

In situ removal of carbon contamination from optics in a vacuum ultraviolet and soft X-ray undulator beamline using oxygen activated by zeroth-order synchrotron radiation

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Carbon contamination of optics is a serious issue in all soft X-ray beamlines because it decreases the quality of experimental data, such as near-edge X-ray absorption fine structure, resonant photoemission and resonant soft X-ray emission spectra in the carbon *K*-edge region. Here an *in situ* method involving the use of oxygen activated by zeroth-order synchrotron radiation was used to clean the optics in a vacuum ultraviolet and soft X-ray undulator beamline, BL-13A at the Photon Factory in Tsukuba, Japan. 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 zeroth-order synchrotron radiation. After the cleaning, the decrease in the photon intensity in the carbon *K*-edge region reduced to 2–5%. The base pressure of the beamline recovered to 10^{-7} – 10^{-8} Pa in one day without baking. The beamline can be used without additional commissioning.

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1. Introduction

When optics are irradiated with synchrotron radiation (SR) in the presence of residual hydrocarbon gases, they are contaminated with carbon (Boller *et al.*, 1983; Rosenberg & Mancini, 1990; Koida & Niibe, 2009). The carbon contamination leads to a significant decrease in the reflectivity in the vacuum ultraviolet and soft X-ray (VSX) region (Koide *et al.*, 1986, 1987*a,b*, 1988, 1989). The interference effects of the carbon contamination can cause strong reflectivity losses even at photon energies around 1000 eV (Chauvet *et al.*, 2011). 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. To reduce carbon contamination, VSX beamlines are usually kept in an ultrahigh vacuum (UHV) of 10^{-7} – 10^{-8} Pa. The contamination, however, is inevitable even though the base pressure is maintained under 1×10^{-8} Pa (Toyoshima *et al.*, 2011). To remove carbon contamination, several methods have been proposed, such as *in situ* cleaning with oxygen activated by zeroth-order SR (Warburton & Pianetta, 1992; Hamamoto *et al.*, 2005), *in situ* cleaning with oxygen activated by a 172 nm excimer lamp (Hamamoto *et al.*, 2005), *in situ* DC oxygen discharge (Koide *et al.*, 1986, 1987*a,b*, 1988, 1989), *in situ* plasma discharge in a mixture of oxygen and argon

gases (Eggenstein *et al.*, 2001) and atmospheric pressure ultraviolet (UV)/ozone cleaning (Harada *et al.*, 1991; Hansen *et al.*, 1993, 1994*a,b*). In the first method, *in situ* SR-activated oxygen cleaning, the optics are cleaned by exposing them to oxygen at 10^{-2} Pa for 15 h and simultaneously irradiating them with zeroth-order SR (Warburton & Pianetta, 1992). This method seems to be suitable for undulator beamlines because a large amount of SR-activated oxygen can be supplied owing to the high photon flux.

In the present paper we report results of *in situ* SR-activated oxygen cleaning of optics in a new VSX undulator beamline, BL-13A at the Photon Factory (PF) in Tsukuba, Japan. The carbon contamination of the optics was removed by exposing them to oxygen at 10^{-1} – 10^{-4} Pa for 17–20 h and simultaneously irradiating them with zeroth-order SR. The decrease in the photon intensity in the carbon *K*-edge region was reduced to 2–5%. Owing to the low pressure of oxygen, the base pressure of the beamline recovered to 10^{-7} – 10^{-8} Pa in one day without baking. The beamline can be used without additional commissioning.

2. Beamline

The VSX undulator beamline, BL-13A at the PF, has a Monk–Gillieson-type monochromator to cover a wide photon-energy range with a high energy resolution (Amemiya & Ohta, 2004).

