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Irradiation test of Mo- and W-mirrors for ITER by low energy deuterium ions

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Abstract

Metallic materials such as molybdenum and tungsten will be used for plasma facing mirrors in ITER. Test pieces of molybdenum and tungsten mirrors manufactured by different methods were irradiated by high flux (5.2–6.0×10^{19} $m^{-2}\,s^{-1}$), low energy (67–80 eV) deuterium ions. The molybdenum mirror manufactured by sintering and electron beam melting of a thin layer of the surface had a higher reflectivity before irradiation than the mirror made without surface melting, but the reflectivity of the former mirror decreased rapidly with fluence. This rapid decrease was caused by blisters generated on the mirror surfaces. The decrease of the 250 nm–20 μm wavelength reflectivity of the molybdenum and the tungsten mirrors made by sintering without surface melting was less than 10% at fluence of 1.2–1.3×10^{25} m^{-2}.

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1. Introduction

In ITER, optical components, such as mirrors mounted close to the plasma, will experience higher levels of radiation due to neutron, gamma ray and particle irradiation than in present devices. It has been confirmed that metallic mirrors, such as molybdenum and tungsten, have a strong resistance to neutron and gamma ray irradiation [1,2]. On the other hand, the effects of energetic particle (charge exchange atoms) bombardment on the mirror should still be studied [3], especially for the lower energy particle bombardment expected on mirrors located near the divertor where the plasma has a much lower temperature. In order to study the effect on the optical properties, test pieces of molybdenum mirrors manufactured by different methods, and a tungsten mirror, were irradiated by high flux (5.2–6.0×10^{19} $m^{-2}\,s^{-1}$), low energy (67–80 eV) deuterium ions up to a fluence of 1.3×10^{25} m^{-2} using the SLEIS (super low energy ion source with high ion flux) facility [4] at a temperature near 100 °C. These flux and fluence levels are representative of those that will be experienced by mirrors in ITER [5].

2. Materials and test methods

Test pieces of molybdenum-mirrors (Mo-mirrors) and a tungsten-mirror (W-mirror) with size 25 mm × 25 mm × 5 mm were irradiated by deuterium ions using the SLEIS facility as shown in Fig. 1. The manufacturing methods and the irradiation conditions for each test piece are summarized in Table 1. Test piece Mo-1 was manufactured by polishing a sintered molybdenum substrate. Test pieces Mo-m1 and Mo-m2 were manufactured by electron beam melting a thin layer of the surface before polishing, in order to eliminate small holes on the surface formed in the sintering process. Test piece W-1 was manufactured by polishing a sintered tungsten substrate. Mo-m2 was irradiated by deuterium ions with energy 80 eV and the other test pieces were

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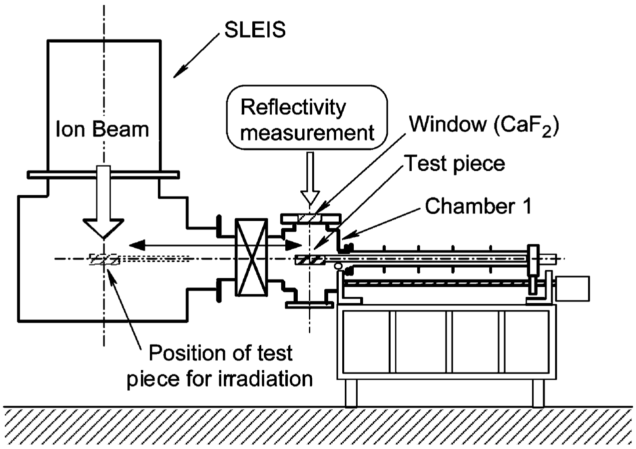

Fig. 1. Experimental setup for irradiation and reflectivity measurement of mirror test pieces.

Table 1
Materials, surface treatments and experimental conditions for each mirror test piece

Symbol of test piece	Mo-1	Mo-m1	Mo-m2	W-1
Material	Sintering Mo	Sintering Mo	Sintering Mo	Sintering W
Surface treatment	Polishing[a]	Melting and polishing[a]	Melting and polishing[a]	Polishing[a]
Irradiation particle	Deuterium	Deuterium	Deuterium	Deuterium
Acceleration voltage (V)	200	200	240	200
Particle energy (fraction)	67 eV (92%) 100, 200 eV (8%)	67 eV (90%) 100, 200 eV (10%)	80 eV (90%) 120, 240 eV (10%)	67 eV (90%) 100, 200 eV (10%)
Particle flux (m^{-2} s^{-1})	5.2×10^{19}	5.4×10^{19}	6.0×10^{19}	5.6×10^{19}
Particle fluence (m^{-2})	Up to 1.3×10^{25}	Up to 3.7×10^{24}	Up to 3.7×10^{24}	Up to 1.2×10^{25}
Temperature (°C)	–	~110	~100	~100

