HowTo: Alignment of in – and off-plane reflexes

Alignment of in – and off-plane reflexes in 6-circle difractometer setup at P10

In case of a *symmetric* reflection, when diffraction planes of a reflection are *parallel* to the surface of a crystal, finding the reflection is quite easy. Let us consider the vertical scattering geometry when the ‘omega’ circle is used for angular scanning of the sample crystal and the ‘delta’ circle is used for positioning the detector.

1) First step is to align the crystal in the direct beam. For that both ‘omega’ and ‘delta’ should be set at 0. The detector slits should be open to a size of 5x5 mm^2 and the detector arm (‘delta’) has to be scanned (without sample in the beam) to define the detector zero-position d_0. For further finding zero-‘omega’ position, the vertical size of detector slit has to be reduced down to a size of 1 mm.

2) The sample (here we suppose Si crystal slab with <111> orientation of the surface) should be set by ‘cryoz’ (vertical sample translation) to a half-intensity position; then the rocking scan by ‘omega’ should be done and ‘omega’ position has to be corrected to the peak intensity position. Above steps (starting from setting a sample to half-intensity position) should be repeated several times with decreasing stepwidth until the exact zero-‘omega’ position (w_0) is defined.

3) Next is to set the ‘omega’-circle to the position w_0+q_b and the ‘delta’-circle to the position d_0+2q_b , where q_b is the Bragg angle for the aimed reflection [example Si(111)] at the working X-ray energy.

4) In order to find the reflection, one has to scan ‘omega’ in a reasonable angular range with a step matching the expected width of the rocking curve; normally the reflection is found after scanning ‘omega’ within ±0.1° and a step of 0.001°.

In case of an *asymmetric* reflection, when diffraction planes of a reflection are *not parallel* to the surface of a crystal, finding the reflection is a bit more elaborate than for symmetric case. The first step is to find out whether the desired reflection is possible for the actual surface orientation and applied photon energy. As it is illustrated in Figure 1, the reflection will be possible to measure (in reflection geometry) if its Bragg angle is larger than the angle j between the diffraction planes and the surface (otherwise the reflection can be measured only in Laue (transmission) geometry).

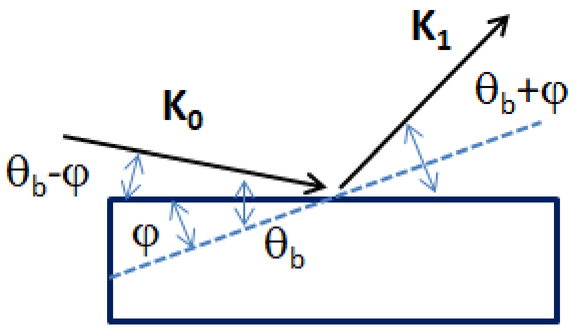


Figure 1: Example of lattice planes.

For a cubic lattice the angle j between two planes having Miller indexes $h_1k_1l_1$ and $h_2k_2l_2$ is defined as:

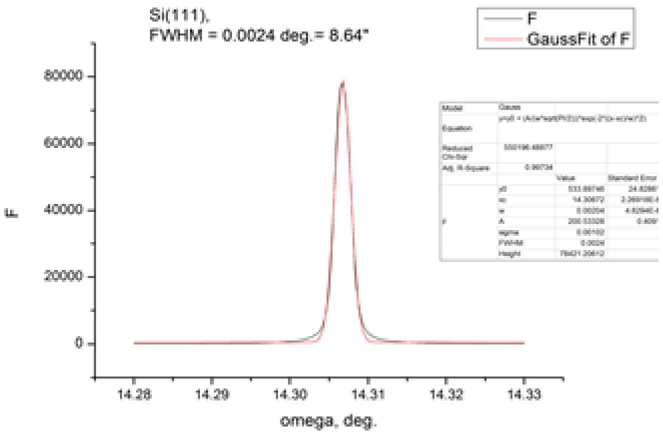
$\cos(j)= (h_1*h_2 + k_1*k_2 + l_1*l_2)/(sqrt(h_{1**}2 + k_1**2 + l_1**2)*sqrt(h_2**2 + k_2**2+ l_2**2))$

For example, for the Si(311) reflection and a <111> crystal surface orientation we have j=29.5°. For X-ray energy of 8 keV the Bragg angle of Si(311) reflection is q_b = 28.2°, which means that this reflection is not accessible in Bragg (reflection) geometry at this X-ray energy.

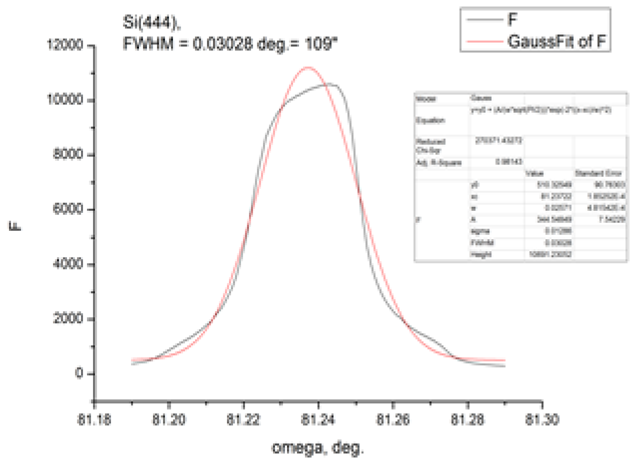
After the measurable reflection is selected, the steps 1) and 2) should be applied. As before, in step 3) the detector arm has to be places at d_0+2q_b. But the ‘omega’ circle should be positioned either at q_b-j or at q_b+j (see Figure 1). The case of incident angle equal to q_b-j is favourable since the intrinsic width is becoming larger due to asymmetry factor; and hence the reflection will be easier to find. After the ‘delta’ and ‘omega’ circles are brought to their positions, one has to start azimuthal scans of the sample using the ‘phi’ circle. Scanning of ‘phi’ motor should be performed in a wide angular range of 180° with reasonably small step of 0.01°. After the reflection is found, one can measure its rocking curve by scanning ‘omega’ as in step 4).

Example: Si(111) & Si(444) reflexes

As an example, the rocking curve of Si(111) and Si(444) at E=8keV are displayed.



Si(111),
FWHM = 0.0024 deg.= 8.64"
F
GaussFit of F
80000
60000
40000
20000
0
F
14.28
14.29
14.30
14.31
14.32
14.33
omega, deg.



Si(444),
FWHM = 0.03028 deg.= 109"
F
GaussFit of F
12000
10000
8000
6000
4000
2000
0
F
81.18
81.20
81.22
81.24
81.26
81.28
81.30
omega, deg.