BL-13B/Beamline commissioning

In Situ Removal of Carbon Contamination from a Chromium-Coated Mirror: Ideal Optics to Suppress Higher-Order Harmonics in the Carbon *K*-Edge Region

Akio Toyoshima,[1] Takashi Kikuchi,[1] Hirokazu Tanaka,[1] Kazuhiko Mase,[1,2,*] and Kenta Amemiya[1,2]
[1]Institute for Materials Structure Science, High Energy Accelerator Research Organization 1-1 Oho, Tsukuba 305-0801, Japan
[2]SOKENDAI (The Graduate University for Advanced Studies), 1-1 Oho, Tsukuba 305-0801, Japan

Carbon-free chromium-coated optics are ideal in the carbon *K*-edge region (280–330 eV) because the reflectivity of first-order light is larger than that of gold-coated optics while the second-order harmonics (560–660 eV) are significantly suppressed by chromium *L*- and oxygen *K*-edge absorption. We adopted chromium-, gold-, and nickel-coated mirrors in the vacuum ultraviolet and soft X-ray branch beamline BL-13B at the Photon Factory. Carbon contamination on the chromium-coated mirror was almost completely removed by exposure to oxygen at a pressure of 8×10^{-2} Pa for 1 h under irradiation of non-monochromatized synchrotron radiation. The pressure in the chamber recovered to the order of 10^{-7} Pa within a few hours. The reflectivity of the chromium-coated mirror of the second-order harmonics in the carbon *K*-edge region (560–660 eV) was found to be a factor of 0.1–0.48 smaller than that of the gold-coated mirror [1].

1 Introduction

Carbon contamination of optics is a serious issue in synchrotron radiation (SR) facilities because it decreases the quality of experimental data such as near-edge X-ray absorption fine structure (NEXAFS), resonant photoemission, and resonant soft X-ray emission spectra in the carbon *K*-edge region. Recently, we developed an *in situ* method to remove carbon contamination from gold-coated optics in a vacuum ultraviolet and soft X-ray (VSX) undulator beamline [2]. Chromium-coated optics are better than gold-coated optics for the carbon *K*-edge region (280–330 eV) because of the larger reflectivity and because second-order harmonics (560–660 eV) are significantly suppressed by chromium *L*- and oxygen *K*-edge absorption.

Here we report on carbon removal from chromium-coated optics. Carbon contamination on a chromium-coated mirror was almost completely removed by exposure to oxygen at a pressure of 8×10^{-2} Pa for 1 h under irradiation of non-monochromatized SR. The reflectivity of the chromium-coated mirror was observed to be a factor of 1.3 larger than that of a gold-coated mirror in the carbon *K*-edge region (280–330 eV). On the other hand, the reflectivity of the chromium-coated mirror in the second-order harmonics region of the carbon *K*-edge (560–660 eV) was a factor of 0.1–0.48 smaller than that of the gold-coated mirror.

2 VSX Undulator Beamline, BL-13A/B

The VSX undulator beamline BL-13A at PF has a Monk–Gillieson-type monochromator to cover a wide photon-energy range at a high energy resolution [3]. Carbon contamination of the optics in BL-13A was removed almost completely by exposing the optics to oxygen gas at a pressure of 10^{-1}–10^{-4} Pa for 17–20 h under irradiation of non-monochromatized SR [2]. Recently, we developed a branch VSX beamline for surface chemistry (BL-13B, Fig. 1). A branching plane mirror (Mp) with a grazing angle of 2° is used to reflect SR to BL-13B. The Mp substrate is coated with 1000-Å-thick chromium, gold, and nickel layers as shown in Fig. 2. The chromium-, gold-, and nickel-coated mirrors can be easily selected by adjusting the z-position of Mp. Chromium-coated optics are ideal in the carbon K-edge region (280–330 eV) because of the large reflectivity and suppression of higher-order harmonics (Fig. 3). In contrast, nickel-coated optics are suitable in the nitrogen *K*-edge region (395–445 eV) because higher-order harmonics are suppressed (Fig. 3). The Mp and M3B/M3B' chambers are pumped with oil-free turbomolecular pumps and non-evaporable getter (NEG) pumps. No ion sputtering pumps are used because carbon-contaminated ion sputtering pumps are thought to produce hydrocarbons by collision with ionized residual hydrogen gas [4] Typical base pressures of the Mp and M3B/M3B' chambers are 3×10^{-7} and 4×10^{-8} Pa, respectively. Carbon contamination on the gold-coated Mp, M3B, and M3B' were removed with oxygen activated by non-monochromatized SR. Typical photon intensities and energy resolutions ($E/\Delta E$) of the BL-13A/13B are shown in Fig. 4.

