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Surface modification of titanium by the exposure to helium plasma
ヘリウムプラズマ照射によるチタンの表面構造変化

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The formation of micron-sized helium bubbles/holes and fibreform nanostructure on metals due to helium plasma exposure has been investigated. Because these nanostructures have very large surface areas, they have potential to increase the effective surface area and improve catalytic properties. Thus, the application of these nanostructure to catalytic materials is suggested. In this study, we investigated the surface modification of titanium under helium plasma exposure using the liner divertor simulator NAGDIS-I. And we explore the necessary condition, such as the incident ion energy and surface temperature, for the fibreform nanostructure formation in titanium by helium irradiation.

1. Introduction

Titanium dioxide (TiO_2) has attracted much interest from the scientific and industrial fields, such as for light-induced water splitting, dye-sensitized solar cells, and self-cleaning surfaces [1]. Due to its unique electronic and catalytic properties, the photo-degradation of organic contaminant becomes one of the most important application of TiO_2 [2]. Because these catalytic reaction occurs on the material surface, fabrication of large effective surface areas are necessary for improving the photo-catalytic properties.

The irradiation of helium to metals leads to microstructural evolution on the surface region, such as helium holes/bubbles and fibreform nanostructure [3]. These structures have potential to increase the effective surface area and improve photo-catalytic properties [4]. However, the necessary conditions for the fibreform nanostructure formation of metals, except for tungsten, have not been investigated yet.

In this study, the change of the titanium surface nature under helium plasma exposure is investigated by the measurement of the relative reflectance and the surface analysis using a scanning electron microscope (SEM). Furthermore, we explore the condition for the fibreform nanostructure formation in titanium by helium irradiation.

2. Experiments

The substrate materials were titanium provided by Nilaco Co. with 99.5 % purity and 0.5 mm in thickness. Samples were polished to a mirror finish. Irradiation of helium plasma was carried out using the liner divertor plasma simulator NAGDIS-I. Steady state high-density plasma is produced by a dc arc discharge using a LaB_6 cathode heated with a filament. The magnetic field strength was 0.05 T. In order to control the incident ion energy to the samples, they were electrically biased in some experiments. Fig. 1 shows the schematic of the experimental setup in the NAGDIS-I. The reflectivity of samples was measured by using a He-Ne laser, whose wavelength is 632.8 nm, and a photodiode with a band-pass optical filter to exclude the emission from the plasma. The surface temperature of the samples was measured by radiation thermometer. After the helium plasma irradiation, the surface modification of samples was observed by SEM.

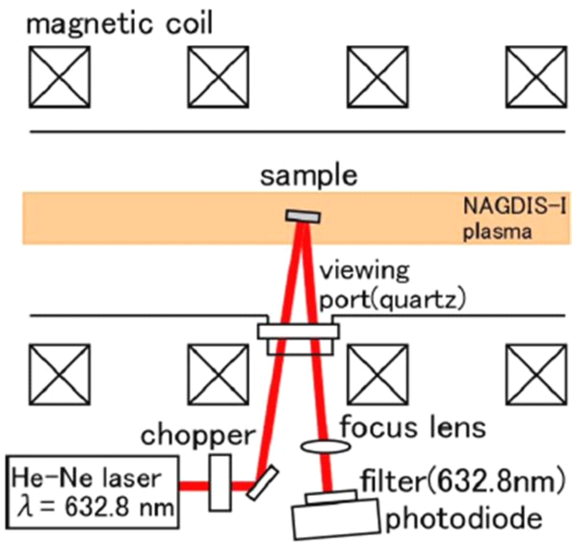

Fig. 1. A schematic of relative reflectance measurement setup

3. Results and discussion

The exposure conditions of helium plasma are summarized in Table 1. The electron density n_e and temperature T_e were from 2.5×10^{18} to 4.3×10^{18} m^{-3} and 8 eV, respectively. These plasma parameters were measured with a Langmuir probe. The samples were biased to -70 V (Ti1), -60 V (Ti2), -75 V (Ti3), and -85 V (Ti4), respectively. Fig. 2 shows the relative reflectance of samples exposed to the helium plasma. For Ti1 and Ti2, the relative reflectance was kept high. On the other hand, the reflectance decreased to around zero for Ti3 and Ti4; further the reflectance decreased during shorter period of time for Ti4. Even though the ion flux did not change so much in Ti3 and Ti4, the reduction time in reflectance decreases significantly with increasing the incident ion energy. Although the surface temperature slightly increases with the incident ion energy, the surface damages should strongly depend on the incident ion energy.

