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X-ray diffraction on Si single crystal with a W-shaped longitudinal groove

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The measurement of the sagittal deviation of an X-ray beam diffracted on the inclined surface of an Si(111) single crystal was performed on beamline BM5 at the ESRF, with λ = 0.1 nm and an inclination angle, β, of 70°. The measured value agrees with the theory developed in previous papers. The topographic picture of the longitudinal edge shows a structure that can be explained in terms of the properties of inclined diffraction.

Keywords: X-ray optics; X-ray crystal monochromators; inclined diffraction.

1. Introduction

In previous papers (Hrdý, 1998; Hrdý & Siddons, 1999) it has been shown that sagittal focusing of an X-ray beam may be achieved by machining a longitudinal groove with a controlled cross-sectional profile into the surface of a single-crystal monochromator. Furthermore, it has been shown recently (Hrdý & Hrdá, 2000) that meridional focusing may be obtained in a similar way, using a transversal groove. These phenomena are connected with X-ray refraction. The theory of this kind of sagittal focusing is based on an approximate formula for inclined diffraction (Hrdý & Pacherová, 1993; Hrdý, 1998) describing the dependence of the sagittal deviation δ of a diffracted beam from a diffraction plane (determined by an incident beam and a normal to the diffracting crystallographic planes) on the angle of inclination β,

$$\delta = K \tan \beta. \tag{1}$$

Here $K = (2r_eF_0/\pi V)\lambda d$, where r_e is the classical electron radius, V is the volume of the unit cell, F_0 is the structure factor for 'zero' reflection, d is the net plane spacing and λ is the wavelength of the diffracted radiation. For silicon, $K = 1.256 \times 10^{-3}\lambda d$ (nm^2). For the sake of clarity, the sagittal deviation is the deviation in the direction perpendicular to the diffraction plane. The situation for monochromatic radiation (Hrdý & Pacherová, 1993; Hrdý, 1998) is depicted in Fig. 1. The impinging boundary beams 1 and 2 determine the accepted angular region ω_s, the centre of which is deviated from a Bragg angle θ_B by an angle $\Delta\theta_s$ (see, for example, Matsushita & Hashizume, 1983). The diffracted beams 1 and 2 are deviated such that the centre of the diffracted angular region (the sagittal projection of which is ω_δ) is deviated from the diffraction plane by the angle δ. The variation of the sagittal deviation along the reflection profile is proportional to the departure from the Bragg angle; then the reflected beams are coplanar. Although the experimental results of Hrdý & Siddons (1999) support the correctness of (1), the direct experimental verification of this formula has not been performed yet. [The ray-tracing program *SHADOW* (Lai & Cerrina, 1986) also calculates δ but it gives values that are about 30% higher than those obtained from (1)]. To test the degree of validity of the approximation used in (1), we performed the precise calculation based on the dynamical theory of diffraction taking into account the real shape of the dispersion surface. Furthermore, we performed the direct measurement of δ on beamline BM5 at the ESRF, with β = 70°, λ = 0.1 nm and an Si(111) crystal.

As suggested by Hrdý (1998), a possible method to determine δ is to measure the splitting of the beam diffracted on an edge created by two inclined crystal surfaces oriented in opposite ways. [In fact, such a splitting has been observed by Hrdý *et al.* (1998)]. In practice, such a crystal was produced by cutting two parallel longitudinal grooves into a (111) diffraction surface, creating a W-shaped profile with the edge at its centre (Fig. 2). Both lateral parts, *i.e.* two 'V'-shaped grooves, create slightly convergent beams because the beams diffracted on both walls of the groove are sagittaly deviated in opposite directions. (This device to create slightly convergent beams is compact and very simple. A large change of inclination angle results in only a small change of beam convergence. Our original idea was to generate interference fringes in this way using a crystal with small β. Unfortunately, the

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experimental conditions did not allow us to realize this goal; nevertheless, we observed some other interesting phenomena, as described below.)