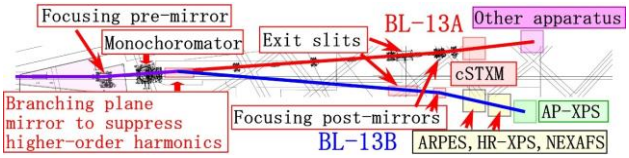

Fig. 1 Floor layout of BL-13A/B [1].

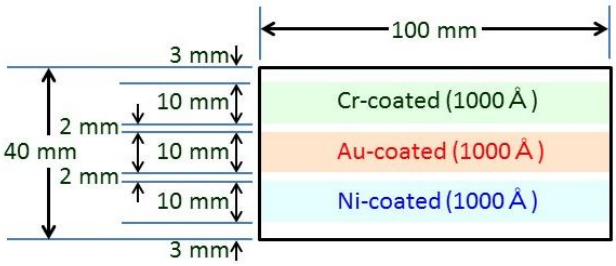

Fig. 2 Branching plane mirror to suppress higher-order harmonics [1].

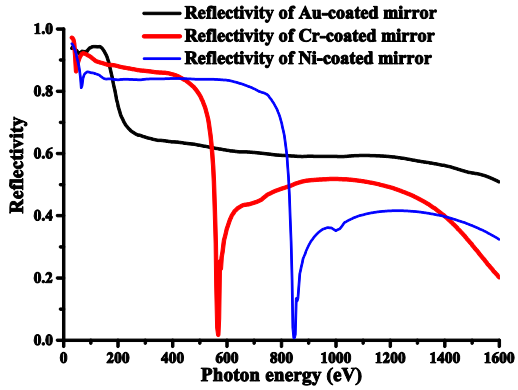

Fig. 3 Reflectivity of chromium-, gold-, and nickel-coated mirrors for the grazing angle of 2˚ and *p*-polarization calculated using the Center for X-Ray Optics website [http://henke.lbl.gov/optical_constants/mirror2.html] [1].

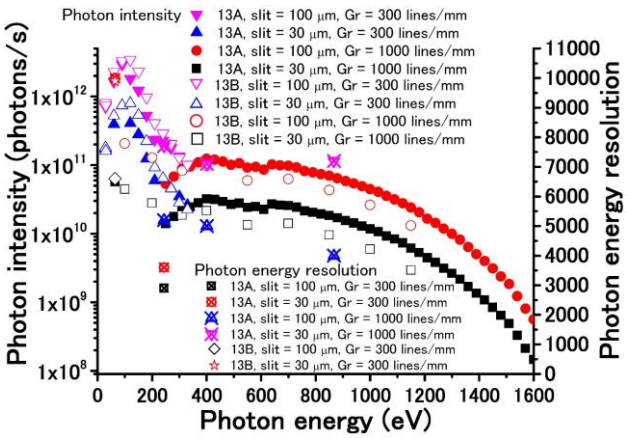

Fig. 4 Typical photon intensity and photon energy resolution ($E/\Delta E$) of the BL-13A [5] and 13B in the photon energy region of 30–1600 eV [1].

