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Development of contamination-free X-ray optics for next-generation light sources

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Abstract. We studied typical forms of contamination on X-ray mirrors that cause degradation of beam quality, investigated techniques to remove the contaminants, and propose methods to eliminate the sources of the contamination. The total amount of carbon-containing substances on various materials in the vicinity of a mirror was measured by thermal desorption-gas chromatography/mass spectrometry and thermal desorption spectroscopy. It was found that cleanliness and ultra-high vacuum techniques are required to produce the contamination-free surfaces that are essential for the propagation of high-quality X-ray beams. The reduction of carbonaceous residue adsorbed on the surfaces, and absorbed into the bulk, of the materials in the vicinity of the mirrors is a key step toward achieving contamination-free X-ray optics.

INTRODUCTION

Next-generation sources of light such as diffraction-limited synchrotron radiation place severe requirements on the X-ray optics to preserve wave-fronts while transporting high photon fluxes of more than 10^{13} photons/s [1]. However, intense irradiation of $10^{12\text{-}13}$ photons/s on the optical surfaces frequently induces serious degradation of the beam in a short time because of various forms of contamination. For example, milky substances, which are deposited on the surface of a mirror in the air, can diffuse the X-rays and increase the minimum focal spot size. Dark-brown contamination observed on an X-ray-irradiated area in vacuum reduces reflectivity and increases the scattering of the X-rays. A particle on an optical surface frequently causes speckle in a spatially coherent beam. To prevent these modes of degradation, contamination-free X-ray optics are required. This paper describes the elimination of each contaminant on an X-ray mirror as well as the pollution sources. Environmental requirements for contamination-free X-ray optics are also proposed.

PARTICLE CONTAMINATION

Illumination by fully spatially coherent X-rays such as that from an X-ray free-electron laser (XFEL) responds to a tiny particle on a mirror by producing speckle in the image. For example, Figure 1(a) shows speckles on a reflected image at a distance of 26 m from a flat mirror installed in the SPring-8 Angstrom Compact free electron LAser (SACLA) [2]. Adhered particles of 5–50 μm diameter were observed on the mirror by an optical microscope and resemble airborne dust that cannot be removed by a gas filter gun. Ultrasonic cleaning in water can, however, reduce the number of particles, resulting in the speckle-free image shown in Fig. 1(b).

To prevent these particles from sticking to the surfaces of X-ray optics, it is important to control the cleanliness of the environment. An open clean-bench, employed to improve cleanliness while installing an X-ray mirror, can achieve a class-1 particle level within a few minutes after being turned on. Visualization of particles by means of a high intensity discharge (HID) bulb in the wavelength range of 500–600 nm is also useful to raise awareness of cleanliness while working around high-precision optics.

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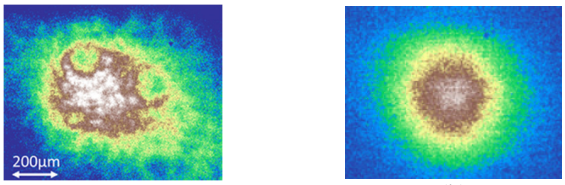


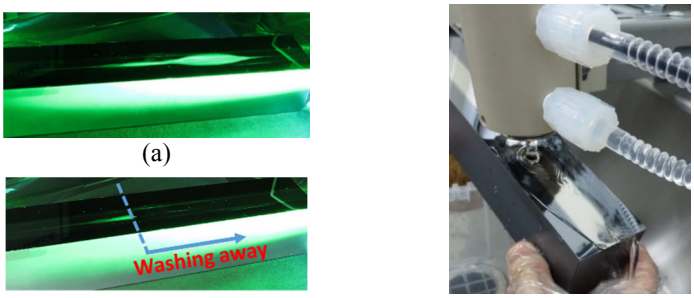
(a) (b)

FIGURE 1. Reflected X-ray free-electron laser images from a flat mirror (a) before and (b) after ultrasonic cleaning in water.

SOLUBLE CONTAMINATION

Milky contamination is often observed on X-ray focusing mirrors installed in an atmosphere of air or He, and used at a flux of 10^{11} photons /s or higher. In the case of a typical mirror system for He flow, an atmosphere of oxygen concentration was reduced to 0.08% when He gas was used instead of air. On the assumption that the chamber was not to be evacuated, an O-ring seal was employed for the main flange, and clean grease was used in the mirror manipulators. However, after 3 weeks of operation with a typical flux of 10^{11} photons/s at 10 keV, scattering of the x-rays gradually increased and the focused beam was spread by a few tens of percent; subsequently, a milky film was found to be widely deposited on the mirror. The contamination on the Rh-coated Si substrate, presented in Fig. 2(a), was soluble in water during ultrasonic cleaning (430 kHz, 45 W) as shown in Fig. 2(b) and (c). After washing in pure water, no damage to the metal coatings such as Rh and Pt was observed, and the surface roughness had not increased. However, this milky contamination was totally impervious to ozone-ashing.