The details of BL-13A have been described elsewhere (Mase *et al.*, 2010a; Toyoshima *et al.*, 2011). Briefly, it was constructed in 2009 for the study of organic thin films grown on surfaces, and it was opened for users on 29 January 2010 (Toyoshima *et al.*, 2011). BL-13A consists of a focusing pre-mirror (M1), a plane mirror (M2), two varied-line-spacing plane gratings (VLSGs, 300 and 1000 lines mm^{-1}), an exit slit, and two focusing post-mirrors (M3, 2 m:2 m focusing; M3', 2 m:6 m focusing) (Toyoshima *et al.*, 2011). The specifications of BL-13A are as follows: photon-energy region: 30–1200 eV; photon flux: 10^9 – 10^{11} photons s^{-1} ; photon-energy resolution ($E/\Delta E$): 10000 at a photon energy of 401 eV (Toyoshima *et al.*, 2011).

M1, M2, the VLSGs, M3 and M3' are coated with a gold film of thickness 1000 Å or 500 Å. M1, M2 and the VLSGs are cooled with water at room temperature (Toyoshima *et al.*, 2011). The surface roughnesses of M1, M2, M3 and M3' under the delivery conditions are 0.20, 0.32, 0.28 and 0.17 nm (root mean square, RMS), respectively. While the SR is horizontally reflected by M1, M3 and M3', it is vertically reflected by M2 and the VLSGs. The grazing angle is 2° for M1, M3 and M3', and the including angles are 174.9° and 171.3° for the 300 and 1000 lines mm^{-1} VLSGs at 290 eV. A planar undulator is used as the source point (Sasaki *et al.*, 1989). The undulator gap is fixed at 154 mm during cleaning and photon intensity measurements, unless otherwise stated. The first and third undulator peaks appear at 113 and 339 eV for this gap. The M1, M2/VLSGs and M3/M3' chambers are pumped with oil-free turbomolecular pumps (TMPs), non-evaporable getter (NEG) pumps, and ion sputtering pumps. Cold cathode vacuum gauges are used for monitoring the pressure in the chambers. The base pressure of every chamber is maintained below 1×10^{-8} Pa. Between the front end and the M1 chamber there are the following components: a quadruple-mask chamber, masks, a square duct, movable water-cooling quadruple masks, a photon position monitor, and a shutter chamber (Mase *et al.*, 2010b; Tanaka *et al.*, 2011). These components constitute an efficient differential pumping system. Even when the M1 chamber is filled with oxygen gas at 9×10^{-4} Pa, the pressure in the front end is 1.3×10^{-7} Pa. Therefore, the change in the vacuum level in the storage ring was negligible during *in situ* SR-activated oxygen cleaning.

3. Results and discussion

3.1. *In situ* SR-activated oxygen cleaning of M2, VLSGs, M3 and M3'

First, we carried out *in situ* SR-activated oxygen cleaning of M2, the VLSGs, M3 and M3'. Dry oxygen with a purity of 99.9% was supplied by opening a variable leak valve attached to a UHV chamber placed downstream of the M3/M3' chamber. This geometry is suitable for cleaning the VLSGs because the long narrow duct between the M2/VLSG and M3/M3' chambers guides the oxygen beam to the VLSGs. The oxygen pressures in the M1, M2/VLSG and M3/M3' chambers were in the range 1×10^{-6} to 3×10^{-6} Pa, 1×10^{-4} to 3×10^{-4} Pa and 1×10^{-1} to 2×10^{-1} Pa, respectively. M1, M2, the

300 lines mm^{-1} VLSG and M3 were irradiated with zeroth-order SR for 19 h. The averaged ring current was 315 mA.

Fig. 1(a) shows photon intensity spectra measured with the 300 lines mm^{-1} VLSG and M3 before and after *in situ* SR-activated oxygen cleaning. The exit slit was fixed at about 30 μm to realise the best energy resolution during the measurements, unless otherwise stated. The photon intensity was measured by using a silicon photodiode (AXUV-100; International Radiation Detectors). The uncertainty in the photodiode measurement was 22% (communication from International Radiation Detectors). The higher-order light was not taken into account to determine the photon intensity. The photon energy was calibrated on the basis of the carbon *K*-edge absorption spectrum of gaseous CO_2 (Adachi *et al.*, 2005). The mirror alignments were the same before and after the cleaning.

