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Comparison of boronized wall in LHD and JT-60U

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Abstract

Boronization has been performed in many fusion plasma devices as one of several effective wall conditioning techniques. In LHD and JT-60U, boronization is operated as a typical wall conditioning method. Using material probes stayed in LHD during an experimental campaign, characteristics of a boronized wall are discussed in each device. Due to the complex three dimensional structure of the vacuum chamber, the thickness of boron film on the material probes depends on the distance from the anodes for glow discharge in LHD. On the other hand, such dependence is not clear in JT-60U due to thick carbon layers deposited on the material probes. To consider long-term operations, a deposited thick carbon layer with high boron-oxide concentration cannot be eroded completely by glow discharges, this layer is expected to be a saturated capacity of oxygen gettering. To keep the capacity of oxygen gettering, an additional boron layer is needed on the thick deposited layer. Since thin deposited layer on boron-coated area of LHD is eroded by glow discharge and flesh boron layer appears, a capacity of oxygen gettering is kept during long-term experiments. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

Boronization has been performed in many fusion plasma devices as one of the most effective wall conditioning techniques [1-7]. It is well known that the major advantages of boronization are (1) covering high Z first wall with low Z materials, (2) oxygen gettering, and (3) quick starting up of wall conditioning after opening the devices to air. The behavior of boronized walls is fairly complicated under physical and chemical processes such as mass transfer and composition changes due to sputtering and deposition of other materials. Therefore, an operation scenario of boronization for next generation devices such as ITER is not optimized. For example, in test chambers, Yamage et al. [8] and Noda

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et al. [9] measured a capacity of oxygen gettering of B and B/C films exposed to He/O_2 glow discharges. But these exposing times are too short to consider actual fusion devices such as the large helical device (LHD) and JT-60U, and an eroded thickness of boron film is quite different between test chambers and actual fusion devices.

In this paper, we discuss an optimized method of coated film uniformity in a wide area and a capacity of boron film as an oxygen getter using exposed material probes during long-term experimental campaigns in LHD and JT-60U.

2. Experimental setup

The large helical device (LHD) is a large-scale superconducting heliotron system with a set of l/m = 2/10 helical coils [10]. Fig. 1 shows a midplane cross-section of LHD. Wall conditioning operations in LHD are a glow discharge cleaning (GDC) with two anodes, a titanium coating and boronization with three nozzles [11].

The first wall is made of stainless steel-316L (SS 316L) and divertor plates are made of graphite (IG430U). Usually wall temperature during plasma operation, GDC, and boronization is room temperature and it rises up to 368 K when wall baking by hot water is conducted [11].

In LHD, the first boronization was conducted in the 5th experimental campaign (2001–2002). In this

campaign, diborane (B_2H_6) diluted by helium (He) was injected from only one nozzle. In the 6th campaign (2002–2003), two injection nozzles were added and the boronization was carried out three times using these nozzles with a typical operation time of 7 h. As a result, oxygen in plasma was reduced and plasma stored energy was increased for the extension of the operational density limit in LHD [12]. SS 316L samples were installed as material probes on the first wall inside the vacuum vessel and they were exposed to only glow discharge. After about 8 month's exposure, one experimental campaign, these samples were taken out after a vacuum vent.

Fig. 2 shows a schematic midplane cross-section of JT-60U. Two anodes are installed for a GDC on the first wall and 12 gas injection nozzles for the boronization are installed at the outer side of poloidal cross-section in JT-60U [6]. A current operation of boronization using deuterated-decaborane $(B_{10}H_{14})$ with He gas started since 2000. A container for polished decaborane is heated to ~380 K during operation of boronization. A diluted decaborane gas is made from blending He and D₂ [5].

Divertor targets are made of carbon fiber composite (CFC) and other plasma facing components are made by graphite tiles. Typical wall temperature during plasma operation is from 523 to 573 K.

As material probes, SS 316L and silicon (Si) samples were installed on the outer port inside the vacuum vessel of JT-60U exposed to only glow



Fig. 1. A schematic drawing of the LHD midplane with boronization systems of three diborane supply nozzles and two GDC anodes.



