Ion implantation for manufacturing bent and periodically bent crystals

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Ion implantation is proposed to produce self-standing bent monocrystals. A Si sample 0.2 mm thick was bent to a radius of curvature of 10.5 m. The sample curvature was characterized by interferometric measurements; the crystalline quality of the bulk was tested by X-ray diffraction in transmission geometry through synchrotron light at ESRF (Grenoble, France). Dislocations induced by ion implantation affect only a very superficial layer of the sample, namely, the damaged region is confined in a layer 1 μm thick. Finally, an elective application of a deformed crystal through ion implantation is here proposed, i.e., the realization of a crystalline undulator to produce X-ray beams. © 2015 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4928553]

Bent crystals can be advantageously employed for several applications. Owing to the strong electric field generated by the ordered atoms in a crystal, it is possible to manipulate charged-particle trajectories via coherent effects such as channeling and volume reflection.1 Bent crystals demonstrated to work as primary collimator and beam manipulator in particle-beam accelerators, such as Tevatron2 and the Super Proton Synchrotron (SPS).3 Radiation emission due to curved trajectories of charged particles in bent crystals was studied in order to produce a photon beam through bremsstrahlung, channeling radiation, parametric X-ray radiation (PXR), and crystalline undulator (CU).4 Bent crystals were also tested as optical elements for focusing hard X- and γ-rays for astrophysics experiments with high efficiency and resolution.5 An γ-ray concentrator based on curved crystals can also be used for high-quality imaging in nuclear medicine.6

The simplest technique to fabricate a bent crystal consists on using an external holder capable of imparting a mechanical moment to a crystal plate. However, an external holder cannot be employed for the applications where payload or miniaturization is severe constraints. As an example, observations of the sky in the 100–1000 keV energy range must necessarily be performed through satellite-borne experiments to avoid absorption by the atmosphere. For any space mission, the payload is a crucial parameter dictating that the curved crystal must be self-standing. In nuclear medicine, there are no weight constrains; however, the need for miniaturization of the optical components requires that no assistance be taken by mechanical devices. If a periodic deformation is needed, as for the realization of a crystalline undulator, an external holder would be not suitable.

Manufacturing of self-standing bent crystals with a curvature that is adjustable and reproducible is still an open issue. A series of techniques have been proposed. A self-standing bent crystal can be obtained by applying a thermal gradient to a perfect single crystal7 or by concentration-gradient techniques, i.e., by growing a two-component crystal with graded composition along the growth axis.8 However, these two methods are energy consuming and do not assure the reproducibility needed for production of a large number of samples required by applications. A self-standing curved crystal can also be obtained by imparting controlled surface damage through a mechanical lapping process or by scratching one side of a crystal plate.9,10 However, these processes cause non-negligible damage in the crystal, generating into the bulk defects and cracks. Thick self-standing bent crystals were recently obtained through the deposition of a thick film composed of carbon fibre.11 However, this process does not permit producing small bent crystals, and thus it is not suitable for the applications where miniaturized samples are required.

Here, we propose to use ion implantation to produce self-standing bent crystals. Ion implantation has been used in the semiconductor industry for several decades.12 A drawback of this process for semiconductor manufacturing was the production of stress in the implanted material.13 This early disadvantage was later turned into a technology for the correction of stresses in thin films and substrates.Implanting high-energy ions into a substrate imparts compressive stresses, causing controllable deformation of the substrate. For example, ion implantation has been used for the correction of shape errors in X-ray stepper masks,14 X-ray mirrors,15 and MEMS deformable mirrors.16

In this paper, we describe a macroscopic monocrystalline Si plate uniformly bent by ion implantation, with its curvature being self-standing. The sample is 10 × 10 mm large and 0.2 mm thick. Si is an anisotropic material, thereby the deformation due to ion implantation may result in a

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nonuniform curvature even if the induced stress field was uniform. Nevertheless, (111) Si lattice planes have isotropic elastic constants. For this reason, a (111) oriented wafer was chosen for the production of the sample.

The sample was implanted using He\(^+\) at the INFN Laboratories of Legnaro (Padova, Italy). Helium ions were accelerated to an energy of 150 keV and directed normally toward the sample surface. The current density of the ion beam was 1 µA/cm\(^2\). The beam flux was found to be temporally and spatially uniform within 5% over an area of 150 mm of diameter. The dose implanted into the sample resulted 2 × 10\(^{16}\) atoms/cm\(^2\).

