Characterization of bent crystals for Laue lenses

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ABSTRACT

In the context of the LAUE project devoted to build a long focal-length focusing optics for soft $\gamma$-ray astronomy (80 – 600 keV), we present the results of reflectivity measurements of bent crystals in different configurations, obtained by bending perfect or mosaic flat crystals. We also compare these results with those obtained using flat crystals. The measurements were performed using the K\textalpha\ line of the Tungsten anode of the X-ray tube used in the LARIX facility of the University of Ferrara.

These results are finalized to select the best materials and to optimize the thickness of the crystal tiles that will be used for building a Laue lens petal which is a part of an entire Laue lens, with 20 m focal length and 100–300 keV passband. The final goal of the LAUE project is overcome, by at least 2 orders of magnitude, the sensitivity limits of the current generation of $\gamma$-ray telescopes, and to improve the current $\gamma$-ray imaging capability.

Keywords: Laue lenses, X-ray, Focusing telescopes, Gamma-rays, Astrophysics, Bent crystals

1. INTRODUCTION

Experimental hard X-/soft $\gamma$-ray (10-1000 keV) astronomy is moving from direct sky viewing telescopes to focusing telescopes. With the forthcoming focusing telescopes in this energy range, a big improvement in sensitivity is expected: a factor of 100-1000 with respect to the best non-focusing instruments of the current generation, either using coded masks or not. A significant increase in angular resolution will be also achievable from the $\sim$ 10 arcmin of the mask telescopes to less than 1 arcmin. The first generation of soft $\gamma$-ray (> 100 keV) focusing telescopes will make use of the Bragg diffraction technique from crystals in a transmission configuration (Laue lenses).

The astrophysical issues that are expected to be solved with the advent of these telescopes are many and of fundamental importance (see, e.g., Ref. \cite{1}). We have already developed two Laue lens prototypes, in the framework of the Hard X-ray TELEscope (HAXTEL) project, devoted to develop and test a technology for building a broad passband Laue lens for small focal length (<10 m)\cite{Frontera08,Virgilli11}. After the successful results of HAXTEL, a new LAUE project is ongoing, devoted to build broad band (80–600 keV) focusing lenses with longer focal lengths. The final goal of the LAUE project is to build a lens with flux high sensitivity. This implies that the focal spot has to be as small as possible, not only to improve the angular resolution but mainly to reduce the background under the spot. To achieve this goal, we moved from flat mosaic crystals employed in the HAXTEL project, to bent crystals. The are provided by the ”Laboratorio Sensori e Semiconduttori (LSS – Ferrara)” and by ”Istituto dei Materiali per l’ Elettronica ed il Magnetismo (IMEM – Parma)”, both institutions involved in the LAUE project.

In this paper, after having shortly described the LAUE project, a description of the bent crystals tested and their properties is given with a comparison of the corresponding properties of flat perfect and mosaic crystals. Finally, the experimental results concerning bent Si and GaAs crystal samples are reported.

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2. THE LAUE PROJECT

The Laue project, supported by the Italian Space Agency (ASI), is devoted to development of an advanced technology for building a Laue lens with a broad energy passband (80–600 keV) and long focal length (up to 20 m and beyond) for space Astrophysics (for a detailed overview of the entire project see Ref. 2,3). From a technical point of view, the massive production of crystals with the demanded characteristics, and the development of a technology for a fast positioning with an accuracy better than 10 arcsec, represent a tough challenge. Only a very tight collaboration between scientific institutions and industries has made possible to employ the most advanced technologies in crystals manufacturing and the ongoing installation of a new facility, in which the crystals will be tested and assembled, with the required accuracy, on a lens petal. The adopted technology for assembling the crystals in the lens is to fix the crystal tiles on its place on the lens frame after properly orienting them under the control of a γ-ray beam. The correct fixing of the crystal tiles to the lens frame is ensured by gluing them to the frame kept in the same position during the assembling phase. Given this assembling strategy, both the X-ray source and the collimator have to be moved together in order that the beam axis is parallel to the lens axis and all crystal tiles can be properly fixed to the lens frame. Such a procedure ensures that each crystal focuses the incident photons in the lens focus, within the equipment uncertainties.

As a result of the LAUE project, a lens petal with 20 m focal length and 100–300 keV passband will be built.

