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Experimental study on grooved Si and Ge crystals for Laue lens application

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Summary. — An experimental study on the method of indentations for bent crystals to realize a hard X-ray Laue lens has been done. We tested the diffraction properties of indented Si and Ge crystalline plates at European Synchrotron Radiation Facility (Grenoble, France). The samples were analyzed by diffraction of their (111) planes with hard X-rays from 150 to 600 keV. Crystals have shown significantly high diffraction efficiency, *i.e.* a Si crystal has exhibited up to 80% at 300 keV. A Ge crystal has confirmed the observation for a Si one, though the diffraction efficiency was about 60%. In both cases rocking curves showed flat-toped rectangular shapes, which demonstrates that the method of indentations evenly bends the crystals. Moreover, measured angular spread was always very close to the morphological curvature of the sample under investigation, showing that this method offers high reproducibility and, thus, easy control of diffraction properties of the crystals.

PACS `61.05.cp` – X-ray diffraction.
PACS `81.40.Lm` – Deformation, plasticity, and creep.

1. – Introduction

Modern physics shows increasing interest in sky observation within X- and soft gamma-ray band. In this energy domain many celestial events emitting high-energy photons awaits to be deeply investigated. Nevertheless, this part of the electromagnetic spectrum is not properly exploited because of the absence of focusing optics. Multilayer grazing incidence optics allows focusing X-rays up to 80 keV [1], but above this limit their efficiency strongly decreases. Past and present instruments operating at energy higher than 80 keV reconstruct incidence direction of photons either by the use of coded masks

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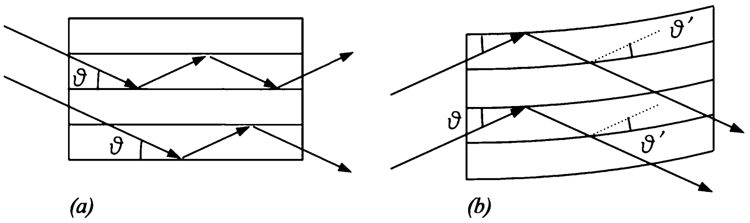

Fig. 1. – X-ray diffraction in Laue geometry in case of an unbent (*a*) and of a bent crystal (*b*). Multiple reflections in case (*a*) results in maximum 50% diffraction efficiency, while in case (*b*) diffraction efficiency can reach 100%.

or by tracking multiple interactions of photons in a sensitive volume. This results in low signal-to-noise ratio due to the fact that background roughly scales with the detector volume. Despite years of technology development, gamma-ray telescopes operating in the 100 keV–1.5 MeV energy range are still lacking the sensitivity that could allow a real breakthrough in the understanding of the physics at the origin of observed emission.

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 [2]. This optics relies on Bragg diffraction (in Laue geometry) through a large number of accurately oriented crystals to deviate and concentrate radiation. Crystals for Laue lens have to fulfill a number of requirements: they have to be available with a controlled passband, to be highly reproducible, and yield the highest possible reflectivity.

There are two kinds of crystals that potentially fulfill these requirements. Mosaic crystals are the most commonly found, however, they exhibit a Gaussian passband and a diffraction efficiency limited to 50%. On the other hand, bent crystals do not undergo this limitation. Due to continuous change of the incidence angle, a single diffraction onto curved crystalline planes is allowed, so that no re-diffraction can occur subsequently (fig. 1). Moreover, a bent crystal offers a continuum of possible diffraction angles, leading to a rectangular-shaped energy passband directly given by its curvature.

There are several different methods to induce a curvature in a crystalline plate. One such method is the mechanical indentation of one of its largest surfaces [3]. The curvature is permanent and adjustable by changing the parameters of indentation, like blade characteristics and speed, geometry and size of the indentations and others [4]. Reproducible deformation of (111) Si and Ge crystals was recently attained through experimental work at Sensor and Semiconductor Laboratory (Ferrara, Italy). A Si sample bent by indentations was tested through X-ray diffraction, proving that the method of indentations allows a well-controlled curvature of crystalline planes to be realized [5]. Samples fabrication is rather simple and more reproducible, cheaper and faster than for mosaic crystals. Moreover, crystals with indentations maintain their curvature without any external holder.

