

Deuterium Retention in Tungsten-Coated Reduced Activation Ferritic/Martensitic Steel^{*)}

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(Received 22 November 2012 / Accepted 7 May 2013)

In order to evaluate tungsten (W) coating performance, thin W film was deposited onto reduced activation ferritic/martensitic steel (F82H) by using rf magnetron sputtering device, then was irradiated at room temperature by 1.7 keV D⁺ ions. After the irradiation, the deuterium retention of the coated sample (W/F82H) was evaluated by thermal desorption spectroscopy (TDS) and compared with those of the F82H and pure W. During TDS measurements, D₂ was desorbed in all materials but HD was only found in significant amount in W/F82H and related to the increase of effective surface area due to the porous film structure. D retention increased with increasing fluence and was accompanied by growth of circular blisters. The size of blisters was limited by the F82H substrate structure.

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Keywords: deuterium retention, tungsten film, coating, magnetron sputtering, ferritic steel

DOI: 10.1585/pfr.8.2405103

1. Introduction

Tungsten is considered to be a good candidate as plasma-facing material of blanket in fusion reactor because of its good thermal properties and high atomic number (*Z*). Among many high-*Z* materials, tungsten has the most relatively short decay time [1]. It also has high melting temperature, high threshold energy of physical sputtering, and does not undergo chemical sputtering [2]. Despite those advantages, tungsten is a very heavy material. Reactor support structures are likely to receive considerable high load if bulk tungsten tiles are used [1]. In order to solve this problem, tungsten coating on plasma-facing components (PFCs) is being developed. However, tungsten film may suffer gas release and film exfoliation during plasma discharge [3].

Ferritic steel has better compatibility with both liquid metal or solid breeder, and all candidate coolants of fusion reactors, compared to austenitic steel [4]. It also has low thermal expansion coefficient and high thermal conductivity [5]. Moreover, the reduced activation ferritic-martensitic steel F82H (Fe-8Cr-2W) has been developed as candidate structure material for ITER blanket because of its advantages [5, 6].

Hydrogen isotope retention in PFCs affects surface properties, plasma fuelling, and wall conditioning of a fusion reactor. This phenomenon greatly depends on the material while there are still few studies on tungsten coating performance. Therefore there is a need to evaluate the deuterium retention/desorption behavior of tungsten coating, especially on F82H.

In the present study, tungsten was deposited onto the F82H as the substrate, then the deuterium retention and desorption properties of the material were investigated.

2. Experimental

2.1 Film deposition

Tungsten (W) was deposited onto reduced activation ferritic/martensitic steel (F82H) by using rf magnetron sputtering device. The F82H substrates, 30 mm × 5 mm × 0.2 mm in size, were manufactured by JAEA, and the tungsten sheet (99.95%) for the sputtering target was manufactured by The Nilaco Corporation. The sputtering gas was Argon with a flow rate of 0.02 m³/h. The power of sputtering was 400 W and the working pressure was 3 Pa.

The film thickness was 50±5 nm, estimated using Auger Electron Spectroscopy (AES). According to analysis of atomic composition, the produced film contained 80-81 at.% of W, 11.5-12.4 at.% of C, 3.2-3.5 at.% of N, and 4.3-4.7 at.% of O. This sample hereafter will be indicated as W/F82H.

2.2 Deuterium retention analysis

The W/F82H was irradiated at room temperature by 1.7 keV D⁺ ions with fluence of 0.5, 1, and 2 × 10¹⁸ D/cm² using ECR ion irradiation apparatus [7]. No mechanical polishing and heating treatment was done before the irradiation. After the irradiation, the deuterium retention was evaluated by thermal desorption spectroscopy (TDS) [8]. The sample was heated up to 1073 K with a heating rate of 0.5 K/s. During the heating, desorption of gases such as HD, D₂, HDO, and D₂O was quantitatively measured. For comparison, the deuterium retention of F82H and pure W

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^{*)} This article is based on the presentation at the 22nd International Toki Conference (ITC22).