3. CRYSTAL TEST APPARATUS

The test of samples of candidate crystals for the LAUE project has been performed with the small LARIX facility shown in Fig. 1. By means of a motorized crystal holder, it is possible to translate the crystal samples along 2 directions perpendicular to the beam and rotate them around three orthogonal axes. The X-ray beam coming from the source (an X-ray tube with a maximum voltage of 150 kV) travels across two collimators with adjustable size, the former at 80 cm from the X-ray source, the latter at a distance of 83 cm from the crystal to be tested. The distance between the collimators is 580 cm. The direct and reflected beam is analyzed by means of two detectors, an X-ray imager with spatial resolution of 300 µm, and a cooled HPGe spectrometer with a 800 eV spectral resolution at 100 keV, both positioned on a detector holder that can moved back and forth along the beam axis.

Figure 1

4. MEASUREMENT SET-UP

The angular distribution of the diffracting planes and the main properties of each sample are obtained by measuring the FWHM of the Rocking Curve (RC) and the reflectivity value. The measurements have been performed using a monochromatic beam at 59.2 keV obtained from the fluorescence Kα line of the Tungsten anode of the X-ray tube (Fig. 2).
The limited length of the experimental set-up (see Fig. 1) and the X-ray source size (0.6 mm radius) give rise to a divergent beam impinging on the crystal sample. An accurate estimate of this divergence is thus crucial, to separate this effect from the response function of each tested crystal.

A divergence measurement has been carried out, using a flat Silicon crystal (111) in Bragg configuration, by measuring the rocking curve of the crystal for different collimator sizes and comparing it with the theoretical value, geometrically estimated.

A compromise between brightness of the beam that is impinging on the crystal and divergence level has been reached, by setting the horizontal aperture of the collimator 1 to 0.3 mm and that of the collimator 2 to 0.2 mm, keeping the vertical aperture of both collimators to 2 mm. With this configuration the resulting divergence value (Full Width at Half Maximum, FWHM) was estimated to be $FWHM_{\text{div}} = 11 \pm 2$ arcsec, in agreement with the geometrical estimate (see Fig. 3).

In order to estimate the sample reflectivity of perfect crystals at 59.2 keV, we compare the incident beam intensity (naturally integrated over all divergence angles) with the crystal Rocking Curve, obtained by rotating the sample in Laue configuration. For the mosaic samples, the effective $FWHM$ of the mosaic spread (crystal mosaicity $\eta$, see Ref. [1]) is instead obtained from the Gaussian spread $FWHM_{\text{meas}}$ of the measured diffraction peak through the following equation [6]:

$$FWHM = \sqrt{FWHM_{\text{meas}}^2 - FWHM_{\text{div}}^2}$$  \hspace{1cm} (1)  

5. CRYSTAL SAMPLES AND TESTED GEOMETRIES

The crystal samples (see Table [1]) have been tested by setting the X-ray tube voltage to 120 kV with a current of about 1.2 mA. For each sample, both the intensity of the transmitted beam through the crystal and the Rocking Curve are measured. The direct beam is also periodically monitored for possible intensity variations with time.
Table 1. Tested samples

| Number of tiles | Material   | Dimensions mm x mm x mm | Geometry                      |
|-----------------|------------|--------------------------|-------------------------------|
| 1               | Silicon    | 15 x 15 x 0.75           | Flat perfect                  |
| 1               | Silicon    | 15 x 15 x 0.75           | Perfect, bent 60 m curvature radius |
| 1               | Silicon    | 15 x 15 x 0.75           | Perfect, bent 27 m curvature radius |
| 1               | Silicon    | 10 x 15 x 10.5           | Flat mosaic                   |
| 5               | Gallium Arsenide | 15 x 15 x 2        | Flat mosaic                   |
| 1               | Gallium Arsenide | Rounded shape x 2     | Bent mosaic                   |
| 1               | Silicon    | 25 x 25 x 1              | Flat                          |
| 1               | Silicon    | 25 x 25 x 1              | Bent via indentation          |
| 1               | Silicon    | 45 x 10 x 3              | Stack of 3 elements, bent via indentations |

Two configurations of the crystals have been investigated: *geometry* 1 and *geometry* 2. In *geometry* 1 the beam is impinging on the lateral side of the crystal tile, i.e., the surface in which one of the two sizes is the crystal thickness. Instead, in *geometry* 2, the X-ray beam is impinging on the main face of the crystal tile. The *geometry* 2 is used for testing the flat and bent mosaic crystals, and the bent perfect crystals in Laue configuration, where a secondary internal curvature of the lattice planes (quasi-mosaic) arises due to the induced external bending.

Two crystal holders are available. The first is a clamp support, which is employed for testing squared or rectangular crystals tiles 10 ÷ 20 mm long each side and 0.5 ÷ 3 mm thick. This crystal holder is used for *geometry* 2 configuration. The other holder is suitable for testing crystals in *geometry* 1. The holder is made of a steal base, on which it is positioned an aluminum wall (see right panel of Fig. 9) that can be moved back and forth along the base depending on the crystal dimensions, in order to rotate the analyzed crystal around the axis of the holder base and to get it within the beam axis.

