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Cleaning of carbon-contaminated optics using O_2/Ar plasma

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Abstract Cleaning of carbon-contaminated beamline optics was studied by RF plasma discharge process using O_2/Ar. Carbon-coated samples were prepared, and through their cleaning processes key parameters were determined, such as the optimal RF output power, mixing rates of O_2/Ar, and chamber vacuum. Considerations were made against possible adverse effects in cleaning the beamline optics, such as comparing the roughness of samples before and after cleaning, and possible detrimental kinetic effects on cable insulation. Under the cleaning parameters to clean the beamline optics, the thickness of removed carbon film and the change in beamline photon flux were analyzed.

Keywords RF plasma cleaning · Carbon-coated samples · Roughness · Cable's conductivity · Beamline photon flux

1 Introduction

Carbon atoms can be cracked down from hydrocarbons by photoelectrons emitted from irradiated optical surface in light source beamline, and the carbon atoms will constitute a film [1–3] on the optical surface. Carbon contamination is a serious problem to the beamline, especially in soft X-ray beamline. Carbon film deposits on a reflecting surface result in a substantial loss of flux and severe efficiency degradation at or above K edge, amounting to orders of magnitude in highly contaminated cases.

The Phase II beamlines of Shanghai Synchrotron Radiation Facility (SSRF) will employ more wigglers and undulators, producing photon beams of much higher brightness than those from bending-magnet beamlines. This in turn results in more carbon contamination, as the deposition rate of contaminant film is proportional to photon flux on the optical components [4, 5], especially for a soft X-ray beamline [6]. It's essential to remove carbon contamination from these optical components.

The methods proposed to clean the beamline optics include RF discharge cleaning, synchrotron radiation cleaning [7–9], UV lamp cleaning in oxygen atmosphere, DC discharge cleaning, glow discharge cleaning [10, 11], and laser pulse cleaning [12, 13]. RF discharge and synchrotron radiation cleaning are good choices for in situ beamline optics cleaning. In this work, RF discharge cleaning was used during the shutdown period of SSRF. The RF plasma (a mixture of oxygen and argon) produces a large quantity of oxygen radicals [14, 15], which oxidizes carbon contaminate into CO and CO_2. Then, the reflection of mirrors increases [16], and beamline photon flux becomes higher.

Before cleaning the contaminated optics, key cleaning parameters were determined, such as the optimal RF output power, mixing rates of O_2 and Ar, and chamber vacuum. Sample mirrors of Si substrate coated with carbon film were made, and beamline optics cleaning experiments performed to determine the cleaning parameters and to ensure that possible adverse effects will not happen while cleaning the real beamline optics. Roughness of the samples before and after cleaning was measured to check gentleness of the RF plasma removal of the carbon films.

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Also, possible detrimental kinetic effects of RF plasma on the Kapton films covering the wires were examined by checking conductivity of the Kapton film.

Using the optimal parameters, the RF plasma cleaning of SSRF beamline optics was carried out. Photodiodes were used to detect the beamline photon flux before and after cleaning. According to the photon flux improvement ratio, thickness of the removed carbon film was estimated.

2 Experimental

2.1 Experimental setup

As shown in Fig. 1, the experimental cleaning equipment mainly consists of the vacuum chamber, gas flow controller, liquid nitrogen tank, RF generator, vacuum pump and gauge, and residual gas analyzer (RGA). The vacuum chamber, sized at 1200 mm × 400 mm × 400 mm (in similar size of a beamline optics chamber), is made of 304 L stainless. It has different kinds of flange interface to install RF power, vacuum pump, vacuum gauge, and RGA. Oxygen and argon release from the bottles is controlled by the gas flow controllers, so as to obtain the optimal mixing rates and the chamber pressure. The water and oil contents in the gas mixture passes are frozen off by liquid nitrogen, and the purified gas mixture arrives at the RF generator, which is a GV10× plasma source, in maximum RF power of 100 W [17, 18], installed on the vacuum chamber. The chamber is pumped at the opposite end to establish a constant flux of oxygen plasma along its longitudinal axis.

Residual gas analyzer (RGA) was used to detect elemental ratio of the residual gases. The cleaning process should be accompanied by increasing carbon ratio. If other elements ratios increased, the feedstock gases would not be pure enough.

Experiments with carbon-coated samples were performed to determine the optimal O_2/Ar rate, gas pressure, and RF power and to check safety of the cleaning process. These optimized parameters were the basis for installing the vacuum pump, vacuum gauge, and RGA on the beamline optics chamber and cleaning the beamline optics.

