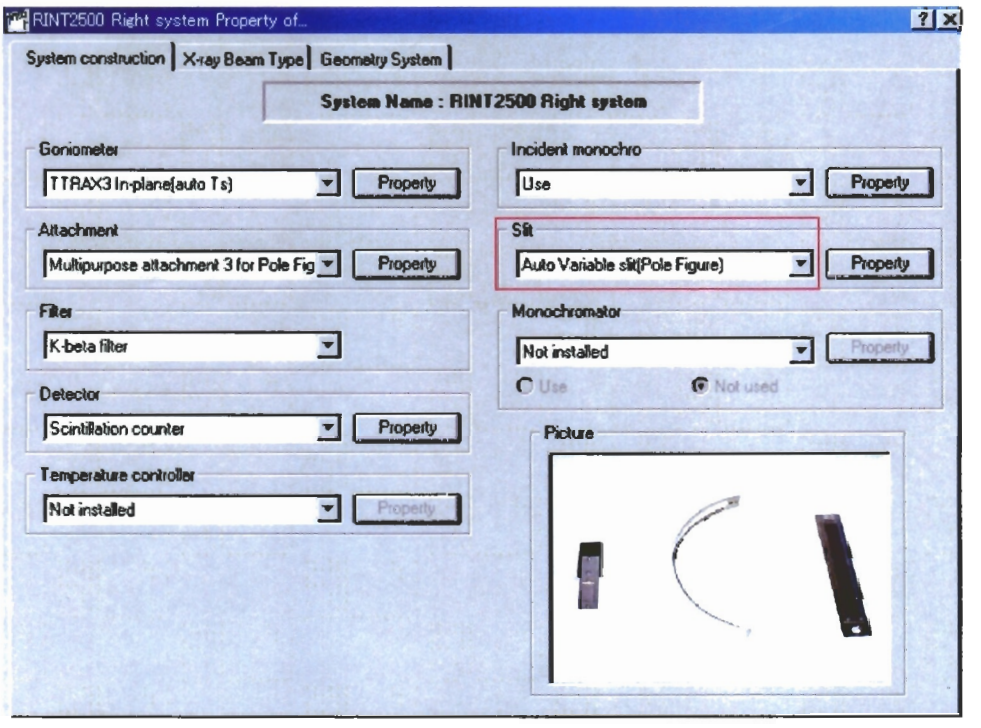




**A-2.** Press "Start measurement".

**A-3.**
Phase identification is performed. And, the intensity ratio of measured peak and database is compared.
If there is no difference between measured peak and database card, it seems that there is no orientation in the sample. If there is the difference, the sample is regarded as texture, pole figure measurement is performed.

