One-dimensionally curved Si and Ge single crystal wafers prepared by hot-pressing: potential performance for optical components for X-ray diffraction

Hiroshi Okuda, Shojiro Ochiai, Kozo Fujiwara* and Kazuo Nakajima*

International Innovation Center, Kyoto University Kyoto 606-8501, Japan
*Institute for Materials Research, Tohoku University, Sendai 980-8577 Japan.

okuda@iic.kyoto-u.ac.jp

Abstract. By using hot pressing, plastically bent Si and Ge wafers in cylindrical shape with keeping mirror surfaces have been obtained. In the present paper, the experimental results obtained by diffractometer with channel-cut monochromater have been discussed from the viewpoint of requirements necessary for curved monochromaters for X-ray optics. It was made clear that by choosing appropriate conditions, the present method provides cylindrical crystals for X-ray monochromaters for practical use.

1. Introduction
Bending semiconducting single crystals to improve efficiency of monochromating X-ray devices has long tradition beginning from Johann[1] and Johansson[2] in the 1930s. Since these initial trials, many attempts have been made to realize both efficient and precise focusing monochromator. However, these two aims, i.e., gathering X-ray flux as efficient as possible, and maintaining precise angular resolution, are generally not compatible. Therefore, elastically bent semiconducting crystals have been used to obtain monochromaters with good angular resolution at a cost of less solid angle to collect flux[3,4]. For better efficiency, bent crystals with mosaicity and therefore having rocking curve profiles with large full-width at half-maximum (FWHM) have been used.

In the present work, we examined semiconductor wafers plastically deformed into cylinders with a designed radius of curvature of 50 mm. Potential performance of the present crystals is discussed based on rocking curve measurements.

2. Experimental
2.1. Sample preparation The samples were prepared by hot pressing of single crystalline semiconductor wafers. The condition of the deformation was described in ref. [5-7]. After the deformation in Ar, the samples were evaluated by X-ray diffraction with Cu-Kα1 radiation. Si and Ge (111) wafers with 38 mm in diameter were deformed into a cylindrical shape with a designed radius of 50 mm. These wafers maintained mirror surfaces after the deformation.

2.2. X-ray measurements Samples plastically deformed in cylindrical shape with a radius of 50 mm have been examined by rocking scans on a diffractometer with an incident Ge 220 channel-cut monochromater. Experimental setup is schematically shown in Fig.1. The size of the incident beam was 0.05 x 0.5 mm². Since the shape of the crystal strongly deviates from flat plate which is normally...
expected for diffraction measurements, we fully opened the receiving slits and measured the peak position of the samples by rocking scans, with a well-defined incident beam and as a function of horizontal distance from the center of the sample, x, which was controlled by horizontal movement of the sample holder. As schematically shown in the figure, the diffraction peak position moves with the distance, x, according to the curvature of the sample. However, since the sample position is not placed at the rotation center of the goniometer when the sample is not at x=0, correction on the rotation, α, is necessary due to off-centered rotation of the sample shown as h in the figure. The correction is given by:

$$\Delta \omega = \tan^{-1}(x/R) + \alpha$$

with

$$\alpha = \tan^{-1}\left(\frac{\cos^2 \theta_0 (R^2 \tan^2 \theta_0 + x^2 (1 + \tan^2 \theta_0))^{1/2} - R \tan \theta_0)}{(R^2 + x^2)^{1/2}}\right)$$

where $\theta_0$ is the Bragg angle of 333 diffraction, and R is the radius of curvature of the deformed wafer. The rocking curves for 333 diffraction as a function of the position of the sample, x, were measured at the center, along the curvature of the sample. No peak shift was observed in the direction perpendicular to the curvature in the present measurements.

3. Results and discussions

Figure 2 shows the measured peak shift as a function of expected peak shift obtained for Si (111) bent crystal. The expected peak shift corresponds to the amount of peak shift for the rocking scan with the correction described in equation (2) when the radius of curvature of the crystal is 50 mm. Therefore, it corresponds to the accuracy that the (111) lattice planes are bent at the designed curvature. As shown in the figure, a good linear relationship was observed for 30 degrees with the standard deviation of 0.132 degree. The slope is slightly smaller than unity, meaning that the real radius is about 2% larger than the designed radius of 50mm. Therefore, Fig. 2 suggests that the cylindrically deformed Si (111)
crystal used in the present measurements has been uniformly bent with an accuracy of 0.132 degree as standard deviation. However, it does not certify the uniformity in the crystal quality. Figure 3 gives distribution of FWHM of the rocking scan and that of integrated intensity as a function of x. The FWHM is not constant, suggesting that the quality of the crystal is not uniform, in other words, the deformation microstructure is not the same. The lower limit of the FWHM is about 0.1 degree, and the maximum is about 0.3 degree. The origin of such large FWHM may be explained either by a localized deformation with high dislocation density or polygonization. If plastic deformation is localized in a small region, large FWHM and small integrated reflectivity should be observed. However, Fig. 3 implies that large FWHM does not correspond to low integrated reflectivity. Therefore, the origin of scattered values of FWHM is attributed to inhomogeneous polygonization of the sample. Figure 4 gives an example of a rocking scan which is well fitted by multiple Gaussian approximation. The width of each Gaussian is about the same as the uniformly deformed hemispherical Si.