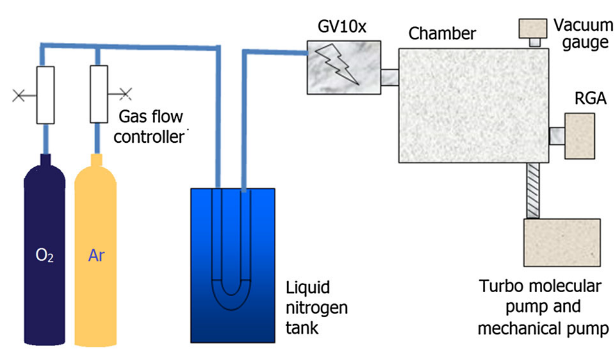

Fig. 1 Schematics of the experimental cleaning system

2.2 Sample preparation and characterization

Samples mirrors were prepared with Φ30 mm Si wafers. They were coated with 50-nm Au film and then 10, 30, or 50-nm carbon film, through chemical vapor deposition (CVD) at Tongji University [19, 20]. As shown in Fig. 2, the carbon films of different thicknesses are different colors (golden, red, and blue for 10, 30, and 50-nm films, respectively, due to the difference in visual light refraction of the carbon films) [21, 22].

Zygo NewView 600 white color interferometer was used to measure roughness of the samples. Most samples did not change in roughness before and after carbon coating, with the RMS of <3 Å (Fig. 3). A few carbon-coated samples became worse in roughness, due to the non-uniformity of carbon film CVD.

2.3 Cable insulation experiments

Cables for adjusting the beam optics mirrors are insulated by Kapton films, which should not be damaged by the beam optics cleaning process. To confirm this, a sample cable was placed near the sample mirror, so as to suffer the same amount of RF plasma as the mirror. This cable resistance was measured before and after cleaning process.

3 Results and discussion

3.1 Cleaning experiments

3.1.1 Optimized cleaning parameters

The RF plasma cleaning parameters optimized through the experiments are summarized in Table 1. The data almost coincide with those given in Ref. [1], except the RF power of 40–50 W, due to probably the difference in chamber sizes. Ref. [17] suggested RF power of 150 W. In our experiments, however, the RF generator always

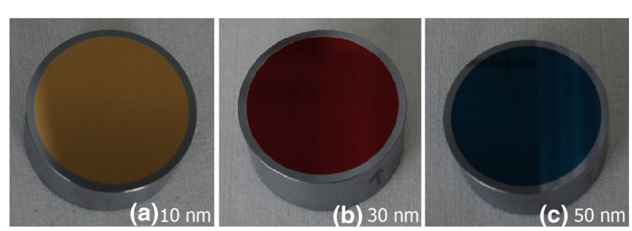

Fig. 2 Sample mirrors with carbon films of different thicknesses

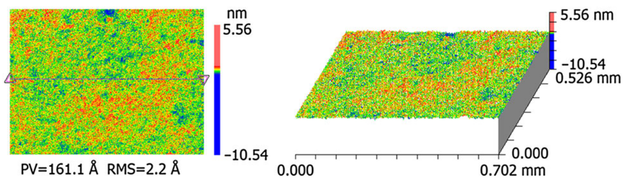

Fig. 3 Roughness of the sample surface

Table 1 Optimal cleaning parameters

Parameters	Values
RF power (W)	75
Ratio of O_2/Ar	10:1
Vacuum (mbar)	0.005–0.03
Flow rate of O_2/Ar gas mixture (sccm)	≤5

stopped work at ≥100 W. Therefore, we reduced the RF power to 75 W.

3.1.2 *Roughness of samples before and after cleaning*

RF plasma can remove carbon film, but violent removal of the carbon contamination film would damage the Au layer on the mirrors surface. Three samples (×13, C1, and d30-1) were used to measure the surface roughness before and after cleaning. The results are given in Table 2. Before and after RF plasma cleaning, samples ×13 and C1 showed no obvious changes in surface roughness, indicating that the beamline optics could stay safety in the RF plasma cleaning process. Sample d30-1 changed in carbon-coating roughness, but its greater roughness before cleaning suggested non-uniform deposition of carbon film. After RF plasma cleaning, roughness of d30-1 became better again.

In Ref. [14], RF plasma effects on roughness and reflectivity of vacuum ultraviolet optics were studied. The sample roughness was measured by interferometer, scanning tunneling microscope (STM), and mechanical profile meter (Talystep) before and after cleaning. No changes in roughness were found. So RF plasma does not damage the mirror surfaces.

Table 2 Carbon-coating roughness of samples before and after cleaning

Samples	Before (Å)	After (Å)	Carbon thickness (nm)
x13	2.3	2.8	30
C1	2.3	2.6	10
d30-1	4.3	2.5	10

3.1.3 *Measurement of cable resistance*

The original cable resistance was 99.9 GΩ. After RF plasma cleaning for five times, in an accumulated time of ∼200 h, its resistance was still 99.9 GΩ and hence no RF plasma effect on cable insulation.

