Bent crystals by surface grooving method for high-efficiency concentration of hard x-ray photons by a Laue lens

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ABSTRACT

We present an experimental study on the method of surface grooving for bending crystals for the realization of a hard x-ray Laue lens. Bent Si and Ge crystalline plates were analyzed by x-ray diffraction of their (111) planes at the European Synchrotron Radiation Facility. Crystals diffracted photons from 150 to 700 keV with efficiency peaking 95% at 150 keV for Si. Measured rocking curves of the samples showed flat-topped profiles with their FWHM equal to the crystal bending, i.e., the method of surface grooving proved to evenly bend the crystals, their energy passband being very well controlled. Surface grooving technique has been found to offer both high reproducibility and easy control of diffraction properties of the crystals. Besides, this method is cheap, simple and compatible with mass production, making it a reliable technique for fabrication of a Laue lens, where serial production of crystals should be envisaged. A Laue lens made of crystals bent by surface grooves can lead to significant detection improvement in astrophysical applications.

Keywords: bent crystals, Laue lens, x-ray diffraction

1. INTRODUCTION

Nowadays the study of x and soft gamma-ray bands represents a challenge for the astrophysics scientific community because of the wealth of celestial sources emitting within these domains. The lack of focusing optics at photon energies higher than 80 keV calls forward the usage of coded masks or quantum optics. Since collection area is smaller than or equal to the detection area, it results in low signal-to-noise ratio because the background is roughly proportional to the volume of the detector. A space-borne Laue lens telescope would allow a sensitivity leap by more than an order of magnitude with respect to existing telescopes within the hard x-ray energy band. Bragg diffraction (in Laue geometry) through a large number of accurately oriented crystals is used to deviate and concentrate radiation from cosmic sources towards a common focus. Crystals for a Laue lens have to fulfill several requirements, i.e., their passband must be adjustable and yield the highest reflectivity.

There are two major classes of crystals satisfying these requirements. Mosaic crystals have been firstly considered and their fabrication methodology has been highly perfected over the years. However, they exhibit a non-uniform (typically Gaussian) passband and diffraction efficiency is limited to 50%. A second method consists in curved crystals, for which such two limitations do not hold true. Indeed, due to continuous change of the incidence angle of an x-ray trajectory with the crystalline planes, re-diffraction within the crystal is prevented and the 50%-limit overcome. Moreover, a uniform distribution of the energy passband is accomplished for an evenly curved crystal and its width is strictly bound up to its curvature.

For realization of a curved crystal several methods have been developed. Reproducible deformation of (111) self-standing Si and Ge crystals was recently achieved and characterized at Sensor and Semiconductor Laboratory (Ferrara, Italy). Curved crystals were achieved by bending through mechanical grooving of one of their largest surfaces. Curvature held permanently and was adjustable by simple change of the parameters of grooving, such as blade features and speed, geometry and size of the grooves and others. A Si sample bent by surface grooving was tested through x-ray diffraction, proving that the method of surface grooving allows a well-controlled curvature of the crystalline planes. Samples fabrication is simple and reproducible, cheaper and faster than for mosaic crystals. For implementation in a Laue lens, cylindrically deformed crystals due to
Figure 1. Grooves were indented on the surface of a Si plate along x direction (a). The probe x-ray beam enters the sample parallel (b) to the grooves.

...the grooves can be piled up to form a stack with their diffracting planes parallel to the major surfaces of the crystal. Photons enter nearly parallel to the diffracting planes, suffer diffraction and are thereby focused onto the detector. Proper welding of neighboring plates would be realized to ensure a good alignment of the grooved plates.

This paper reports an experimental study on the method of grooves for realization of Si and Ge bent crystals. After a brief description of sample preparation, their diffraction properties will be presented and discussed.

2. EXPERIMENTAL

The method of surface grooving is based on irreversible compression of the crystal beneath and beside the grooves. This region is highly defected and partly amorphous and its extent is generally limited to some microns, depending on dicing parameters, e.g., grit and advance speed of the blade. Thus, the crystalline material between the grooves is prevented from full mechanical relaxation and, as a result of such a deformation, the remaining crystal below the grooves is bent to a net and uniform curvature without the usage of any external device.

