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Measurement of tritium in tungsten deposition layer by imaging plate technique after exposure to gaseous tritium

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HIGHLIGHTS

- Tritium desorption behavior from W deposition was investigated by IP technique.
- Tritium activity sorbed in W deposition was much higher than that in W bulk.
- Tritium sorbed in W deposition was not released at room temperature in Ar.
- Tritium desorption behavior was different by tritium exposure temperature.

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ABSTRACT

It is important to understand tritium desorption behavior from plasma-facing materials of a fusion reactor in order to discuss effective tritium recovery method from in-vessel components. However, basic behavior of hydrogen isotopes in W deposition layer is not understood completely. In this study, characterized tungsten deposition layer formed by hydrogen plasma sputtering was exposed to gaseous tritium at 300 °C or 500 °C and tritium desorption behavior by vacuum heating was investigated by the imaging plate technique. For comparison, bare tungsten substrates were exposed to gaseous tritium in the same condition. Initial tritium activity in the deposition layer was much higher than that in the bare substrate. Tritium desorption behavior from tungsten deposition layer was different by the temperature of the layer during tritium exposure process. By heating at 500 °C for 1 h, 97.5% of tritium was desorbed from the layer exposed to tritium at 300 °C. On the other hand, by heating at 500 °C for 2 h, only 44.6% of tritium was desorbed from the layer exposed to tritium at 500 °C. To recover most tritium from W deposition layer and W substrate, heating at above 700 °C is required.

1. Introduction

Tungsten (W) is a candidate material for plasma-facing components because of low sputtering yield, high melting point and low solubility for hydrogen isotope. During fusion plasma discharges, the W surface will be modified due to sputtering and re-deposition. The deposited W layer has a structure different from the original W material. Therefore, the investigation of tritium behavior not only in original materials but also in deposited layers is necessary. It has been observed that after sputtering-deposition processes, W deposition layer formed by hydrogen plasma sputtering retains a large amount of hydrogen [1–6]. This means that W deposition layer has a large tritium sorption capacity and its influence on tritium behavior in the vessel may not be negligible.

From a viewpoint of tritium management during maintenance of in-vessel components such as blanket and divertor, tritium desorption rate and tritium recovery ratio at the in-vessel baking temperature are key parameters. When the baking temperature for tritium recovery is higher, tritium recovery ratio becomes higher generally. However, the in-vessel baking temperature is limited, for instance, wall baking temperatures are 95 °C in LHD [7], 118 °C in KSTAR [8], 300 °C in JT-60U [9], 240 °C in ITER first wall and 350 °C in ITER divertor [10]. Therefore the understanding of tritium desorption behavior at these moderate temperatures is important. On the other hand, from a viewpoint of recovery of fuel tritium retained in the in-vessel components, the components are heated as high as

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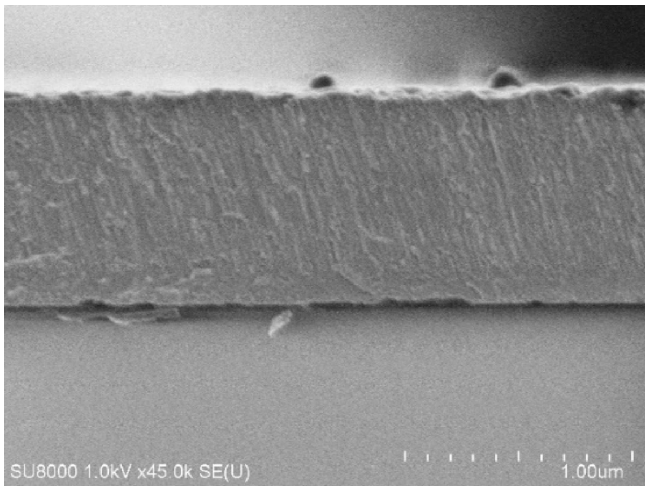

Fig. 1. SEM image of the cross-section of W deposition layer.

possible to release almost all tritium. To determine an appropriate heating temperature for each component is important. For instance, thermal treatment at 800–1000 °C are suggested for detritiation from plasma facing components of ITER [11].

Although experimental data on tritium desorption behavior from bulk W were obtained by several researchers, that from W deposition layer is insufficient. In this study, characterized W deposition layer formed by hydrogen plasma sputtering was exposed to the tritium-deuterium (T-D) gaseous mixture together with W bulk material and tritium desorption behavior by heating in vacuum was investigated by the imaging plate (IP) technique. From the obtained results, tritium behavior in W deposition layer and optimal detritiation condition were discussed.