The photon intensity spectra reflect the NEXAFS of the optics. The sharp dips at 285.1 and 291.5 eV in the photon intensity spectrum before the cleaning suggest that the carbon contamination has an ordered structure, because the NEXAFS of amorphous carbon show more broad peaks (Tang *et al.*, 2001). Graphite-like carbon is likely to be responsible for these dips because graphene layers are known to grow on metal surfaces with a flat-on configuration through the surface decomposition of carbon-containing molecules (Wintterlin & Bocquet, 2009). Since SR emitted from a planar undulator is linearly polarized in the horizontal plane, the incident SR is *p*-polarized for M1, M3 and M3' and *s*-polarized for M2 and the VLSGs. On the basis of the NEXAFS spectra of graphite

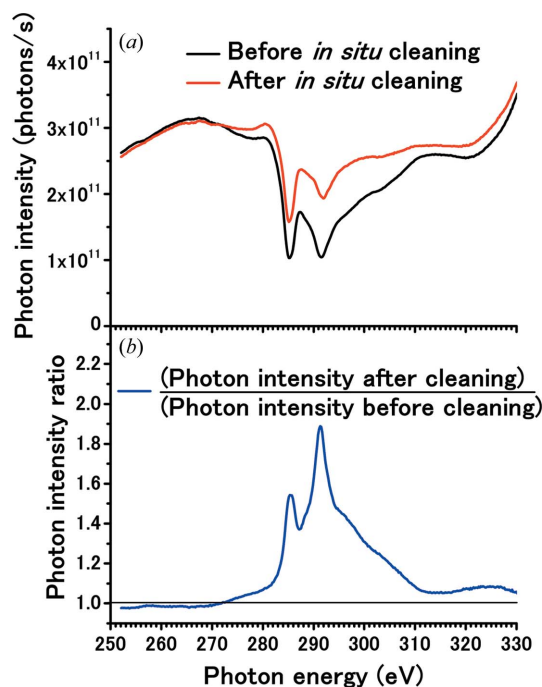


Figure 1
(a) Photon intensity spectra measured with the 300 lines mm^{-1} VLSG and M3 before and after *in situ* SR-activated oxygen cleaning of M2, the VLSGs, M3 and M3'. The photon intensity is normalized for a ring current of 450 mA. (b) The ratio of the photon intensity after the cleaning to that before the cleaning.

(Rosenberg *et al.*, 1986) 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. The dip at 291.9 eV, which remained after the cleaning, indicates that the oxygen pressure of 1×10^{-4} to 3×10^{-4} Pa for 19 h was not enough to remove oxygen contamination of M2 and the 300 lines mm^{-1} VLSG.

We estimated the thickness of the carbon contamination on the optics before the cleaning on the basis of the data in Fig. 1(a) and calculations of the reflectivity performed by using the Center for X-ray Optics web page (http://henke.lbl.gov/optical_constants/layer2.html). Since the oxygen pressure in the M3 chamber was high, the carbon contamination on M3 was assumed to be almost completely removed after the cleaning. Thus, the 46% dip at 285.1 eV, which remained after the cleaning, can be ascribed to the carbon contamination of M1. We estimated the thickness of the carbon contamination on M1 to be about 4 nm on the basis of the calculated reflectivity. Since the 65% dip at 285.1 eV before the cleaning can be ascribed to the carbon contamination of M1 and M3, the thickness of the carbon contamination on M3 was estimated to be about 3 nm. The cleaning rate for M3 was estimated to be more than 0.15 nm h^{-1} . Since the 64% dip at 291.5 eV before the cleaning can be ascribed to the carbon contamination of M2 and the VLSG, the averaged thickness of the carbon contamination on M2 and the VLSG was estimated to be about 3 nm. After the cleaning, a 33% dip appeared at 291.9 eV. The slight shift in the dip position indicates that the chemical state of the upper part of the carbon contamination was slightly different from that of the remaining part. The cleaning rate was estimated to be about 0.06 nm h^{-1} for M2 and the VLSG.