**【B. Investigation of slit condition】**

**B-1.** Change "Slit" to slit for Pole Figure in Rigaku Control Panel.

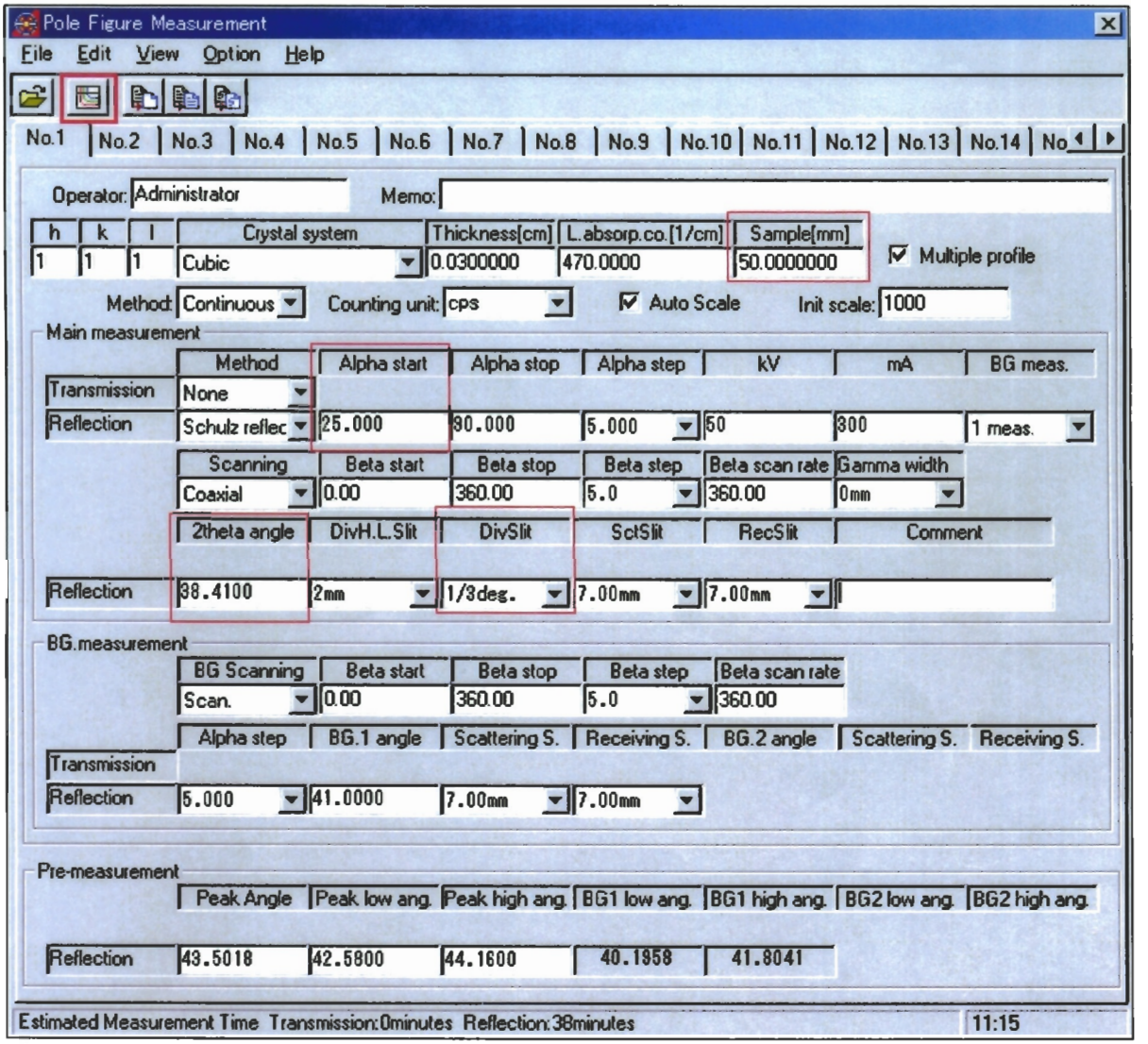


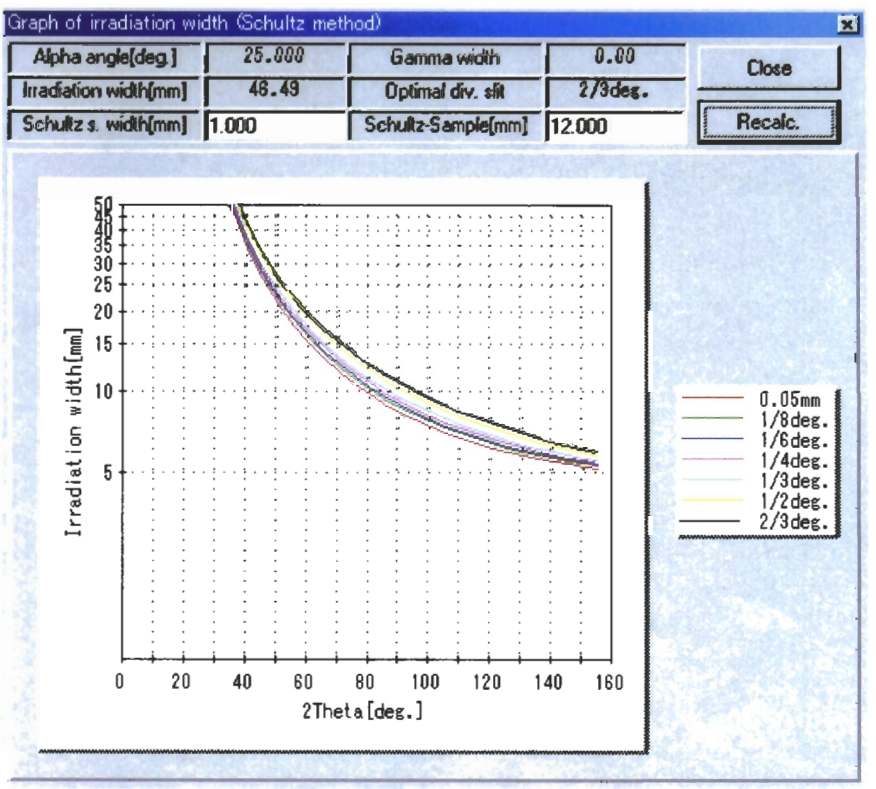

**B-2.** Set 2mm divergence height limit slit, and mount schulz slit.

**B-3.** Click [Pole Figure measurement] in [Rigaku]-[Right Measurement].
Select "None" in [Transmission] and "Schulz reflection" in [Reflection].
Enter sample size, alpha start angle and 2theta angle measured in test scan.

**B-4.** Push "Graph of irradiation width" button.
Optimum divergence slit is calculated when pushing "Recalc" button

**B-5.** Enter the optimum DS in "DivSlit".

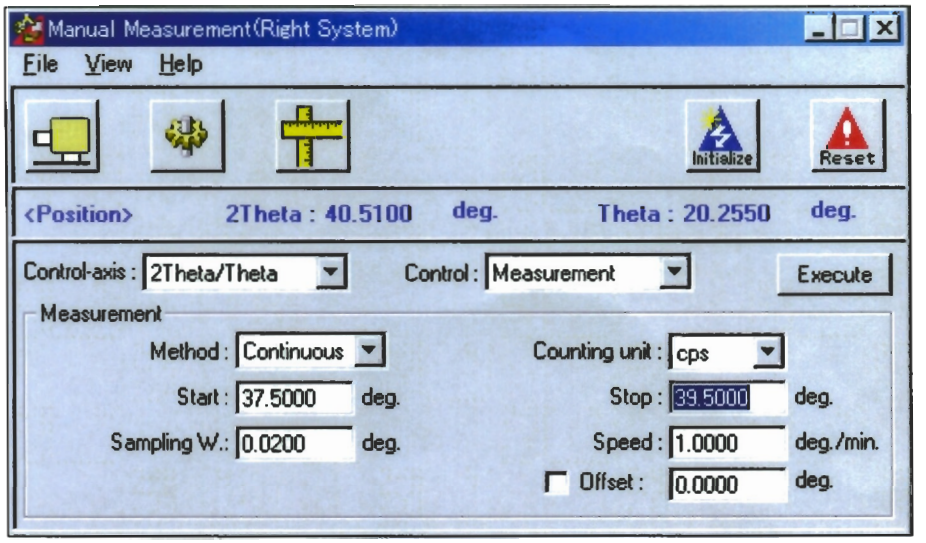

Pole Figure Measurement
File Edit View Option Help
No.1 No.2 No.3 No.4 No.5 No.6 No.7 No.8 No.9 No.10 No.11 No.12 No.13 No.14
Operator: Administrator
Memo:
h k l Crystal system Thickness[cm] L.absorp.co.[1/cm] Sample[mm]
1 1 1 Cubic 0.0300000 470.0000 50.0000000
Multiple profile
Method: Continuous
Counting unit: cps
Auto Scale
Init scale: 1000
Main measurement
Method Alpha start Alpha stop Alpha step kV mA BG meas.
Transmission None
Reflection Schulz reflec 25.000 80.000 5.000 50 300 1 meas.
Scanning Beta start Beta stop Beta step Beta scan rate Gamma width
Coaxial 0.00 360.00 5.0 360.00 0mm
2theta angle DivH.L.Slit DivSlit SctSlit RecSlit Comment
Reflection 38.4100 2mm 1/3deg. 7.00mm 7.00mm
BG.measurement
BG Scanning Beta start Beta stop Beta step Beta scan rate
Scan. 0.00 360.00 5.0 360.00
Alpha step BG.1 angle Scattering S. Receiving S. BG.2 angle Scattering S. Receiving S.
Transmission
Reflection 5.000 41.0000 7.00mm 7.00mm
Pre-measurement
Peak Angle Peak low ang. Peak high ang. BG1 low ang. BG1 high ang. BG2 low ang. BG2 high ang.
Reflection 43.5018 42.5800 44.1600 40.1958 41.8041
Estimated Measurement Time Transmission:0minutes Reflection:38minutes
11:15


Graph of irradiation width (Schultz method)
Alpha angle[deg.] 25.000 Gamma width 0.00
Irradiation width[mm] 48.49 Optimal div. slit 2/3deg.
Schultz s. width[mm] 1.000 Schultz-Sample[mm] 12.000
Close
Recalc.
Irradiation width[mm]
2Theta[deg.]
0.05mm
1/8deg.
1/6deg.
1/4deg.
1/3deg.
1/2deg.
2/3deg.