2. Experimental

2.1. Preparation of W deposition layer

Samples of W deposition layer were formed on W substrates and quartz substrates by hydrogen RF plasma sputtering. The W substrates were fully recrystallized after cutting and polishing in advance. A W plate, 50 mm × 50 mm in size and 1 mm thickness, 99.95% in purity (Nilaco Co.), was mounted on an RF electrode. The W substrates, 10 mm × 10 mm in size and 1 mm in thickness (A.L.M.T. Co.), and quartz substrates, 5 mm × 10 mm in size and 1 mm in thickness, were mounted on the ground electrode. A plasma chamber was evacuated to about 10^{-3} Pa by a vacuum pump and pure hydrogen gas was introduced via a mass flow controller. The hydrogen gas pressure was controlled at 10 Pa. The RF power of 250 W was supplied to the RF electrode with 13.56 MHz to ignite hydrogen plasma. The sputtering-deposition process was continued for 140 h.

The structure of W deposition layer formed in this condition was analyzed by TEM and XPS. The deposition layer consists of fine grains [1] and relatively large porosity [12]. This means that the deposition layer has high grain boundary density. Oxide W exists only at the top surface and W exists as metal W in the deeper region. A certain amount of oxygen was detected but carbon was not detected [13]. To evaluate the thickness of the deposition layer, the quartz substrate was broken in half and its cross section was observed by SEM as shown in Fig. 1. The thickness was evaluated to be 913 nm.

Table 1
Heating condition in tritium gas exposure process and tritium desorption process.

Experiment 1		Experiment 2	
Temp [°C]	Time [h]	Temp [°C]	Time [h]
Tritium gas exposure process			
300	3	500	3
Tritium desorption process			
30	189	30	528
95	8	95	2
150	7	150	2
300	2	300	2
500	1	500	2
700	0.5	700	1

2.2. Tritium gas exposure

The T exposure apparatus is shown in Fig. 2(a). A set of the W deposition layer formed on W substrate and a bare W substrate was put in a quartz tube and heated by an electric furnace in vacuum. The temperature for degassing was kept for 3 h at 300 °C in experiment 1 and at 500 °C in experiment 2. Tritium gas diluted with deuterium gas was introduced in the quartz tube from a ZrNi bed heated. The samples were exposed to the T-D gaseous mixture (7.2% T/D) at total pressure of 1.2 kPa for 3 h, at 300 °C in experiment 1 and at 500 °C in experiment 2. After tritium exposure was completed, the electric furnace was quickly removed and the quartz tube was cooled with water and then with liquid nitrogen to inhibit the migration of hydrogen isotopes. The T-D gas mixture was reabsorbed into the ZrNi bed. During cooling by liquid nitrogen, the quartz tube was evacuated by a vacuum pump.

2.3. Tritium measurement by IP technique

Tritium activity in the samples were evaluated by IP technique. This technique was widely applied for understanding tritium behavior in plasma facing materials [14,15]. IP can detect β electrons emitted from tritium existing within the depth of 1 μm in metals [16]. IP and IP reader used in this work were BAS-TR2025 and FUJIFILM BAS2500 (Fuji Photo Film Co. Lit.), respectively. Tritium activity is expressed as the photo-stimulated luminescence (PSL) intensity. IP measurement was performed in a glove box filled with argon to avoid contacting samples with air as shown in Fig. 2(b). After tritium gas exposure, the closed quartz tube containing samples was transported to the glove box. Samples were taken out in argon atmosphere and IP was put on samples for 1 h at room temperature.

2.4. Thermal treatment

After the first IP measurement for evaluation of initial tritium retention, samples were stored in the glove box and sometimes IP measurement was carried out to investigate tritium desorption at room temperature. Then, samples were put into a quartz tube different from the tube used in tritium gas exposure. The quartz tube was connected to the vacuum heating apparatus and heated in vacuum at the preset temperature as shown in Fig. 2(c). Heating and following IP measurement were repeatedly performed. Heating conditions are summarized in Table 1.

3. Results and discussion

IP image for W deposition layer (left) and W substrate (right) exposed to gaseous tritium at 300 °C is shown in Fig. 3. Tritium activity in W deposition layer was much higher than that in the bare W substrate. In spite of heating in vacuum, a remarkable reduction of tritium activity in W deposition layer was not observed

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