Fig. 1(b) shows the ratio of the photon intensity after the cleaning to that before the cleaning. This ratio spectrum reflects the NEXAFS of the carbon contamination removed from the optics. The ratio spectrum in Fig. 1(b) resembles the NEXAFS spectrum of graphite at the incidence angle of 0° (Rosenberg *et al.*, 1986). This result provides evidence for our conclusion that the carbon contamination on M2 and the VLSG is composed of graphite-like carbon with a flat-on configuration. The ratio reaches 1.54 at 285.5 eV and 1.89 at 291.3 eV.

Fig. 2(a) shows photon intensity spectra measured with the 1000 lines mm^{-1} VLSG and M3' before and after *in situ* SR-activated oxygen cleaning. The oxygen pressures in the M1, M2/VLSG and M3/M3' chambers were in the range 1×10^{-5} to 7×10^{-5} Pa, 2×10^{-3} to 1.2×10^{-2} Pa and 1×10^{-1} to 2×10^{-1} Pa, respectively. M1, M2, the 1000 lines mm^{-1} VLSG and M3' were irradiated with zeroth-order SR for 17 h. The averaged ring current was 350 mA. The photon intensity dip at 291.9 eV almost disappeared after the *in situ* cleaning. These results indicate that *in situ* irradiation with zeroth-order SR in oxygen at 2×10^{-3} to 1.2×10^{-2} Pa for 17 h was sufficient to remove carbon contamination from M2 and the VLSGs. The dip at 285.1 eV improved from 51% to 43% after the *in situ*

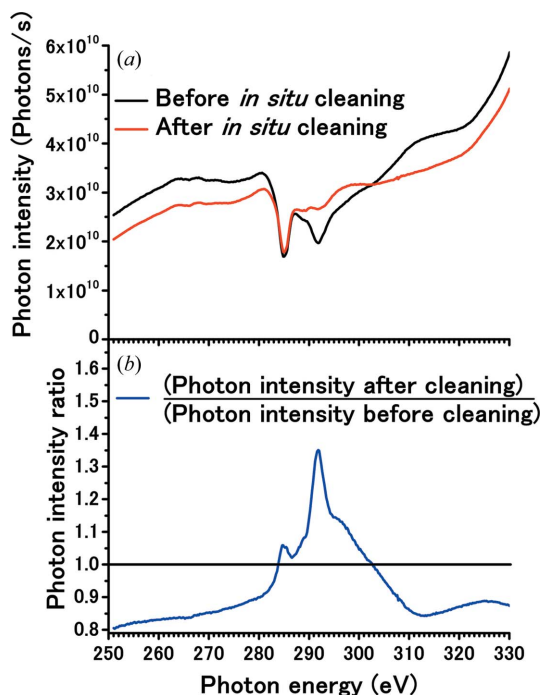


Figure 2

(a) Photon intensity spectra measured with the 1000 lines mm^{-1} VLSG and M3' before and after *in situ* SR-activated oxygen cleaning. The photon intensity is normalized for a ring current of 450 mA. (b) The ratio of the photon intensity after the cleaning to that before the cleaning.

