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Degradation of reflectivity in stainless steel mirrors under irradiation with low-energy helium ions

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ABSTRACT

To evaluate the reflectivity change of first mirrors for plasma diagnostics, in situ measurement of the reflectivity in stainless steel has been done under the bombardment of low-energy helium ions. The damage structure associated with the degradation of the reflectivity was also examined by in situ transmission electron microscopy. It was found that the degradation behavior of optical reflectivity showed two stages; a rapid reduction stage occurred at a relatively low fluence of up to 1×10^{21} He/m², and the following one decreased monotonically without saturation. The eventual reduction in the reflectivity reached about 30% of the initial value by irradiation with 8×10^{22} He/m² at room temperature. The degradation of the reflectivity depended on the irradiation energy of helium in the order of 1, 5 and 3 keV, and its deep correlation with the radiation damage in the penetration depth of the laser light near the surface was suggested.

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1. Introduction

In order to control and evaluate ITER performance, it is necessary to measure a wide range of plasma parameters. Optical diagnostic system using a first mirror (FM) is one of the conventional and important diagnostic systems [1]. The FM faces plasma and is heavily bombarded with plasma particles and the surface is damaged. Decrease in the optical reflectivity for some candidate materials of FMs, such as Al, Cu, Mo Ta, and W [2–5], was investigated under long-term sputtering with hydrogen or deuterium ions. Balden et al. also studied surface roughening of stainless steel by hydrogen bombardment [6]. The effects of fast neutrons on the optical properties of multilayer materials were studied [7] and simulated using high-energy ions in stainless steel, Cu and Be [8].

In the case of DT burning fusion device, FM is bombarded with a fairly high flux of particles such as hydrogen isotopes and helium. Damaging of the materials by helium is much stronger and serious than that by hydrogen isotopes [9,10]. Therefore, studies on the effects of helium irradiation on the reflectivity and radiation damage in FM materials are necessary. However, a little information is available at present [9].

Therefore, in the present work, it was aimed to investigate the degradation of the reflectivity and its correlation with the damage structure in stainless steel irradiated with low-energy helium ions. 2. Experimental

Specimens used in the present study were made from base materials of solute annealed type 316L stainless steels (SUS316L, Fe - 0.015 wt% C - 0.52 wt% Si - 0.96 wt% Mn - 0.02 wt% P - 0.004 wt% S - 12.39 wt% Ni - 16.28 wt% Cr - 2.12 wt% Mo - 3 wtppm B). The specimens carved out from the base materials were cut to a size of 10 \times 10 \times 0.5 mm³.

After mirror-polishing mechanically with 50 nm alumina powder, the specimens were irradiated with 1, 3 and 5 keV helium ions at room temperature, respectively, up to a fluence of about 1×10^{23} He/m². Irradiation was performed with a newly built device for the present study. This device enabled in situ measurements of the reflectivity change during irradiation. The schema of the device is shown in Fig. 1. The reflectivity change under irradiation with a flux of 5×10^{17} He⁺/m²s was measured with a stable semiconductor laser with a wavelength of 670 nm and a photodetector, which were placed at an angle of 45° from the normal direction on the specimen surface, as shown in Fig. 1. The reflectivity in the range from 190 to 2500 nm in the wave length was also measured with a spectrophotometer after irradiation.

In addition to the reflectivity measurement, the microstructure evolution induced by irradiation was examined by SEM and TEM to obtain the information about the correlation with the reflectivity.

3. Results

* Corresponding author. E-mail address: miyamoto@riko.shimane-u.ac.jp (M. Miyamoto). The reflectivity obtained under irradiation decreased drastically with the increase in the fluence, as shown in Fig. 2, where the



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Fig. 1. Schematic view of the experimental setup.



Fig. 2. The fluence dependences of reflectivity in SUS316L irradiated with 1, 3 and 5 keV-He^+ at room temperature.

reflectivity change in SUS316L irradiated with 1, 3, and 5 keV He⁺ ions at room temperature, respectively, is plotted as a function of the fluence. One should note that the reflectivity continues to decrease up to a rather high fluence without saturation in the present

experiment. In the worst case obtained during irradiation with 3 keV-He⁺, the reflectivity fell to less than 20% at a fluence of 8.7×10^{22} He/m², whereas the initial value was 66%. As seen from the figure, two stages in the reflectivity degradation appeared at the three energies of irradiation. First, a rapid reduction occurred at a relatively low fluence region up to 1×10^{21} He/m², and then a gradual decrease stage followed without saturation. In comparison with the irradiation energies of 1, 3 and 5 keV, peculiarly, the most considerable reduction was observed during 3 keV irradiation. As discussed in the following sections, the radiation damages that occurred and their depth distribution in the irradiated specimens seemed to play an important role in the degradation of the reflectivity.