Fig. 2. A schematic diagram with boronization systems of 20 decaborane supply holes and two GDC anodes in JT-60U.

discharge for about 17 months, two experimental campaigns, and then taken out after a vacuum vent. These samples were then taken out.

The elemental depth profiles were measured with two methods. One is the X-ray photoelectron spectroscopy (XPS) with sputtering by argon (Ar) ions and the other is the auger electron spectroscopy (AES) [13]. For hydrogen and deuterium, a qualitative analysis was performed using secondary ion mass spectroscopy (SIMS) [14].

3. Results

3.1. Comparison of boron film LHD and JT-60U

In LHD, an SS 316L sample was installed on the movable sample stage at 4.5L port to the second boronization in the 6th experimental campaign (2002). This stage was set to level with the first wall.

In JT-60U, an SS 316L sample was installed and exposed during the 13th operation of boronization (as a serial number of operation in JT-60U) in 2000 using the inserting holder on the vacuum flange at P-4 section. This stage was set roughly at the level of the first wall.

The atomic composition ratios of boron film on the material probes measured with the XPS are shown in Fig. 3. In LHD, a boron concentration of 75% is observed in a boron film at the depth of 55 nm, and oxygen of 20% and carbon of 5% at the depth of 25 nm. A depth profile of oxygen shows two peaks of concentration. The first peak is located near the base material, and it indicates a trapped oxide impurity at the initial phase of boronization. The second peak is located at the top surface which indicates trapped oxygen at an additional glow discharge of 3 h, just after boronization. Each gettering process has been studied using the test chambers [8,9,15].

In JT-60U, a boron concentration of 90% is observed in a boron film at 145 nm, and 2% oxygen and 5% carbon are also observed at 50 nm. In JT-60U, the boron concentration just after boronization is higher than that in LHD and the oxygen concentration is lower than that in LHD.

3.2. Toroidal distribution of coated boron thickness in LHD

Toroidal distributions of boron film by material probes in the 7th (2003) and the 8th (2004) cam-



Fig. 3. Depth profiles using XPS with Ar^+ sputtering for O (\bigcirc), B (\square), C (\diamondsuit) and Fe (\times) of material probes (a) in LHD with a boron thickness of 55 nm at 4.5L port and (b) in JT-60U with a boron thickness of 145 nm at P-4 section per one boronization.

paigns are shown in Fig. 4. Since low conductance of gas nozzle was not adjusted in the 7th campaign, only the areas of sections 1 and 10 were coated thickly with boron film. To improve uniformity of the boron film, the gas flow rates of these nozzles were adjusted before the 8th campaign (2004). After the 8th campaign, an improvement of uniformities were observed at areas of sections 4, 6, and 9. However, thick boron films were not observed near the nozzle at 7.5L port. During the 8th campaign, an erosion rate due to glow discharge was measured using a material probe which pre-coated with a certain thickness of boron. Two hundred fifty nanometers of boron at section 5 was eroded by exposing to glow discharge for 814 h. The erosion rates are different at each toroidal section due to non-uniform current density of glow discharge. Boron thickness before erosion is analyzed using



Fig. 4. Toroidal distribution of boron thickness on material probes using AES analysis in LHD.

these erosion rates and shown in Fig. 4 as estimated thickness. This estimated thickness shows two peaks of boron film. One is around the area of sections 10, 1, and another is around sections 4, 5 and 6. This result indicates a boron film that coated thickly near the GDC anodes. In the test chamber [15], the boron thickness shows a dependence on distance from gas injection nozzle. However, a clear dependence on distance was not observed in LHD. This reason is considered the non-uniform current density of glow discharge and the non-uniform diborane density by the complicated vacuum chamber structure.

3.3. Coated boron and deposited carbon layer in JT-60U

Toroidal distributions of coated boron thickness and deposited carbon thickness during 2003/09 to 2005/01 are shown in Fig. 5. A thickness of boron film was analyzed using XPS and SIMS. Thick boron coated areas were P-5 and P-8 sections and thick carbon deposited areas were P-15 and P-17 sections. A clear dependence between the GDC anode position (P-3 and P-15) and toroidal dependence of boron thickness is not observed due to the influence of localized thick carbon layer.

