Damage study of optical substrates using 1-µm-focusing beam of hard X-ray free-electron laser

T Koyama[1], H Yumoto[1], Y Senba[1], K Tono[1], T Sato[2], T Togashi[1], Y Inubushi[2], J Kim[3], T Kimura[4], S Matsuyama[3], H Mimura[5], M Yabashi[2], K Yamauchi[3], H Ohashi[1], T Ishikawa[2]

[1]JASRI/SPring-8, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan
[2]RIKEN SPring-8 center, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5148, Japan
[3]Department of Precision Science & Technology, Faculty of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan
[4]Research Institute for Electronic Science, Hokkaido University, Kita 21 Nishi 10 Kita-ku, Sapporo 001-0021, Japan
[5]Graduate School of Engineering, The University of Tokyo, 7-3-1, Hongo, Bunkyo-ku, Tokyo 113-8656, Japan

E-mail: koyama@spring8.or.jp

Abstract. We evaluated the ablation threshold of silicon and synthetic fused silica, which are widely used as optical substrates such as those in X-ray mirrors. A focusing XFEL beam with a beam size of approximately 1 µm at a photon energy of 10 keV was used. We confirmed that the ablation thresholds of these materials, which were 0.8 µJ/µm^2 for the silicon and 4 µJ/µm^2 for the synthetic fused silica, approximately agreed with the melting dose.

1. Introduction

X-ray free-electron lasers (XFELs), such as the Linac Coherent Light Source (LCLS) [1] and the SPring-8 Ångstrom Compact free electron LAser (SACLA) [2], deliver intense ultra-short pulses and fully transverse coherent photons in the hard X-ray region. Such intense beams possibly induce damage in optical elements, which can be a serious problem with degraded beam quality. Deformation of the surface of optical elements, such as total reflection mirrors that function as focusing optics and/or low-pass filters and crystals of monochromators, causes particularly disturbing wavefronts of XFEL beams. It is important to obtain information on the damage properties of silicon and amorphous SiO_2, which are used as the substrates of total reflection mirrors.

Damage by FEL irradiation is very actively being investigated in the XUV region [3-5]. Irradiation tests in the hard X-ray region have also been reported to evaluate focusing beam properties [6] since the advent of XFEL facilities.

In this work, we investigated the ablation threshold of optical substrates by using focused an XFEL beam, which had sufficient power density to study ablation phenomena, to obtain knowledge on the damage properties of X-ray optics. The samples used in this work were silicon and synthetic fused silica (amorphous SiO_2), which are widely used substrates in X-ray mirrors.

2. Performance of focusing beam

The experiments were performed at the beamline BL3 of SACLA. Fully transverse coherent X-rays were generated from a 400-m long linear accelerator and a 120-m long undulator section. The SACLA source was operated at a mean pulse energy of 130 μJ, a pulse duration of 20 fs, and a pulse repetition rate of 10 Hz. An X-ray photon energy of 10 keV was chosen. Unwanted contributions of light from higher-order harmonics and gamma-rays were largely suppressed by passing it through a double-mirror system under a grazing incidence of 2 mrad in an optics hutch. The X-ray beam was collimated both vertically and horizontally by a four quadrant slit to a size that was slightly smaller than the focusing mirror aperture to prevent the mirror substrate edges from being irradiated. The focusing mirror consisted of two carbon coated elliptical mirrors aligned in a Kirkpatrick–Baez configuration. The mirror parameters are summarized in Table 1. The focusing mirrors were developed in collaboration with Osaka University, SPring-8, and JTEC Corp. High quality long X-ray mirrors (400 mm in length) were fabricated and their diffraction limited performance was confirmed [7, 8]. The focusing mirror system was located 115 m downstream from the exit of the undulator. The size of the incident beam in front of the focusing mirror was approximately 200 μm in diameter at full-width at half maximum (FWHM). The spatial acceptance of the focusing mirror was 600 × 600 μm^2, and reflectivity through the two mirrors was 97%. A knife-edge scanning method was applied to measure the focusing beam profile. A 200-μm diameter Au wire was used as the knife-edge. The beam intensity was reduced with a silicon attenuator during the scan to prevent the knife-edge surface from being deformed. The knife-edge was scanned step by step and 20 XFEL pulses were averaged in each step to reduce shot to shot fluctuations in the normalized intensity. The beam size was measured as 0.95 × 1.2 μm^2 at FWHM, as shown in figure 1. The pulse energy at the focal spot was available up to 100 μJ.

