Study on effective laser cleaning method to remove carbon layer from a gold surface

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Abstract. Hydrocarbon cracking and carbon contamination is a common problem in soft x-ray Synchrotron Radiation (SR) beam lines. Carbon contamination on optics is known to absorb and scatter radiation close to the C K-edge (284 eV) spectral region. The purpose of this work is to study and develop a laser cleaning method that can effectively remove the carbon contaminations without damaging the underneath gold-coated optics. The laser cleaning process is non-contact, accurate, efficient and safe. Nd:YAG laser of 100 ns pulse duration is used for carbon cleaning. The effect of laser pulse duration, laser fluence, number of laser passes, angle of incidence and spot overlapping on the cleaning performance is studied. Cleaning effect and subsequent film quality after laser irradiation is analyzed using x-ray photoelectron spectroscopy (XPS) and soft x-ray reflectivity (SXR) techniques.

1. Introduction
In extreme ultraviolet (EUV) spectrometers and Synchrotron Radiation (SR) beam lines, carbon contamination is a severe problem. The EUV and X-ray optical elements get contaminated with carbon due to photo induced hydrocarbon cracking phenomena. The carbon contamination deteriorates the optics performance and therefore an effective cleaning method is required [1-2]. Usually the contaminated optics are replaced with new optics or re-coated after appropriate treatment; the former one is expensive whereas the later one is a time consuming process. Earlier, several reports are published on removal of carbon layer from optical elements of beamlines using conventional cleaning techniques like plasma cleaning, chemical cleaning, glow discharge in oxygen atmosphere, etc. [3-5]. These techniques have some drawbacks as surface figure quality and roughnesses are modified during the cleaning process. In the present study a Nd:YAG laser of ns pulse duration is used for laser cleaning process. The laser cleaning process has several advantages like it is a non-contact, efficient and useful for the cleaning of optical elements without any damage to surface shape and quality [6]. The Nd:YAG laser is more suitable for laser cleaning process because of its relatively low cost, high reliability, availability of high energy and variable pulse duration and a fibre optic beam delivery options. The Nd:YAG has high absorption in carbon (50%) compared to gold bulk material (2%). Thus it can clean the carbon contamination without damaging the substrate.
Laser cleaning process requires a certain minimum fluence called ablation threshold fluence. Lasers of short pulse duration in the microsecond to nanosecond time scale are preferred for the cleaning
process. The short duration pulses generally limit the heat deposition on the surface of the material and avoid the conduction of the heat inside the material.
In this paper we analyse the potential advantages of ns pulsed Nd:YAG laser for precise removal of carbon layer from a gold coated sample. Process parameters like pulse duration, laser fluence, number of laser passes, angle of incidence and spot overlapping during the laser cleaning are optimized to obtain best cleaning effect. The cleaning effect on carbon film and gold layer are analyzed by XPS and angle-dependent reflectivity measurements.

2. Experimental Procedure
A pre deposited test sample of gold film (200nm) on BK7 glass substrate was used to study the laser cleaning effects. For optimizing the laser parameters for carbon removal a thin carbon layer of 20 nm thicknesses was deposited on top of the gold film. During the deposition of the carbon layer a small portion of the sample was covered with the mask to keep the gold film intact in that region.
Schematic of laser cleaning setup is shown in Fig. 1. Experimental parameters of laser cleaning setup are given in Table-1. In this setup an orthogonal pair of galvanometer scanners is located at a suitable distance from the target. Galvanometer scanners typically consist of a mirror mounted to an electric motor, with positional feedback arrangement. The scanner mirror can move the laser beam in the X-Y direction. The laser beam always remains at focus in the plane using a flat field lens. The flat field lens is used such that the laser beam strikes normal (perpendicular) to the sample. The typical laser spot diameter at the focal plane is ~ 500 μm. In the laser cleaning process the pulse energy is varied from 1mJ- 10mJ , angle of incidence from 10 degree to 45 degree, number of passes from 1 to 10 and percentage of spot overlapping from 60 % to 90 % for a fixed area of 4800 mm². For different percentage of the laser spot overlapping a CAD drawing is generated and then data is transferred to X-Y scanner. For the cleaning experiment sample was kept in a sealed chamber. The sample holder was fixed on a linear stage to get the precise repositioning of the sample. During the laser cleaning process the debris of the carbon layer was removed using a suction pump of 200 lpm capacity.
Laser cleaning experiments was performed on a small area of the sample as shown in the Fig. 2. In the figure region-A is corresponding to intact gold film, and region-B is carbon coated gold film. These two areas were kept under the mask during the laser cleaning. Region-C was used for laser beam exposure to remove the carbon layer.

