First boronization in KSTAR


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Abstract

First boronization in KSTAR is reported. KSTAR boronization system is based on a carborane (C2B10H12) injection system. The design, construction, and test of the system are accomplished and it is tested by using a small vacuum vessel before it is mounted to a KSTAR port. After the boronization in KSTAR, impurity levels are significantly reduced by factor of 3 (oxygen) and by 10 (carbon). Characteristics of a-C/B:H thin films deposited by carborane vapor are investigated. Re-condensation of carborane vapor during the test phase has been reported.

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1. Introduction

Wall conditioning is an important procedure for better plasma performance [1]. It reduces impurities released from walls to plasmas and control hydrogen recycling. The main ideas of wall conditioning is to treat walls by DC glow discharge (GD) with H2/D2 or He for impurity removal and to coat wall surfaces of plasma facing components (PFCs) for the particle flux control from the wall. Various wall coatings such as boronization (Bz), carbonization, and siliconization are used to getter oxygen impurity effectively [1].

Boronization in GD is a well-established technique and widely used in many tokamaks: B2H6 (B2D6) gas is used which is easy to control the flow rate, but it is toxic and explosive [1]. Therefore, a boronization system with an expensive safety control system should be equipped. On the other hand, decarborane (B10H14/B10D14) [2] or carborane (C2B10H12) [3] solid powders have been used for boronization that are not toxic, and easy to handle.

KSTAR has chosen carborane as boronization source material. First boronization in KSTAR has been performed successfully in its second campaign. In this paper, we describe the KSTAR boronization system and the improvement of wall conditions after the boronization of KSTAR wall.

This paper is organized as follows. In Section 2, we will describe the KSTAR boronization system in detail. In Section 3, we will describe the KSTAR boronization and discuss the improvement of wall condition after boronization. Characteristics of a-C/B:H thin films deposited by carborane and that exposed to plasmas will be shown in Section 4. We would like to discuss re-condensation problems that we are dealing with during the boronization in Section 5. Finally, a conclusion will be given.

2. KSTAR boronization system

KSTAR boronization system is based on a carborane injection system. Carborane vapor is produced by heating carborane solid powders up to 120 °C by using a dedicated evaporator oven. The oven is a cylindrical-shaped metal box with a container equipped with a heater and a temperature sensor (see Fig. 1). Because KSTAR is a superconducting machine, access to the machine area is strictly prohibited once superconducting magnet is cooled down. Furthermore, maintenance of a system installed inside the machine during a plasma operation campaign is almost impossible: KSTAR has a cryostat structure outside the vacuum vessel. Thus, the boronization system is installed outside the cryostat of the KSTAR vessel for easier access to the boronization system during a plasma campaign. Carborane powders are loaded into the container which can be pulled out from the oven (upper part of Fig. 1a). Then the oven is heated up. Carborane vapor is introduced into the vacuum vessel through a guiding pipe system which is also heated up to 120 °C to avoid the re-condensation of the carborane vapor in the pipe system before it reaches the vacuum vessel. The flow rate of carborane vapor is controlled by oven temperature and a gate valve.

Before we inject the carborane vapor into the KSTAR vacuum vessel, we have measured the injection rate (vapor pressure)

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depending on the oven temperature for a calibration procedure by using a small vacuum chamber. As the oven temperature increases, the pressure increases exponentially first, then it increases with a constant $dP/dt$ (pressure change rate) of about 0.001 mbar/s (not shown here). Carborane injection rates using 1 g and 10 g carborane as a function of oven temperature, together with reference values measured by Buzhinskij et al. [4] are shown in Fig. 2. As the temperature increases, carborane evaporates and the injection rate increases as the pressure in the carborane oven increases. The injection rate in KSTAR boronization system is an order of magnitude smaller than that in ref. [4], since the injection rate depends not only on the temperature of the oven, but also on both the volume of the oven and amount of carborane loaded in the container. Nevertheless, similar gradient of the injection rate in ref. [4] and this work at low temperature (35–50 ºC) indicates that the carborane evaporation rate as a function of oven temperature in both cases is almost similar. However, in the case of the KSTAR oven, a large volume inside the oven has to be filled by the carborane vapor that builds up relatively small partial pressure. This is the reason for the small injection rate compared to that in ref. [4].

On the other hand, injection rates in KSTAR boronization system show two different evaporation phases. Using 1 g of carborane, the injection rate decreases at first, then it increases further. Using 10 g, it increases fast, and then it increases slowly. This is due to the re-condensation of evaporated carborane at colder parts of walls of carborane box (see Fig. 1a). As the entire carborane box is heated up, re-evaporation of re-condensed carborane at the carborane box walls begins. At a temperature of 150 ºC using 10 g, the maximum injection rate is about $10^{-3}$ Torr l/s. Thus, during the KSTAR boronization, we have heated the heating jacket which is rolled outside the carborane box up to 50 ºC, and then the main oven heater was turned on.