4. Discussion

From a ratio of signal intensity of D/B by SIMS, this deposited carbon layer including deuterium is shown in Fig. 6(a). In this co-deposited layer, chem-



Fig. 5. Toroidal distribution of boron thickness on material probes using XPS and SIMS, carbon thickness using XPS in JT-60U.

ical bonding of B for four depths is shown in Fig. 6(b). A B_2O_3 bond locates at 193 eV, B–C bond as boron carbide at 189 eV and B–B bond as fresh boron at 188 eV [16,17]. Just after boronization in JT-60U, fresh boron was kept and boron-oxide concentration is only 2% [18]. But on this exposed sample, from the top surface to 20 nm, the boron-oxide bond is a dominant peak and at a depth of 37.4 nm the peak of boron carbide was increasing.

At this depth of 37.4 nm, a major bond is boron carbide at 189 eV, and the intensity of B–B bond is less. For the actual divertor target tile in JT-60U, it was reported that fresh boron did not remain and had shifted to boron carbide [14].

In LHD, a major boron-oxide peak of chemical bond is observed at the top surface of boron film as same as JT-60U. In deeper region (>3 nm), a fresh boron peak at 188 eV can be observed as shown in Fig. 6(c). Yamage et al. reported the difference between pure B and B/C of oxygen gettering in the test chamber. This result shows that gettering effect of pure B is two times higher than that of B/ C [8]. A potential of oxygen gettering in LHD is higher than that in JT-60U. But in the actual experimental devices, an unestimated effect such as a thick carbon layer influences the capacity of oxygen gettering. A difference in boron film between LHD and JT-60U is the thickness of boron-oxide with high concentration. This boron-oxide layer in JT-60U cannot be eroded by glow discharge cleaning during night. On the other hand, thin boron-oxide layer in LHD can be eroded easily by GDC. Due



Fig. 6. (a) Depth profiles of carbon, boron, oxygen and iron using XPS on the left hand and depth profile of signal intensity ratio of D/B using SIMS on the right hand of material probe at P-15 section in JT-60U. (b) Binding energy of boron film on material probe for depths of 3.74, 11.22, 20.57, and 37.4 nm at P-15 section in JT-60U. (c) Binding energy of boron film on material probes for depths of 1, 3, 5, 7, and 9 nm in LHD. A B₂O₃ bond is indicated at 193 eV, B–C bond as boron carbide is indicated at 189 eV and B–B bond as fresh boron is indicated at 188 eV.

to this reason, the lifetime of oxygen gettering effect is long in LHD.

The lifetime of oxygen gettering effect in LHD was roughly estimated about one experimental campaign using the test chamber [9]. In this reference, two kinds of factors, diffusion and deposition were

pointed out, but erosion rate by glow discharge was not included. In this paper, the importance of erosion was discussed. This balance between erosion and deposition rate matches a coated thickness of boron film in LHD. An experimental result of capacity is longer than the estimated value in the test chamber.

In JT-60U, P-15 and P-17 sections have a thick carbon layer, and these layers have a high concentration of boron-oxide, about 90%, and at these areas the capacities of oxygen gettering are expected to be very low. From plasma performances, a saturation of oxygen gettering was observed at every 200 shots by a spectrometer in JT-60U [1].

5. Conclusions

Using the analyzed data of long-term material probes in LHD and JT-60U, the characteristics of boronized wall were discussed and significant results are as following:

- (1) The thickness of boron film in toroidal direction depends on the position of GDC anode in LHD. In JT-60U, such dependence is not observed clearly due to deposited thick carbon layers. Just after boronization, more fresh boron film is produced in JT-60U, but the concentration of boron-oxide increased during the experimental campaign, and these thicknesses are motr than these in LHD.
- (2) For long-term operations in JT-60U, high B-O concentration of 90% was observed at P-15 section and it is expected to be the saturation capacity of oxygen gettering.
- (3) For thick B–O layer with a high B–O concentration on boron film, an additional boronization is needed such area as P-15 in JT-60U. In LHD, the deposited layer on boron-coated area on the first wall is thin, and the next fresh boron can be exposed by erosion of glow discharge. Then the lifetime of oxygen gettering effect is retained during long-term experiment, and at present a limitation of this lifetime is not observed in LHD.

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