2. Determination of δ

The geometry of the Si(111) single-crystal sample with the W-shaped longitudinal groove is shown in Fig. 2. For the experiment we chose the position of the crystal such that the diffracting (111) planes were vertical and the edge of the groove was horizontal. This reduces the smearing of the splitting on the edge because the vertical dimension of the source is smaller than the horizontal one. We also chose the vertical divergence of the beam such that the beam slightly overfilled the groove. The divergence in the perpendicular (horizontal) direction was delimited such that the horizontal dimension of the beam at the sample was 1 mm. The diffracted beam was detected on high-resolution X-ray Kodak film at a distance of 2 m from the sample. Without any refraction effect, the deviation δ would be zero and the cross section of the diffracted beam would follow the W shape of the groove. In the real case ($\delta \neq 0$), however, we should observe a splitting in the central part of the picture, corresponding to the diffraction on the central edge, and a half splitting on the upper and lower parts of the picture, corresponding to the diffraction on the edge between the inclined and symmetrical part of the crystal. As follows from Fig. 1, it is not possible to see directly the splitting 2δ (at the central part of the picture) because of the finite value of ω_δ, which is comparable with δ. However, it should obviously be possible to see the gap $w = 2\delta - \omega_\delta$ (visible gap). To maximize the splitting, we used a relatively high value of β. As the tip of the edge is not perfect, we should add to the above-estimated splitting a value t equal to the width of this imperfect part, which in our case was about 10 (15) µm. Finally, we should take into account smearing caused by the size of the radiation source. Practically, we should subtract the size of the demagnified source s_d. Then the observed splitting at a distance l from the crystal edge is

$$w = l(2\delta - \omega_\delta) + t - s_d. \quad (2)$$

The width of the diffraction region ω_δ may be calculated from the relation which follows from the work of Hrdý (1998), as well as from Fig. 1,

$$\omega_\delta/\delta = \omega_s/\Delta\theta_s. \quad (3)$$

Here $\Delta\theta_s$ is the deviation of the centre of the single-crystal diffraction pattern (Darwin–Prins curve or crystal function) from the Bragg angle and ω_s is the width of the single-crystal diffraction pattern.

The measurement was performed on beamline BM5 at the ESRF. To take advantage of the small vertical size of the source (80 µm), we placed the crystal sample such that the diffracting (111) planes were vertical, as mentioned above. The radiation was monochromatized by a channel-cut Si(111) crystal, oriented in the same way as the sample and placed before the sample. The beam diffracted by the sample was registered at distances of 0.2, 0.5, 1.0 and 2.0 m. The wavelength used was 0.1 nm. The measured inclination angle of the W sample was β = 70.25°.

The value of δ calculated from equation (1) for our experimental arrangement is 1.096×10^{-4}. The value of ω_δ/δ from (3) is 1.066 ($\omega_s = 1.44 \times 10^{-5}$ and $\Delta\theta_s = 1.351 \times 10^{-5}$) and thus the theoretical value of ω_δ is 1.168×10^{-4}. As was mentioned above, in our case $t \simeq$ 10 µm. The smearing as a result of the vertical size of the source, at a distance of 2 m from the sample, is 80 µm × (2 m/40 m) = 4 µm, which is negligible. Substituting these values into (2), for the distance l = 2000 mm we obtain the expected value of the splitting w = 0.2143 mm (visible gap). This value is indicated in the photograph (blue lines) taken at a distance of 2 m (Fig. 3), which shows that the calculated and measured gaps coincide fairly well. The measured value of w is 0.21 ± 0.04 mm, which gives an experimental value $\delta = (1.07 \pm 0.2) \times 10^{-4}$.

Expression (1) was derived on the basis of dynamical theory using a simplified shape of the dispersion surfaces. Together with the values calculated from (1), Fig. 4 presents values of δ calculated using the correct shape of the

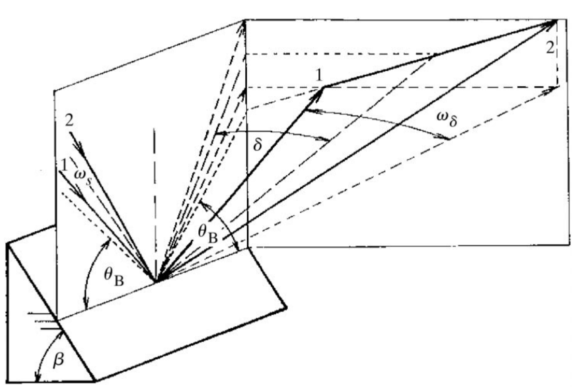

Figure 1
The geometry of inclined diffraction. Beams 1 and 2 are the boundary beams delimiting the diffraction region of the accepted and diffracted monochromatic beam. The variation of the sagittal deviation of the reflected beam along the reflection profile is proportional to the departure from the Bragg angle.