[a] Polishing was made mechanically using abrasive containing diamond paste.

irradiated by deuterium ions with energy 67 eV. Since the beam current density of the ion source is almost uniform (uniformity $\leqslant 5\%$) in the area (25 mm × 25 mm) of the test pieces [6], the deuterium flux F on the test piece was derived by measuring the current I_T on the test piece using the equation $F = I_T \times (3f_{D_3^+} + f_{D^+})/A_T/e$, where, $f_{D_3^+}$ and f_{D^+} are the fractions of D_3^+ and D^+ in the ion beam. Those fractions were measured by a beam analyzer. A_T is the area of the test piece and e is the electron charge. The variation of the flux was maintained within 10% during the irradiation by controlling the ion current. The temperature of the test piece was measured by a thermocouple in contact with the side of the test piece.

The test piece on the holder is moved to below the ion source for irradiation. The surrounding deuterium pressure of the test piece was 1.6×10^{-1} Pa. After the irradiation, the test piece was translated to the vacuum chamber 1 and the reflectivity was measured without any vacuum break (Fig. 1). The reflectivity measurements were carried out in the wavelength range 250–2000 nm. In addition, the reflectivity of the test piece Mo-1 was measured in the wavelength range of 2.5–20 μm by using a Fourier transform-infrared (FT-IR) spectrometer at Nagoya University after the irradiation. The surface condition was observed using a scanning electron microscope (SEM) and a laser microscope.

3. Results

Fig. 2 shows the reflectivity of the test piece Mo-1 before and after the irradiation (fluence: 1.3×10^{25} m^{-2}) in the wavelength range 250 nm–20 μm at the incident angle of 45°. The drop of the reflectivity was less than 5% for wavelengths > 2 μm. The reflectivity as a function of fluence was measured at the various wavelengths in the range 250–2000 nm with the incident angles of 45° (Fig. 3(a)) and 10°. For both incidence angles, the reflectivities decreased with the fluence. The decreases

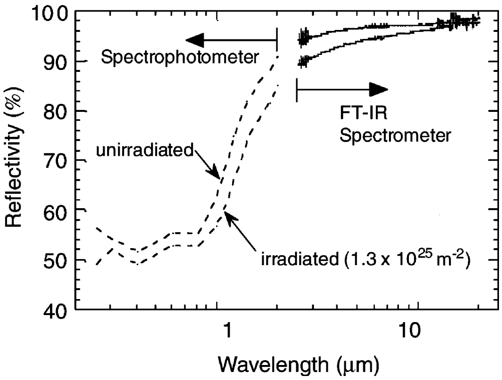

Fig. 2. Reflectivity of the unirradiated and the irradiated molybdenum mirror (Mo-1) in the wavelength range 250 nm–20 μm measured by the spectrophotometer and the FT-IR spectrometer at the incident angle of 45°.

were less than 10% at the fluence of 1.3×10^{25} m^{-2}. The reflectivity of the test piece Mo-m1 is shown in Fig. 3(b) as a function of fluence. The reflectivity was larger than that of Mo-1 before irradiation. However, the decrease rate of the reflectivity of Mo-m1 vs. fluence was larger than that of Mo-1 for the same fluence levels. The decreases for Mo-m1are 25–30% at a fluence of 3.7×10^{24} m^{-2} for the incident angle of 45°. The reflectivity of Mo-m2 irradiated with energy 80 eV (90%) also decreased with the fluence, and the decrease rate was larger than that of Mo-m1 irradiated with the lower energy of 67 eV.

Fig. 3(c) shows the reflectivity of W-1 as a function of fluence at the various wavelengths measured with incident angle 45°. The reflectivity decreases with the fluence but the decrease is less than 10% in the wavelength range of 250–2000 nm at a fluence of 1.2×10^{25} m^{-2}.