Such milky contaminants have not been observed on any mirrors in vacuum. Therefore, we developed a new focusing mirror system, compatible with ultra-high-vacuum (UHV) operation. An Al wire seal was adopted for the main flange, and no lubricant was used inside the vacuum system of the new manipulator (Flexure hinges were employed, and all of the actuators were installed outside of the vacuum region.) The pressure in this chamber could be maintained below 10^{-7} Pa without the need for any baking procedures. Consequently, no degradation of X-rays was measured, even after one full year of operation, and milky substances were not observed.



(a) (b) (c)

FIGURE 2. Typical milky contamination on a focusing mirror consisting of an Rh-coated Si substrate. (a) After 3 weeks of use under He flow. (b) The same surface after the residue has been partially removed by washing. (c) The mirror being washed by water from an ultrasonically activated spot shower.

CARBON CONTAMINATION

Dark-brown substances suggestive of carbon contamination are frequently observed on the X-ray-irradiated area of mirrors in vacuum, and even operating in an ultra-high vacuum environment below 10^{-7} Pa is not sufficient to prevent the formation of these contaminants. It is well-known that, when an optical surface is abruptly illuminated by X-rays after having reached equilibrium in a vacuum chamber, the total chamber pressure dramatically increases by one or two orders of magnitude depending on the incident photon flux and surface treatment, and then gradually drops back; subsequently, carbon contamination is observed on the surface. The rapid rise of the pressure indicates photo-induced reactions, rather than a thermal reaction. The fact that the carbon-rich deposits are concentrated in the irradiated area indicates that the photo-reactions occur directly on the surface of the mirror. Some recent studies have shown that carbon contamination was almost totally removed *in situ*, in the case of the soft x-ray beamline, by

inletting oxygen gas [3-4]. However, when this is done, the beryllium window, which is an indispensable part of the X-ray beamline, becomes oxidized; this could easily reduce its strength and could also degrade the beam quality. Hence, we used semi-quantitative techniques to verify where most of the carbon actually comes from, and we will propose ways to remove these sources of carbon.

The presumptive sources are broadly divided into three types, including: (a) residual gases, (b) initially adsorbed species on the mirror, and (c) foreign matter on the surface of, and embedded in the bulk of, the materials around the optics as illustrated in Fig. 3. First a residual gas analyzer was used to show that the main carbon-containing species in the UHV chamber are CO and CO_2, and that their partial pressure was less than 10^{-9} Pa when the total pressure was 10^{-7} Pa. Even assuming that all of these residual species at 10^{-9} Pa were to become contaminants on the mirror, the estimated deposition rate would be only about 1 monolayer (ML)/100 h, which is very low in comparison with the severe carbon contamination actually observed. Subsequently, the density of initially adsorbed organic species on the surface of the optics was estimated to be about 5 ng/cm^2 for the Au-coated mirror to be described later; once again, this small quantity cannot provide an adequate explanation for the amount of contaminants that is actually observed. Finally, we measured how much carbonaceous residue could be attributed to the materials in the vicinity of the mirror, in addition to that on the mirror itself.

Various organic compounds adsorbed on a metal-coated sample and some materials around the optics were comprehensively analyzed by using thermal desorption-gas chromatography/mass spectrometry (TD-GC/MS), which is widely employed for the investigation of organic contamination on semiconductor wafers. Organic residues that were thermally desorbed at 400 °C from 50×50 mm test samples, were semi-quantitatively analyzed by GC/MS with a sensitivity better than 1 ng/cm^2. Table 1 describes typical results for the total organic substances on popular vacuum materials such as SUS304 and A5052 as well as for typical optics of Au-coated Si, which were carefully treated for installing in UHV according to the procedures described below. In the cases of SUS304 and A5052, two sets of test samples were prepared to reveal the process in which most organic substances adsorb on the samples. One set of the samples were deliberately wiped with oil, then degreased with organic solvent and a neutral detergent, followed by a final cleaning with volatile solvents. As an alternative to the solvent cleaning, another set of samples was cleaned up by a thermal bake-out at 400 °C, followed by a 24 h soak in air at room temperature. Even though both of these cleaning procedures have been demonstrated in practice to achieve base pressures of less than 10^{-8} Pa when the surfaces are not being illuminated by X-rays, it must be emphasized that a considerable amount of organic residue was still adsorbed on the surface that had been washed at room temperature in comparison with the surface that had been exposed to an air environment.

The amounts of low molecular weight, carbon-containing species desorbed from SUS304, A5052, Si, and Au-coated Si were measured by thermal desorption spectroscopy (TDS) as listed in Table 2. The mass-to-charge ratio ranged from 1 to 200 amu. It is well-known that stainless steel contains a large amount of carbon; remarkably large amounts of CO and CO_2 have been detected in SUS304 in comparison with other materials.

