DUV cleaning of aluminium optics left at the atmosphere

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Abstract. The DISCO beamline at SOLEIL synchrotron radiation facility [1] is a low energy beamline (1-20 eV) with three end-stations. The imaging branch is the lowest energy of the three, ranging between 1 and 5 eV [2], for which ultra high vacuum is not mandatory. Therefore, the branch is separated from the beamline vacuum by a DN63CF suprasyl window protected for colour centre formation by suprasyl plate moveable under vacuum. In order to maximise the reflectivity in the photon energy domain of interest, all the mirrors specific to the branch have been coated with Al-MgF2. Commissioning and first year of exploitation were performed under primary vacuum conditions in the imaging branch line. During that period, the optics got heavily polluted, which induced a strong global loss in photon flux. Al-MgF2 optics cannot be cleaned using oxygen plasma without heavy damages and loss of reflectivity. Therefore, the mirrors had to be removed and cleaned ex situ. Following this maintenance, the branch had been re-exploited under high vacuum conditions assured by a turbomolecular pumping to provide what we expected to be better protection of the optical elements. Although the very first photometric measurements showed a gain in flux, very soon the photon flux starts decreasing.

1. Introduction

The DISCO beamline at SOLEIL synchrotron radiation facility possess three branches. Two of them are maintained under ultra high vacuum. The third one is a near-UV imaging branch, and it uses photons only between 1 and 6.52 eV. This branch is equipped with two DUV microscopes optimized for fluorescence imaging of tissues and living cells [3]. The commissioning and the first years of exploitation were performed under primary vacuum conditions (0.017 mbar) on the branch line. During this period of time, the optics got heavily polluted inducing a strong global loss in photon flux. The nature of the mirrors’ coating does not permit classical in situ plasma and low oxygen cleaning of the optics. Daily flux was monitored with an AXUV-100 calibrated photodiode fitted with an interferometric filter at 350 nm with 30 nm bandwidth was positioned just before the monochromator of the microscopes.

Flux at the microscope entrance was measured with another photodiode directly before the sample stage; the monochromator was scanned to check whether the flux reduction was homogeneous as a function of the photon energy.
2. Losses due to contamination

2.1. Transmittance loss

The losses of transmittance of the suprasyl plate and of the suprasyl vacuum-tight window were observed by measuring the transmittance of these elements along several points (figure 1). It appeared that contaminated area might loose up to 90% of transmittance in the deep ultraviolet (DUV). It was therefore necessary to clean the windows and mirrors and to find a way to prevent from further pollution. Because of the nature of the mirrors coatings (AlMgF₃) treatment of the optics with reactive oxygen species or with ozone [4] was not possible without damaging the coatings.

2.2. Visualisation of carbon deposition

Carbon deposition is clearly visible on the window between the ultra-high vacuum of the beamline and the primary vacuum of the imaging branch (Figure 1), the first mirror under primary vacuum M52 (Figure 2) and the last mirror under primary vacuum M54 (Figure 3).
2. 3. High vacuum conditions

We decided to increase the vacuum in the branch from 0.017 mbar down to 10^{-6} mbar, which should normally slow and reduce contamination of the optics by lowering the levels of carbon species in the residual vacuum. Before removing the optic for cleaning and re-coating, the photon flux delivered to the microscope was gradually decreasing, impairing the experiments recorded (Figure 4).

![Figure 4. Flux measured at microscope entrance](image)

Cleaning of the optics permitted to recover the initial photon flux on the microscopes (light blue curve). However, rapidly after placing the entire branch line under high-vacuum, dramatic reduction of the photon flux was observed within the first days (Figure 5 and 6).

![Figure 5. Loss of flux under 10^{-6} mbar and beam exposition](image)
3. Going to air

It appeared that the enhanced vacuum conditions, with a low partial pressure of oxygen, could be responsible for the extremely high contamination of the optics. Hence, we decided to operate the branch line at the atmospheric pressure.

![Figure 6. Flux at the microscope entrance just after cleaning of the optics, after 10 days under strong vacuum and beam irradiation and after 5 days under beam at the atmosphere.](image)

Surprisingly, initial photon fluxes were recovered within few days under those conditions (Figure 6). We hypothesise that reactive oxygen species produced by the SR between 1 and 6.52 eV at the atmosphere, react with carbon deposit and remove it, thus preventing from further pollution of the optics to appear. With low oxygen pressure alone, however, the ROS degrade the mirrors coatings. Air permits to keep an oxygen partial pressure with a buffer of nitrogen that prevents further degradation of the optics coating. Therefore the right mixture of oxygen and nitrogen would benefit for low carbon contamination of DUV optics.

References

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