Removal of Surface Contamination by Atomic Hydrogen Annealing

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The surface treatment using atomic hydrogen generated by a heated tungsten mesh was investigated for the cleaning of the optical elements used in the synchrotron facility. We call the surface treatment by the atomic hydrogen annealing (AHA). The Au and Ni mirrors and Ni and Mo diffraction gratings with carbon contamination were cleaned by the chemical reaction and thermal effect due to the recombination of the atomic hydrogens during AHA. The carbon contamination was removed and the reflectance of the Au and Ni mirrors was recovered by AHA. The AHA conditions could be optimized for cleaning of Mo/Si multilayer mirrors from the relationship between the treatment condition and degradation. In addition, to clarify the removal reaction of the carbon contamination, the two-types of amorphous carbon (a-C) films were used. The etching rate of the a-C film by AHA was strongly related to the hydrogen content, atomic density and sp²/sp³ component of a-C film.

Keywords: Atomic hydrogen annealing, Carbon contamination, Removal, Mirror, Mo/Si multilayer

1. Introduction

The fabrication technology of the metal-oxide-semiconductor field effect transistor (MOSFET) has been investigated to improve the device performance. The device performance and cost are defined by the line width during lithography. Recently, the novel light source has been introduced to the 7 nm FinFET technology [1]. In this case, the extreme ultraviolet (EUV) light with a wavelength of 13.5 nm is used. Optical elements such as Mo/Si multilayer mirrors installed in semiconductor lithography equipment are contaminated with the carbon during the EUV light irradiation and the reflectance of the mirrors decreases. Therefore, the removal method of the carbon contamination is required for reduction of maintenance costs.

We proposed the surface treatment method using the atomic hydrogen, named as the atomic hydrogen annealing (AHA) [2-8]. In AHA, the hydrogen molecules are decomposed by the catalytic cracking reactions on the heated tungsten (W) catalyst placed near the sample. By contrast, in the H₂ plasma treatment, the atomic hydrogen is generated by collision between H₂ molecule and an accelerated electron in gas phase. The efficiency for the generation reaction of atomic hydrogen is not so high and temporally atomic hydrogen density is the order of 10¹⁷ m⁻³ [9]. On the other hand, in AHA, the atomic hydrogen is generated on a large area W surface and the atomic hydrogen density is at least 10 times higher than that generated by the plasma method. It was reported that the highly atomic hydrogen densities of 1.5 × 10²⁰ m⁻³ [10] and 10²³ m⁻³ [11] were realized.

The surface treatment using the atomic hydrogen generated on a heated metal catalyst have been widely investigated. The photoresist removal by the atomic hydrogen treatment using the heated W catalyst without plasma has been reported [12-18]. In this method, even a highly doped ion-implanted photoresists are removed by the atomic hydrogen [18]. In addition, the chemical reactions between the atomic hydrogen and photoresists were reported [14]. In addition, the cleaning of mirrors had been also
investigated. Mo/Si multilayer mirrors are used in hydrogen ambient for EUV light lithography. However, the Mo/Si multilayer mirrors are degraded by the blister formation due to the hydrogen radicals, ions, or thermal damage [19,20]. Therefore, the AHA conditions have to be optimized to suppress the blister formation. It was reported that the Ru coated Mo/Si multilayer mirror oxidized by EUV irradiation was subjected to the atomic hydrogen and the reflectance was recovered by reduction of RuOx [21,22]. We had investigated the removal of carbon contamination on Ni mirror and brief examination had already published in AIP conference proceedings [23]. However, the other metal mirrors and the Mo/Si multilayer mirrors without a Ru coating have been never investigated yet.

In this study, the removal of surface contamination by AHA was investigated using Au, Ni and Mo/Si multilayer mirrors used in NewSUBARU. In addition, to clarify the removal reaction of carbon contamination, the two types of amorphous carbon (a-C) films were used.

