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Hydrogen incorporation into tungsten deposits growing by hydrogen plasma sputtering

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HIGHLIGHTS

- Tungsten deposits were formed by hydrogen plasma sputtering.
- The hydrogen reflected from the sputtering target was mainly contributed to hydrogen incorporation.
- A small amount of W deposit was formed even in the shadow region from the sputtering target.

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ABSTRACT

Understanding of hydrogen accumulation behavior in tungsten deposits is important from viewpoints of tritium economy and tritium safety in fusion reactors. Some reports indicate that a large amount of hydrogen isotope is incorporated into tungsten deposits growing by hydrogen plasma sputtering. However, the mechanism of hydrogen incorporation is not clarified yet. In this work, tungsten deposits were formed at different circumstances in the sputtering device and the amount of incorporated hydrogen was measured. The implantation of hydrogen reflected from the sputtering target was mainly contributed to the hydrogen incorporation. The contribution of the implantation of reactive hydrogen from plasma was smaller than that of the reflected hydrogen in the experimental condition. A detectable amount of tungsten deposits was formed even in the shadow region from the sputtering target. This suggests that a certain amount of tungsten atoms, which lost its initial energy by collision with molecular hydrogen, diffuses in the gas phase and adheres in the shadow region.

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1. Introduction

Plasma facing wall in fusion reactors is modified by sputtering and re-deposition during plasma operation. It is commonly known that tritium inventory is greatly increased when graphite materials are used as the plasma-facing wall because carbon deposit, which is widely formed on surface and gap of graphite tiles, can retain a large amount of hydrogen isotope. Tungsten (W) is a candidate material for the plasma facing wall because of low solubility and low sputtering yield for hydrogen isotope. Although the sputtering rate of W wall is slower than that of graphite wall, hydrogen retention in W deposit should be evaluated because fusion DEMO and commercial reactors are continually operated for a long time. Some reports indicated that a large amount of hydrogen isotope is incorporated into the W deposition layer growing by hydrogen plasma sputtering [1–3]. However, the mechanism of hydrogen incorporation is not understood completely.

During the sputtering-deposition process by hydrogen plasma, three kinds of hydrogen imping on the growing surface of W deposits. First one is molecular hydrogen (H_2), second one is reactive hydrogen such as ionized hydrogen (H_2^+) and atomic hydrogen (H) in plasma, and third one is reflected hydrogen from the sputtering target (H). It was observed in our previous work that deuterium retention in the W deposits by gaseous deuterium exposure is much smaller than that during the sputtering-deposition process [4]. This means that the contribution of gaseous hydrogen to hydrogen retention in the sputtering-deposition process is small. The reactive hydrogen such as ionized hydrogen impinging on the W deposit with plasma space potential is considered to contribute hydrogen retention because relatively high deuterium retention in the W deposit was observed by deuterium plasma exposure [4]. The reflected hydrogen hits on the growing surface of W deposits with highest energy among three kinds of hydrogen. The reflected hydrogen probably contributes hydrogen retention.

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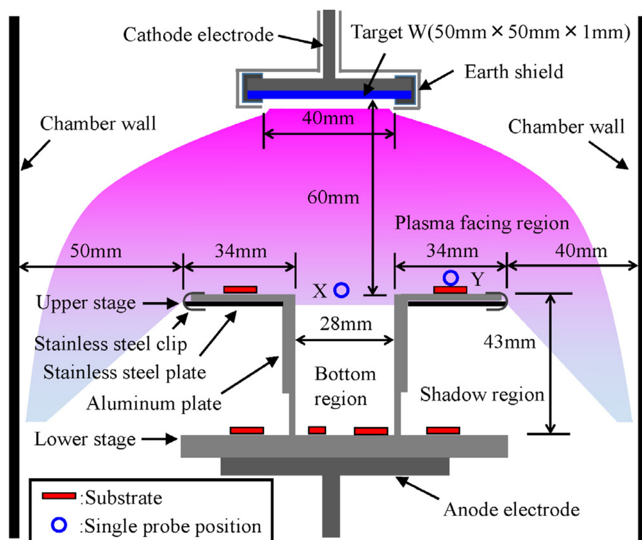

Fig. 1. Positional relation of a sputtering target, sample stage and substrates.

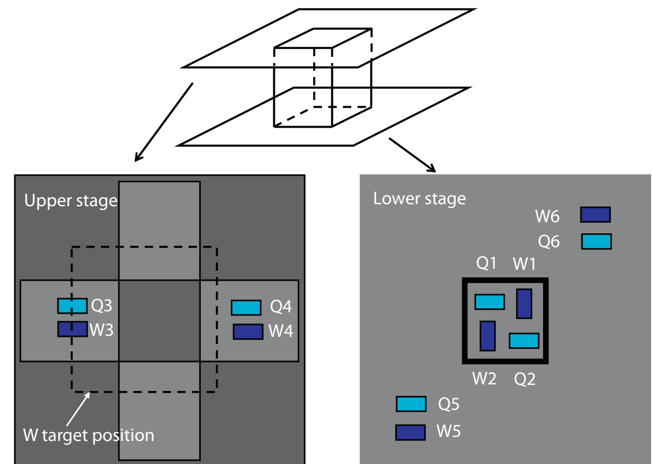

Fig. 2. Position of each substrate on upper stage and lower stage. "W" means tungsten substrates and "Q" means quartz substrates.

