

In situ Removal of Carbon Contamination from the Whole Optics in a Vacuum Ultraviolet and Soft X-Ray Undulator Beamline Using Oxygen Activated by Non-Monochromatized Synchrotron Radiation

We developed an *in situ* method to remove carbon contamination from the whole optics in a vacuum ultraviolet and soft X-ray (VSX) beamline. The carbon contamination of the optics was removed by exposing them to oxygen at a pressure of 10^{-1} - 10^{-4} Pa for 17–20 h and simultaneously irradiating them with non-monochromatized synchrotron radiation. After a series of operations to remove carbon contamination, the decrease in the photon intensity in the carbon *K*-edge region was reduced from 64–65% to 2–4%. The base pressure of the beamline recovered to 10^{-7} - 10^{-8} Pa in one day without baking. The beamline can be used without adjusting the optics.

When optics are irradiated with synchrotron radiation (SR) in the presence of residual hydrocarbon gases, they are contaminated with carbon [1]. The carbon contamination causes a significant decrease in the reflectivity of the optics. The photon flux loss in the carbon *K*-edge region is especially critical because it reduces the quality of experimental data, such as near-edge X-ray absorption fine structure (NEXAFS), resonant photoemission, and resonant soft X-ray emission spectra. Therefore, it is crucial to remove carbon contamination from optics in synchrotron radiation facilities. To solve this issue, we focused on an *in situ* SR-activated oxygen cleaning method proposed by Warburton and Pianetta [2]. Applying this method we succeeded in removing carbon contamination from the whole optics in a VSX undulator beamline, BL-13A [3]. BL-13A is equipped with a Monk-Gillieson-type monochromator to cover a wide energy range of 30–1600 eV [4, 5]. BL-13A consists of a focusing pre-mirror (M1), a plane mirror (M2), two varied-line-spacing plane gratings (VLSGs, 300 and 1000 line/mm), an exit slit, and two focusing post-mirrors (M3 and M3') as shown in Fig. 1. M1, M2, VLSGs, M3, and M3' are coated with gold. The base pressure of BL-13A is less than 1×10^{-8} Pa. However, the photon intensity in the carbon *K*-edge region had decreased by as much as 64–65% after 300 days of operation. The thickness of the carbon contamination on M1, M2, VLSGs, M3, and M3' was estimated to be 3–4

nm based on calculations of the reflectivity performed by using the Web page of the Center for X-Ray Optics (http://henke.lbl.gov/optical_constants/layer2.html).

In situ removal of carbon contamination from the optics was carried out four times. Figure 2 shows photon intensity spectra measured with the 300-line/mm VLSG and M3 before and after the third *in situ* removal of carbon contamination. For the spectrum before the carbon removal we ascribed the sharp dip at 285.1 eV to the $C 1s \rightarrow \pi^*$ transition of graphite-like carbon with a flat-on configuration on M1 and M3, and the sharp dip at 291.5 eV mainly to the $C 1s \rightarrow \sigma^*$ transition of graphite-like carbon with a flat-on configuration on M2 and the VLSG on the basis of the polarization-dependent NEXAFS spectra of graphite [6]. The oxygen pressures in the M1, M2/VLSG, and M3/M3' chambers were maintained in the ranges of 2×10^{-4} to 6×10^{-4} Pa, 5×10^{-3} to 1.5×10^{-2} Pa, and 1×10^{-5} to 4×10^{-5} Pa, respectively. M1, M2, the 300-line/mm VLSG, and M3 were irradiated with non-monochromatized SR for 20 h. The averaged ring current was 450 mA. The sharp dips due to the carbon contamination were reduced to 2–4% after the carbon removal as shown in Fig. 2. The base pressure of BL-13A recovered to 10^{-7} - 10^{-8} Pa in one day without baking. The carbon contamination on the 1000-line/mm VLSG and M3' was removed in a similar manner. BL-13A can be used without adjusting the optics.