Fig. 4 shows the surfaces of test pieces observed by the SEM after the irradiations. The holes and blisters were observed on the surface of Mo-1. Those holes seem to be formed in the sintering process since the same holes were observed before irradiation. There was no significant change on the surface before and after the irradiation except blisters. The depth of the holes and the heights of the blisters were measured by a laser microscope to be near 0.5 and 1–2.5 μm, respectively. Many larger blisters were observed on the surfaces of Mo-m1 and Mo-m2 manufactured by melting the thin layer of the surface by electron beam, in order to eliminate holes observed on the surface of Mo-1. The number of blisters on Mo-m2 was larger than those on Mo-m1. On the other hand, the smaller blisters were observed on the surface of tungsten mirror W-1. The size of the blisters on the surface of W-1 was less than a few μm. The elemental analysis of the blisters by an electron probe micro analyzer (EPMA) confirmed that they had the same composition as the material of each mirror. There was no compositional change in the irradiated surface. No damage by sputtering was observed on the mirrors tested.

4. Discussion

Fig. 5 compares the reflectivity of Mo-1, Mo-m1, Mo-m2 and W-1 as a function of fluence at a wavelength of 800 nm. The reflectivity of Mo-m1 and Mo-m2 were higher than Mo-1 before irradiation but the decrease rates were larger than that of Mo-1. The decrease rate of Mo-m2 was larger than that of Mo-m1. The decrease rate of W-1 was not so significant as that of Mo-1. Similar results were recorded at other wavelengths. The results were supported by surface observations using the SEM. We found no surface damage due to sputtering, which was expected from the very low sputtering yield by deuterium ions at the low energy range [7], but many blisters were observed on the surfaces. The reduction of reflectivity with fluence seems to originate from the increase of the blisters on the surfaces. More and larger blisters, which seem to be generated by gas pressure, were observed on the molybdenum mirrors (Mo-m1,

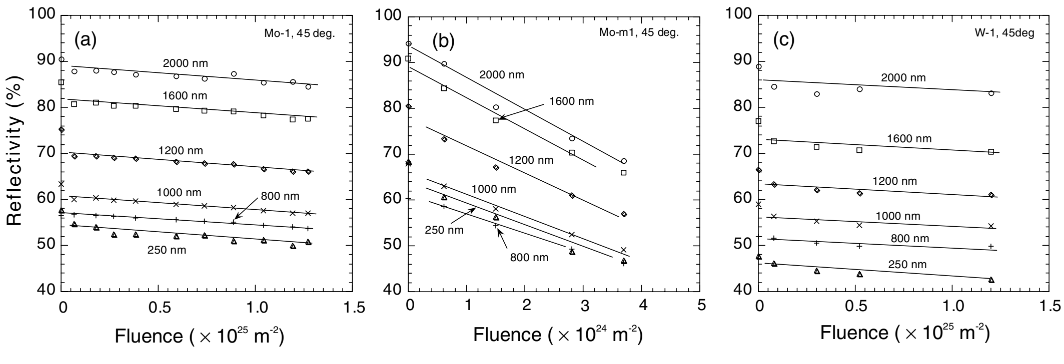

Fig. 3. Reflectivity of test pieces (a) Mo-1, (b) Mo-m1 and (c) W-1 as a function of fluence measured at various wavelengths in the range 250–2000 nm with the incident angle of 45°.