2. Experimental
2.1. AHA apparatus
The AHA apparatuses are shown in Fig. 1. In this study, we used two AHA apparatuses. One is for large size samples such mirrors used in synchrotron facility, called the chamber 1. In the chamber 1, W mesh in size of 55 × 55 mm² with a wire diameter of 0.03 mm and have 50 holes/inch was used. The other is for the evaluation of distance dependence, called chamber 2. Sample was set at a top edge of transfer rod. Therefore, the sample size was suppressed up to 20 × 20 mm². The distance between the W mesh and sample (Dms) could vary from 10 to 500 mm. In the chamber 2, W mesh in size of 10 × 55 mm² with a wire diameter of 0.1 mm and have 30 holes/inch was used.

2.2. Property evaluation of carbon contamination
To clarify the property of carbon contamination generated on the Au mirror during soft X-ray irradiation, the carbon contamination on Au/Cr/c-Si substrate was measured by X-ray photoelectron spectroscopy (XPS).

2.3. AHA of Au and Ni mirrors and Ni and Mo diffraction gratings
The contaminated Au and Ni mirrors were subjected to AHA. Because the mirrors were too large to be introduced into the XPS chamber, we used a dummy Au and Ni-coated mirrors for XPS that has slightly contaminated with carbon and has been placed with the SR mirror during the AHA treatment. The Au film and Ni film on a Cr film coated c-Si substrates were treated in chamber 1. The Dms was fixed at 80 mm. The mesh temperature (T mesh) was also fixed at 1700 °C. The flow rate of H2 and gas pressure were 150 sccm and 30 Pa, respectively. The AHA treatment time (tAHA) was 60 min. In the case of a Ni mirror, UV-O3 ashing [24] was also carried out at the room temperature for 120 min as a reference.

The evaluation of the mirrors before and after the AHA treatment was carried out with visual observation, surface profile measurement by optical interferometer, surface roughness measurement by atomic force microscope (AFM), evaluation of the thin film structure for depth direction using hard X-ray reflectometry at grazing incidence (XRR), and surface composition analysis by XPS. The soft X-ray reflectance of the mirror before and after the treatment was measured using the soft X-ray reflectometer of NewSUBARU BL-10 [25]. The reflectometer can measure a wide in-plane reflectance distribution of over 50 mm. However, in order to measure the distribution at a shallow grazing angle of incidence of 3°, we developed a new dedicated mirror holder. The measurement was performed at a grazing angle of incidence of 3°, and the reflectance and its in-plane distribution at a photon energy of 200 to 600 eV were investigated.

In addition, to confirm the removal of the carbon contamination on a Mo diffraction grating, the Mo
diffraction grating used in NewSUBARU BL10 was subjected to AHA in the chamber 1.

2.4. AHA of Mo/Si multilayer mirror

The Mo/Si bilayer period, thickness ratio of Mo/Si and total thickness were 8.3, 0.4/0.6, and 35 nm, respectively. The Mo/Si multilayer on c-Si substrate was subjected to AHA. In this case, the value of $T_{\text{mesh}}$ were at 1700, 1800, and 1900 °C. The $D_{\text{ms}}$, the flow rate of H$_2$ and gas pressure were 30 mm, 150 sccm and 30 Pa, respectively. The blistering was evaluated by an optical microscope.

2.5. Etching of carbon film by AHA

To measure the etching rate of a-C film, two types of a-C films were used. The a-C films were deposited by pulsed plasma-enhanced chemical vapor deposition (pulsed PECVD) [26] and filtered arc deposition (FAD) [27,28]. The pulsed PECVD-a-C:H film was deposited at 3 Pa using C$_2$H$_2$ gas. The FAD-a-C was deposited using a graphite target (99.999%) at substrate bias voltage of 200 V and 3 Pa for 15 min. The hydrogen content of the FAD-a-C film was larger than that of the pulsed PECVD-a-C:H film [28].

The a-C and a-C:H films were subjected to AHA using chamber 1. To measure the etching rate, the part of the carbon film was covered by metal mask with holes of 2 × 2 mm$^2$ in size. The step formed at boundary between masked and unmasked region was measured by a stylus surface profiler and white-light interferometer systems. The $D_{\text{ms}}$, $T_{\text{mesh}}$, the flow rate of H$_2$, and gas pressure were 80 mm, 1700 °C, 150 sccm, and 30 Pa, respectively. The $t_{\text{AHA}}$ were 10, 30, 60, and 120 min.