This paper reports on a comprehensive experimental study on the method of indentations, for bending crystals. After briefly describing the theory of indentation technical details of the experiment will be given. Then experimental results on Si and Ge grooved crystals will be presented and their diffraction properties discussed. Finally, a discussion compares experimental data to theoretical expectations.

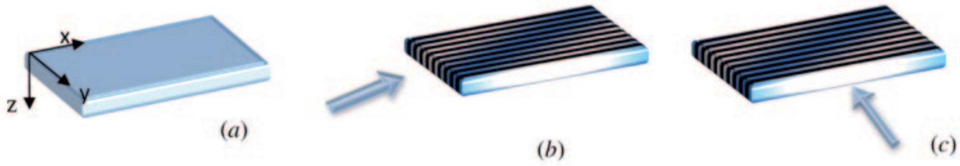

Fig. 2. – Grooves were indented on the surface of a Si or Ge plate along one direction, either x or y (a). The pencil X-ray beam enters the sample parallel (b) or perpendicular (c) to the grooves.

2. – Experimental method

The method of indentations for bending crystals relies on irreversible deformation of the material beside and beneath the indentations themselves. This amorphous region, being full of dislocations, acts as a solid wall for the crystalline material between the indentations. Thus this latter, which is elastically compressed and prevented from relaxation, leads to a permanent and uniform curvature within the crystal.

Pure Si and Ge wafers were diced to form plates using a high precision dicing saw (DISCO$^{\text{TM}}$ DAD3220), equipped with rotating diamond blades of various width and grain size. Grooves were manufactured on the surface of the plates along one direction, either x or y (fig. 2a). Si and Ge plates were 1 mm and 2 mm thick, respectively, their orientation being the (111). Fabrication parameters of all samples are reported in table I.

The curvature of every sample was measured through an optical profilometer (VEECO$^{\text{TM}}$ NT1100) with 1 μm lateral and 1 nm vertical resolution and equipped with a stitching system. Profilometric characterization was carried out on the face without indentations 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. 3. Both production and optical characterization of all samples have been carried out at SSL Ferrara.

All samples were tested through X-ray diffraction during a 6-day run at ESRF beamline ID15A. A highly monochromatic and quasi-parallel beam was set to an energy ranging from 150 to 600 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 diffracted or transmitted 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 [6].

TABLE I. – *Fabrication parameters of the samples under analysis.*

Code	Size (mm^3)	Number of indentations	Grooves direction	Grooves spacing (μm)	Depth of grooves (μm)
S72	$25.5 \times 36.6 \times 1$	25	[211]	1000	400
S81	$54.2 \times 30.6 \times 1$	30	[110]	1000	400
2_G32	$18.6 \times 9.8 \times 2$	11	[110]	800	1000