cleaning. The dip which remained after the cleaning was ascribed to the carbon contamination of M1. Fig. 2(b) shows the ratio of the photon intensity after the cleaning to that before the cleaning. The ratio reaches 1.06 at 284.9 eV and 1.35 at 291.9 eV. The removal of carbon contamination from M2, the VLSG and M3' seems to be responsible for the decrease in the photon intensity in the region outside of the carbon K-edge. This explanation is supported by calculations of the reflectivities of a thick Au mirror covered with a carbon ultrathin film and an uncovered thick Au mirror; the calculations were performed by using the Center for X-ray Optics web page (http://henke.lbl.gov/optical_constants/layer2.html). The surface roughening of the optics resulting from the oxygen cleaning is thought to be negligible because no energetic ions are produced by the photodissociation of oxygen (Lu *et al.*, 1998). By activating the NEG pumps, the base pressures of the M1, M2/VLSG and M3/M3' chambers recovered to 3×10^{-8} , 2×10^{-8} and 6×10^{-8} Pa, respectively, in two days without baking.

Fig. 3(a) shows a photograph of M2 during *in situ* irradiation with zeroth-order SR in oxygen at 2×10^{-3} to 1.2×10^{-2} Pa. The area irradiated with the SR emitted an intense green light. We ascribed the light to the green line emission at 557.7 nm arising from the O($^1S \rightarrow ^1D$) transition of atomic oxygen (Slanger *et al.*, 2001). The green light emission from the path of the zeroth-order SR was much weaker than that from M2's surface (Fig. 3a). The carbon contamination was observed to be removed only in the area irradiated with zeroth-order SR (Fig. 3b). These results indicate that O atoms are generated mainly by the interaction of oxygen molecules with photo-

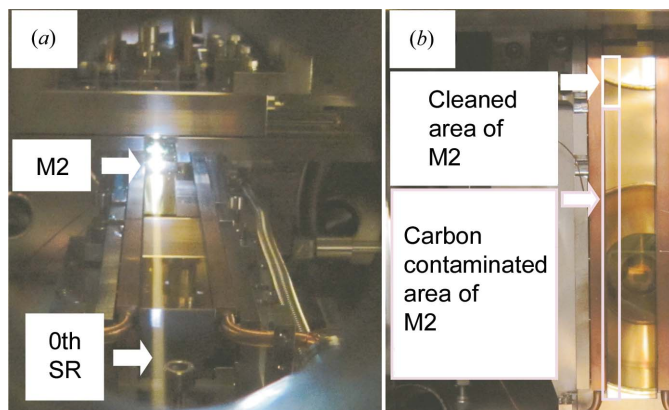


Figure 3
Photographs of M2 (a) during and (b) after *in situ* irradiation with zeroth-order SR in oxygen at 2×10^{-3} to 1.2×10^{-2} Pa. Since M2 can slide horizontally, we used the left area first in order to leave the other area clean. The area of M2 irradiated by zeroth-order SR depends on the photon energy.

electrons emitted from the optics. The O atoms and ions seem to play a dominant role in the removal of carbon contamination.

3.2. Atmospheric pressure UV/ozone cleaning of M1

Next, the carbon contamination of M1 was removed by atmospheric pressure UV/ozone cleaning at 363 K for 10 min. The cleaning was carried out by Dr H. Ohashi and Dr T. Miura at SPring-8. The carbon contamination was observed to be negligible during a visual inspection after the UV/ozone cleaning (Fig. 4). The surface roughness of M1 improved from 0.20 nm (RMS) to 0.17 nm (RMS) after the UV/ozone cleaning (Ohashi & Miura, 2011). Fig. 5 shows photon intensity spectra measured before and after the UV/ozone cleaning. The photon intensity dip at 285.1 eV is found to have reduced considerably. This result indicates that the carbon contamination of M1 was removed to some extent. We ascribe the dip that remained after the cleaning to the carbon contamination of M1 deposited during the course of commissioning of M1.



Figure 4
Photographs of M1 (a) before and (b) after atmospheric pressure UV/ozone cleaning at 363 K for 10 min. The light brown area on M1 before the cleaning was speculated to be graphite-like carbon with a flat-on configuration and a thickness of about 4 nm (see §3.1).