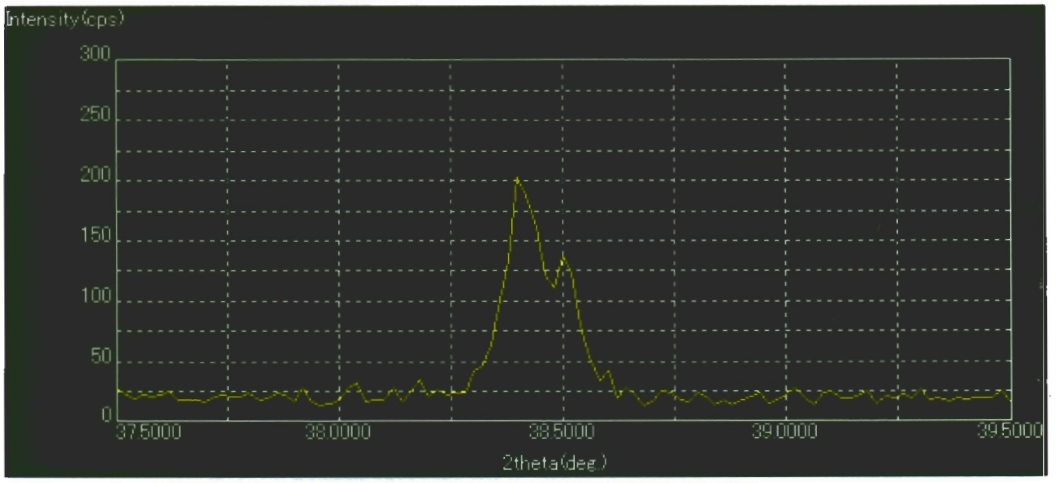


Rigaku

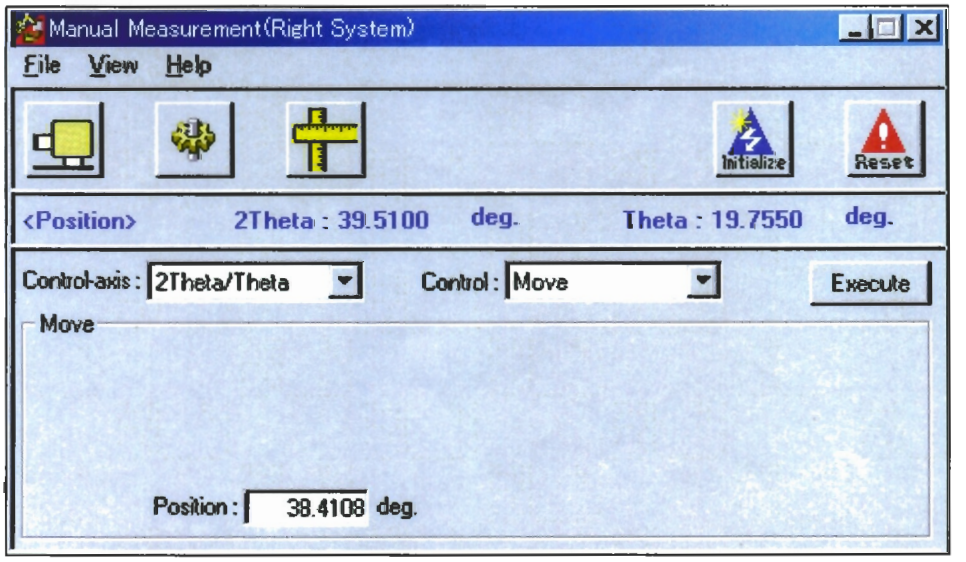
**B-6.** Click [Manual measurement] in [Rigaku]-[Right Measurement].
Set [DivSlit] to optimum DS, [RecSlit] to 0.45mm. The value of [SctSlit] is same as [DivSlit].
Profile measurement is performed.