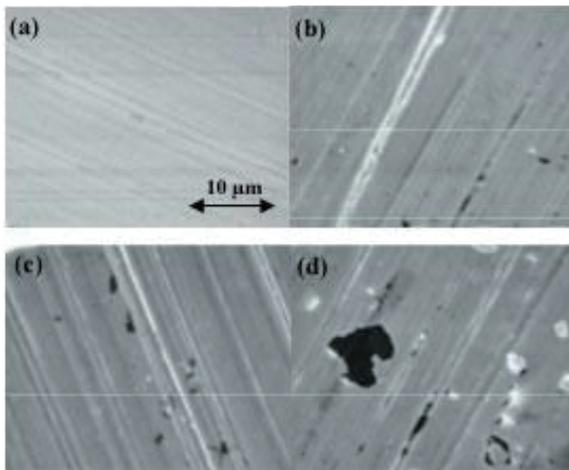


Fig. 1 Surface morphologies of (a) F82H, (b), W/F82H before irradiation, (c) W/F82H after irradiation at 1×10^{18} D/cm², and (d) W/F82H after irradiation at 2×10^{18} D/cm².

were also measured.

3. Results and Discussions

Figures 1 (a) and 1 (b) show the surface morphologies of the F82H and W/F82H. The linear pattern of W/F82H follows the original structure of the F82H substrate. Small cracks were observed on the surface of W/F82H (Fig. 1 (b)) which shows the porous structure of the film. According to Thornton's model, deposition of W at RT will produce porous film structure for Argon pressure ranging from 1-6 Pa [9]. Figures 1 (c) and 1 (d) show the W/F82H surface after irradiation. The existing cracks grew larger after irradiation due to the blisters. Blister formation will be discussed in another part of this section. Film crack size was up to 7 μm after irradiation at 2×10^{18} D/cm².

Retained deuterium was desorbed in the form of HD, D₂, HDO, and D₂O. Figure 2 shows the spectra of D₂ desorbed from F82H, pure W, and W/F82H at fluence of 0.5, 1, and 2×10^{18} D/cm². The peaks appeared around 500 K. Deuterium retention in tungsten is commonly known to exist at impurities, dislocations, vacancies, and voids. Modelling of deuterium release from tungsten by Poon *et al.* [10] revealed that deuterium release at around 900 K is subjected to deuterium trapping inside voids and deuterium release at 500-650 K is trapping at vacancies. Ogorodnikova *et al.* [11, 12] defined two types of deuterium trapping in tungsten, low-temperature (450 K) and the high-temperature traps (600 K). The low-temperature traps occur because of impurities and dislocations, and the other are related to vacancy clusters in the bulk. The 500-520 K peak of pure W in the results corresponds to vacancies.

The W/F82H spectra also showed the evidence of low-temperature traps most probably due to impurities of the film, but W/F82H had peaks at slightly lower temperature than pure W which was around 480 K. Porous thin film causes D₂ peak to shift to low-temperature region. Some