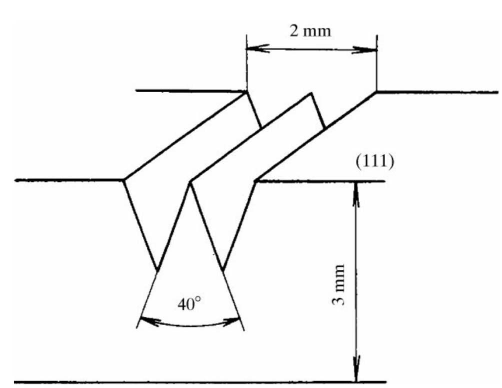

Figure 2
The geometry of the W-shaped sample.

dispersion surfaces, for λ = 0.1 nm and diffraction on Si(111) planes. Our experimental value is also included. From this picture it is seen that (1) gives reliable values of δ in the region $\beta < 85°$ and that at least for $\beta = 70°$ the value agrees well with the experimental result. On the other hand, the existence of ω_δ introduces some uncertainty in the determination of δ because the splitting is not sharp enough; furthermore, the distance l could not, in this experiment, be longer than 2 m. It is obvious that a more precise measurement is needed. As follows from the work of Hrdý & Siddons (1999), the sharpness of the splitting should be substantially increased by using two or four grooved crystals in the dispersive (−,+,+,−) arrangement. In this case, the horizontal broadening of the diffracted beam as a result of ω_δ should be cancelled and the value of the splitting (visible gap) should be $4\delta l$ in the case of two edges in dispersive position or $8\delta l$ in the case of edges on all four diffracting surfaces.

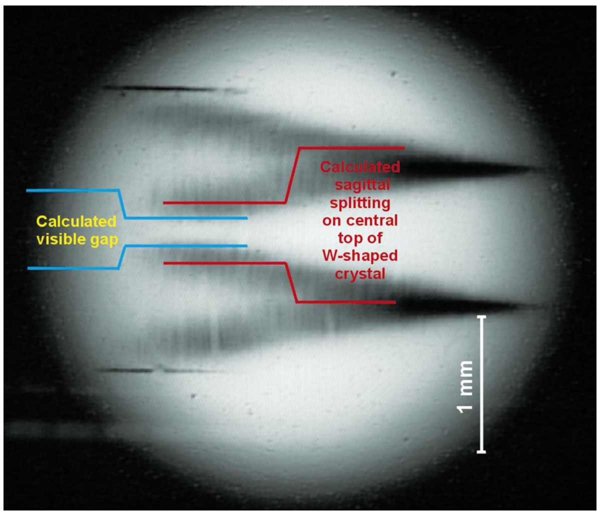

Figure 3
Photographic representation of the diffracted radiation on the W-shaped Si(111) sample, showing the splitting of the diffracted radiation (12.4 keV) on the central edge of the groove (two inclined surfaces) and also on both the upper and the lower edges (inclined and symmetrical surfaces). The blue lines show the theoretical value of the visible gap, w [$w = l(2\delta - \omega_\delta) + t - s$] using the theoretical value of δ, whereas the red lines show the hypothetical sagittal splitting $2\delta l$ (provided that $\omega_\delta = 0$) using the theoretical value of δ.

3. Topography

The topographic picture (Fig. 3) consists of many almost vertical lines, creating a 'Christmas tree'-like structure, which may be explained in terms of the inclined diffraction properties depicted in Fig. 1. As can be seen in Fig. 1, the monochromatic diffracted beam from a certain point on the inclined surface is imaged on the X-ray film as a tilted line. The angular width ω_δ of this line is, in our experiment, about 5.5 times larger than would be found for the single-crystal diffraction pattern (ω_s) in the case of symmetrical (not inclined) diffraction. Thus the almost vertical lines in the left part of Fig. 3 are the images of the well diffracting micro-regions close to the edge. This is better seen in Fig. 5, which shows a magnification of the left central part of Fig. 3. These lines are, at the same time, the images of the magnified crystal function resulting from the inclined diffraction. It is in agreement with the position of the red lines in Fig. 3, which show the positions of the beams diffracted at the points close to the edge and deviated by the angle δ. Thus, this topographic picture obviously supports the model shown in Fig. 1 (note that the