![FIGURE 1: Schematic diagram of laser cleaning setup](image1)

![FIGURE 2: Schematic diagram of sample used](image2)

After the laser cleaning experiments the XPS measurements were carried on each region using an Omicron EA-125 photo electron spectrometer using Al Kα radiation at base pressure of ~5×10⁻¹⁰ Torr. A concentric hemispherical analyzer of resolution 0.8 eV and pass energy 50 eV was used.
Soft x-ray reflectivity experiments were performed using λ=9 nm on three regions A, B and C (see Fig 2) using reflectivity beamline at Indus-1 synchrotron radiation source [7]. Reflectivity analysis
reveals the details of carbon and gold layer separately as two have a distinct critical angle in the soft x-ray region.

Table-1: Parameters of Nd:YAG laser

| Parameter                  | Value          |
|----------------------------|----------------|
| Pumping method             | Flash Lamp     |
| Average output power       | 1-20 W         |
| Pulse energy               | 1-10 mJ        |
| Peak power                 | 100 kW         |
| Pulse duration             | 100 ns         |
| Repetition rate            | 1-5 kHz        |
| Linear scan speed          | 20 mm/s        |
| Focus spot diameter        | 0.1-0.5 mm     |

At the reflectivity beamline, the experimental chamber consists of two rotary stages driven by stepper motors used to accomplish \(\theta-2\theta\) scan and a linear translation stage for sample mounting. A soft x-ray silicon photodiode detector (International Radiation Detector Inc., USA) was used in direct current mode for measuring both incident and reflected beam intensity. An electrometer amplifier is used to measure the current output of the detector. Angle scan was performed in the 0-40 degree incident angle at 9nm wavelength. The experimental data obtained were fitted using the Parratt formalism [8]. Effect of roughness was taken into account using Novet-Croce model [9]. To refine the fit parameters a nonlinear least square refinement routine based on the Genetic algorithm was used [10].

3. Results and Discussions

XPS and soft x-ray reflectivity measurements corresponding to region A, B and C (as described in Fig 2) are shown in Fig. 3 and Fig. 4 respectively. XPS spectrum of C 1s binding energy of the three regions: A) Intact gold film, B) carbon coated gold film and C) laser cleaned film region (after carbon removal) are shown in Fig. 3a-c respectively. The large width of the C 1s peak is indicative of the presence of different phases of carbon in the sample. Spectrum unfolding by Gaussian de-convolution method suggests the presence of C sp3 peak at 285 eV and graphite peak at 283.7 eV in the carbon coated gold film region (Fig. 3b) [11]. Presence of atmospheric carbon on the gold film (region-A) give rises a C 1s peak as shown in Fig. 3a [12]. After laser treatment the carbon film is removed from the sample but the presence of C 1s peak in Fig. 3c is corresponding to atmospheric carbon. Intensity and shape of C 1s peak of gold film region (region-A) and laser cleaned carbon film region (region-C) are almost same.

Measured and fitted SXR data are shown in Fig. 4. SXR data corresponding to region-B having carbon on gold film shows two critical angles corresponding to carbon and gold layer (curve b). SXR curve corresponding to pure gold film (region-A) shows a single critical angle (curve a). SXR curve of region-C which is corresponding to carbon removal area shows a single critical angle (curve c). SXR pattern of intact gold film region-A and carbon removed gold film region-C are found in close agreement. Comparison of SXR curve A and C suggest that the laser cleaning process has removed the 20 nm carbon layer and underneath gold layer remained intact. Our detailed study of carbon removal from the gold surface suggests that the laser cleaning method has 98% efficiency for 30 degree incident angle. Results of detailed study will be published elsewhere. Laser based cleaning technique can be used to effectively remove carbon contamination from actual optics.
4. Conclusions
Experiments of laser cleaning of carbon layer from the gold film sample has been performed using Nd:YAG laser of 100 ns pulse duration. The maximum cleaning efficiency was found 98% at 30 degree incident angle. XPS and SXR data confirms that Nd:YAG laser can effectively remove carbon contamination on gold coated substrate without damaging the underneath gold film and substrate.

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