He\(^+\) was chosen because light ions interact with the substrate nuclei only near the stop point, thereby minimizing the lattice damage. Indeed, considering the mass and the initial energy of the ions, the main mechanism through which the ions lost energy was the interaction with the electrons.\(^\text{17}\) SRIM\(^\text{18}\) simulations indicate for 150 keV He\(^+\) ions in Si an initial electronic stopping of about 250 eV/nm and a nuclear stopping of 2 eV/nm. During this slowing-down process, ions are deflected very little and move in an almost straight line, causing few dislocations in the crystalline lattice. The energy lost per unit of path is described by the Bragg curve,\(^\text{19}\) which has a peak near the final point of the trajectory where the ion velocity is low and the transferred momentum is maximum. At this point, the interactions of the implanted ions with the nuclei of the substrate become significant and the number of dislocations in the crystalline lattice increases. Under the conditions of implantation aforementioned, the depth distribution of implanted He\(^+\) ions was simulated through the program SRIM (see Fig. 1). The ions projected range resulted \(R_p = 0.88 \mu m\) with a straggling of \(\sigma = 0.14 \mu m\). When stopped, the ions cause the amorphization of the substrate and thus its swelling.\(^\text{20}\) As a result, the implantation process is capable of imparting a sub-surface stress, creating a tensile layer buried in the substrate structure.\(^\text{21}\)

The buried layer of amorphous material extends below the sample surface within \(\pm 3\sigma = 0.84 \mu m\). This layer represents 0.42% of the total volume of the crystal, namely, it is a very thin layer. For this reason, the crystals can be considered “defect-free” for the applications that require a high lattice quality of the crystal bulk.

Fig. 2(a) shows the deformation of the sample as measured using an optical profilometer (VEECO NT1100). The sample resulted uniformly bent with a curvature radius of 10.5 ± 1.0 m. This curvature is the largest achieved in literature for a macroscopic Si sample implanted with ions under the MeV energy.

The amorphous-Si phase is metastable and may transform into crystalline-Si. The transformation rate is strongly dependent on temperature and presents an Arrhenius-like behaviour with an activation energy of 2.7 eV. At temperatures below ∼300 °C, the amorphous to crystal transition is kinetically inhibited.\(^\text{22}\) Thus, to prove the stability of the obtained curvature, we submitted the sample to a 300 °C annealing process for 3 h long and then we measured again the curvature, founding it unchanged. This operation was performed at SSL using a Lenton ECF 12/6 chamber furnace.

Since the amorphized portion of the sample acts as a tensile film, the Stoney formalism for an equi-biaxial plane stress regime can be applied\(^\text{23}\)

\[\sigma_f = \frac{E_s}{6(1 - \nu_s)} \frac{h_s}{h_f} \frac{1}{R} \]

(1)

where \(h_s\) and \(h_f\) are the thickness of the substrate and of the tensile film, respectively. \(E_s\) and \(\nu_s\) are the Young’s modulus and Poisson’s ratio of the substrate, \(\sigma_f\) is the film tensile stress, and \(R\) is the local radius of curvature. Since the exact value of the film thickness is not known, the tensile force per unit length in the film \(S = \sigma_f h_f\) has been used in the computation. Since \(R = 10.5 \text{ m}\), using the Stoney’s formula it can be inferred that ion implantation induced an integrated stress \(S = 145.4 \pm 14.0 \text{ Pa} \times \text{m per unit length}\).

The effect of ion implantation was then simulated through Straus7 finite element (FE) package.\(^\text{24}\) An equivalent Si layer 1 µm thick and with a tensile stress of 145.4 MPa bonded to a Si crystal 10 × 10 × 0.2 mm with the same crystallographic orientation as the manufactured sample was simulated. A net and spherical curvature was obtained, with the radius of curvature being 10.7 m (see Fig. 2(b)).

FIG. 2. Square sample bent to a spherical curvature using ion implantation. The surface that did not undergo the implantation process is displayed. (a) Morphological surface of the sample measured through interferometric profilometry. (b) Same sample simulated through FE analysis.

FIG. 1. Depth distribution of implanted He\(^+\) ions in a monocrystalline Si substrate simulated through the program SRIM.
The effect of ion implantation was finally analytically calculated through AntiCryDe,\textsuperscript{25} imposing a couple of perpendicular moments

\[ M_{[110]} = M_{[211]} = \sigma_f h_f h_y \frac{h_x + h_y}{2}, \]  

namely, 0.0146 N × mm per unit length to the crystal plate. In this case, the radius of curvature turned to be 10.4 m.

The depth reached by the implanted ions is very small compared to the crystal thickness, that is, 200 μm. Then, defects and dislocations do not affect the crystal bulk. In order to verify that the whole crystal structure is not altered and uniformly bent, the sample was tested by X-ray diffraction using a beam that passed through the entire crystal thickness, namely, in transmission (Laue) geometry. The characterization was carried out by performing rocking curves (RCs), i.e., by recording either the transmitted and diffracted beam intensity, while the crystal was being rotated around the position where the Bragg condition was satisfied. The ratio between diffracted and transmitted photons is called diffraction efficiency.