**B-7.** Read off peak position.

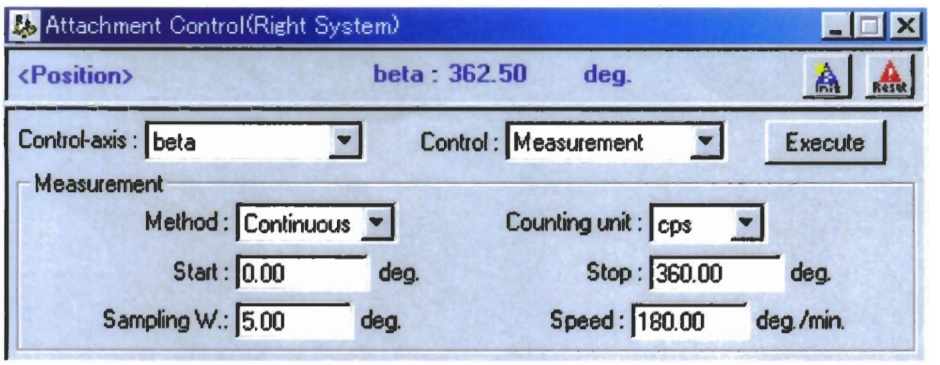


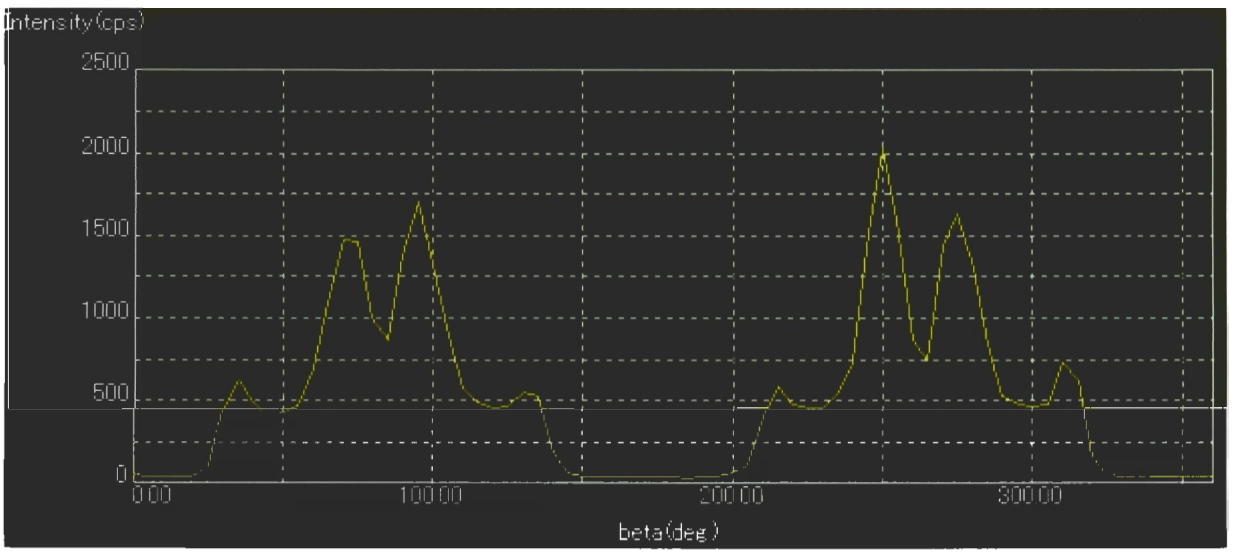

**B-8.** Set to the position with "Move".



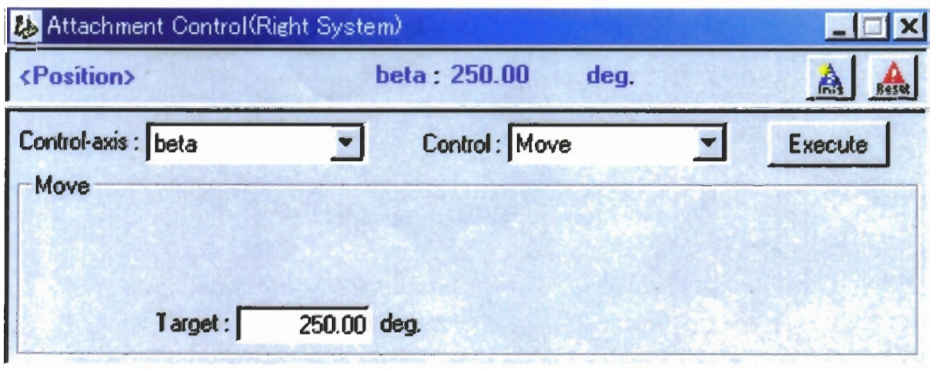
**B-9.** Open [Attachment Control] window. Move to the lowest alpha angle to be measured in [Alpha-R]. The max range of Alpha-R is 15deg to 90deg. As alpha angle is lower, the peak width broadens.