3. Results and discussion

3.1. Property of carbon contamination

The XPS wide and C 1s spectra of the carbon contamination and the Au film are shown in Figs. 2 (a) and (b), respectively. The optical image of the carbon contamination on the Au film is inserted in Fig. 2 (a). Brown region of 0.3 × 4 mm$^2$ in size corresponded to the soft X-ray beam pattern. Therefore, the brown region was attributed to be the carbon contamination generated during soft X-ray irradiation. The only absorption peaks of C 1s and O 1s core levels were observed in the brown region. The thickness of carbon contamination was above 2 nm because the Au peak was not observed and the detection limit of XPS was approximately 2 nm in depth [29]. The C 1s peak was decomposed to 5 peaks attributed to C-C, C-OH, C-O-C, C=O, and O=C-OH were observed at 284.6, 285.5, 286.6, 287.5, and 288.9 eV, respectively [30]. The area ratio of various chemical bonds to C-C bond of carbon contamination generated by soft X-ray irradiation were almost the same as the normal region of Au film without carbon contamination. This indicates that the contamination was formed from absorbed hydrocarbon (C$_x$H$_y$), CO, and H$_2$O in air exposure. It was reported that the build-up of carbon contamination layer was caused by cracking process of C$_2$H$_2$ via photoelectrons emitted photons [31-33]. It was considered that atomic density of carbon in contamination generated by soft X-ray irradiation was so higher than carbon contamination. Therefore, it is difficult to remove the carbon contamination generated during the soft X-ray irradiation on optical mirrors.

3.2. Removal of carbon contamination on Au mirror

The optical images of the Au mirror before and after AHA are shown in Fig. 3. The carbon contamination on the Au mirror indicated by an
arrow in Fig. 3 disappeared after AHA. The reflectance of Au mirror before and after AHA are shown in Fig. 4. The reflectance at the region of carbon contamination was low as 0.02 at 290 eV and was 20 times lower than normal region before AHA. The reflectance at the position of carbon contamination was increased from 0.02 to 0.18 by AHA. The reflectance in normal region was not changed by AHA. This indicates that the Au mirror was cleaned without damage.

3.3. Removal of carbon contamination on Ni mirror and Ni and Mo diffraction gratings

The XPS spectra and AFM images of the Ni/Cr/c-Si substrate with and without AHA are shown in Figs. 5 and 6, respectively. In this case, the carbon contamination was not formed by soft X-ray irradiation, but native contamination was generated by air exposure. The peak of C 1s was decreased by AHA. The mean surface roughness (mean Rms roughness) of the Ni surface were 0.32 and 0.33 nm before and after AHA. The roughness was almost the same as that before AHA. The reflectance spectra of the Ni mirror subjected to the AHA and UV-O3 ashing are shown in Fig. 7. In UV-O3 ashing, the reflectance around 540 eV related to O 1s core level was decreased by the oxidation of the Ni surface. On the other hand, the reflectance around 540 eV was improved by AHA because of the
removal of the native oxide on the Ni mirror.

Next, the Ni mirror with the carbon contamination was treated by AHA. The optical images of the Ni mirror before and after AHA are shown in Fig. 8. It was confirmed that the carbon contamination was removed by AHA. The change in reflectance of the Ni mirror before and after AHA are shown in Fig. 9. The low reflectance in the range from 280 to 310 eV due to the carbon contamination was observed before AHA. The reflectance of the Ni mirror was improved from 0.14 to 0.64 at the photon energy of 291 eV by AHA without any surface damage.

In addition, the Ni diffraction grating with variable line space (1200 lines/mm, Ni thickness of 80 nm) was also treated by AHA. It was confirmed that the carbon contamination on the Ni diffraction grating was removed by AHA. Furthermore, the Mo diffraction grating with lamina grating (1000 lines/mm, Mo film thickness of 50 nm) was also treated by AHA. The optical images of the Mo diffraction grating before (a) and after (b) AHA are shown in Fig. 10. The carbon contamination on the Mo diffraction grating was decreased by AHA. However, the carbon contamination remained after AHA.