Pure Si and Ge wafers were diced to form plates using a high precision dicing saw (DISCO\textsuperscript{T M} DAD3220), equipped with rotating diamond blades of various width and grain size. Grooves were manufactured on the surface of the plates along x direction (Fig. 1a). Si and Ge plates were 1 mm and 2 mm thick, respectively, their orientation being the (111). Fabrication parameters of the samples under analysis are reported in Tab. I.

The curvature of every sample was measured through an optical profilometer (VEECO\textsuperscript{T M} NT1100) with 1 µm lateral and 1 nm vertical resolution and equipped with a stitching system which allows scanning over as wide an area as 10×10 cm\textsuperscript{2}. Typically, a wafer exhibits non zero bowing, i.e., its surfaces are not perfectly flat. Thus, in order to take into account exclusively of the deformation due to grooves, subtraction of the profile before and after the process was done. Moreover, since the profile of a surface with grooves is altered by their presence, profilometric characterization was carried out on the back face of each sample. As a result of the process, an elliptical curvature appeared, with the shortest radius of curvature perpendicular to the grooves. A typical profilometric measurement is shown in Fig. 2.

All samples were tested through x-ray diffraction during a 6-day run at beamline ID15A of ESRF (Grenoble, France). A highly monochromatic and quasi-parallel beam was set to an energy ranging from 150 to 700 keV thanks to a two-reflection Laue Si (111) unbent monochromator. The characterization was carried out by recording the Rocking Curves (RCs), i.e., by rotating the crystal exposed to the beam, while either the diffracted or transmitted beam intensity was recorded as a function of the incidence angle. Angular distribution of diffracting planes (hereinafter referred to as angular spread) is quantified by the FWHM of the RC, which also highlights diffraction efficiency of the sample under investigation. Efficiency has been calculated as the ratio of diffracted beam intensity over the transmitted one, this latter being recorded when the crystal is not subject to diffraction conditions.\textsuperscript{9} All samples were analyzed by diffraction of their (111) planes, the probe beam entering the sample at different depths from the grooved surface (coordinate z) and in quasi-parallel (hereinafter referred to as parallel) configuration with respect to the grooves (Fig. 1b).

3. RESULTS AND DISCUSSION

Si crystal S71 was initially measured at 150 keV with the beam penetrating the sample through its 25.5×1 mm\textsuperscript{2} surface at different depths from the grooved side and parallel to the grooves. Bending angle of the sample, as
Table 1. Fabrication parameters of the samples under analysis.

<table>
<thead>
<tr>
<th>Code</th>
<th>Size (mm$^3$)</th>
<th>Number of groove grooves</th>
<th>Groove direction</th>
<th>Groove pitch (µm)</th>
<th>Groove depth (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S71</td>
<td>25.5×25.5×1</td>
<td>31</td>
<td>[110]</td>
<td>790</td>
<td>500</td>
</tr>
<tr>
<td>2_G32</td>
<td>18.6×9.8×2</td>
<td>11</td>
<td>[110]</td>
<td>800</td>
<td>1000</td>
</tr>
</tbody>
</table>

Figure 2. Optical profilometry scanning of the surface without grooves for crystal S71 (a). False-color representation of deformation is highlighted. Cross sections of the deformation pattern along y = [211] (b) and x = [110] (c) directions as taken on the center of the sample measured by optical profilometry, averaged 15.7 arcsec along [110] direction. Figs. 3a, 3b and 3c show both diffracted and transmitted RCs as normalized to transmitted beam intensity, so that diffraction efficiency is readily displayed.

All RCs exhibited flat-topped rectangular and uniform shapes with a FWHM of 14.1 arcsec, very close to the optically determined crystal bending. This sample features a significantly high efficiency, about 93.4% over the whole depth. Hence, a bent crystal was shown to amply overcome the 50%-efficiency limit, that holds for a mosaic crystal.