**B-10.** Beta scan is performed to find the strongest point.



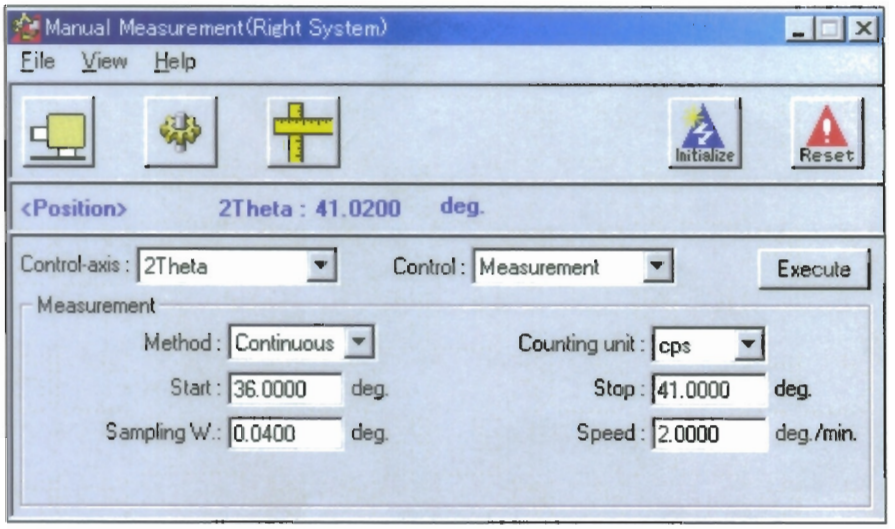

**B-11.** Move to the strongest beta angle.



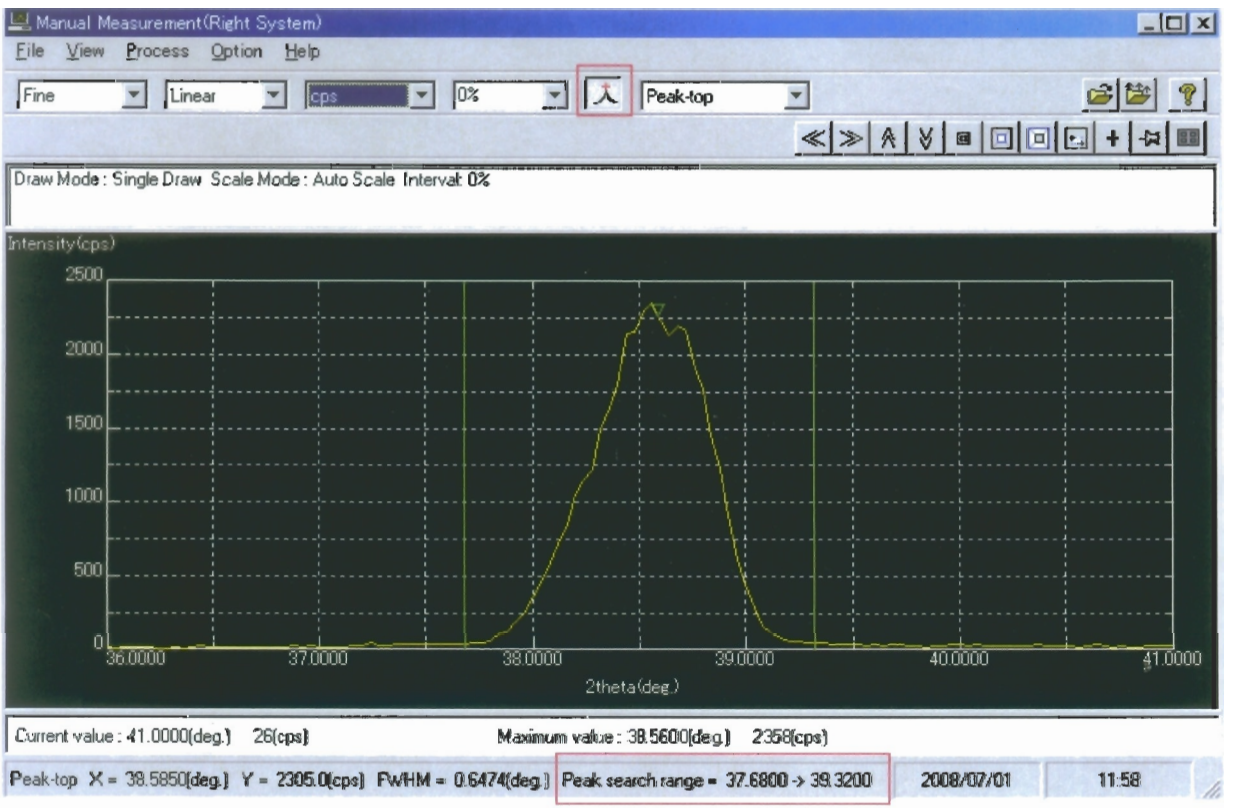



Rigaku

**B-12.** 2theta measurement is performed to decide low and high position of peak and BG range.



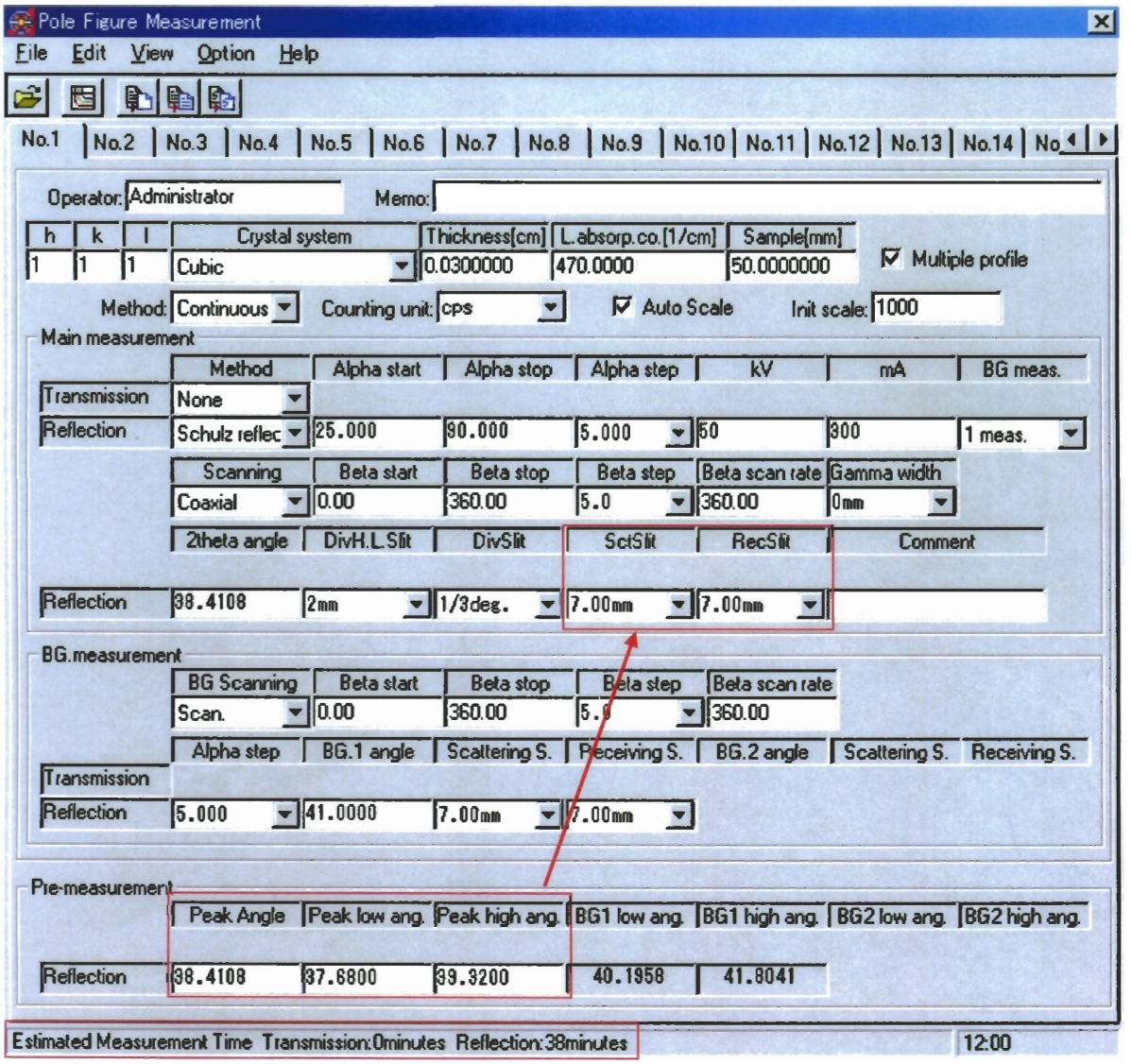

**B-13.** Press "peak search" button, left click in low position and right click in high position. The peak position is displayed on the bottom of window.






Rigaku

**B-14.** Enter "Peak Angle" measured in alpha90deg, low angle and high angle. The software selects the optimum RS automatically from registered RS value in "Rigaku Control Panel".