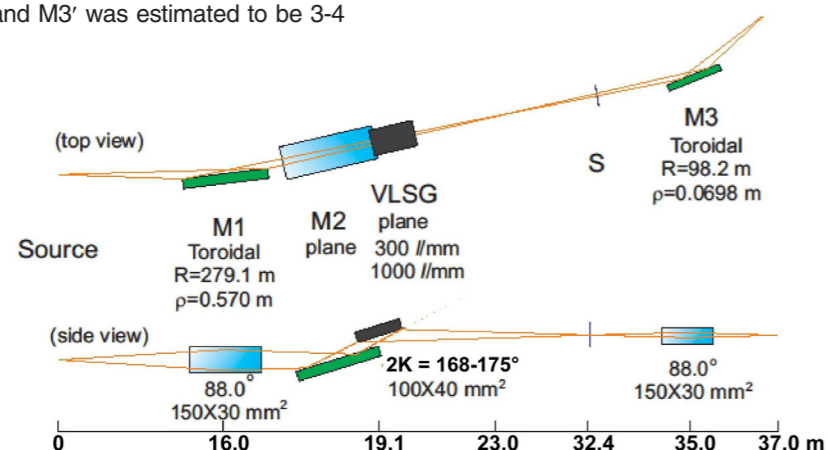


Figure 1: Optics of BL-13A.

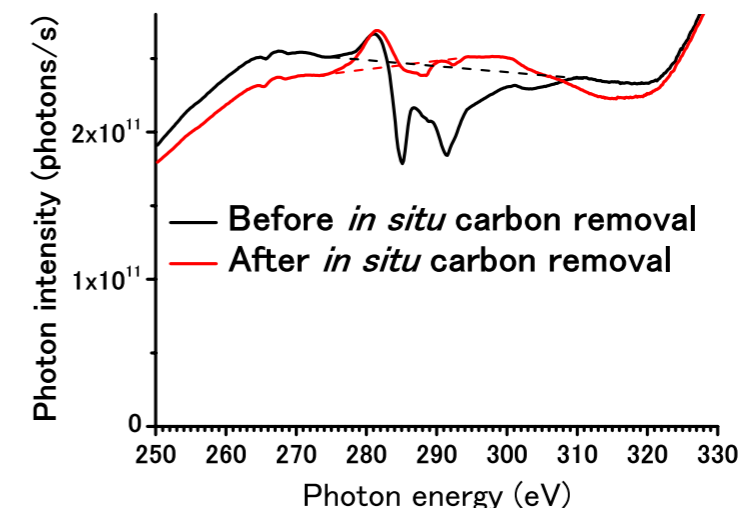


Figure 2: Photon intensity spectra measured before and after the third *in situ* carbon removal; the measurements were made with the 300-line/mm VLSG and M3 [3]. The dashed lines represent the baselines.

The key requirements for the present *in situ* carbon removal method are as follows. 1) The surface of the optics must be made of a material that is inert to activated oxygen. 2) The vacuum in the optics chambers must be hydrocarbon-free. The base pressure of the chambers should be less than 5×10^{-8} Pa. 3) A differential pumping system and an interlock system must be installed to prevent the degradation of the vacuum of the storage ring. 4) Oil-free turbomolecular pumps should be attached to the optics chambers to remove oxygen and reacted gases. 5) The recommended oxygen pressure is 10^{-3} - 10^{-4} Pa for the first mirror and 10^{-2} - 10^{-3} Pa for the other optics when the thickness of the carbon contamination is 3–4 nm. The typical SR irradiation time is less than 20 h. 6) Non-evaporable getter pumps should be used to facilitate the rapid recovery of the base pressure.

We propose the following model for the mechanism of the carbon removal. The first step is the interaction of oxygen molecules with electrons emitted from the optics under SR irradiation, generating active oxygen species such as oxygen atoms, excited oxygen molecules, and ions. Then the active oxygen species react with the carbon contamination and produce carbon oxides. The advantages of the present carbon removal method are as follows. 1) The carbon contamination of the whole

optics in a VSX beamline can be removed within 20 h. 2) The base pressure of the beamline can be recovered to 10^{-7} - 10^{-8} Pa in one day without baking. 3) The beamline can be used without adjusting the optics. 4) This method is especially suited for undulator beamlines because the carbon removal rate is expected to be proportional to the photon flux.

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BEAMLINE

BL-13A

A. Toyoshima¹, T. Kikuchi¹, H. Tanaka¹, J. Adachi¹, K. Mase¹ and K. Amemiya¹ (KEK-PF)