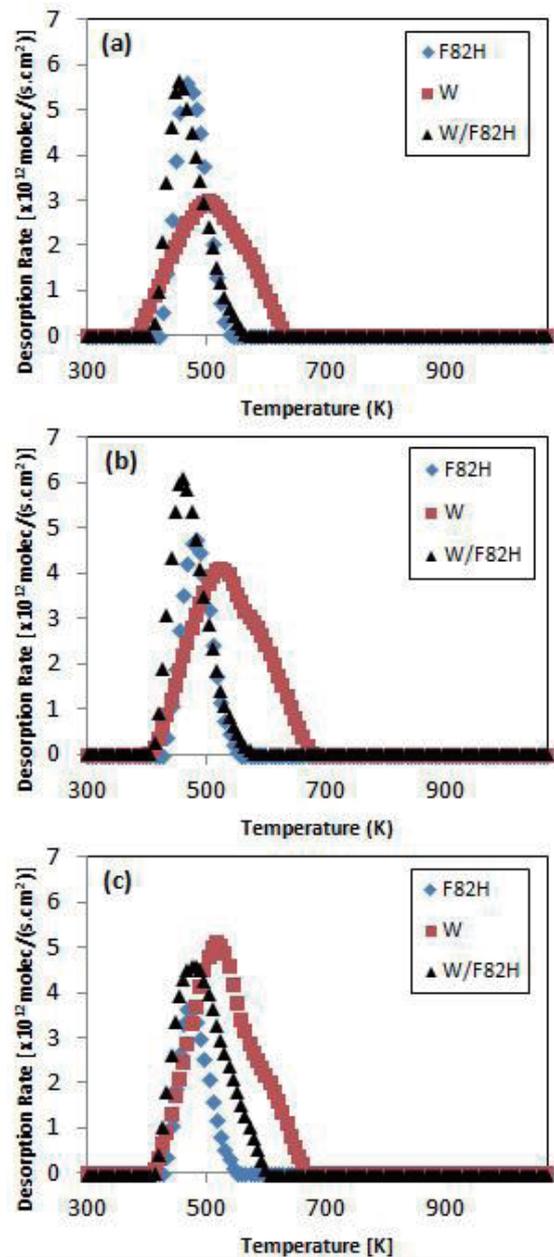


Fig. 2 D₂ TDS spectra of samples irradiated at fluence of (a) 0.5×10^{18} (b) 1×10^{18} (c) 2×10^{18} D/cm².

results of W coating on graphite also showed peaks around low-temperature region after being exposure to D plasma at RT [12, 13]. While W/F82H clearly showed different spectra from pure W, the spectra of W/F82H were similar with the spectra of F82H.

The total retention of D in all samples are shown in Fig. 3. The increase of total retention in all samples is not significantly different. However, there was a change in distributions of retained D and impurities inside different samples. The contribution of each gas form to deuterium retention is shown in Fig. 4.

The desorption amount of HD for pure W and F82H was quite small, compared to other gases, such as D₂ and

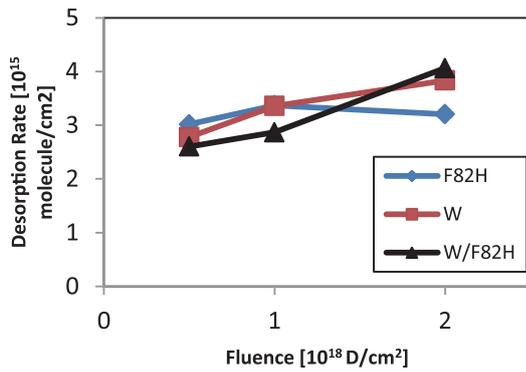


Fig. 3 Total D retention in F82H, pure W, and W/F82H.

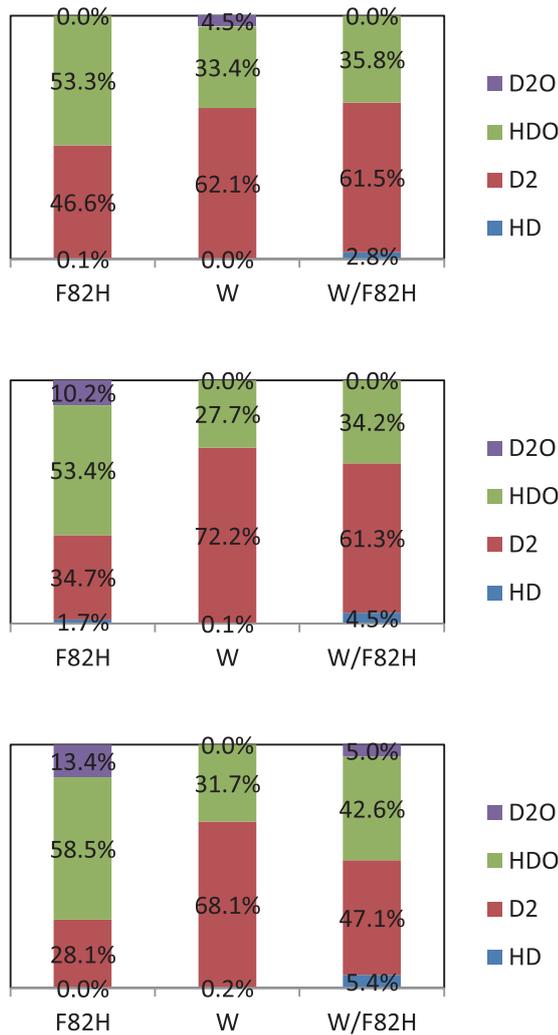


Fig. 4 Contribution of desorbed gases to total D retention at fluence of (a) 0.5×10^{18} (b) 1×10^{18} (c) 2×10^{18} D/cm 2 .

HDO. On the other hand, HD was only clearly released from W/F82H. In addition, the amount of desorbed HD increased proportional to the fluence. The desorption of HD characterizes a porous surface. This led to the increase of the effective surface area, which results into the increase of the amount of hydrogen adsorption at the surface.