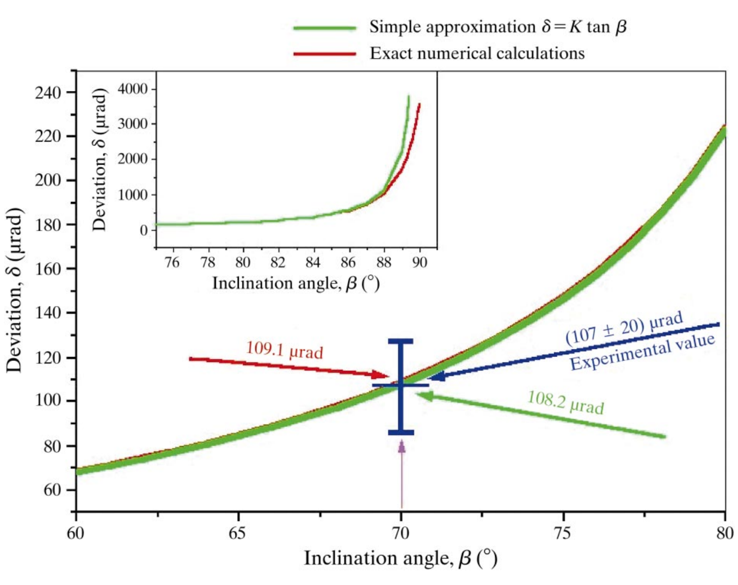

Figure 4
The dependence of δ on the inclination angle β for the Si(111) crystal and λ = 0.1 nm. The calculated curve taking into account the correct shape of the dispersion surfaces and Ewald spheres is shown in red. The curve calculated according to (1) is shown in green. The experimental value is indicated in blue.

diffracting planes in Fig. 1 are horizontal whereas in the experiment they are vertical). It is interesting to note that the neighbouring points on the crystal along the edge create (almost) vertical lines which *do not* overlap (they are arranged parallel). On the other hand, the same points in the case of symmetrical diffraction would create horizontal lines which would overlap. This means that a structure like that seen in the left part of Fig. 3 would represent (for monochromatic radiation) a topographic image of the edge region (or corresponding part on crystals located upstream), which would have higher resolution than is reachable with a symmetrically cut crystal. On the other hand, it is obvious that for neighbouring points arranged perpendicularly to the edge, the resolution should be much worse because of the large ω_δ. This should be kept in mind when recording topographic pictures of crystals utilizing diffraction planes that are not parallel to the surface.

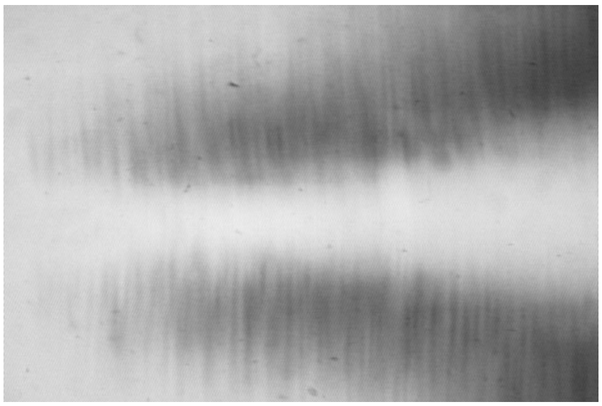

Figure 5
Magnification of the left part of Fig. 3 showing the splitting on the central edge.

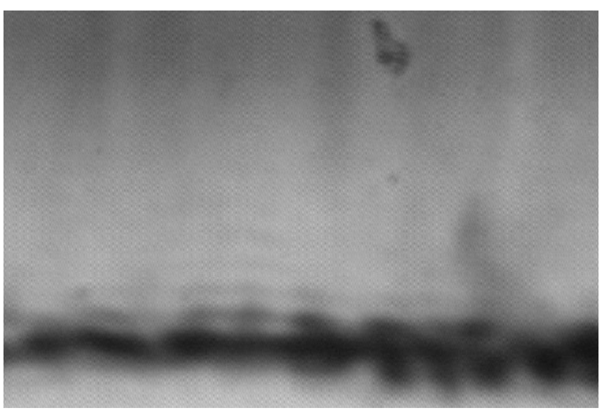

Figure 6
Magnification of the lower part of Fig. 3 showing the diffraction on the edge between the inclined and the symmetrical part of the crystal surface.

To check that the 'Christmas tree'-like structure shown in Fig. 3 actually exists, we had the same picture recorded on beamline ID19. Similar results were obtained. In Figs. 3 and 5 it is possible to see the images of both the symmetrical and the inclined part of the crystal. The images of the symmetrical part of the crystal (thick horizontal lines on both sides of the picture, which may only be the result of overexposure) show no structure, while the image of the inclined part does.

Fig. 6 is a magnification of the lower part of Fig. 3, showing the diffraction on the edge between the symmetrical and the inclined part of the crystal. It is possible to see interference patterns along the edge. The most probable explanation is that these fringes arise from the interference on the edge caused by the secondary slit located close before the sample which delimited the vertical divergence of the radiation. A more precise explanation would require an additional experiment.

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