6. RESULTS

6.1 Samples provided by IMEM

Bent crystals are obtained by lapping one of the main crystal cross sections with sandpaper that introduces defects in a superficial layer undergoing a highly compressive strain. The samples provided by IMEM are shown in the upper part of Table 1.

6.1.1 Flat Silicon, Bent Silicon 60 m and 27 m curvature radius in geometry 2

Both the flat Si sample and the two bent Si samples have a dimension 15 x 15 x 0.75 mm³ with diffracting planes in *geometry* 2 being the (100). They all come from the same ingot, therefore the measurements provide a good comparison of their properties. As shown in Fig. 4, the reflectivity at 59.2 keV increases linearly as a function of the crystal curvature, demonstrating that the lapping technique can improve the diffraction efficiency of a flat perfect crystal. For a 60 m and 27 m curvature radius the reflection efficiency increase by a factor 2 and 3, respectively, with respect to that of the flat crystal.

6.1.2 GaAs flat mosaic crystal in transmission configuration

Two samples were tested, using the clamp crystal support (Fig. 5). The tested samples are known to have a mosaicity of about 25 arcsec. Their cross-section is square with dimensions of 15 x 15 mm² and thickness of 2 mm. The measurements are found to be consistent with the expectations with a value of the reflectivity at 59.2 keV of about 35% and a mosaicity of about 25 arcsec.
6.1.3 Bent mosaic crystal of GaAs(111) in transmission configuration

The sample has a rounded shape with a radius of 35 mm while the bending radius is 40 m. We mounted the tile on a support and analyzed it in the central region in Laue configuration, being (111) the diffracting planes. The measured efficiency (see Fig. 8) is about 40%, slightly greater than the flat sample, consistent with a small increase of the angular spread with respect to the flat GaAs crystal (25–30 arcsec). The curvature does not affect the internal structure and the local mosaic spread does not change, while it influences the global behaviour of the sample that allows the focusing effect.
6.1.4 Stack of bent Silicon (111) crystals in geometry 1

We have also tested a stack of 14 Si(111) crystals (Fig.7) in geometry 1 with a bending radius of 55 m. Each crystal has a size of $10 \times 15 \text{ mm}^2$ and a thickness of 0.75 mm, with the stack showing a surface, on which the beam is impinging, of $10 \times 10.5 \text{ mm}^2$.

![Figure 7. The Silicon stack on the crystal support during the test.](image)

The sample has been analyzed in two different regions. Although the stack was assembled manually and the tiles were not glued among them, it showed similar diffraction properties, with a peak efficiency at 59.2 keV of about 30%.

Although stacks made of bent silicon have been discarded due to technical difficulties in producing a large quantity of stacks with the desired alignment, the opportunity of testing the crystals in this configuration is equally important for possible future applications.

6.2 Crystals provided by LSS

The LSS laboratory at the University of Ferrara provided us bent Silicon crystals with lattice planes (111) perpendicular to the main crystal surface. The bending is achieved by indentation of a mesh of grooves of one of the surfaces of the crystal\textsuperscript{7}. The obtained curvature is permanent and can be finely tuned by changing the grooving parameters, such as blade features and grooving speed, geometry and size of the grooves. It can be demonstrated that for some of the lattice planes used as diffraction planes in Laue configuration, when a primary curvature is provided to the crystal, a secondary (quasi-mosaic) curvature of the diffraction planes occurs (Fig. 13). This secondary curvature can be properly exploited for increasing the diffraction efficiency. The final bending is verified by means of a profilometer with an uncertainty of 5% (2 meters in our case, being the lens curvature radius 40 meters).

6.2.1 Silicon bent crystals in geometry 1

The sample has a volume of $25 \times 25 \times 1 \text{ mm}^3$, with 1 mm being its thickness. It was analyzed in geometry 1, with the beam penetrating the crystal through the lateral surface $25 \times 1 \text{ mm}^2$, perpendicular to the grooved plane.

Table 2 shows the FWHM of the RCs obtained rotating the crystal around the axis of the main surface of the crystal, taking into account the divergence effect of the beam. A relation seems to be present between radiated point position and FWHM, maybe due to the fact that photons impinging on the crystal cross-section close to the edges can escape from the lateral sides instead of escaping from the back surface, giving rise to a different angular spread.

The results are consistent with previous tests performed by LSS at the ESRF synchrotron radiation facility in Grenoble, confirming that LARIX facility is suitable to determine, with good confidence, the crystals diffraction properties.
Figure 8. Sketch of a bent perfect crystal in quasi-mosaic geometry.