3.4. Change in Mo/Si multilayer mirror by AHA

The optical images of the Mo/Si multilayers subjected to AHA are shown in Fig. 11. The circular projections with 20 - 30 µm in diameters, named blisters, were observed in a $T_{\text{mesh}}$ of 1900 °C (see Fig. 11(h)). The XPS spectra of the Mo/Si multilayers are shown in Fig. 12. At a $T_{\text{mesh}}$ of 1700 °C, the XPS spectra was not changed by AHA. On the other hand, the Mo signal appeared at $T_{\text{mesh}}$ of 1900 °C for 60 min after AHA. It was reported that the Mo/Si multilayer was broken in H$_2$ ambient above 200 °C [19]. Under condition of $T_{\text{mesh}}$ of 1900 °C, the temperature was over 200 °C. On the other hand, no the damage was confirmed even long $t_{\text{AHA}}$ (200 min) from optical images and XPS spectra under conditions of long $D_{\text{ms}}$ (80 mm) and low $T_{\text{mesh}}$ (1700 °C).

The atomic hydrogen density was increased by increasing $T_{\text{mesh}}$. It is considered that the atomic hydrogen density and the sample temperature are important for optimize the treatment condition of AHA. The reduction effect of graphene oxide did not depend on the $D_{\text{ms}}$ [8]. On the other hand, the sample temperature strongly depended on $D_{\text{ms}}$. The chamber 2 is useful for optimization of AHA condition.

The reflectance spectra of the Mo/Si multilayers subjected to AHA are shown in Fig. 13. The reflectance decreased to 0 under condition of $T_{\text{mesh}}$ of 1900 °C for 60 min. The Mo/Si multilayer was broken by the atomic hydrogen and/or thermal radiation from the heated W mesh. The decrease in reflectance corresponded to the results of optical microscope and XPS.

In addition, the reflectance spectra were changed by AHA. The maximum reflectance and peak position as a function of $t_{\text{AHA}}$ are shown in Fig. 14. The peak position is related to shrinkage and expansion of the Mo/Si multilayer. The peak position was decreased by AHA at $T_{\text{mesh}}$ of 1800 and
1900 °C. The shrinkage of the Mo/Si multilayer occurred by the thermal effect due to recombination energy of the atomic hydrogen and thermal radiation during AHA. The atomic hydrogen can diffuse into Mo and Si easily. The blisters are formed by the influence of the thermal H-atom irradiation and are caused by the layer delamination occurring exclusively at the outermost Mo on Si interface [19]. In the case of AHA, it was afraid that the blisters were formed because the atomic hydrogen density was high. However, it was confirmed that the reflectance was not degraded at $T_{\text{mesh}}$ of 1700 °C, $D_{\text{mm}}$ of 80 mm, and $t_{\text{AHA}}$ of 90 min.

3.5. Properties and etching rate of a-C films by AHA

The a-C:H and a-C films prepared by pulsed PECVD and FAD, respectively, were evaluated by Raman measurement and XPS. The peak position of the G and D bands and the intensity ratio of D band to G band ($I_D/I_G$) in Raman spectra are summarized in Table 1. As the intensity ratio of sp$^3$ to sp$^2$ increases, the $I_D/I_G$ ratio decreases [34]. The film density of a-C is related to the $I_D/I_G$ ratio [35]. Therefore, the atomic density of FAD-a-C film was higher than that of pulsed PECVD-a-C:H film. In addition, the sp$^3$/sp$^2$ area ratio of the pulsed PECVD-a-C:H and FAD-a-C in film inside were 10.5 and 0.55 in C 1s XPS spectra, respectively.