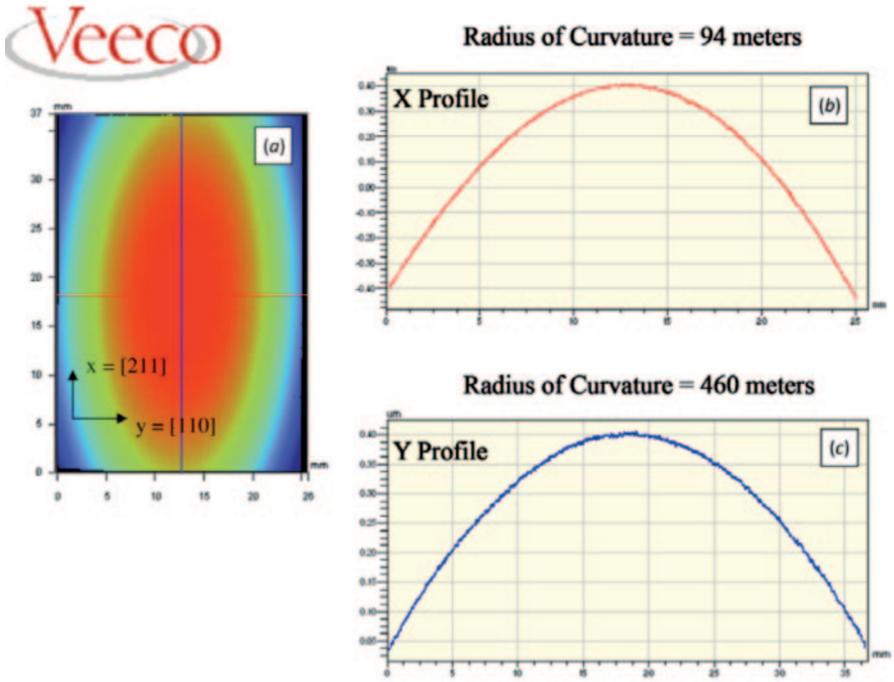

Fig. 3. – Optical profilometry scanning of the surface without indentations of crystal S72 (*a*). False-color representation of deformation is highlighted. Cross-sections of the deformation pattern along x (*b*) and y (*c*) directions as taken on the center of the sample with indications of the two main curvature radii.

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). Two different configurations were used, *i.e.* the beam was set quasi-parallel (hereinafter referred to as parallel) or perpendicular to the grooves (figs. 2*b* and 2*c*).

3. – Results and discussion

Si crystal S81 was measured at 300 keV with the beam penetrating the sample through its $30.6 \times 1\,\mathrm{mm}^2$surface, at fixed depth from the grooved side (coordinate z) and at different coordinates y. The beam was set parallel to the grooves. Bending angle of the sample, as measured by optical profilometry, averaged 29.5 arcsec along [110] direction. Figures 4*a*, 4*b* and 4*c* show both diffracted and transmitted RCs as normalized to transmitted beam intensity, so that diffraction efficiency is readily displayed.

RCs exhibited flat-topped rectangular and uniform shapes with a FWHM of 26 arcsec, close to the optically determined crystal bending. This sample features a significantly high efficiency, about 82% constantly over the whole sample, showing that a curved crystal can amply break the 50%-efficiency limit, which holds true for an unbent crystal. No dependence on coordinate y was recorded, meaning that curvature is homogeneous throughout the sample.

With the beam perpendicular to the grooves of sample S72, efficiency showed different behavior from the previous case. This sample was measured at 150 keV with the beam