Table 1. Focusing mirror parameters.

Surface profile	Elliptical cylinder
Substrate material	Synthetic fused silica
Mirror substrate size	420 × 50 × 50 mm^3
Surface coating	Carbon 50 nm
Graz. incidence angle	(H) 1.5 mrad, (V) 1.55 mrad
Spatial acceptance	(H) 615 μm, (V) 632 μm
Focal length	(H) 1.55 m, (V) 2.00 m

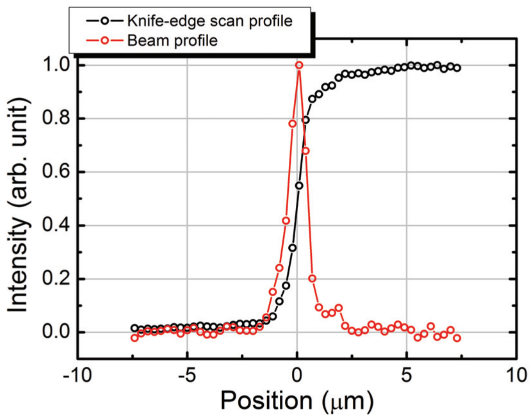

Figure 1. Typical beam profile measured with knife-edge scanning method in the horizontal direction.

3. Evaluation of ablation threshold

A series of focused single pulses irradiated the samples. Silicon and synthetic fused silica were used as the samples, which are widely used as optical substrates such as those in X-ray mirrors. The surface roughness of the silicon and the synthetic fused silica were 0.2 nm (rms) and 0.5 nm (rms). The pulse energy was controlled with silicon attenuators of various thicknesses placed in front of the focusing mirror. The random shot-to-shot fluctuations in the XFEL pulse energy were monitored by using a scattering based gas monitor [9]. A pulse energy ranging from 0.01 to 100 μJ was applied in this experiment. The ablation thresholds of the samples were evaluated by measuring the imprint diameters of the irradiated area [10] by means of scanning probe microscopy (SPM) and scanning electron microscopy (SEM).

Figure 2 shows imprint SEM images of irradiated samples of (a–c) silicon and (d–f) synthetic fused silica. The imprint diameters increased with increased fluence. Molten materials were ejected from the craters.