3.2 Cleaning of beamline optics

According to the optimal cleaning parameters in Table 2, M1 mirror of the soft X-ray beamline was cleaned. Beamline photon fluxes before and after cleaning were detected, and thickness of the removed carbon film could be estimated from the improvements in photon flux ratio.

3.2.1 *Improvement in photon flux*

The M1 mirror of the soft X-ray beamline was cleaned, twice during two shutdown periods of times in an interval of about six months. Before and after shutdown of the beamline, photon flux was measured with the same beam intensity, gap size of the undulator magnet pole, slit size, and grating. In the first shutdown, the mirror was cleaned for 20 h at the RF output power of 75 W and vacuum pressure of 0.008 bar. The electron beam current before and after the shutdown was 240 mA. The photon flux after cleaning increased obviously (Table 3). In the second shut down, the mirror was cleaned for 50 h at the RF power of 75 W and vacuum pressure of 0.02 bar. At the beam current of 210 mA, the improvement of photon flux ratio was more obvious (Table 3).

3.2.2 *Evaluation of removed carbon film*

Figure 4 shows the beamline photon flux as function of photon energy, after the second cleaning process. Before cleaning, the flux ratio of carbon edge to the flux without contamination was 3%, while the ratio after cleaning increased to 13%. According to the ratios, the thickness of removed carbon film can be estimated. The photon flux ratio can be calculated by $I/I_0 = e^{-ut}$, where I and I_0 are flux of carbon edge from the mirrors with and without carbon contamination, respectively; $u = 1.1 \times 10^{-5}$ cm^{-1} is the carbon edge absorbance coefficient; and t is the carbon film thickness.

Assuming that contamination of every mirror is proportional to the thermal power, from the grazing angle of M1 mirror, its carbon film thicknesses are 6 nm before cleaning and 2.8 nm after cleaning, i.e., the removal of 3.2-nm carbon film.

The second cleaning process continued for 50 h; thus, the carbon removal rate is 0.011 Å/min. The result is far

Table 3 Photon fluxes before and after (Φ_B and Φ_A) the first and second cleaning

Photon energy (eV)	Gap (mm)	Φ_B (μA)		Φ_A (μA)		$(\Phi_A - \Phi_B)/\Phi_B$	
		1st	2nd	1st	2nd	1st (%)	2nd (%)
Fe							
723	82.5	2.20	0.52	3.92	3.33	78	540
670	79.7	2.10	0.51	3.27	3.01	56	490
O							
543	72.9	1.67	0.17	2.98	1.49	79	776
N							
410	65.8	0.83	0.08	1.97	0.52	138	550
C							
296	58.8	0.08	0.01	0.22	0.08	175	700
284	57.8	0.05	0.01	0.08	0.05	60	400

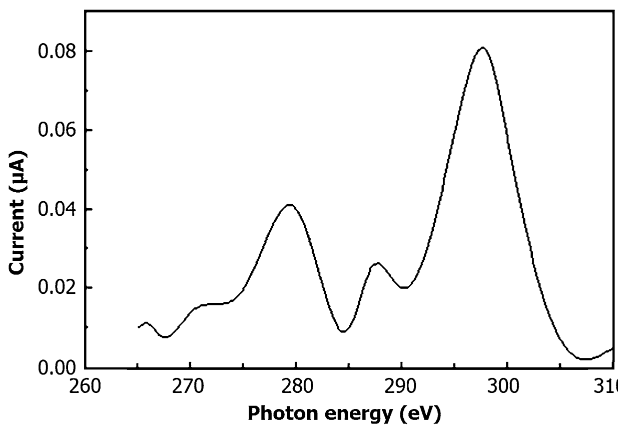

Fig. 4 Beamline photon flux with different energy after the second cleaning process

smaller than that of Ref. [17], probably due to the fact that the RF power in this work is only half of that in Ref. [17]. Another reason is that the M1 mirror chamber differs in structure from the experimental cleaning system, on which the GV10× plasma source and the vacuum pump are installed at the same side of chamber. Thus, the oxygen plasma could not reach the mirror surface efficiently and hence the reduced carbon removal rate.

4 Conclusion

RF plasma cleaning of beamline optics was conducted on the experimental system and the M1 mirror chamber of a soft X-ray beamline at SSRF. Possible detrimental kinetic effects due to the RF plasma were studied. Under the RF power of 75 W, the O_2/Ar mixing ratio of 10:1 and flow rate of ≤ 5 sccm, and the chamber pressure of 0.005–0.05 mbar, the SSRF beamline optics cleaning can be efficient and safe, without obvious changes in roughness of the cleaned mirror surface, and in insulation of the cable for adjustment of the mirrors. Further efforts shall be made to improve the removal rate of carbon contamination.

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