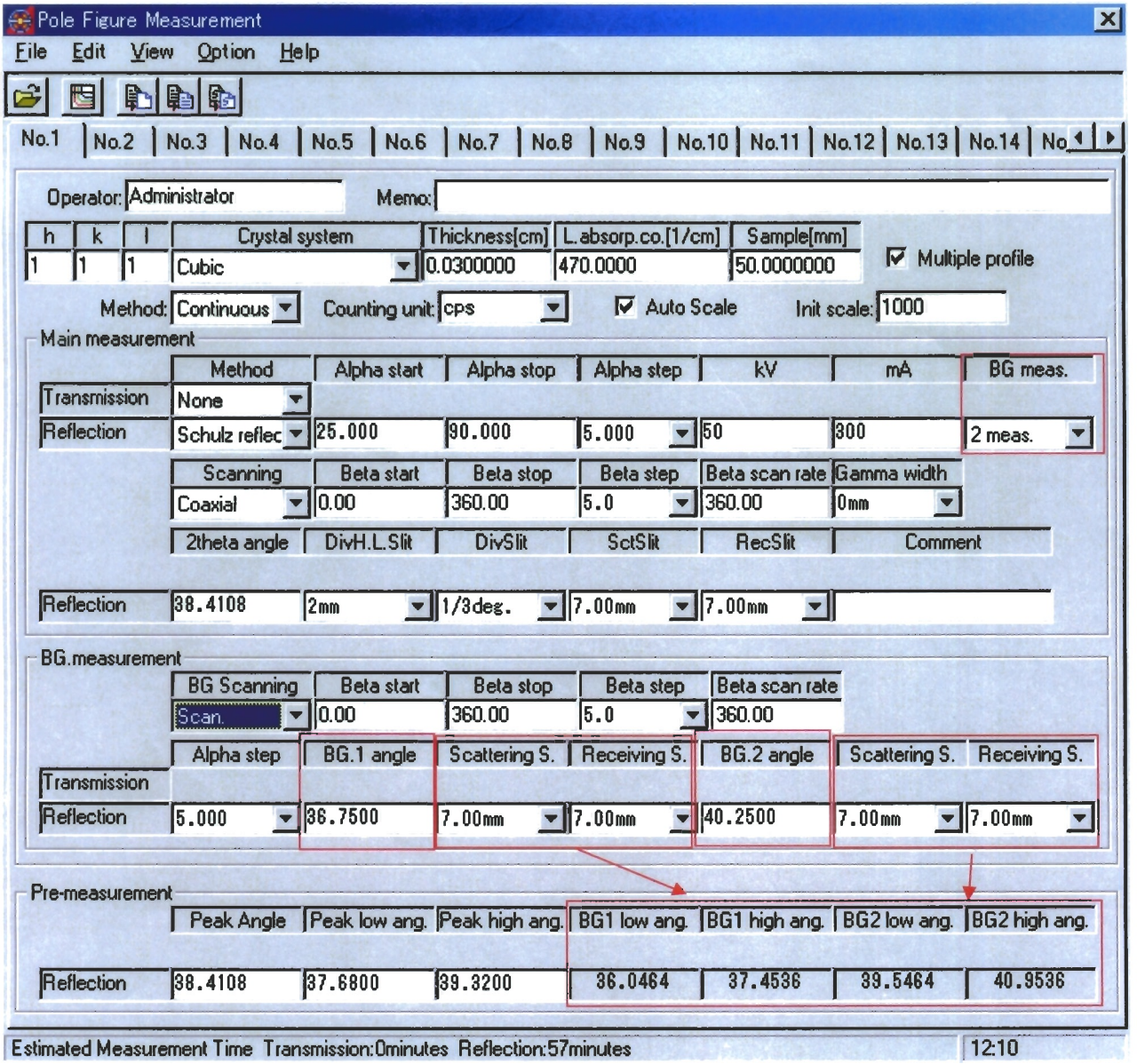


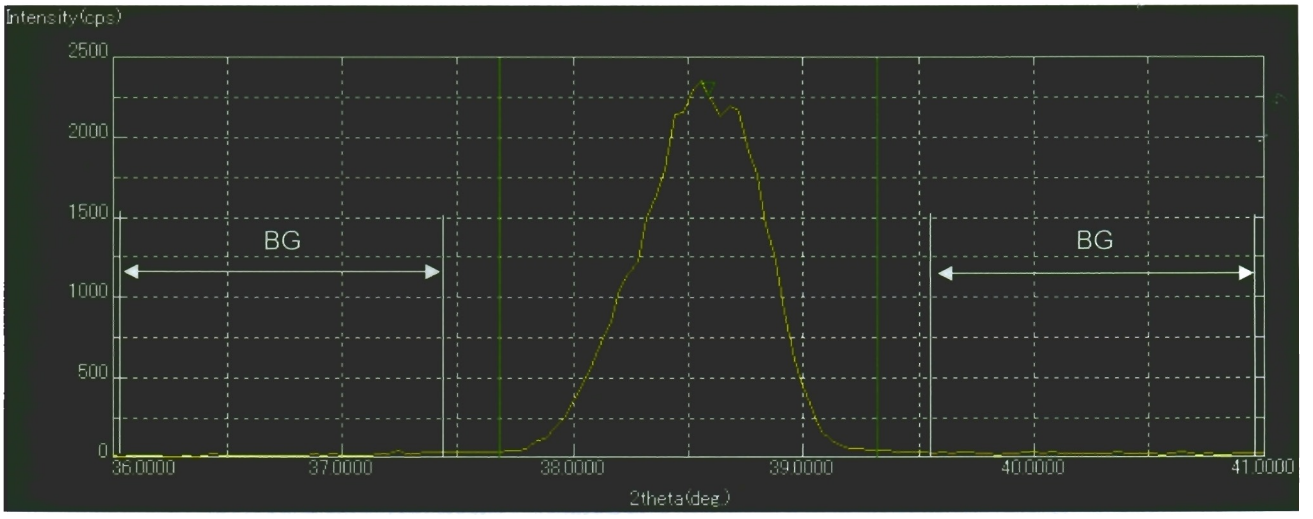

There are two types in BG measurement. [Scan] measures specified beta range. However, only beta point in which the intensity is the lowest is saved. [1point] performs BG measurement in beta point that is the lowest intensity in main measurement.

Measurement time depends on the rate of beta scan. In most cases, beta scan rate is 120 to 360 deg/min. Estimated measurement time is displayed on the bottom of window.

Gamma oscillation is used when grain size is large. Although 5,10 and 20mm can be selected on software, oscillation width depends on the size of cum equipped with MPA in practice