F82H had the most variety of D-containing gas forms. HDO was the most dominant form. Increasing the fluence resulted into the decrease of D $_2$ percentage due to the interaction. Different from F82H, D $_2$ was more dominant than HDO in pure W. At lower fluences, the variety was quite similar to that of pure W except the appearance of small amount of HD. At high fluence, the percentage of HDO and D $_2$ O increased. Because W/F82H did not release HDO and D $_2$ O as much as F82H but quite the same as pure W, W film was able to modify the surface of F82H substrate.

The total D retention in W/F82H was lower than that in pure W at low fluence. Many deuterium paths exist within porous structure of W film [14]. The deuterium desorption during irradiation through these path might be responsible for the small total deuterium retention. Another possible reason for the small retention of W/F82H is C and O impurities inside the W film. Roszell *et al.* [15] reported that W foil contaminated by C and O on the surface showed deuterium retention less than pure W foil. Surface impurities made W surface become amorphous so that some of the bombarded deuterium may be easily re-emitted, thus lowering the retention amount inside material. Therefore impurities create a barrier that blocks inward diffusion of deuterium.

On the other hand, the total D retention at high fluence in W/F82H was a bit larger than that in pure W and F82H because of the significant increase of HDO and D $_2$ O desorption. Because the estimated eroded depth was less than the film thickness and the impurities tend to create barrier to block diffusion into the bulk, it is unlikely for deuterium to interact with oxygen content inside F82H substrate. The significant increase of HDO and D $_2$ O at high fluence might be caused by the oxygen impurity. High fluence irradiation may break the W-O bonds in the film so that D was able to interact with O during desorption. Further investigation on the deuterium depth distribution is needed.

Figure 5 shows surface morphologies of the W/F82H irradiated at different fluence. After irradiation at fluence of 0.5×10^{18} D/cm 2 , blister was hardly found as shown in Fig. 5 (a). Blisters were seen on W/F82H surface after irradiation at higher fluences. Figures 5 (b) and 5 (c) show large blisters which support the large amount of retention observed at higher fluences.

The shape of blisters found on W/F82H surface is small circular shape. Figure 5 (d) also shows that the blister size is limited by the linear pattern of the film. This limitation on blister size causes the lower D retention compared to pure W. Blisters on pure W foil were observed to be large low-dome-shaped and they have lamellar structures due to the gas accumulation along the lamellar grain boundary [14]. However, if the fluence is increased, the number of small circular blisters on W/F82H also increases, causing the D retention to be high. Further relation between D retention and growth mechanism of blisters in W film should be investigated.

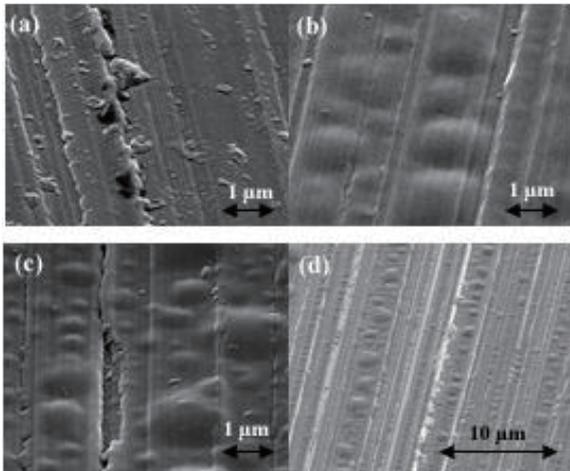


Fig. 5 Surface morphologies of W/F82H after irradiation at fluence of (a) 0.5×10^{18} (b) 1×10^{18} (c) 2×10^{18} , and (d) 1×10^{18} D/cm².

4. Conclusions

Deuterium retention of tungsten-coated low activation ferritic/martensitic steel (W/F82H) was investigated. Comparison with F82H and pure W showed that the contribution of D-containing gases inside three samples was different. D₂ was desorbed in all materials but HD was only found in significant amount in W/F82H and related to the increase of effective surface area due to the porous film structure.

D retention in W/F82H increased with increasing fluence. Impurities such as C and O inside the film enhanced the re-emitting ability, reducing the retention in WF82H compared to pure W. The increase of D retention was accompanied by growth of circular blisters. The size of

blisters is limited by the F82H substrate structure. Relation between D retention and growth mechanism of blisters in W film should be investigated.

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