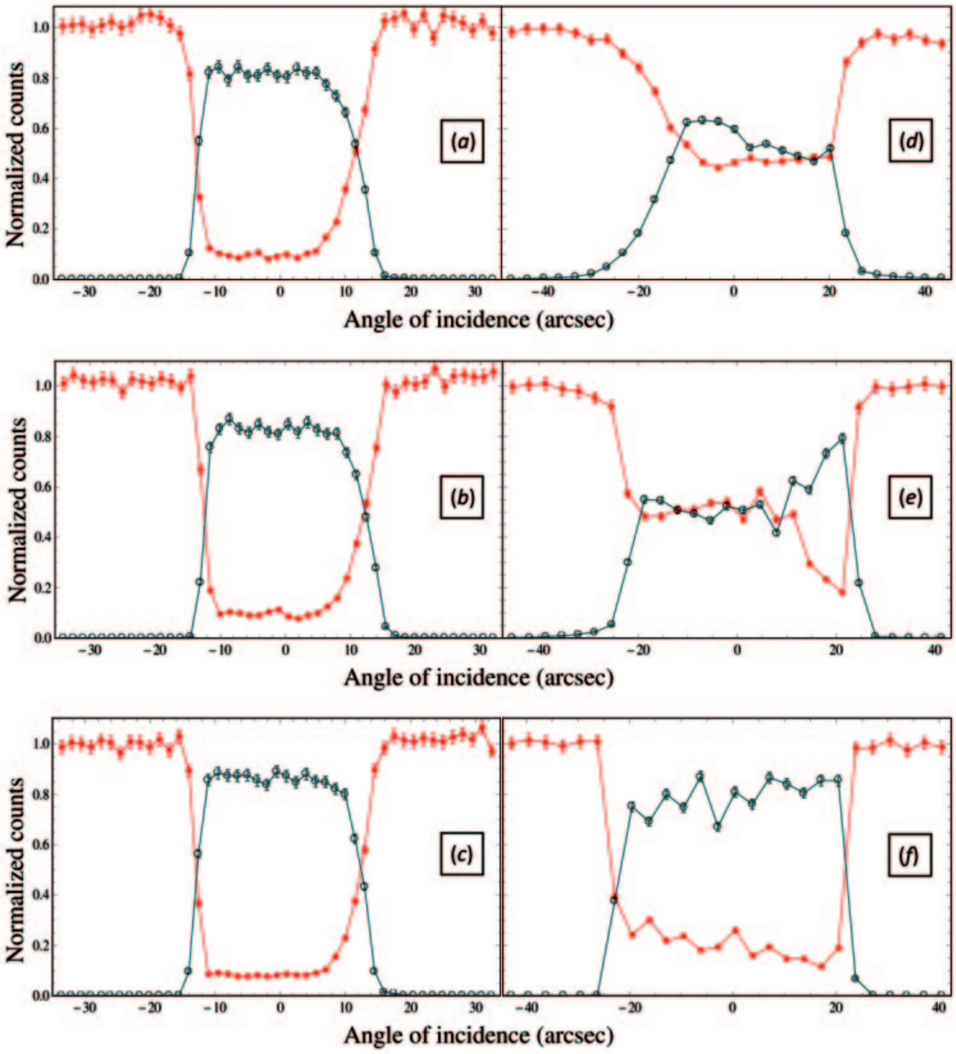

Fig. 4. – The left-hand side shows RCs of crystal S81 with the beam parallel to the grooves at a fixed distance from the grooved face and at $y = 5.0\,\text{mm}$ (a), $y = 5.9\,\text{mm}$ (b), $y = 6.8\,\text{mm}$ (c). The right-hand side shows RCs of crystal S72 with the beam perpendicular to the grooves at different depths from the grooved surface, *i.e.* at $z = 0.05\,\text{mm}$ (d), $z = 0.55\,\text{mm}$ (e), $z = 0.75\,\text{mm}$ (f). RCs with rectangular and homogenous shapes were achieved in all cases, with an energy passband of the order of crystal bending (about 26 arcsec for the sample S81 and 55 arcsec for S72). Efficiency is significantly high in all cases and up to 82% for sample S81.

entering the Si crystal through its $25.5 \times 1\,\text{mm}^2$at different depths from the upper grooved side. Bending angle of the sample was measured by optical profilometry to be 55 arcsec along [211] direction. The RCs in figs. 4d, 4e and 4f still highlight flat-topped and uniform shapes though efficiency varies over the crystal depth, *i.e.* it is nearly 50% close to the grooved region, and raises up to 79% deeper into the crystal. This feature can be ascribed to the fabrication process of indentations. In fact, generation of mosaicity

