Fabrication of a Bragg beam splitter for hard x-ray free-electron lasers

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Abstract. We report a fabrication method of an ultrathin silicon crystal as a beam splitter for the hard x-ray regime based on the Bragg diffraction operated in the symmetric Bragg geometry, and evaluation results of crystalline perfection at SPring-8. A sub-10-μm thick Si(511) crystal was fabricated with a reactive dry etching method using atmospheric-pressure plasma. Following the evaluation of topography and diffractometry, the crystal was found to be strain-free, and capable of splitting a monochromatic x-ray beam into two branches with almost 1:1 splitting ratio.

1. Introduction

Recently, the Linac Coherent Light Source (LCLS) [1] at the SLAC National Accelerator Laboratory and SPring-8 Angstrom Compact free electron LAser (SACLA) [2] started operation of hard x-ray free-electron lasers (XFELs), which provide brilliant and coherent femtosecond x-ray pulses. Innovating XFEL applications require the development of new optical devices, in particular, a broad range of opportunities exist for developing beam splitters. For example, beam splitters are essential components in split-delay optics, that is, an autocorrelator that can be used to control the time delay between two XFEL pulses with extremely high accuracy up to the attosecond range.

Bragg beam splitters based on the Bragg diffraction operated in the Bragg geometry is useful to avoid deteriorating wavefronts and temporal structures of two replica pulses. In addition, the Bragg angle is larger than the critical angle of total reflection, which facilitates to provide larger delay time for a crystal-based autocorrelator [3] than that for a mirror-based one [4].

To date, there were only a few reports of Bragg beam splitters because of the difficulties in fabrication processes. The most severe requirement is that the crystal must be sufficiently thin to decrease absorption in the refraction branch without lattice bending, because thin crystals are easily bent by weak stresses due to holding or residual strain. Although ultrathin near-perfect silicon single crystals are available using the silicon-on-insulator technology, the crystal orientation is limited to the (111) [5]. In this paper, we report a new method to fabricate ultrathin Si single crystals as Bragg beam splitters by a reactive dry etching method using atmospheric-pressure plasma, and evaluation results with coherent x-rays at SPring-8.
2. Fabrication

Our target is to fabricate thin and bending-free crystals with uniform thickness distributions over the illuminated area. Towards this goal, we designed the beam splitter as described below. To avoid bending, a frame-shaped crystal was designed, as shown in Fig. 1. The thin area for beam splitting is supported by a thick tapered frame. This taper angle sets the minimum Bragg angle $\theta_{\text{min}}$ for which the transmission beam is usable. The corresponding maximum photon energy $E_{\text{max}}$ is given by $E_{\text{max}} = \frac{hc}{2d \sin \theta_{\text{min}}}$, here $h$ is the Plank's constant, $c$ is the speed of light, and $d$ is the lattice spacing of the reflection plane. We applied an angle of $20^\circ$ for the taper angle, which corresponds to $E_{\text{max}}$ of 17.34 keV for the (511) reflection.

![Fig. 1](image1)

Fig. 1 Schematic of a frame-shaped crystal.

The crystals were fabricated by a combination of mechanical grinding and plasma chemical vaporization machining (PCVM) [6] that is a reactive dry etching method using atmospheric-pressure plasma. Owing to the high pressure, ions with low kinetic energy and short mean free path are generated in the plasma, which avoids inducing lattice strains. Another advantage of this method is a capability to localize the processing area to within the diameter, down to 500 $\mu$m. By using fluorine radicals, we empirically found that PCVM etches isotropically with a processing speed that is independent of crystal orientation, e.g., (111) or (511).

The fabrication process consists of the following elements. First, the frame-shaped figure is roughly prepared by mechanical grinding from a 1.5-mm-thick silicon plate. Next, PCVM is applied to remove the damaged layer created during the mechanical grinding and reduce the thickness of the thin area. Finally, variations in thickness were corrected with computer-controlled raster scanning of the highly localized plasma. Note that the corrected thin area has a rectangular shape because of the raster scanning whereas the initial thin area has a circular shape. More details are shown in [7]. As samples, we fabricated a sub-10-$\mu$m thick (511) crystal with a surface roughness of below 0.15 nm rms in a 64 $\mu$m $\times$ 48 $\mu$m area on both sides.