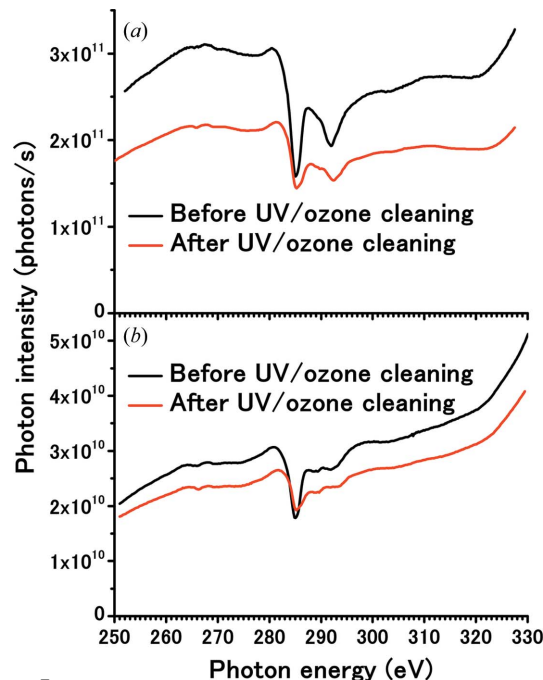


Figure 5
Photon intensity spectra measured (a) with the 300 lines mm^{-1} VLSG and M3 and (b) with the 1000 lines mm^{-1} VLSG and M3' before and after atmospheric pressure UV/ozone cleaning at 363 K for 10 min. The photon intensity after cleaning was weak because the position of the light source changed in the interval between the two sets of measurements (*i.e.* before and after cleaning). Since the storage ring suffered damage during the Great East Japan Earthquake on 11 March 2011 (magnitude: 9.0; intensity on the Japanese seven-stage seismic scale in Tsukuba area: 6–) (Honda *et al.*, 2012), its commissioning resulted in a considerable change in the position of the light source of BL-13A.

Since oil-sealed rotary pumps are used in the experimental hall of the PF, the air around the M1 chamber is not free from oil vapor. Thus, replacement of optics inevitably introduces a considerable amount of hydrocarbons in the chamber.

3.3. *In situ* SR-activated oxygen cleaning of M1, M2 and VLSGs

Finally, we carried out *in situ* SR-activated oxygen cleaning of M1, M2 and the VLSGs. Dry oxygen with a purity of 99.9% was supplied by using variable leak valves attached to the M1 and M2/VLSG chambers. Fig. 6(a) shows photon intensity spectra measured with the 300 lines mm^{-1} VLSG and M3 before and after *in situ* SR-activated oxygen cleaning. The oxygen pressures in the M1, M2/VLSG and M3/M3' chambers were in the range 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 lines mm^{-1} VLSG and M3 were irradiated with zeroth-order SR for 20 h. The averaged ring current was 450 mA. The photon intensity dips at 285.1 and 292.3 eV almost disappeared after the cleaning. The small increase at 281.4 eV is a non-integer-order undulator peak. The ratio of the photon intensity after the cleaning to that before the cleaning (Fig. 6b) shows sharp peaks at 285.1 and 291.5 eV. These results indicate that *in situ* irradiation with zeroth-order SR in oxygen at 2×10^{-4} to 6×10^{-4} Pa for 20 h is sufficient to remove the