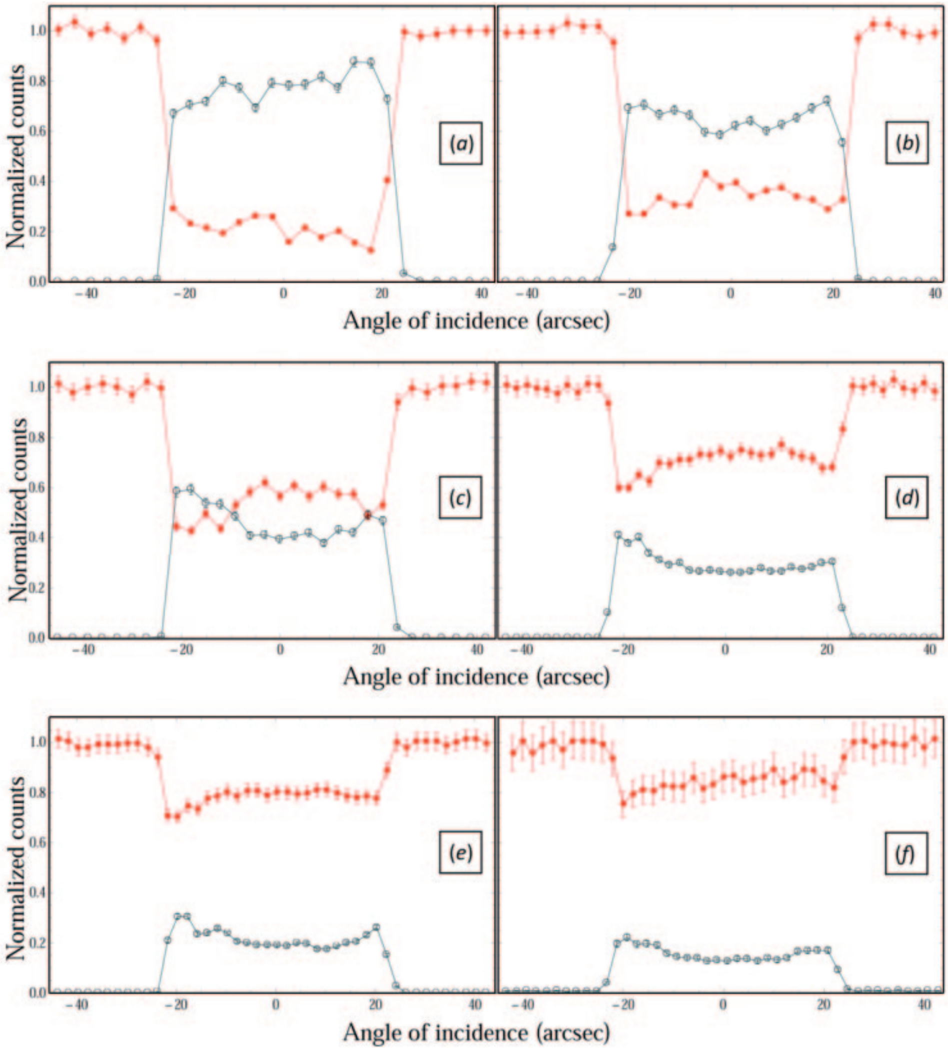

Fig. 5. – RCs of crystal S72 with the beam perpendicular to the grooves, measured at $z = 0.75\,\text{mm}$ and $x = 0.15\,\text{mm}$. Beam energy was set at 150 keV (*a*), 200 keV (*b*), 300 keV (*c*), 400 keV (*d*), 500 keV (*e*) and 600 keV (*f*). Efficiency falls off with photon energy according to dynamical theory of diffraction though a rectangular shape of the distribution is preserved.

perpendicularly to the advance speed of the blade is easier to form than longitudinally because of the stronger action exerted by the blade on the sidewalls of the groove. As a result, the angular spread, and in turn the energy bandwidth, increases across the depth of the grooves and decreases outside. Hence, this leads to an efficiency decrease throughout the whole depth of the grooves. Outside the grooved area, high efficiency is restored, meaning that crystal structure is not significantly affected by mosaicity.

Sample S72 was characterized *vs.* energy, the beam entering the crystal far from the grooved region and perpendicular to the grooves (fig. 5). The sample features significant

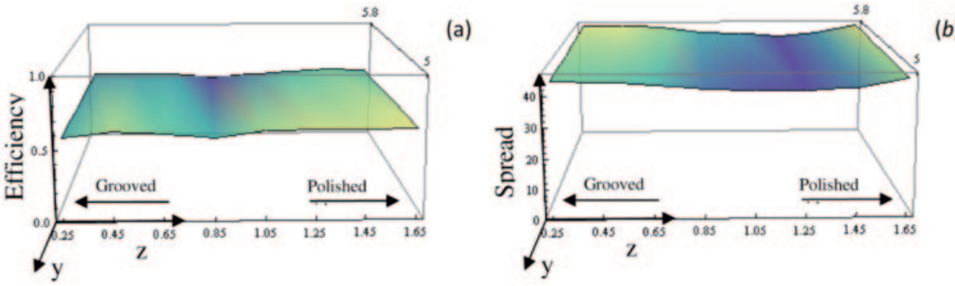

Fig. 6. – Diffraction efficiency of crystal 2_G32 *vs.* coordinate z (mm) at several positions within the crystal (coordinate y) with the beam perpendicular to the grooves (a). Same dependence of angular spread is shown. No dependence on coordinate y (mm) was recorded in any case. Efficiency is about 60% over the whole crystal depth.

diffraction efficiency up to 600 keV, ranging from 80% down to nearly 20%. In the next section a comparison of experimental performance to theoretical expectations is presented. It shows that the decrease in efficiency with energy is completely in agreement with dynamical theory of diffraction.

Ge crystal 2_G32 was measured at 300 keV with the beam penetrating the sample through its $9.8 \times 2\,\text{mm}^2$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. Figure 6a shows diffraction efficiency as a function of coordinates y and z, which averages 58% and is quite uniform throughout the crystal. Though this is still a good performance, it is less than the 93% theoretically expected, this being probably due to a non-perfect crystalline quality of the base material. Figure 6b shows that the angular spread is very homogeneous throughout the crystal, which is a good point.