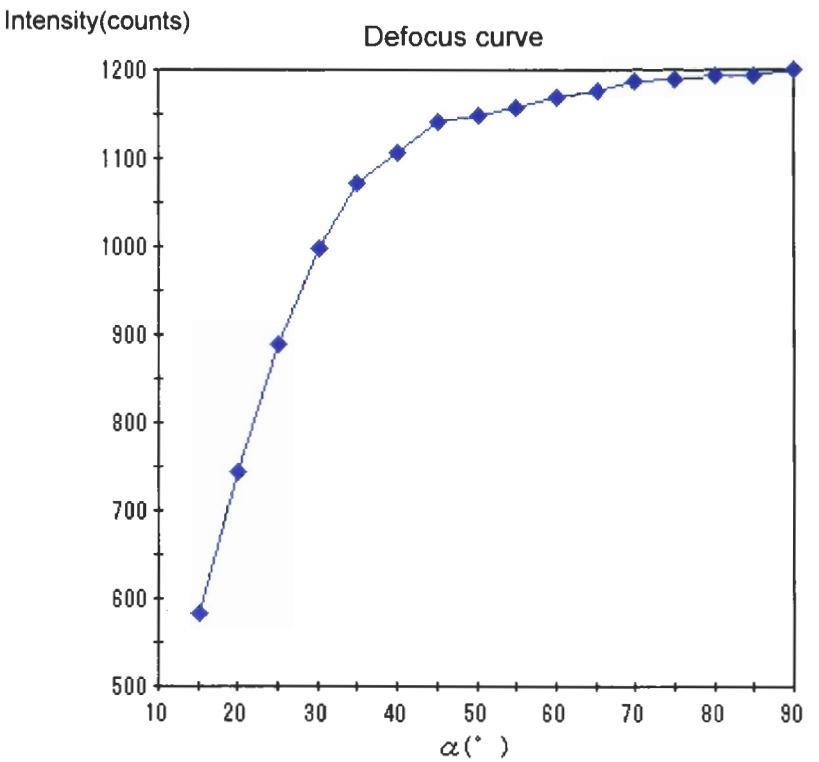
**B-15.** 1 measurement or 2 measurement can be selected in BG measurement.
Enter center BG position to be measured in BG.1 or BG.2 angle. Adjust the value not to overlap low and high of peak. In case there is close peak, change RS to smaller. Even if RS value of BG is different from RS of main measurement, software can correct the difference of intensity due to different RS.



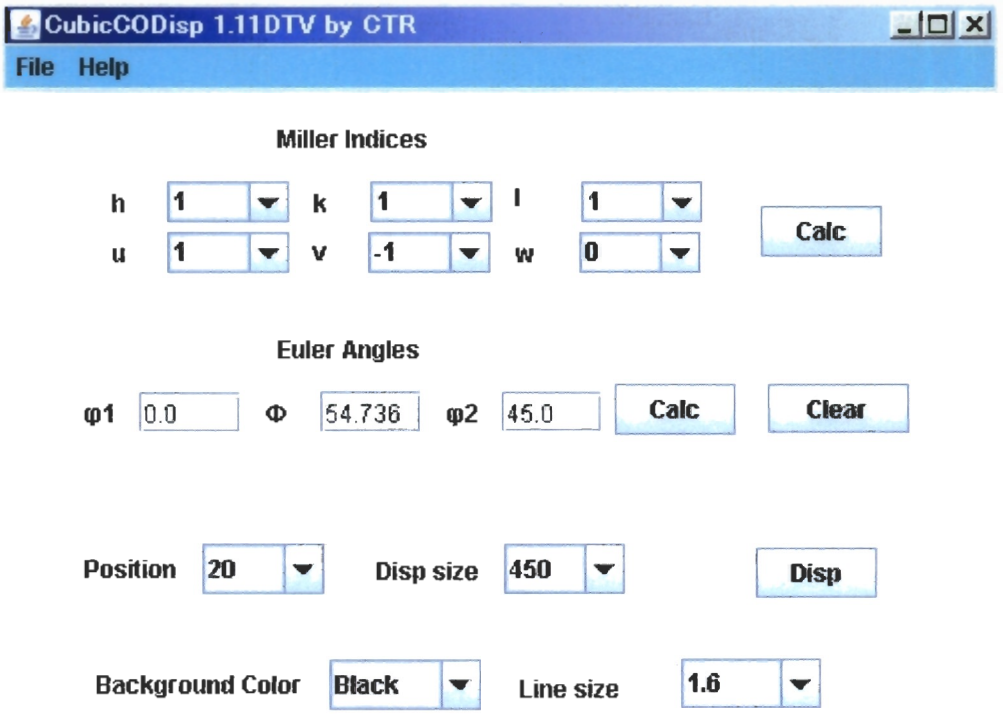



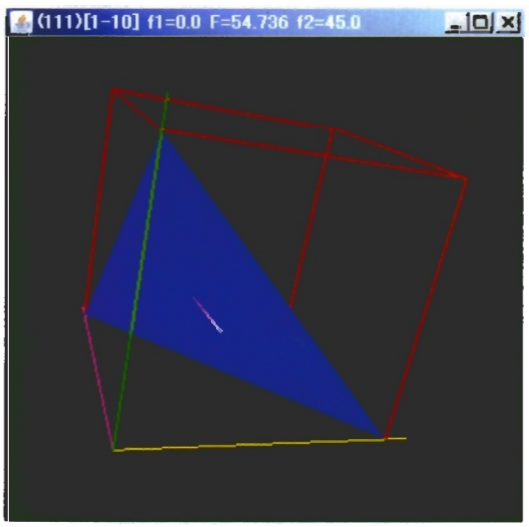
***Defocus correction**

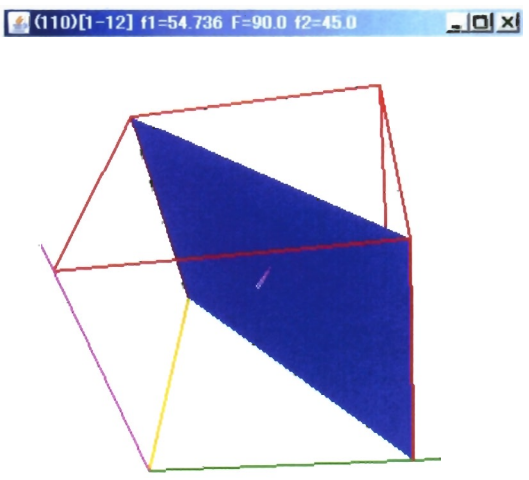
It is important to perform "defocus correction" to draw pole figure correctly in reflection method. As alpha angle is smaller, the intensity decreases due to defocus. Non-oriented bulk sample or powder sample are used for defocus correction. Basically, the shape of defocus curve depends on goniometer radius, 2theta angle and the width of RS. DS has little affect on the intensity decrease. Therefore, when measuring non-oriented sample, it need to use the same RS as real sample.