Figure 9. Left: Silicon bent crystals analyzed in geometry 1. Right: Pattern of the crystal regions tested.

Table 2. Rocking curve width as a function of the tested crystal region (Fig. 9)

| Analyzed region | Distance with respect to the grooved face (mm) | FWHM (arcsec) |
|-----------------|-----------------------------------------------|---------------|
| 1               | 0.03                                          | 25.4          |
| 2               | 0.22                                          | 43.9          |
| 3               | 0.32                                          | 51.6          |
| 4               | 0.52                                          | 52.2          |
| 5               | 0.43                                          | 67.8          |

6.2.2 Stack of bent Silicon (220) crystals

The stack is composed of three Si bent crystals with 110 m bending radius, a front surface of $45 \times 10 \text{ mm}^2$ and thickness of 3 mm. The corresponding plane curvature is 84.4 arcsec. The stack was tested in order to misalignments of single crystals of the stack with each other.

Figure 10. The Silicon stack and the regions tested.
Geometry 1 test

The diffractivity of the (111) planes ($\theta_b \approx 1.92^\circ$) was tested in different points of the lateral surface $10 \times 3$ mm$^2$ (see Fig. 10).

The incident X-ray beam enters the stack at different distances from the grooved surface (regions 1, 2, 3, see Fig. 10 and Table 3). The measured RCs are very broad and are consistent with the sum of three Gaussian (Fig. 11). We expected to see a broad peak with FWHM of the order of the crystal bending, about 84 arcsec. The best agreement with the expectations is found when the central region (2) is irradiated. These results can be explained in terms of depth of the stack traveled by the diffracted beam.

![Rocking Curve](image1)

Figure 11. RCs (Geometry 1) of the Silicon stack measured in the regions 1 (left), 2 (center), and 3 (right). The RCs have been fit using 3 Gaussian functions, due to the imperfect alignment of the crystals in the stack and to the depth traveled by the diffracted beam.

| Region analyzed | Distance from the grooved surface (mm) | FWHM (arcsec) |
|-----------------|----------------------------------------|---------------|
| 1               | 0.4                                    | 45.87         |
| 2               | 1.5                                    | 87.46         |
| 3               | 2.3                                    | 79.12         |

Geometry 2 test

The stack in geometry 2 has been also tested. In this case the beam is incident on the grooved surface. In this case the diffracting planes are the (220), which do not show the quasi-mosaic structure of the (111) planes. The thickness in this case is only 3 mm. Five regions of the cross-section have been irradiated.

The test results are shown in Fig. 12 that layers of the stack are not perfectly aligned with each other, with the maximum misalignment being more relevant in the external regions (C and A).

6.2.3 Quasi-mosaic Silicon (111) samples in geometry 2

We have characterized two samples (n5 and n6) with a square surface of $20 \times 20$ mm$^2$ and thickness of 2 mm, with two different external curvature radii $R_e$: 60 and 8 meters, respectively (Fig. 13). The diffracting planes (111), in transmission configuration, are orthogonal to the square surface. From the dynamical theory of diffraction of bent crystals the ratio between the internal curvature radius $R_i$ and $R_e$ is equal to 2.6, with a consequent internal curvature of the diffracting (111) planes of 160 and 20 meters, respectively.

From the measured rocking curve we derived an angular spread of $11 \pm 2$ arcsec for sample n5. This value is in agreement with the convolution of the expected intrinsic RC width of the sample (2.5 arcsec) with the beam divergence. The measured reflectivity at 59.2 keV is about 80%. For the sample n6 we measured a RC width of $21 \pm 2$ arcsec and a reflectivity value of 27% (Fig. 14).
7. CONCLUSIONS

In this paper, various bent and flat crystals have been tested for their possible use in Laue lenses. For each sample, angular spread and diffraction efficiency at 59.2 keV have been measured as a function of the crystal thickness and curvature radius and compared with those obtained using flat crystals. We have also shown that the LARIX facility is suitable to perform these tests.

All the analyzed GaAs samples provided by IMEM show a high-efficiency diffraction, with an average angular spread that satisfies the requirements of the LAUE project. The curvature radii are in agreement with the theoretical expectations.

The test of Silicon crystals provided by LSS with different curvature radii and thicknesses have allowed us to establish that Silicon crystals could be successfully employed for LAUE project. However current technological
limitations to bend crystals with thickness greater than 2 mm prevent to use them. Hence, Germanium tiles are more appropriate to be used for the LAUE project, given that 2 mm thickness of this material is sufficient to achieve the needed diffraction efficiency. Similarly, bent crystals of GaAs(111) in mosaic configuration can be also used for the LAUE project.

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