In order to compare the result with the deformation of softer crystal, the curvature of the cylindrically bent Ge (111) crystal is also evaluated by the rocking scan. Figure 5 gives the curvature of (111) plane in the cylindrically bent Ge crystal plastically deformed into R=50 mm. The least-square fit of the peak position suggests that the accuracy of the curvature of (111) plane is 0.133 degree as standard deviation, which is almost the same as that for the Si crystal shown above. The slope of the least square fit was 0.96, i.e., about 4% larger radius than the designed one. To compare the deformation microstructure of Ge with that of Si, the distributions of FWHM and the integrated intensity for Ge crystal are shown in Fig. 6. The distribution given in the figure is different from that given in Fig. 3, in that both the FWHM and the integrated intensities are more uniform. For example, the FWHMs of the rocking curves lie between 0.09 and 0.12 degrees, whereas those for Si crystal lie between 0.1 and 0.36 degrees. The integrated intensity is more uniform as shown in Fig 6. Gradual change in the integrated intensity may rather be explained in terms of small misalignment of the direction of surface normal from the scattering plane, resulting from small twist of the crystal due to residual strain.
Figure 7 is an example of rocking curve measured for the Ge (111) crystal. The FWHM of the peak is about 0.11 degree. Considering that the FWHM of each Gaussian fit in the multiple Gaussian fit given in Fig.4 is about 0.1 degrees, we may conclude that the deformation microstructure in the present Ge crystal is characterized by a uniform distribution of dislocations, with the local crystal quality similar to that of the Si sample, but without existence of polygonization at the length scale used in the present measurements. When we consider the application of these curved monochromating crystals to a focusing optics, the microstructural difference whether the crystal has a well-developed (coarse) polygonization or uniformly dispersed structure becomes quite important, since well-defined focusing optics need to use small source size, leading to more strict diffraction conditions. In this viewpoint, development of coarse polygonization is harmful for focusing optics when the characteristic size of the region becomes comparable to the source size of a generator. The origin of polygonization observed in the present sample is not conclusive yet. However, we may point out two possibilities. One is a formation of the microstructure during recovery of once-formed dense dislocations. The other is related to a formation of slip bands during bending. For example, if the Schmidt factor is favorable for some specific slip systems during plastic deformation, dislocation motion on such systems may become dominant to form visible slip bands. These phenomena are often observed in the tensile test of annealed pure metals or single-crystalline salts. If the latter happens, the direction of surface normal, \( \mathbf{n} \), and the direction of reciprocal lattice disagree after deformation due to crystal rotation. In the present measurements, however, no clear disagreement between the radius of curvature obtained from optical (surface) reflection and that obtained from diffraction was observed. Therefore, we may conclude that the splitting rocking curve observed for Si (111) crystal should be due to rearrangement of dislocation during deformation at high temperature.

We have shown that both Si (111) wafers and Ge (111) wafers deformed into cylindrical shape with a designed radius of 50 mm gave a cylindrical (111) plane within an accuracy of 0.13 degree of standard deviation for a large angle of more than 30 degrees as shown in Figs.2 and 5. However, when we examine more in detail, the Si crystal had a microstructure with polygonization.
When we apply these crystals to focusing monochromater, there are several important points to consider provided that the accuracy of curvature on deformation is enough.

One is the balance between the accuracy of lattice curvature, $\sigma_0$ and the FWHM of the diffraction, $\Delta$, that $\sigma_0 < \Delta$. For the present crystals, Ge crystal does not satisfies the condition, suggesting that some part of the crystal does not satisfy the diffraction condition when the crystal is used for focusing optics without further modifications.

The other point is that we need to figure out the critical condition that determine the size of focused beam. Figure 8 gives a model calculation of focus size for Cu-K\(\alpha\) radiation and for 333 diffraction. The results suggest that for Cu radiation with 333 diffraction, the broadening of the incident beam due to penetration of X-ray into the monochromating crystal is relatively small even for Si, although it is unacceptably large for Mo K\(\alpha\) radiation, about 0.6 mm. Therefore, for Cu K\(\alpha\) radiation, it turned out that the focusing error may be governed by the accuracy of lattice plane curvature when the radius of curvature is large. The triangle shown as Johann error in the figure is calculated for the deviation if all the Johann crystal can satisfy diffraction condition. However, this is not practical for small R, since the FWHM of the diffraction can not reach such large angles. Therefore, we conclude that the most important point to realize curved crystal for focusing crystal is to control the deviation of lattice plane curvature as small as possible, with FWHM of the rocking curve larger than the deviation. Fig.8 tells us that to obtain reasonable size, e.g., 0.2 mm, of focus at moderate camera length, we need to control the deviation of curvature of the lattice plane within about 0.1 degrees. Present deviation of about 0.13 degree implies that the quality of the crystal is now approaching to a practical level if we avoid polygonization. For the radius of curvature used in the present deformation, 50 mm, Figure 8 shows that the focusing accuracy obtained from $\sigma_0 = 0.1$ degree and penetration error for Si give several tens microns, which is comparable to conventional microfocus X-ray generators. Considering the fact that it is possible to broaden FWHM during further polishing to prepare Johann crystal by controlling surface damage layer, present cylindrical crystal has sufficient quality to be used for focusing monochromating crystals. Further refinement of the deformation conditions and preparation of Johannsson crystal is now under way.

4. Conclusions

Si and Ge (111) crystals plastically deformed into cylinder with R=50 mm have been examined by X-ray diffraction. In spite of the same accuracy in lattice-plane curvature, quite different deformation microstructures were suggested. By assessing focusing errors, it is concluded that the present Si and Ge crystals can be used for focusing monochromaters, although the microstructure of Si with polygonization is not ideal. For larger camera length, i.e., larger radius, calculation suggests that the most important factor to maintain well-focused diffraction is the accuracy of lattice plane curvature. Present accuracy of lattice plane curvature, about 0.13 degrees, corresponds to focusing size of 0.2
mm up to about 120 mm of R. With present microstructures, curved crystals prepared by the present method is found to best fit for very compact focusing monochromaters.

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