The etching depth of pulsed PECVD-a-C:H and FAD-a-C films as a function of $t_{\text{AHA}}$ is shown in Fig. 15. The measurement limit of etching depth was estimated to be about 2 nm. The etching depth of the pulsed PECVD-a-C:H film was linearly increased with increasing $t_{\text{AHA}}$. Although the etching depth of FAD-a-C film until $t_{\text{AHA}}$ of 60 min was measured to be 0 nm, it was considered that the etching depth of
the FAD-a-C film was also increased with increasing $t_{\text{AHA}}$. The etching rate estimated from the etching depth for $t_{\text{AHA}}$ of 120 min are also summarized in Table 1. It indicated that the etching rate depended on the properties of carbon film. In general, the H contents of pulsed PECVD-a-C:H films are higher than that of FAD-a-C films because the source material of C$_2$H$_2$ includes hydrogens. The etching rate of the carbon film is strongly related to the H content and sp$^2$/sp$^3$ ratio. Therefore, the etching rate of the pulsed PECVD-a-C:H film was 4 time higher than that of the FAD-a-C film.

The etching rate of the pulsed PECVD-a-C:H film was almost the same as the etching rate of carbon contamination on the Ni mirror. It is considered that the carbon contamination on the optical elements was formed from C$_2$H$_2$ during soft X-ray irradiation. Therefore, the etching rate of the carbon contamination generated by soft X-ray irradiation and the pulsed PECVD-a-C:H film was similar. The pulsed PECVD-a-C:H films used in this study are expected to be the standard carbon film to investigate the reaction between atomic hydrogen and carbon contamination and optimization of AHA conditions.

As mentioned above, the degradation of the Mo/Si multilayer was negligible under the condition at $T_{\text{mesh}}$ of 1700 °C, $D_{\text{ms}}$ of 80 mm, and $t_{\text{AHA}}$ of 90 min. It is expected that the 10-nm-thick carbon contamination could be removed by AHA without damage (blister formation) from the etching rate of the pulsed PECVD-a-C:H film.

3.6. Reaction between atomic hydrogen and carbon contamination on mirrors

The etching of carbon-related materials like photoresist due to atomic hydrogen had been widely investigated. As mentioned above, the chemical component of the carbon contamination generated during X-ray irradiation was almost the same as carbon contamination generated by air exposure. Therefore, it is considered that the reaction of between atomic hydrogen and carbon contamination on mirrors for synchrotron radiation was almost the same as carbon contamination. However, the properties of carbon films strongly affected the etching rate as shown in Fig. 15. If the etching rate depends on only atomic density, the atomic density of FAD-a-C film was 4 time higher than that of pulsed PECVD-a-C:H film. Therefore, this implies that the component of the sp$^2$ network like graphite and the sp$^3$ network like diamond also influences the etching reaction between atomic hydrogen and carbon network. In addition, the hydrogen contents in the carbon films also influenced to etching rate.

![Fig. 15. Etching depths of the pulsed PECVD-a-C:H and FAD-a-C films on the c-Si substrates by AHA as a function of $t_{\text{AHA}}$.](image)

| Carbon film     | Peak position (cm$^{-1}$) | FWHM (cm$^{-1}$) | $I_D/I_G$ | sp$^2$/sp$^3$ area ratio | Etching rate* (nm/min) |
|-----------------|---------------------------|-----------------|-----------|-------------------------|------------------------|
| Pulsed PECVD a-C:H | 1569 1341                | 185 276         | 0.56      | 10.5                    | 0.11                   |
| FAD a-C         | 1545 1360                | 218 237         | 0.41      | 0.55                    | 0.03                   |

*Estimated from the etching depth at $t_{\text{AHA}}$ of 120 min.

4. Conclusion

The surface treatment using the atomic hydrogen, named AHA, was investigated for the removal of the carbon contamination on the optical elements. The Au and Ni mirrors and the Ni and Mo diffraction gratings with the carbon contamination were cleaned by the chemical reaction and thermal effect due to the recombination of the atomic hydrogens during AHA. The reflectance of the mirrors used in synchrotron facility was recovered by AHA.

Although the Mo/Si multilayer mirror was degraded by AHA under the conditions of high $T_{\text{mesh}}$, short $D_{\text{ms}}$, and long $t_{\text{AHA}}$, it was estimated that the 10-nm-thick carbon contamination on the Mo/Si multilayer can be removed by AHA without damage.

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