![Fig. 2](image2)

Fig. 2 Schematic of experimental setup. See text.
3. Evaluation method
To evaluate the degree of crystalline perfection, we used plane-wave topography and diffractometry measurements at the 1-km-long beamline BL29XUL of SPring-8 [8] with 8.39 keV photons. A schematic of the setup is shown in Fig. 2, which is similar to that employed by Sutter et al. [9]. First, the x-ray beam from an undulator is reflected by a high-heat-load Si(111) double crystal monochromator (DCM). Then, the x-ray beam is monochromatized with a four-bounced Si(444) reflection ($\theta_B = 70.5^\circ$) arranged in the (+, −, −, +) geometry in experimental hutch 1 (EH1), 52-m downstream from the source. We achieved high monochromatization with an energy resolution $\Delta E / E = 4.8 \times 10^{-6}$ [which was smaller than the intrinsic energy width of $8.3 \times 10^{-6}$ for the (511) reflection]. Furthermore, we accomplished high angular resolution (less than 1 $\mu$rad) at experimental hutch 3 (EH3) owing to the 1 km beam transport. The x-ray probe was introduced on the samples at the Bragg angle $\theta_B = 45.0^\circ$. In this scheme, both the reflected and refracted x-ray-beam intensities were detected by PIN photodiodes PIN1 and PIN2, respectively. Topographs of the reflected x-rays were taken by a CCD camera with 6 $\mu$m $\times$ 6 $\mu$m pixel size.

4. Results and Discussion
Measured topograph on the Bragg condition and rocking curve are shown in Fig. 3. The topograph (Fig. 3a) shows almost uniform intensity distribution in the corrected thin area, which indicates the crystal does not have any lattice bending nor defects. Although Fig. 3a also shows two black regions originated by the refraction effect at the upper and lower edges of the corrected thin area, they do not critically influence the beam splitting performance because they exist at sufficiently far area from the center of the corrected thin area. Furthermore, the rocking curve (Fig. 3b) agrees well with the calculations. Fringes in the rocking curve are called *Pendellösung* beats that are observed for a thin crystal because of interference between x-rays reflected from the entrance and the backside surface [10]. The period of the *Pendellösung* beats susceptibly depends on the crystal thickness. The thickness of the crystal at the illuminated area was evaluated to be 8.1 $\mu$m. The splitting ratio for almost monochromatic x-ray beam was approximately 5:4. Thickness variation over the corrected thin area was measured to be 8.0–9.2 $\mu$m. Although the thickness variation is not fully suppressed, we found

![Fig. 3](image-url)
that the variation in an area illuminated by XFEL pulse (400–600 μm in the optical direction) is up to 500 nm peak-to-valley (P-V), which leads reflection intensity variation and wavefront distortion of transmission XFELs up to 10% and 1/30 λ, respectively. The thickness variation will be corrected to below 100 nm P-V by sophisticating fabrication process. Quantitative evaluation of crystalline perfection is difficult from these results due to non-uniform incoming x-rays and uncertain symmetric property of the reflection plane while the crystal has excellent crystalline perfection qualitatively. In the near future, we will investigate thermal effect under the FEL condition.

The Si(511) crystals are useful in an autocorrelator for the hard x-ray regime [3]. However, the use of the (511) reflection leads a significantly low throughput below 0.4% for the broad bandwidth of SASE XFEL pulses (ΔE/E > 10−3). A lower order reflection planes, such as (422) or (331) with Darwin widths of 1.38 × 10−5 and 1.34 × 10−5 in energy domain, respectively, may be considered to achieve higher throughputs. The present method, PCVM, has potential application to fabrication of Bragg beam splitters with these reflection planes.

Acknowledgements
This study was partially supported by the Proposal Program of SACLA Experimental Instruments of RIKEN and the Global COE Program from the Ministry of Education, Sports, Culture, Science and Technology, Japan. The use of BL29XUL at SPring-8 was supported by RIKEN.

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