The incorporation mechanism of working gas in the sputtering-deposition process by argon, helium and nitrogen plasma sputtering has been discussed in the field of development of metal thin films [5–7]. These studies indicate the concentration of working gas in metal thin films increases with increasing the incident energy of the ionized working gas, where the incident energy was controlled by the substrate bias voltage, and decreases with increasing the gas pressure. In the literatures, it was described that the working gas incorporation in zero-bias voltage is the results of ions which hit the target and are then reflected as energetic neutrals. Winters and Kay suggested that surface adsorption characteristics greatly influences gas incorporation [6]. The authors speculate from these reports that the high hydrogen incorporation in the W deposit growing by hydrogen plasma sputtering is caused by the implantation of hydrogen reflected from the sputtering target and sorption of implanted hydrogen on W grains. In this work, W deposits were formed at different circumstances in the sputtering device and the amount of incorporated hydrogen was measured to discuss the mechanism of hydrogen incorporation.

2. Experimental

2.1. Production of W deposits

W deposits were formed on W substrates and quartz (Q) substrates by hydrogen plasma sputtering using the capacitively coupled RF plasma device. Parallel plates of anode and cathode were installed in the vacuum chamber. A W plate, 50 mm × 50 mm in size and 1 mm in thickness, 99.95% in purity, were mounted on the cathode. The cathode was surrounded by the earth shield of a stainless steel mesh except the sputtering area of the W plate to avoid erosion of the electrode structure by sputtering. The especial sample stage was fabricated from aluminum plates and stainless steel plates to produce W deposit at different circumstances. Positional relation of the sputtering target, the sample stage and the substrates are schematically shown in Fig. 1. The set of W substrate, about 5 mm × 10 mm in size and 0.05 mm in thickness, and Q substrate, about 5 mm × 10 mm in size and 1 mm in thickness, were mounted on Bottom region (B), Plasma facing region (P) and Shadow region (S). Position of each substrate and its name is shown in Fig. 2. The center of the sample stage deviates from the target center to the 5 mm right direction in this figure. Here, hydrogen existing in the vacuum chamber is categorized into the molecular hydrogen (H_M), the reactive hydrogen such as atomic hydrogen and ionized hydrogen (H_I), and the reflected hydrogen (H_R). The W deposit grows under H_M and H_R incidents in B region, under H_M, H_I and H_R incidents in P region, and under H_M incident in S region.

After setting substrates, the vacuum chamber was evacuated 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 100 W was supplied to the cathode electrode with 13.56 MHz to ignite hydrogen plasma. The sputtering-deposition process was continued for 310 h. The temperature of each region was measured by thermocouples. The surface temperatures of the sample stage in B region and P region rose to 90 °C and that in S region rose to 65 °C. Ion fluxes at position *X* and *Y* shown in Fig. 1 were separately measured by the Langmuir probe method after the experiment. At position *X*, the ion flux was 1.0×10^{19} m^{-2} s^{-1} and the plasma space potential was 15 V. At position *Y*, the ion flux was 1.0×10^{18} m^{-2} s^{-1} and the plasma space potential was 13 V. The DC self-bias generated at the sputtering target was measured to be 1670 V by the high voltage probe.

The weight of the W deposit formed on the substrate was obtained from weight change of the substrate before and after the experiment by using an electric balance with a sensitivity of 0.01 mg. Atomic ratio (at%) in the W deposit was analyzed by an energy dispersive X-ray equipment (EDX:Genesis2000, EDAX Inc.). The surface observation was performed by the scanning electron microscopy (SEM:SU8000, Hitachi High-Tech. Co.). EDX analysis and SEM observation were carried out for W deposits formed on quartz substrates. The quartz substrates were broken in half to observe the cross section of the W deposits and its thickness was evaluated. EDX and SEM used in this work were installed at the Center of Advanced Instrumental Analysis, Kyushu University.

2.2. Measurement of incorporated hydrogen

Each W deposit formed on the W substrate was placed into a quartz tube connected to a gas chromatograph (GC: GC8A, SHIMAZU Co.). The quartz tube was filled with argon gas and heated to 800 °C in 100 °C step by an electric furnace. Argon gas was introduced into the quartz tube every 30 min in order to transport the released hydrogen into GC. This operation was repeated in some times at each temperature until the amount of released hydrogen became significantly small.

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