Fig. 3(a)–(d) shows the SEM micrographs of the samples after the helium plasma exposure. On the Ti1 surface, neither hole nor fibreform nanostructure is observed (Fig. 3(a)). On the other hand, helium bubbles and holes are formed on the Ti2 surface (Fig. 3(b)). For Ti1 and Ti2, incident ion energy was about the same. Thus, the helium babble formation would depends on the surface temperature. Concerning Ti3, and Ti4, the surface color was totally black visually after the helium irradiation. The surfaces of Ti3 and Ti4 are covered with a micrometer fine structured material shown in Fig. 3(c) and (d). Compared with these fibreform nanostructures, the size of nanostructure of Ti4 is larger than that of Ti3. Since the surface temperature of Ti3 and Ti4 were almost the same, it is likely that the size of fibreform nanostructure depends on the incident ion energy.

4. Conclusion

We have investigated the necessary conditions for the fibreform nanostructure formation in titanium by helium irradiation. We have found that the incident ion energy and surface temperature are important parameters to form fibreform nanostructured titanium.

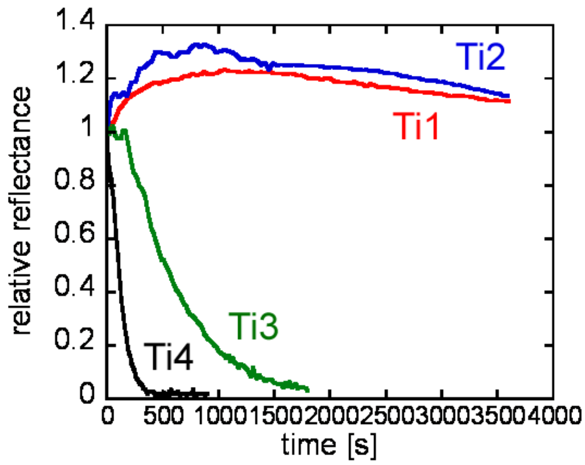

Fig. 2. Measured relative reflectance of titanium under helium plasma exposure; the detail exposure conditions are summarized in Table 1.

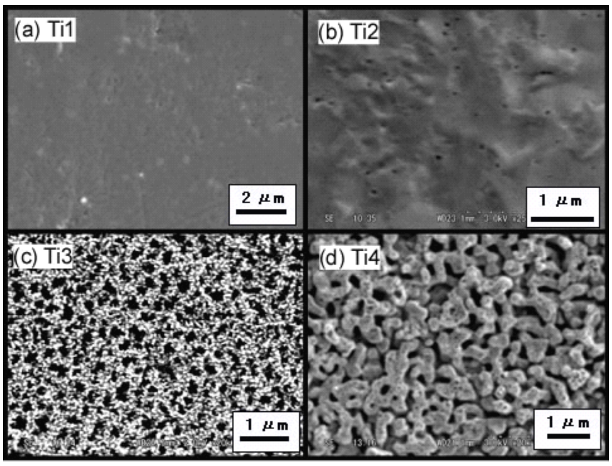

Fig. 3. SEM micrographs of titanium exposed to helium plasma under the conditions shown in Table 1.

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Table 1. Experimental parameters of the helium irradiation experiment for Ti1, Ti2, Ti3, and Ti4

	Ti1	Ti2	Ti3	Ti4
Incident ion enrgy	62 eV	56 eV	73 eV	83 eV
Fluence	8.4×10^{25} m^{-2}	7.6×10^{25} m^{-2}	4.7×10^{25} m^{-2}	2.1×10^{25} m^{-2}
Ion flux	2.3×10^{22} $m^{-2}s^{-1}$	2.1×10^{22} $m^{-2}s^{-1}$	2.6×10^{22} $m^{-2}s^{-1}$	2.3×10^{22} $m^{-2}s^{-1}$
Time	3600 s	3600 s	1800 s	900 s
Temperature	580 K	660 K	750 K	760 K
Surfacemodification	-	He bubble/holes	Fibreform nostructure	Fibreform nanostructure