3 *In Situ* Carbon Removal from Chromium-Coated Mirror

Carbon contamination on the chromium-coated mirror was removed by exposure to oxygen at a pressure of 8×10^{-2} Pa for 1 h under irradiation of non-monochromatized SR. The pressure in the Mp chamber recovered to 10^{-7} Pa within a few hours after the carbon removal. Figure 5 shows photon intensity spectra of BL-13B in the carbon *K*-edge region before and after *in situ* carbon removal from the chromium-coated mirror. We ascribed the small negative peak at 285.5 eV before the carbon removal to the C $1s \rightarrow \pi^*$ transition of graphite-like carbon. After the carbon removal, the peak at 285.5 eV disappeared and the photon intensity in the carbon *K*-edge region (280–330 eV) increased. These results indicate that the carbon on the chromium-coated mirror has been almost completely removed. Since the photon intensity involves higher-order harmonics, the negative peaks at photon energies of 266, 288, and 292 eV are ascribed to oxygen *K*-, chromium L_{III}-, and chromium L_{II}-edge absorption at 532, 576, and 584 eV, respectively. The pressure in the chamber recovered to the order of 10^{-7} Pa within a few hours without baking. The beamline can be used without additional commissioning.

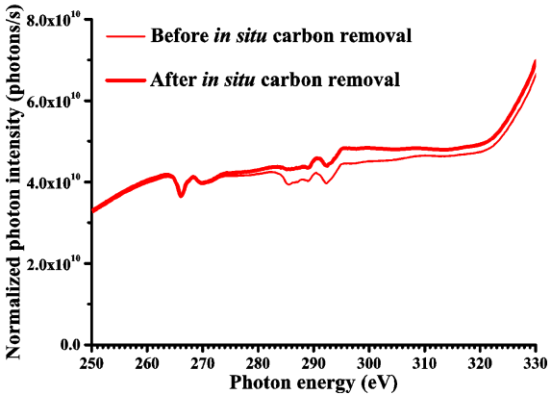

Fig. 5 Photon intensity spectra of BL-13B in the carbon *K*-edge region before and after *in situ* carbon removal from the chromium-coated mirror [1].

4 Comparison between Chromium- and Gold-Coated Mirrors

Figure 6 shows photon intensity spectra measured with a chromium-, gold-, or nickel-coated mirror in the photon energy regions of 250–330, 500–660, and 750–990 eV. The chromium L_{III}- and L_{II}-edge absorption at 576 and 584 eV, respectively, are also observed in the spectra measured with the gold- and nickel-coated mirrors (Fig. 6, dot-dashed lines) because chromium is used to improve the adhesion property between the substrate and the coating. Oxidized chromium seems to be responsible for the oxygen *K*-edge absorption at a photon energy of 532 eV observed in the spectra measured with the gold- and nickel-coated mirrors (Fig. 6, dashed line). Since the photon intensity involves higher-order harmonics, the peak at a photon energy of 282 eV in the spectra measured with the nickel- and gold-coated mirrors is assigned to the fifth-order undulator peak at 564 eV (Fig. 6, dotted line). The peaks at 282 and 564 eV are negligible in the spectra measured with the chromium-coated mirror, suggesting that 564-eV photons are mostly absorbed by oxidized chromium. The photon intensity in the third-order harmonics region of the carbon K-edge (840–990 eV) is one order of magnitude smaller than that

in the second-order harmonics region (560–660 eV) because the grazing angle is 2˚.

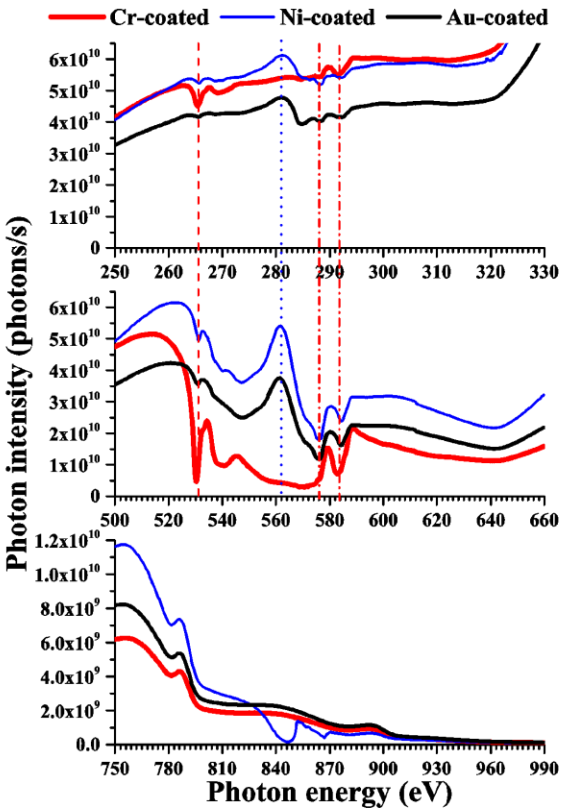

Fig. 6 Photon intensity spectra measured with a chromium-, gold-, or nickel-coated mirror in the photon energy regions of 250–330 (upper), 500–660 (middle), and 750–990 eV (lower). Higher-order harmonics are involved [1].

