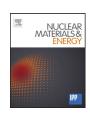
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Surface modification and sputtering erosion of iron and copper exposed to low-energy, high-flux deuterium plasmas seeded with metal species

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ABSTRACT

Four sets of targets were used in this study: (1) Fe targets surrounded with 304 type stainless steel composed of mid-Z elements: Fe, Cr, Ni, and Mn (designated as Fe[304SS] targets), (2) Fe targets surrounded with high-Z tungsten (designated as Fe[W] targets), (3) Cu targets surrounded with mid-Z copper (designated as Cu[Cu] targets), and (4) Cu targets surrounded with high-Z tungsten (designated as Cu[W] targets). The targets were exposed to low-energy (140 and 200 eV), high-flux (about 10^{22} D/m²s) deuterium (D) plasmas at various temperatures in the range from 355 to 740 K. The surface morphology of the Fe and Cu targets is found to be dependent strongly on atomic number of re-deposited species and on the exposure temperature. For the Fe[W] and Cu[W] targets, due to formation of the W-enriched nano-sized structures on the target surfaces, the sputtering erosion yield is lower than that for the Fe[304SS] and Cu[Cu] targets, respectively. For the Fe[304SS], Fe[W], and Cu[W] targets, the sputtering erosion yield is increased distinctly as the exposure temperature rises from 355 to 740 K.

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1. Introduction

Reduced Activation Ferritic/Martensitic (RAFM) steels such as F82H [1], EUROFER [2], RUSFER [3], CLF-1 [4] are candidate materials for first wall and breeding blanket structural application in future fusion power plants [5]. However, a few years ago it was suggested to consider the RAFM steels in certain areas of the main chamber wall in DEMO reactor where the particle and power flux are not prohibitive [6]. To assess the possibility to employ the bare RAFM steels as plasma-facing materials, there is a need to examine a behavior of the steels under plasma exposure. Special attention must be placed on erosion of such steels since it would play a major role in component lifetime assessment.

The erosion processes of metallic materials irradiated with energetic ions have been widely investigated under laboratory condi-

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tions [7–21], however not much is known about temperature dependence of the erosion yield.

Experiments on sputtering of Au, Ag, Cu, Zn, Bi, Ge targets by 45 keV Xe, Ar and Ne ions have shown that the effect of target temperature on sputtering erosion is, in general, small for temperatures not too close to the melting point. The erosion yield rises sharply when the temperature approaches the melting point [15–17].

The overall erosion of 316 stainless steel bombarded with 330 to 6000 eV hydrogen and helium ions at temperatures above 573 K increased to about 1.7 times the yield at room temperature [18, 19]. On the other hand, the total erosion yield for 304 LN stainless steel irradiated with 1 keV deuterium ions was found to be nearly independent of the target temperature in the temperature range between 300 K and 800 K, although the surface structure was drastically changed. At temperatures above 1000 K the erosion yield increased combined with preferential sputtering of Cr and Ni which may be explained by thermal diffusion and segregation [20].

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Particle-induced sputtering of alloys consisting of elements with different atomic number (Z) results in a surface enrichment of element with higher Z due to preferential sputtering of lower Z elements [9]. This effect should be very strong if the energy of incident particles is close to the sputtering threshold of higher-Z element.

It has been reported recently [22–28] that exposure of multicomponent F82H and EUROFER RAFM steels and Fe-W layers (as a surrogate) to low-energy (40–200 eV) high-flux (>10²¹ ions/m² s) deuterium (D) plasmas leads to formation of nanostructured near-surface layers enriched with tungsten.¹ Note that high concentration of tungsten is observed on tips of nano-sized fibers forming the nanostructured near-surface layer [23]. The W-enriched layer reduces the Fe sputtering leading to a decrease of the fluence-integrated sputtering erosion yield² with increasing D fluence [23, 25, 28]. Additionally, it has been observed that the fluence-integrated erosion yield of the F82H steel increases by a factor of about two as the D plasma exposure temperature rises from 403 to 773 K [22].

However, for the F82H and CLF-1 RAFM steels exposed to lowenergy (80 eV) high-flux He plasma, the He fluence-integrated sputtering yields of Fe and Cr do not depend on the exposure temperature in the range of 573–873 K, but start to increase at 900 K [29]. Sputtered Fe and Cr atomic fluxes are found to drop during the plasma exposure that is explained by surface enrichment of W and development of surface morphology [29].

It is pertinent to note that materials with low-*Z* (Be) and high-*Z* (W) are candidate plasma-facing materials, and, therefore, one might expect that W impurities will be deposited on the Be surface. To gain a better understanding of temperature dependences of the sputtering erosion yield and an influence of high-*Z* element impurities on the surface morphology evolution of the mid-*Z* targets, additional sputtering experiments with pure mid-*Z* metal targets are required. The goal of this work was to examine temperature dependences of the surface modification and sputtering erosion yield of mid-*Z* Fe and Cu targets surrounded with mid-*Z* (Fe, Cr, Ni, Mn, Cu) and high-*Z* (W) elements under exposure to low-energy, high-flux deuterium plasma.