3. KSTAR boronization and effect of boronization

For the first boronization of KSTAR, 40 g of carborane powder is loaded. Carborane vapor is injected into pulsed DC He glow discharge to produce a-C/B:H thin films on the walls. He flow rate is fixed at 600 sccm with a pumping speed of 2800 l/s. Duty cycle is set to 0.25 (pulse duration 5 s on/15 s off). 2 h of boronization and 1 h He GDC are performed sequentially. After the injection of carborane, increase of masses around 144 (carborane, C_2B_10H_{12}) and 70 (compounds from carborane) are clearly observed (not shown here). RGA spectra show significant reduction of H_2O and O_2 level in the vacuum vessel after the boronization by a factor of 10. However, H_2 level is increased by a factor of 10 which is originated from carborane molecules in the vacuum vessel. Thus, it is important to control the amount of H_2 in the vacuum vessel after the boronization. After the 1st boronization, about 8 g of re-condensed carborane is recovered from the oven wall.

The effect of boronization in KSTAR is evaluated by optical emission spectroscopy (impurity line emission) and filterscope. Emission spectroscopy indicates also a sudden increase of BII (345 nm, 447 nm) peak just after the boronization, then it is decreasing as a function of time, while oxygen line intensity shows abrupt drop, then increase as a function of time (not shown here). Fig. 3 shows the normalized impurity level (divided by $n_e$) at outer wall before (shot number 2153, 2154) and after the boronization (shot number 2164, 2165). The normalized impurity level were decreased by factors of 3 (OII), and by 10 (CIII) after the boronization. The impurity density in the vacuum vessel was decreased and maintained low. The lifetime of a-C/B:H layers about 100 shots, deduced by the increase of H_2O level in the vacuum vessel (by RGA, not shown here).

4. Characterization of a-C/B:H thin films for KSTAR boronization

Characteristics of a-C/B:H thin films are investigated. Thin films are deposited on silicon wafers in a He DC glow discharge by using
carborane vapor in a small vacuum chamber which is equipped with a copy of KSTAR boronization system. Small amount of carborane (less than 1 g) is loaded. Thin films are characterized by spectroscopic ellipsometry, XPS, and AES will be studied by RBS, NRA.

Optical properties of a-C/B:H thin films are shown in Table 1. Thickness of thin film depends on the amount of source molecules, deposition time, and ion energy impinging on the surface, etc. Samples deposited on silicon wafer (HYU1, HYU2) have low refractive indices: \( n = 1.45 \) and \( n = 1.7 \), while KSTAR sample – deposited on stainless steel coupon installed at M port just below the carborane injection pipe – has much higher refractive index \( n = 2.34, k = 0.22 \). Two interpretations can be given: first, it might be due to the different ion energy impinging on the surface, which makes thin film soft (HYU1, HYU2) or hard (KSTAR). Second, KSTAR sample exposed to plasmas and GDC during the second campaign, thus the characteristics of film would be changed, while HYU1 and HYU2 samples were investigated as they are deposited. Further investigation is needed to identify the differences.

**Table 1**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (nm)</th>
<th>( n )</th>
<th>( k )</th>
</tr>
</thead>
<tbody>
<tr>
<td>HYU1</td>
<td>130</td>
<td>1.45</td>
<td>0</td>
</tr>
<tr>
<td>HYU2</td>
<td>246</td>
<td>1.7</td>
<td>0</td>
</tr>
<tr>
<td>KSTAR</td>
<td>177</td>
<td>2.34</td>
<td>0.22</td>
</tr>
</tbody>
</table>

During the carborane evaporation test by using small vacuum chamber, it is found that carborane re-condensation occurred at colder parts of the boronization system. After 10 g evaporation experiment, about 3.7 g of re-condensed carborane is recovered from the vacuum vessel. Fig. 5 shows that 3.6 g at the lower part of gate valve, 0.01 g at pressure gauge, and 0.09 g inside the pipe to Turbo-Molecular Pump (TMP). Re-condensation occurred also inside the TMP, thus TMP blade was stuck. It is found that carborane molecules stay in the vacuum vessel for a long time, even after the boronization due to (probably) low sticking coefficient. This is clearly observed during a-C/B:H thin film deposition experiments: Shadow areas under silicon wafers are also deposited where ions from the plasma cannot freely enter (not shown here). Therefore, the amount of carborane loaded into the carborane evaporation system has to be carefully controlled and adjusted. Otherwise, the use of carborane vapor in a large vacuum system with TMPs like KSTAR would cause a serious problem: injected carborane molecules have to be consumed in the vacuum vessel before they reach TMPs.

6. Conclusion

First boronization in KSTAR has been successfully performed by using carborane vapor. With boronized wall, the amount of impurities in the KSTAR vacuum vessel is significantly reduced.
Characteristics of a-C/B:H thin films before and after the boronization have been systematically studied. Further investigation is underway. Re-condensation of carborane vapor in the carborane box and in the vacuum system has to be reported. Although this is one of important issues for machines that will/plan to use carborane vapor as boronization material, the re-condensation of carborane is not reported, yet. Furthermore, physical properties of carborane such as vapor pressure, pH, viscosity, boiling point, and freezing/melting point, are still unknown [6]. In order to use carborane vapor more effectively, physical properties of carborane have to be measured. Optimization of KSTAR carborane oven and vacuum system design are needed, and it is underway.

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References