Figure 7 shows the ratio of the photon intensity with the chromium-coated mirror to that with the gold-coated mirror. The measured ratio differs from the calculated ratio because the surface chromium is oxidized and because the measured photon intensity involves higher-order harmonics. The photon intensity with the chromium-coated mirror is a factor of 1.3 larger than that of the gold-coated mirror in the carbon *K*-edge region (280–330 eV), while the former is a factor of 0.1–0.48 smaller than the latter in the second-order harmonics region (560–660 eV). These results demonstrate that chromium-coated optics are more advantageous than gold-coated optics in the carbon *K*-edge region.

5 Conclusions

We constructed a branch VSX undulator beamline BL-13B at the PF and opened it for users in October, 2013. Plane mirrors coated with gold, chromium, or nickel are used to branch SR. Carbon contamination on the chromium-coated mirror was removed by exposure to oxygen at a pressure of 8×10^{-2} Pa for 1 h under irradiation of non-monochromatized SR. The base pressure of the beamline recovered to 10^{-7} Pa in a few

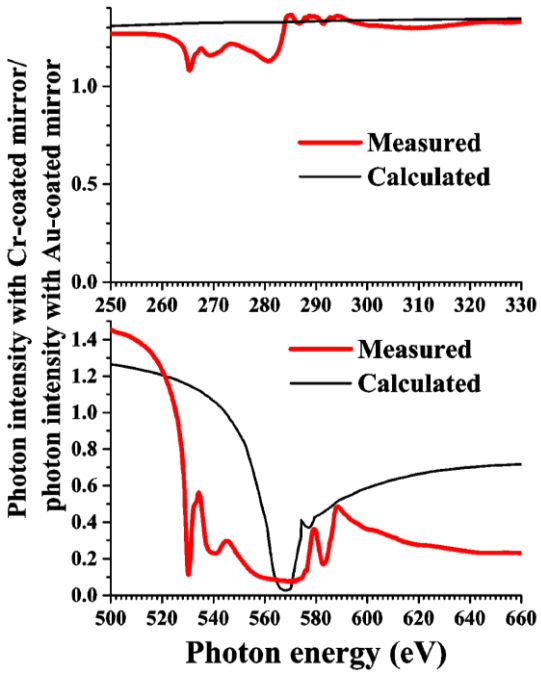

Fig. 7 Ratio of measured and calculated photon intensities with a chromium-coated mirror to those with a gold-coated mirror. Data in Figs. 3 and 6 are used. The measured ratio is different from the calculated ratio because the surface chromium is oxidized and because the measured photon intensity involves higher-order harmonics [1].

hours without baking. The beamline can be used without additional commissioning. The photon intensity with the chromium-coated mirror is a factor of 1.3 larger than that of the gold-coated mirror in the carbon K-edge region (280–330 eV), while the former is a factor of 0.1–0.48 smaller than the latter in the second-order harmonics region (560–660 eV). These results demonstrate that chromium-coated optics are ideal for experiments in which intense SR without higher-order harmonics is required in the carbon *K*-edge region.

Acknowledgement

We express our sincere thanks to the staff of the PF for their valuable support during the course of the experiments. This work was performed during the commissioning of BL-13B.

References

[1] A. Toyoshima *et al.*, *J. Synchrotron Rad.*, submitted.
[2] A. Toyoshima *et al.*, *J. Synchrotron Rad.* **19,** 722 (2012).
[3] K. Amemiya and T. Ohta, *J. Synchrotron Rad.* **11,** 171 (2004).
[4] R. Yamada *et al.*, *J. Nucl. Mater.* **111–112,** 744 (1982).

[5] Toyoshima *et al.*, *J. Phys.: Conf. Ser.* **425,** 152019 (2013).

*mase@post.kek.jp