In order to investigate the causes of the significant degradation, the microstructure observations were carried out by means of electron microscopy. The SEM images of the surface morphology showed little change after irradiation, and a smooth surface was observed in every specimen contrary to expectation. On the other hand, TEM observation of internal microstructure showed the heavy damages induced by helium irradiation. Fig. 3 shows the microstructure evolution under irradiation with 3 keV-He⁺ at room temperature. The sharp black dot contrasts obtained at small s diffraction condition (upper) and circular white contrasts at large s condition (lower) are I-type dislocation loops and He bubbles, respectively. These fine defects clusters started to form at a relatively low fluence of about 1×10^{20} He/m² and increased in density immediately. Because of the dense defects, it became difficult to resolve the microstructure evolution at higher fluences of over $1 \times 10^{21} \text{ He/m}^2$.

4. Discussion

A serious degradation in the optical reflectivity of the mirror occurred under helium irradiation. Furthermore, the decrease continued until a high fluence without a clear saturation in this experimental condition. Taking account of a usage environment under burning plasma such as ITER, we worry that the mirror seems to lose its function in a short time. To avoid unexpected results, it is important to examine the mechanism of the decrease in the optical reflectivity. In this section, the possible factors of the decrease are discussed.

The microstructure observations with SEM and TEM showed much more change in the internal structures rather than at the



Fig. 3. Microstructure evolution in SUS316L under irradiation with 3 keV-He⁺ at room temperature. The sharp black dot contrasts obtained at small s diffraction condition (upper) and circular white contrasts at large s condition (lower) are I-type dislocation loops and He bubbles, respectively.



Fig. 4. Depth distribution of damages induced by irradiation with 1, 3, 5 keV-He⁺ calculated by TRIM-code [12].



Fig. 5. Reflectivity of SUS316L irradiated with 1, 3 and 5 keV-He⁺ to a fluence of $8\times10^{22}\,\text{He/m}^2$ at room temperature. The data of unirradiated specimens are plotted for comparison.

top surface of the specimen. Especially, the drastic decrease in the reflectivity observed until the fluence of $1 \times 10^{21} \text{ He/m}^2$ seems to be strongly associated with the considerable formation of defects clusters such as I-loops and helium bubbles, whose density increased to great quantities until the fluence of 1×10^{21} He/m². In fact, the optical reflection is affected by not only a top surface state but also a sub-surface one in a penetration depth of the laser light. The penetration depth, δ , is given by $\delta = \lambda/4\pi\kappa$, where λ and κ are the wavelength and the substance-specific extinction coefficient, respectively [11]. In this experimental condition, the value of δ is estimated to be about 15 nm for a laser wavelength of 670 nm. On the other hand, damages induced by irradiation with 1, 3 and 5 keV-He⁺ distributed as shown in Fig. 4, where the calculation was performed by TRIM code [12]. It is known that a large number of defects are formed within the range of the penetration depth for the three energies of irradiation. Additionally, this damage distribution, shown in Fig. 4, gives a good explanation for the strange energy dependence of the decreasing reflectivity shown in Fig. 2. The largest amount of damage within the depth of 15 nm occurred during irradiation with 3 keV-He⁺. Corresponding to the amount of the damage, we can consider that the largest reduction rate of reflectivity is observed in the specimen irradiated with 3 keV-He⁺. This relation between the amount of defects and the penetration depth is recognized in the wavelength dependence of the reflectivity. Reflectivity of the specimens irradiated with 1, 3 and 5 keV-He⁺ to a fluence of 8×10^{22} He/m² at room temperature for a wavelength from 200 to 800 nm is shown in Fig. 5. The reflectivity of the specimen irradiated with 1 keV-He⁺ drastically decreases for shorter wavelengths with a shallower penetration, which is consistent with the congestion of defects within the shallower region in the case of 1 keV-He⁺ irradiation as shown in Fig. 4.

In the case of heavy irradiation of over ${\sim}10^{22}~\text{He}^{+}/\text{m}^{2}$, where the reflectivity monotonically decreases, the surface sputtering and damaging should be effective for the degradation of the reflectivity. Actually, the sputtering thickness in the specimen irradiated with 3 keV He^{+} ions to a fluence of 1 \times 10²³ He/m² is estimated to reach about 100nm. The mechanism of the reflectivity degradation in the heavy irradiation stage is being studied by using additional techniques such as STEM-EELS, and the results will be reported in near future.

5. Summary

The optical reflectivity change of mirrors under helium irradiation in SUS316L was examined with a newly built device. In the worst case of this examination, the reduction in the reflectivity reached to 30% of the initial value by irradiation at room temperature. The strong correlation between the amount of radiation damages in the penetration depth of the laser light near the surface and the decreasing of the reflectivity indicates that the damage that occurred in the penetration depth is one of the most important factors in the mechanism of the decrease in the optical reflectivity. Since the more complex factors are expected in the case of heavy irradiation, further investigation into the details is required.

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