Curved crystalline planes were selected for the diffraction experiment because, in this case, the RCs contain information related to the crystalline quality of the bulk. Indeed, the full width at half maximum (FWHM) of the RCs for a perfect bent crystal is equal to the angle subtended by the curved diffracting planes, since the Bragg condition is met within the angular range defined by the diffracting plane curvature. If the crystal quality is deteriorated, the RCs would result broadened and the diffraction efficiency would decrease with respect to the theoretical value.\textsuperscript{26} (311) planes were chosen because they are the curved lattice planes in Laue geometry with the highest diffraction efficiency. Here, (311) planes are in asymmetric configuration, holding an asymmetry angle \( \phi = 58.52^\circ \) from the (111) surface.

Characterizations were performed at beamline ID15A of ESRF (Grenoble, France). A highly monochromatic and collimated beam was tuned to 150 keV by a two-reflection Laue Si (111) unbent monochromator. The monochromaticity was \( \Delta E/E = 2 \times 10^{-3} \), and the beam size was 50 × 50 μm. Diffraction analysis is shown in Fig. 3. The expected diffraction efficiency was calculated taking into account an ideal bent crystal. The good agreement between experimental data and theoretical expectations indicates that the crystallographic planes are homogeneously bent and the crystallographic quality preserved.

As an elective application of ion implantation to obtain a deformed crystal, we envisage the fabrication of a CU. A CU consists of a crystal whose planes are periodically bent with an amplitude much larger than the interplanar spacing. Such an undulator can be exploited as a generator of electromagnetic radiation by ultra-relativistic positrons channeling in the undulated planes of the CU.\textsuperscript{4} Indeed, ion implantation can be used to produce precise bending of perfect crystals, leaving the bulk substantially defect-free, which is a necessary condition for channeling experiments. Moreover, ion implantation can be combined to photolithographic techniques, in order to produce micrometric pattern of implanted regions.

Here, we numerically simulated a Si CU, 5 mm long, 0.2 mm thick, and 1 mm wide; the undulating period was 1 mm. The same CU was realized through the grooving method and successfully tested using a proton beam.\textsuperscript{27} These parameters fulfill the condition for an optimal undulator in the case of 15 GeV positrons.

The Si crystal was modelled through 50 000 brick elements, whereas the implanted layers were modelled through 5000 membrane elements of 1 μm thick. The FE analysis was initialized with the same parameters of the simulation shown in Fig. 2(b), i.e., a tensile pre-stress of 145.4 MPa was imparted to the membrane elements in order to simulate the coactive stresses generated by the implantation process. The output of the simulation is shown in Fig. 4(a). In Fig. 4(b), it is plotted the displacement along the \( x \) axis of the crystallographic planes in the \( xz \) plane at \( x = y = 0 \). Fig. 4(b) shows the displacement taking into account 6 different \( x \) positions, starting from the centre of the sample (\( x = y = 0.0 \) mm) to the crystal surface (\( x = 0.1 \) mm, \( y = 0.0 \) mm), with step 0.02 mm. The undulating amplitude results to be 4.11 ± 0.07 nm, where the uncertainty refers to different \( x \) position considered.

The undulating amplitude was also analytically calculated taking into account a stress \( \sigma = 145.4 \) MPa. In order to assess the out-of-plane deformation of the CU analytically, a plane strain regime has been assumed, since the width of the CU is 5 times larger than its thickness. Moreover, no deformation along \( y \) is expected to occur at \( y = 0 \) mm due to symmetry. For these reasons, a 2D strain state is expected to be adequate to calculate the CU deformation along the \( x \) direction at \( y = 0 \) mm. The amplitude resulted 3.92 nm.

The two results are very similar. Once built an undulator by ion implantation, it will be possible to verify which model is more predictive. In any case, the good correlation between the two theoretical models indicates a positive match with the experimental data. Moreover, the geometrical acceptance

![Graph](image-url)
of the simulated CU is its whole thickness, namely, 0.2 mm. On the contrary, the active thickness for an undulator realized through the grooving method is less than the CU thickness, because the grooves make a portion of the crystal not exploitable for channeling.

Concluding, ion implantation has been demonstrated to be an elective method to produce self-standing bent crystals. Deformation has demonstrated to be stable until 300 °C, which is a temperature high enough for the applications that involve bent crystals. In particular, this temperature would permit to safely operate with a crystalline undulator. Moreover, ion implantation leaves the crystal bulk substantially free from defects, since the superficial amorphized layer is very small with respect to the entire crystal volume, namely, less than 1%.

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