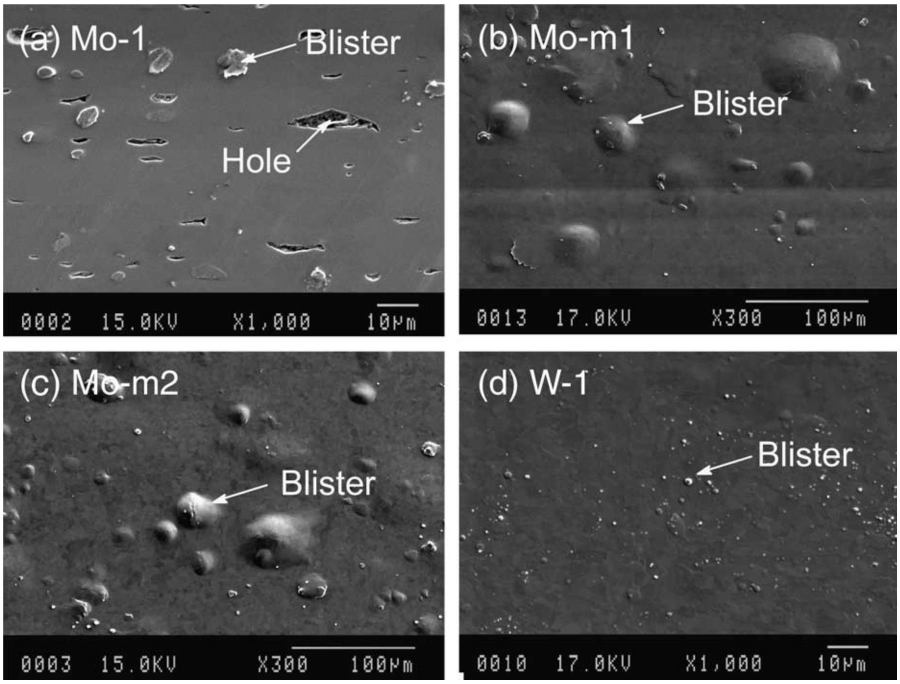

Fig. 4. Surfaces observed by SEM; (a) Mo-1 [fluence: 1.3×10^{25} m^{-2}, irradiation energy: 67 eV], (b) Mo-ml [fluence: 3.7×10^{24} m^{-2}, irradiation energy: 67 eV], (c) Mo-m2 [fluence: 3.7×10^{24} m$^{-2,}$ irradiation energy: 80 eV], and (d) W-1 [fluence: 1.2×10^{25} m^{-2}, irradiation energy: 67 eV].

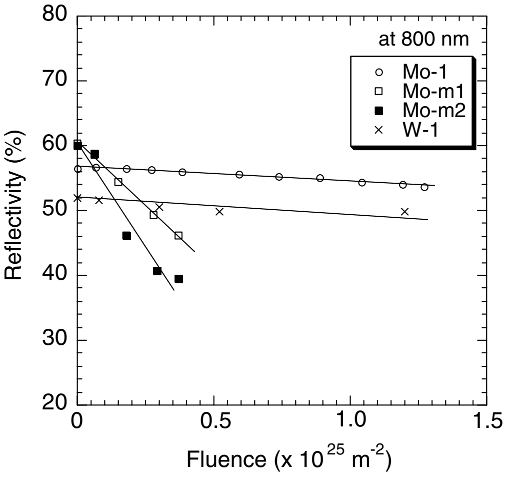

Fig. 5. Reflectivity at 800 nm of test pieces Mo-1, Mo-ml, Mo-m2 and W-1 as a function of fluence. The reflectivity of Mo-ml and Mo-m2 were higher than Mo-1 before irradiation but the decrease rates were larger than that of Mo-1.

Mo-m2) manufactured by melting the thin layer of the surface by electron beam than the mirror Mo-1 manufactured by sintering only. The surface damage of Mo-m2 irradiated with 80 eV deuterium ions was larger than that of Mo-ml irradiated with 67 eV deuterium ions. It seems that the damage becomes larger with the energy of irradiated ions. The surface damage of W-1 was not so significant and blisters on the surface were very small comparing with those of molybdenum mirrors. It is considered that the above results originate mainly from the manufacturing method of the mirrors. The surface melting to eliminate holes on the surface decreased the resistance to blistering.

The reflectivity R can be written as $R = R_0\exp(-(4\pi\delta\cos\theta/\lambda)^2)$, where R_0 is the reflectivity of a perfectly smooth surface, δ is the average roughness, θ is the incident angle, and λ is the wavelength [8]. We calculated the ratio $R_2/R_1 = \exp(-(4\pi(\Delta\delta)\cos\theta/\lambda)^2)$ for various $\Delta\delta = \delta_2 - \delta_1$ in the range (20 μm $> \lambda >$ 1 μm $\gg \delta$), where R_1, δ_1 and R_2, δ_2 are the reflectivity and the average roughness before and after the irradiation, respectively. The calculated ratio agreed with the measured value within 5% for $\Delta\delta$ of 40–60 nm. On the other hand, the $\Delta\delta$ of Mo-1 was estimated from the surface observations by SEM and the laser microscope as 33–53 nm ($\delta_1 = 19$ nm, $\delta_2 = 52$–72 nm). From this result, the decrease of the reflectivity in the IR region of Mo-1 seems to be explained by the increase of the surface roughness. In the visible and ultraviolet region, further investigation is needed.

5. Conclusion

It was confirmed that the mirrors manufactured from sintered molybdenum and tungsten substrates are resistance to degradation by low energy deuterium ion irradiation. The data will be useful for designing the plasma facing diagnostics mirrors in ITER. On the other hand, it will be important to estimate the particle flux and the energy on each mirror located at various positions in ITER to estimate the lifetime of the mirrors. Irradiation tests of mirrors manufactured from single crystal molybdenum, tungsten etc., which have a better optical performance for particle irradiations [3], should be carried out. In addition, it is important to estimate the effect of bombardment by impurity particles, such as beryllium and carbon originating from the first wall and the divertor plate, which can lead to deposition on the mirror surface.

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