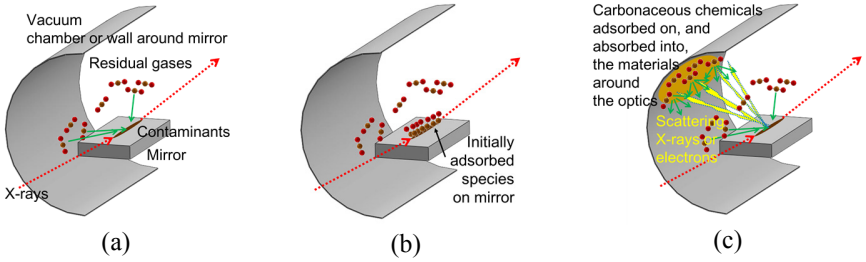


FIGURE 3. Schematic view of carbon sources. (a) Residual gases such as CO and CO_2. (b) Initially adsorbed species on the mirror. (c) Adsorbed carbonaceous chemicals adsorbed on, and absorbed into, the materials around the optics.

TABLE 1. Typical TD-GC/MS results for total organic substances on SUS304 and A5052 treated for installation in UHV

Material	Treatment of the surface	Total organic substances(ng/cm^2)
SUS304	After wiping with oil, and then washing with organic solvent, a neutral detergent, and volatile organic solvents	15
A5052		40
SUS304	After cleaning up at 400 °C, followed by exposure to an air environment for 24 h at room temperature	7
A5052		5
Au coated Si		5

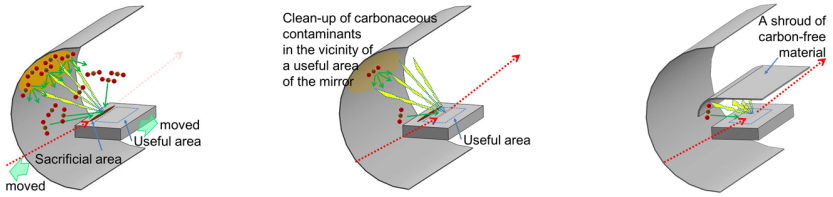
These data imply that an X-ray mirror must cope with a large number of potential carbon sources even if the materials have been treated for compatibility with UHV. To explain the rapid increase of pressure at the beginning of irradiation that was previously mentioned, we suggest that X-rays could produce volatile species containing carbon from contaminants on and below the surfaces of the materials surrounding the optics, as well as initiate surface reactions on the mirror itself that result in the deposition of carbon. It seems quite probable that scattered photons or electrons from the surface of an X-ray-irradiated optical device could dissociate and desorb large amounts of carbonaceous species from the surfaces and/or the bulk of the wall materials as illustrated in Fig. 3(c). This suggests that X-rays induce second-order reactions, confirming the observation of increasingly serious contamination as higher flux irradiates the optical material.

Based on this assumption, we propose X-ray induced cleaning of the surrounding materials as an easy way to reduce carbon contamination; this is illustrated in Fig. 4(a) and (b). After installing a new optical device for the first time, a high flux beam would irradiate the surface alongside of the useful area by translating the mirror or changing the path of the incident beam. Scattered X-rays and electrons from the sacrificial area could clean up carbonaceous residue around the device. As a result, contaminants would be dramatically reduced on the useful area.

Another approach to achieving contamination-free optics would be to surround - or nearly surround - the mirror by a shroud of carbon-free material, for example, ultraclean Si as illustrated in Fig. 4(c). It would, of course, be very important to prevent carbonaceous substances from being adsorbed on the surface of the shroud while installing it.

TABLE 2. Typical results of inorganic species containing carbon from various materials by thermal desorption spectroscopy

Material	Temperature(°C)	Inorganic species containing carbon (10^{15} molecules / cm^2)	
		CO	CO_2
SUS304	50–600	6	4.5
A5052	50–350	0.2	0.5
Si	50–1000	0.1	0.1
Au on Si	50–1000	0.4	0.2



(a) (b) (c)

FIGURE 4. Schematic view of reduction and elimination of carbon sources surrounding a mirror. (a) A high-flux beam irradiates a sacrificial area after moving the optics or the incident beam, resulting in (b) intense bombardment of the surrounding materials by scattered photons and electrons, reducing their carbon content. (c) Carbon-free material shielding the surface of the mirror.

CONCLUSION

We conclude that a very clean UHV environment utilizing carbon-free materials surrounding the X-ray optics are essential to prevent contaminants on the mirror. The elimination of the carbon sources by high-flux X-ray irradiation is a key step toward achieving contamination-free X-ray optics.

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