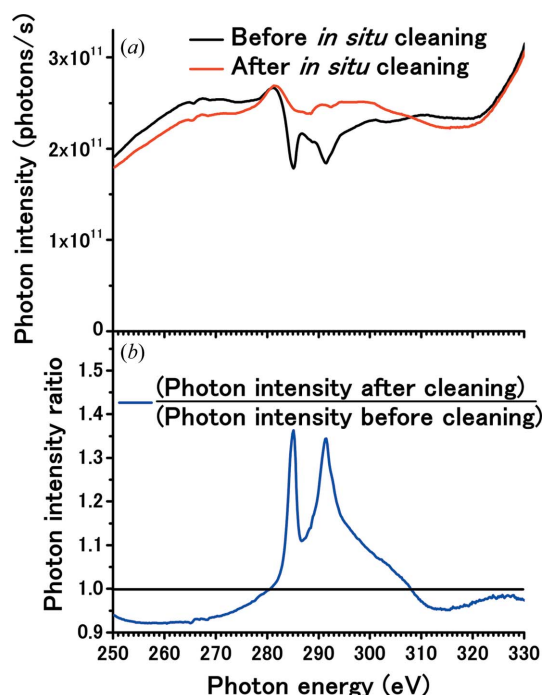


Figure 6
(a) Photon intensity spectra and (b) ratio of the photon intensity measured after *in situ* SR-activated oxygen cleaning to that measured before the cleaning; the measurements were made with the 300 lines mm^{-1} VLSG and M3.

graphite-like carbon on M1. The required oxygen pressure was smaller than that used in the case of M2 and the VLSGs by one order of magnitude (2×10^{-3} to 1.2×10^{-2} Pa). This result is reasonable because the intensity of the zeroth-order SR on M1 was much larger than that of the zeroth-order SR on M2 and the VLSGs. The photon intensity spectrum after the cleaning shows a 4% dip at 288.5 eV and 2% decrease at 292.3 eV. Since these absorption energies are different from those before the cleaning (285.1 and 291.5–291.9 eV), we ascribe them to the carbon contamination in the bulk of the gold film of the optics. The decrease in the photon intensity in the region outside of the carbon *K*-edge is attributed to the removal of carbon contamination from M1, M2 and the VLSG.

Fig. 7(a) shows photon intensity spectra measured with the 1000 lines mm^{-1} VLSG and M3' before and after *in situ* SR-activated oxygen cleaning. The oxygen pressures in the M1, M2/VLSG and M3/M3' chambers were in the range 1×10^{-4} to 9×10^{-4} Pa, 1×10^{-3} to 1.6×10^{-2} Pa and 1×10^{-5} to 2×10^{-5} Pa, respectively. M1, M2, the 1000 lines mm^{-1} VLSG and M3' were irradiated for 18 h. The averaged ring current was 450 mA. The photon intensity dip at 285.1 eV almost disappeared after the cleaning. The ratio of the photon intensity after the cleaning to the one before the cleaning (Fig. 7b) shows sharp peaks at 285.1 and 291.5 eV, and it resembles the NEXAFS spectrum of graphite at the incidence angle of 60° (Rosenberg *et al.*, 1986). This result provides evidence for our conclusion that the carbon contamination on M1 is composed of graphite-like carbon with a flat-on configuration. The photon intensity spectrum after the cleaning shows a 5% dip at 288.7 eV and 2% decrease at 292.7 eV. By activating the NEG

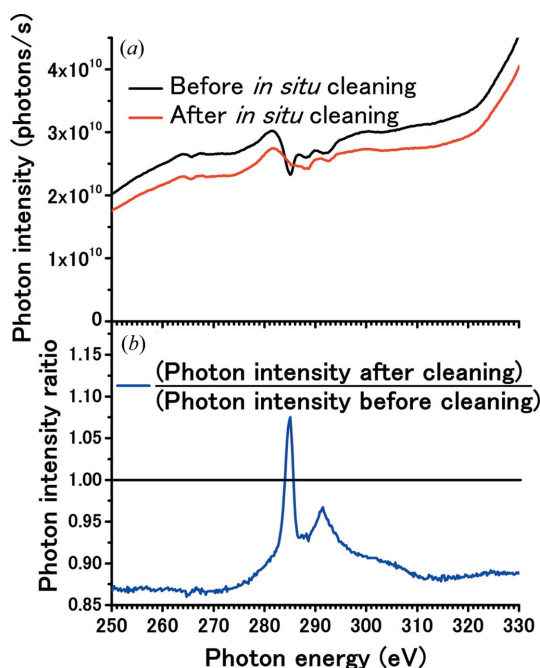


Figure 7
(a) Photon intensity spectra and (b) ratio of the photon intensity measured after *in situ* SR-activated oxygen cleaning to that measured before the cleaning; the measurements were performed with the 1000 lines mm^{-1} VLSG and M3'.