# Cubic Crystal Orientation Display program
# Cubic COD

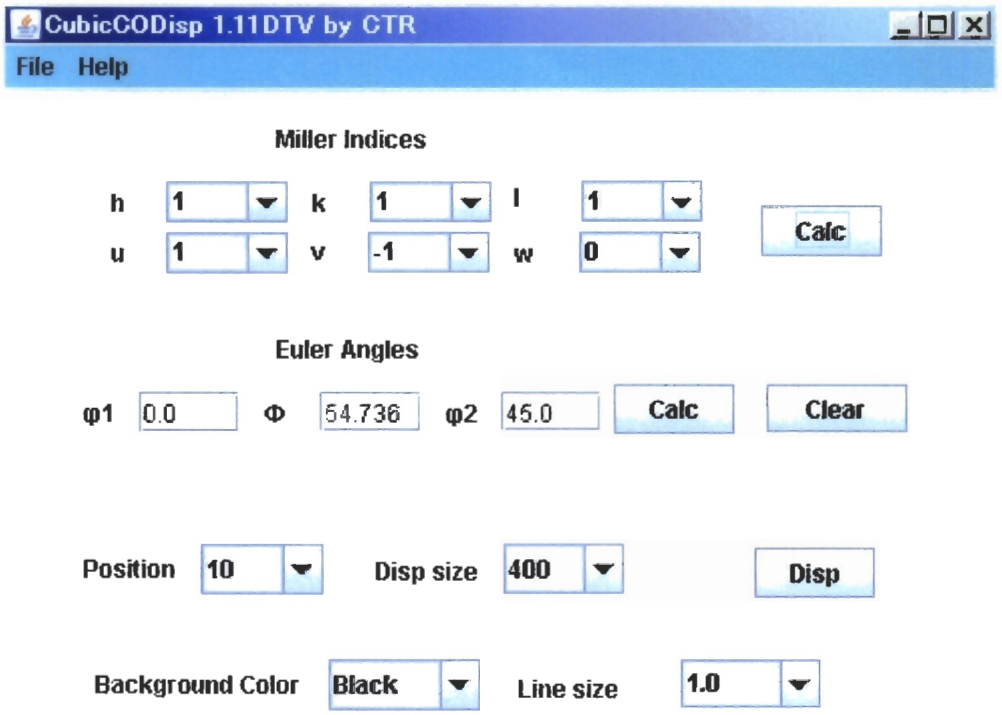


**[operation]**

How to open [Cubic COD]---
Doubleclick "CubicCODIsp.jar" in C:¥CTR¥bin

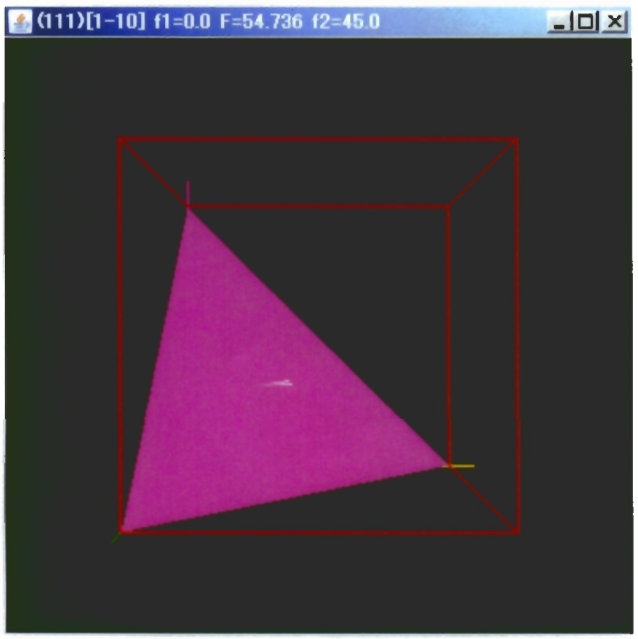



(hkl)[uvw] is selected on [Miller Indices].
Euler angle is calculated on [Euler Angles] by pushing "Calc" button.
Program does not calculate Euler angle and all values of Euler angle display "0.0" when h*u+k*v+l*w does not equal to zero.

Euler angle by Bunge type is entered in $\phi 1$, $\Phi$, $\phi 2$.
Calculated Miller Indices is displayed on [Miller Indices] with "Calc" button.
If h*u+k*v+l*w does not equal to zero, all values of Euler angle display "0.0"
All values of Euler angle display "0.0" by "Clear" button.

[Position]:not used.
[Disp size]:size of 3D orientation window.
[Background Color]: exchange of background color in 3D orientation window.
[Line size]: change of line width in 3D orientation window.
"Disp" button: 3D orientation window is displayed. Enable to display multiple windows.

**[3D orientation window]**




In above window, original point is located at back of left below.
a axis is green, b axis is orange and c axis is magenta.
Purple triangle expresses plane (111)
Direction is expressed with white arrow on purple triangle [1-10]

Movement of lattice : right drag with mouse
Rotate : left drag with mouse
Zoom-in and –out : scroll wheel drag with mouse

**[Summary]**

It is possible to specify crystal orientation from pole figure to some extent. Or, ODF analysis enables to get Euler angle that indicates the relation between material axis(ND, RD)and lattice plane. It is possible to calculate crystal orientation{hkl}<uvw> from this Euler angle.
However, it is difficult to estimate actual Plane-Direction from {hkl}<uvw>.
This program was made to understand crystal orientation quickly by 3D display.
{hkl}<uvw> means identification direction. (hkl)[uvw] is expressed as singularity expression.
This program uses singularity expression. After this, crystal orientation is expressed as (hkl)[uvw].

**[Features]**

1. Can calculate Euler angle from (hkl)[uvw].
2. Can calculate (hkl)[uvw] from Euler angle
3. Can display crystal orientation with 3D.
4. Can display (hkl) plane without Euler angle.
   Program does not calculate Euler angle when h*u+k*v+l*w does not equal to zero.
   In this case, direction is not on plane.
5. It is possible to zoom-in and -out and rotate 3D lattice.

**[Angle calculation]**

Crystal orientation is expressed as (hkl)[uvw].
Euler angle is expressed as $\phi 1$, $\Phi$, $\phi 2$.

$h=n*\sin\Phi\sin\phi 2$
$k=n*\sin\Phi\cos\phi 2$
$l=n*\cos\Phi$
$u=m(\cos\phi 1\cos\phi 2 - \sin\phi 1\sin\phi 2\cos\Phi)$
$v= m(-\cos\phi 1\sin\phi 2 - \sin\phi 1\cos\phi 2\cos\Phi)$
$w=m*\sin\phi 1\sin\Phi$
$\phi 1=\arcsin(w/\mathrm{sqrt}(u*u+v*v+w*w)*\mathrm{sqrt}(\mathrm{sqrt}(h*h+k*k+l*l)/(h*h+k*k))$
$\Phi = \arccos(1/\mathrm{sqrt}(h*h+k*k+l*l)$
$\phi 2=\arccos(k/\mathrm{sqrt}(h*h+k*k)=\arcsin(1/\mathrm{sqrt}(h*h+k*k))$
where h*u+k*v+l*w equals to zero.

**[operation environment]**

It is required that Java runtime and Java 3D are installed.