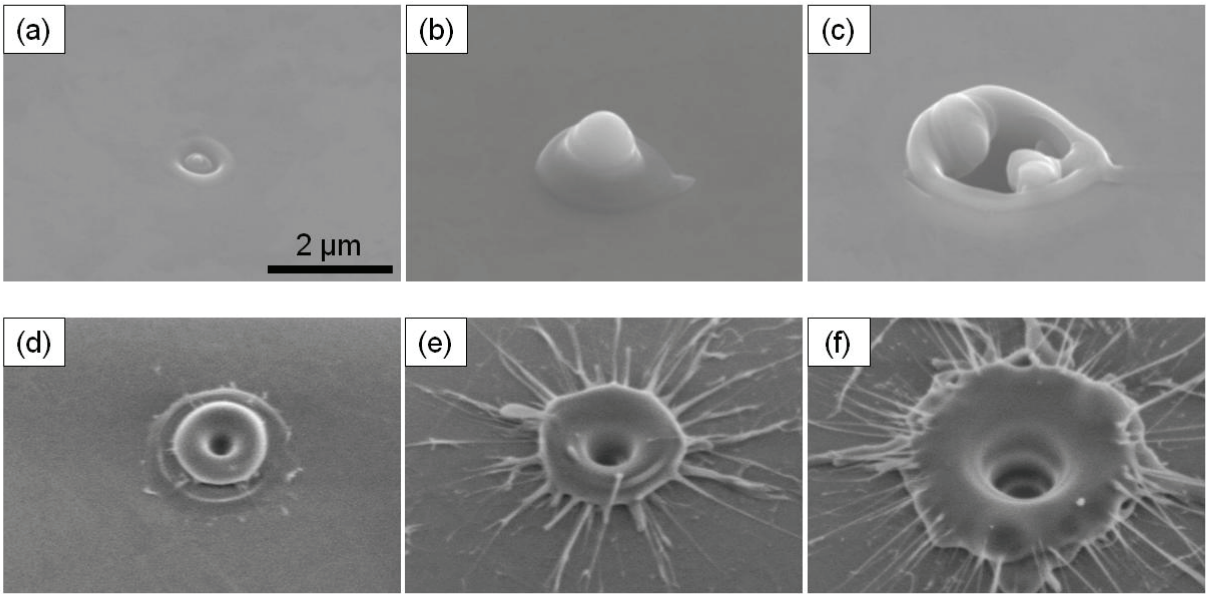

Figure 2. (a–c) Imprint SEM images of silicon under angle of 45° at fluence of 2.7 μJ/μm^2, 4.3 μJ/μm^2, and 6.7 μJ/μm^2. Imprint diameters were 1.1 μm, 2.4 μm, and 3.1 μm. (d–e) Imprint SEM images of synthetic fused silica under angle of 30° at fluence of 6.7 μJ/μm^2, 8.3 μJ/μm^2, and 12 μJ/μm^2. Imprint diameters were 1.7 μm, 2.4 μm, and 3.8 μm.

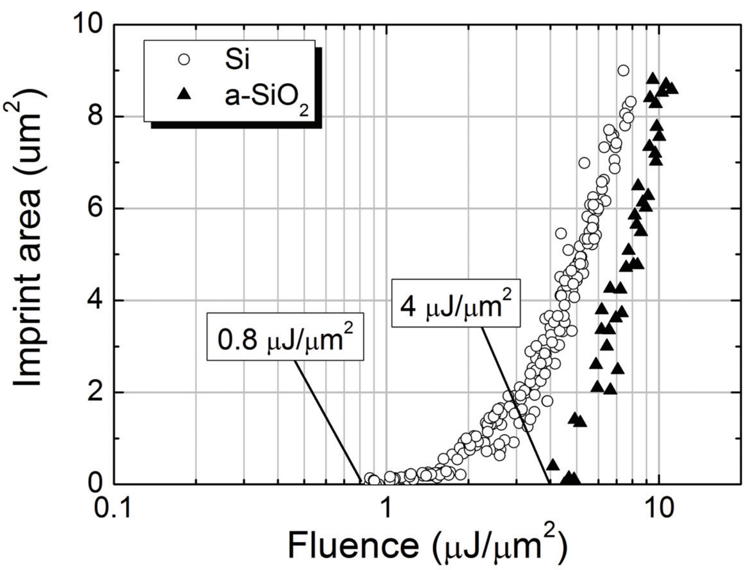

Figure 3. Imprint areas of silicon and synthetic fused silica (amorphous SiO_2) are plotted as a function of fluence. Imprint areas were measured by SPM.

The imprint areas are plotted as a function of fluence in figure 3. The ablation threshold of silicon was evaluated to be 0.8 μJ/μm^2, which was converted to the dose of a single atom [11, 12] as 0.8 eV/atom. This value was within the range of the calculated melting dose of 0.4–0.9 eV/atom. The melting dose of silicon was calculated from the thermodynamic properties [13], which took into consideration the temperature dependent heat capacity and the latent heat of melting. The ablation threshold of synthetic fused silica was evaluated to be 4 μJ/μm^2, which was converted to the dose of a single atom as 1.5 eV/atom. This value was close to the calculated melting dose of 1.1 eV/atom, which assumed a softening point of 1600°C.

We confirmed from these results that the ablation thresholds of silicon and synthetic fused silica approximately agreed with the melting dose in the experiment using a focusing XFEL beam at a photon energy of 10 keV.

4. Summary

We evaluated the ablation threshold of silicon and synthetic fused silica, which are widely used as optical substrates such as those in X-ray mirrors. We confirmed that the ablation thresholds of silicon and synthetic fused silica approximately agreed with the melting dose.

Acknowledgments
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