pumps, the base pressures of the M1, M2/VLSG and M3/M3' chambers recovered to 1.4×10^{-7} , 4×10^{-8} and 6×10^{-7} Pa in one day without baking. The beamline can be used without additional commissioning.

4. Conclusions

We performed *in situ* SR-activated oxygen cleaning of the optics in a VSX undulator beamline, BL-13A at the PF. The carbon contamination of M1, M2/VLSGs and M3/M3' was removed by exposing them to oxygen at pressures in the range 1×10^{-4} to 9×10^{-4} Pa, 2×10^{-3} to 1.2×10^{-2} Pa and 1×10^{-1} to 2×10^{-1} Pa, respectively, for 17–20 h and simultaneously irradiating them with zeroth-order SR. The photon intensity dips in the carbon *K*-edge region reduced to 2–5% after the cleaning. The base pressure of the beamline recovered to 10^{-7} – 10^{-8} Pa in one day without baking. The beamline can be used without additional commissioning. The interaction of oxygen molecules with photoelectrons emitted from the optics seems to play a dominant role in the removal of carbon contamination. Therefore, *in situ* SR-activated oxygen cleaning is suitable for optics in undulator beamlines where a high photon flux is easily achieved.

The key requirements for *in situ* SR-activated oxygen cleaning are as follows. (i) The surface of the optics must be made of material that is inert to activated oxygen. Gold is an ideal material for this purpose. (ii) The vacuum in the optics chambers must be hydrocarbon-free. The base pressure of the chambers should be less than 1×10^{-8} Pa. (iii) Cold cathode vacuum gauges should be used in order to prevent damage by the activated oxygen. (iv) A differential pumping system and

an interlock system must be installed between the front-end and the beamline to prevent the degradation of the vacuum of the storage ring. (v) Variable leak valves should be used to introduce pure oxygen into the optics chambers. (vi) Oil-free TMPs should be attached to the optics chambers to remove oxygen and reacted gases. (vii) The recommended oxygen pressure is 10^{-3} – 10^{-4} Pa for the first mirror and 10^{-2} – 10^{-3} Pa for the other optics. The typical irradiation time is less than 20 h. If such a high pressure is not allowed, oxygen with the maximum allowed pressure should be introduced for a longer time since the product of pressure and duration for which the pressure is applied is an important factor. The effective pressure on the optics can be increased if oxygen is supplied by a beam doser (Bozack *et al.*, 1987; Winkler & Yates Jr, 1988). Green light emission from the optics will be a good indicator of the oxygen pressure on the optics because its intensity is expected to be proportional to the oxygen pressure. (viii) NEG pumps should be used to facilitate the rapid recovery of the base pressure.

Preventing carbon contamination of optics is the next important issue. Since optics are continuously irradiated by SR during beamline operation, continuous exposure to oxygen gas seems to prevent carbon contamination (Warburton & Pianetta, 1992). The carbon contamination shown in Fig. 1 had increased during operation for about 300 days. Thus, the introduction of oxygen with a pressure of 10^{-5} – 10^{-7} Pa is supposed to be enough to suppress carbon contamination. Some preliminary experiments are now under way.

We express our sincere thanks to Dr H. Ohashi and Dr T. Miura (JASRI) for the atmospheric pressure UV/ozone cleaning and the surface roughness measurements of M1 in SPring-8. We are grateful to the staff of the PF for their valuable support during the course of the experiments. This work has been performed during the commissioning of BL-13A at PF.

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