It is known that in the linear cascade regime of sputtering, the higher is the surface energy³ of a metal, the lower is the sputtering yield [13]. In this respect, it will be interesting to examine temperature dependences of the sputtering erosion yields for mid-Z metals with different values of the surface energy, $E_{\rm bind}$, namely, for Fe and Cu. Note that $E_{\rm bind}$ (Cu) \approx 3.5 eV, whereas $E_{\rm bind}$ (Fe) \approx 4.3 eV [13].

2. Experimental

Iron (Fe, 99.99 wt.% purity) and copper (Cu, 99.96 wt.% purity) provided by The Nilaco Corp., Japan, were used in this study. Rectangular-shape samples, $10 \times 10~\text{mm}^2$ in size and about 0.8 mm (Fe) and 0.3 mm (Cu) in thickness, were cut from plates of each material followed by mechanical polishing to a mirror-like finish and cleaning in an ultrasonic bath.

The targets were exposed at various temperatures (355–740 K) to D plasma consisted of $\rm D_2^+$ (about 70% of the ion flux) and $\rm D^+$

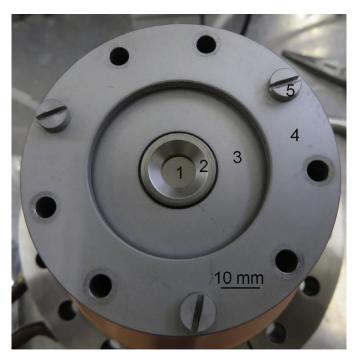


Fig. 1. View of sputtering Fe target (#1) fixed on the holder with the help of type 304 stainless steel rings (## 2, 3, 4) and three molybdenum bolts (5).

(about 30%) in the linear plasma generator (LPG) located at National Institutes for Quantum and Radiological Science and Technology (QST, the former JAEA), Rokkasho, Japan [30]. The incident D particle flux was fixed in the range from 0.7×10^{22} to $1.3\times10^{22}\,D/m^2s,$ whereas the D fluences were fixed at $5\times$ 10^{25} and 10^{26} D/m². It should be noted that the D particle flux was calculated as monitored D ion flux⁴ multiplied by 1.7 (i.e., $0.3 + 2 \times 0.7$). In turn, the D fluence, Φ , was determined by integrating the D particle flux over the exposure time. Bias voltages of -144 and -204 V were applied to the targets, resulting in incident ion energies, $E_{\rm inc}$, of 140 and 200 eV, correspondingly, taking into account the plasma potential of about $-4\,\mathrm{V}$ as measured by a Langmuir probe. The required exposure temperature was set by the thermal contact between the target and the water-cooled holder and by slight variation of the incident D ion flux. The temperature was monitored using a type K thermocouple tightly pressing the rear of the target.

In the first set of experiments, each of the Fe sputtering targets was fixed on the LPG holder with the help of rings (Fig. 1) manufactured from 304 type stainless steel (304SS) of composition 18–20 wt.% Cr, 8–12% wt.% Ni, <2 wt.% Mn, balance Fe. The 304 type stainless steel was chosen to examine influence of mid-Z metals, other than Fe, on the surface morphology of the Fe target. Note that the sputtering erosion yields of these mid-Z elements (Cr, Ni, Mn, Fe) are practically equal in magnitude [7].

However, to reveal an effect of high-Z material deposition both on the surface microstructure and on the erosion yield, in the second set of experiments the small fixing ring No. 2 (Fig. 1) was replaced by the ring manufactured from tungsten. Besides, the surfaces of the 304SS rings Nos. 3 and 4 (Fig. 1) facing the plasma were coated with W. The W coatings were deposited in a commer-

 $^{^1}$ Along with the base material: iron, RAFM steels contain typical mid-Z steel elements (chromium, vanadium, manganese) as well as small amounts of high-Z elements such as tungsten ($\approx\!0.4\,\div\approx\!0.8$ at.% W) and tantalum ($\approx\!10^{-2}$ at.% Ta).

² The fluence-integrated erosion yield is calculated by dividing the total number of sputtered atoms by the integrated number of incident D particles at given D fluence.

 $^{^3}$ The surface binding energy is defined here as the energy, which is necessary to apply in order to remove an atom from the top surface layer in vacuum during the ion sputtering process.

⁴ The D ion flux was determined by measuring ion current through metal target fixed on the special moveable probe and electrically isolated from fixing ring. This probe can be moved into plasma about 3 cm upstream from the target holder. Note that an error in determination of the D ion flux is caused mainly by ion-induced secondary electron emission and, at D ion energies \leq 200 eV, does not exceed 30% [31-33].

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