4. – Simulations

Experimental data have been analyzed and compared to theoretical expectations through a simulation code developed in Python programming language. The software can calculate diffraction efficiency and reflectivity for both bent and mosaic crystals. The reflectivity is calculated according to definition given in ref. [6]. Expected performance is then compared to experimental data.

In fig. 7a experimental diffraction efficiency of sample S81 *vs.* coordinate y is compared to the theoretical efficiency determined for a crystal having an angular spread of 26 arcsec and a thickness of 30.6 mm in both the cases of an homogeneous curvature and a mosaic crystal (crystallite size is considered negligible with respect to the extinction length [6]). Theoretical efficiencies were calculated taking into account the FWHM of the RCs, thus an experimental uncertainty is included. The response is slightly lower than the theoretical efficiency for a bent crystal, meaning that indentations allow a nearly perfect curvature to be obtained. In any case, the 50%-limit yielded by mosaic crystals is overcome.

For sample S72, diffraction efficiency was studied as a function of photon energy (fig. 7b). It follows that the efficiency is always very close to its theoretical limit, falling off with photon energy according to dynamical theory of diffraction [7].

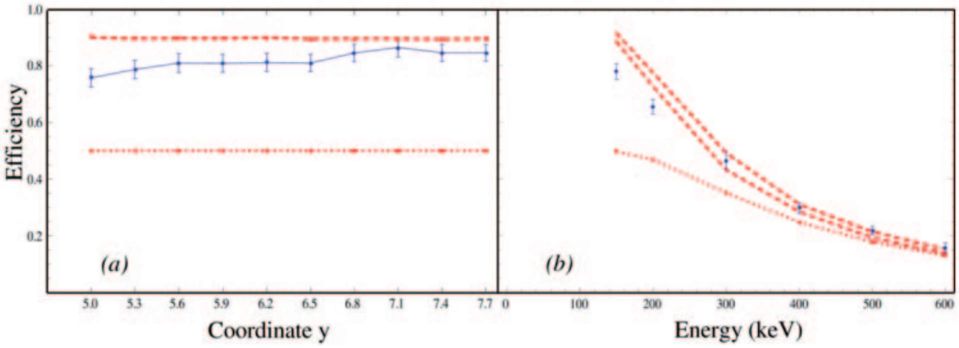

Fig. 7. – (Colour on-line)(*a*) Experimental efficiency (blue circles) *vs.* coordinate y (mm) for crystal S81 with the 300 keV pencil beam parallel to the grooves. Red dashed and dotted lines represent theoretical efficiencies in case of a curved and of a mosaic crystal, respectively. An experimental uncertainty is included in both cases. Experimental efficiency slightly varies within the crystal, being always rather close to the theoretical limit yielded by a bent crystal. (*b*) Experimental and theoretical diffraction efficiencies *vs.* energy, for sample S72. RCs were carried out at $z = 0.75$ mm from the grooved face with the beam perpendicular to the grooves. Theoretical efficiency (red dashed line) was calculated taking into account the FWHM of the RCs, thus an experimental uncertainty is included. Blue circles with their error bars represent measured diffraction efficiency. Red dotted line represents theoretical efficiency for a mosaic crystal with angular spread equal to the FWHM of the RCs.

5. – Conclusions

Indented Si and Ge (111) crystals exhibit significantly high efficiency and broad-band response when subject to X-ray diffraction. For Si, efficiency varies as the beam is parallel or perpendicular to the indentations and this is ascribed to generation of stronger mosaicity perpendicularly to the indentations. However, Si samples proved to diffract efficiently, and far exceeding the 50%-limit, which affects mosaic crystals, up to 600 keV, this domain being considerably wide for astrophysics applications with Laue lens. This latter can be made by stacks of indented crystals set with their diffracting planes parallel to the main surface of the plate. Proper welding of neighbouring plates would be realized to ensure the alignment of such a structure to be done. Though Ge performance is lower than theoretically expected, this fact being probably due to a non-perfect crystalline quality of the base material, 60% of efficiency has been achieved. However, future experiments will use better quality Ge wafers. In all cases measured angular spread is very close to the morphological curvature of the sample under investigation, thus meaning that a homogeneous and controlled curvature in both materials can be obtained and a very well-controlled crystal energy bandwidth can be achieved.

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