Sample S71 was characterized at several energies, the beam entering the crystal far from the grooved region and in parallel configuration with respect to the grooves. RCs are shown in Fig. 4. The sample features a significant diffraction efficiency up to 700 keV, ranging from 92% down to 29%. All the records showed a rectangular shape of the angular distribution at any energy. Next section compares experimental performance to theoretical expectations, showing that the decrease in efficiency with energy is completely in agreement with the dynamical theory of diffraction. More information about on-beam characterization is in reference.\(^7\)

Ge crystal 2_G32 was measured at 300 keV with the beam penetrating the sample through its 9.8×2 mm$^2$ surface at different depths from the grooved side and parallel to the grooves. Morphological bending angle of the sample was 42.4 arcsec along [110] direction. Figs. 3d, 3e and 3f show its measured RCs. The FWHM of the angular distribution is always of the order of crystal bending, i.e., it is nearly 45 arcsec throughout the whole crystal depth. Diffraction efficiency averaged 58% and kept constant as a function of coordinate z. Although this figure of merit is far lower than the theoretically predicted 93%, this is still a good performance. Efficiency drop is probably due to non-perfect crystalline quality of base material.
Figure 3. RCs of crystal S71 with the beam parallel to the grooves at several distances from the grooved face, i.e., at (a) \( z = 0.4 \) mm, (b) \( z = 0.6 \) mm, (c) \( z = 0.8 \) mm; all the RCs were recorded at \( y = 13.9 \) mm. RCs of crystal 2\textsubscript{G32} with the beam parallel to the grooves at different depths from the grooved surface, i.e., at (d) \( z = 0.25 \) mm, (e) \( z = 0.85 \) mm, (f) \( z = 1.25 \) mm. RCs with rectangular and homogenous shapes were achieved in all cases, with an energy passband of the order of crystal bending (about 16 arcsec for the sample S71 and 45 arcsec for 2\textsubscript{G32}). Efficiency is close to the unity for Si while it averages 58% for Ge sample.
Figure 4. RCs of crystal S71 with the beam parallel to the grooves, measured at \( z = 0.8 \) mm and \( y = 13.9 \) mm. Beam energy was set at 200 keV (a), 300 keV (b), 400 keV (c), 500 keV (d), 600 keV (e) and 700 keV (f). The filled red circles plot the intensity of the transmitted beam, whereas the empty blue circles plot the intensity of the diffracted beam. Efficiency falls off with photon energy according to the dynamical theory of diffraction though a rectangular shape of the distribution is preserved.
Experimental data of the samples under investigation have been analyzed and compared to theoretical expectations through a simulation code developed in Python programming language. The software computes diffraction efficiency and reflectivity for both curved and mosaic crystals. Reflectivity, which is a qualifier of the diffraction properties of a crystal, is defined as the ratio of diffracted beam intensity over the incident one. The results of simulation will be shown here only for sample S71. In Fig. 5a, experimental diffraction efficiency is compared to theoretical efficiency as a function of coordinate $z$. Theoretical efficiency was calculated for a crystal with angular spread of 14.1 arcsec and thickness of 25.5 mm, in both the cases of a perfectly curved and a mosaic crystal (the crystallite size is considered negligible with respect to the extinction length). Since theoretical efficiencies were calculated taking into account the FWHM of experimental RCs, a margin of uncertainty has been included. As visible, experimental efficiency is constantly close to the theoretical limit for a perfectly curved crystal, meaning that the method of surface grooving leads to nearly a perfect curvature.

Diffraction efficiency was also studied vs. energy (Fig. 5b). It came out that the response was always very close to its theoretical limit, falling off with the photon energy strictly according to dynamical theory of diffraction.

5. CONCLUSIONS

Mechanical grooving of one of the largest surfaces of a crystal has proven to yield self-standing uniform and controlled curvature in Si and Ge (111) crystals. The samples exhibited significantly high-efficiency diffraction and broad-band response. The Si sample proved to diffract efficiently up to 700 keV, by far overcoming the 50%-limit affecting mosaic crystals. This energy range is sufficiently wide for many astrophysical applications of a Laue lens. The response of Ge was rather high though lower than theoretical prediction, probably due to non-perfect crystalline quality of base material, 60% of efficiency has been achieved. In all cases the morphological curvature measured by optical profilometry was in good agreement with the curvature determined by x-ray diffraction. On the strength of this experimental work, it looks possible to fabricate optical components for a Laue lens by stacking grooved Si or Ge bent crystals, provided that a good alignment of the plates is ensured.
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