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# Oxygen gettering properties of boron film produced by diborane dc glow discharge

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**Abstract**

Boron film coated on plasma facing walls has been utilized to reduce the oxygen impurity level by the gettering action. The boron film is also useful to reduce the hydrogen recycling. In this study, the boronization was conducted by a dc glow discharge with a mixture gas of diborane and helium both for a graphite and a stainless steel (SS) liners. After the boronization, the oxygen glow discharge was carried out to evaluate the gettered oxygen amount. The state of the oxygen in the surface was also examined. The gettered oxygen amount in the case of the graphite liner was about twice larger than that in the case of the SS liner. The oxygen was trapped in the depth range from the top surface to 100 nm or from the top surface to 20–30 nm in the case of graphite or SS, respectively. The oxygen was observed to be chemically bonded with the boron. After the oxygen discharge, the helium discharge was conducted to recover the oxygen gettering ability. After the helium discharge, the oxygen discharge was again carried out. The gettered oxygen amount in the case of graphite was comparable with that in the case of SS. © 1997 Elsevier Science B.V.

## 1. Introduction

The boronization has been widely conducted in fusion devices to reduce the oxygen impurity level in the plasma [1–4] and/or to suppress the fuel hydrogen recycling [5]. The oxygen in the plasma is trapped in the boron film and thus the effective charge number, $Z_{eff}$, can be decreased. The discharge operation is extended to a high density regime. In a case that the boron film is coated on the graphite wall, the erosion of the graphite in form of carbon monoxide is also suppressed and then the carbon impurity level can be reduced.

The boron film can trap the hydrogen considerably, but such that the hydrogen can be desorbed by baking with a temperature of about 300°C. Thus, the hydrogen recycling is suppressed if the wall conditioning is sufficiently carried out before the main discharge shot. In such a case, the energy confinement is well improved.

In order to understand the oxygen gettering capability and the hydrogen retention properties of the boron film, numerous simulation experiments have been conducted so far [6–10]. For the evaluation of these properties, the residual gas analysis (RGA) is often employed. In addition to the RGA, the surface analysis such as Auger electron spectroscopy (AES) are also required. Since the boron film is coated on a metal wall such as stainless steel, SS, or graphite in the device, the boron properties coated on these materials have to be investigated.

In this study, the oxygen gettering properties of the boron films made by the diborane discharge, deposited both on graphite liner and SS liner, were examined by conducting the oxygen discharge. The oxygen gettering capability was measured by the RGA during the discharge. After the oxidation of the boron film, the surface state was analyzed by the AES. The helium discharge was also done

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after the oxygen discharge in order to see the reactivation effect on the oxygen gettering.

## 2. Experiment

For the boronization and the oxygen gettering experiments, a dc glow discharge apparatus called surface modification teststand (SUT) of the National Institute for Fusion Science, was used. This apparatus consists of the discharge chamber and the AES chamber. In the discharge chamber, the anode, the liner made by 304 SS or graphite (IG-110U) and the sample holder were placed (Fig. 1). The liner temperature in the present experiments was kept at RT.

The boronization experiment was conducted by using diborane discharge with a mixture gas of diborane (5%) and helium (95%), a voltage of 360–480 V, the current was 0.2–0.3 A, the pressure 2.7 Pa, the diborane flow rate 24.5 sccm and helium flow rate 15.7 sccm, for the case of the SS liner or the graphite liner. The boronization time was 72 min and the thickness of the boron film was approximately 200 nm. The SEM photograph taken for the boron film deposited on the graphite remained roughly the same as that of the substrate since the surface roughness of the graphite was much larger than the film thickness. The boron film deposited on the SS made the surface smooth. After the boronization, the depth atomic composition of the sample placed on the sample holder was analyzed by AES.

In order to measure the oxygen gettering capability, the oxygen glow discharge was conducted after the boronization. The mixture gas of oxygen (10%) and helium (90%) was used. The voltage, the current, the pressure, oxygen flow rate and helium flow rate were 360–620 V, 0.3 A, 2.7 Pa, 2.4 sccm and 28.0 sccm, respectively. The discharge time was 20 min. During the discharge, the oxygen partial pressure was monitored by the quadrupole mass spectrometer, QMS. The depth atomic composition was analyzed by AES.

For the reactivation of oxygen gettering ability, after the oxygen discharge the helium discharge was carried out using helium gas (100%), a voltage of 360–620 V, the current was 0.2–0.3 A, the pressure 2.7 Pa and the helium flow rate 31.4 sccm. The discharge time was 60 min. After the helium discharge, the oxygen discharge was again conducted to examine the recovery of the oxygen gettering.

## 3. Results

Fig. 2 shows the change of the oxygen partial pressure for the cases of the graphite liner and the SS liner. After the oxygen glow discharge was turned on, the partial pressure rapidly dropped and gradually increased. After the turn off of the discharge, the pressure returned to the value before the discharge. During the discharge, the signal of CO was detected. The oxygen gettering amount was obtained after the subtraction of the contribution of CO. The oxygen gettering amounts for the SS and the graphite liners became $(0.5–1.2) \times 10^{17}$ O/cm$^2$ and $(1.8–2.0) \times 10^{17}$ O/cm$^2$, respectively. The gettered amount of the boron film on the graphite was roughly twice larger than that of the SS. Since the surface area of the boron film on the graphite is much larger than that of the SS, this difference may take place.

Fig. 3 shows the AES depth profiles of the boron films on the graphite and the SS after the oxygen glow discharge. In the case of the SS, the oxygen was trapped in

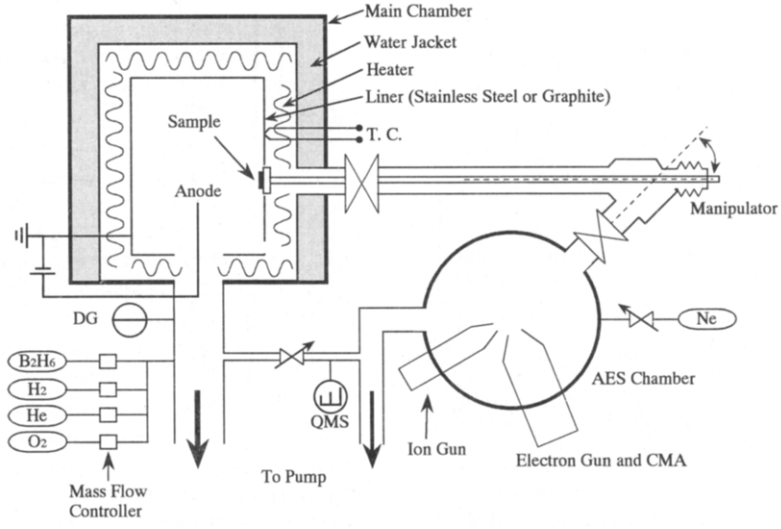


Fig. 1. Schematic diagram of surface modification teststand, SUT.

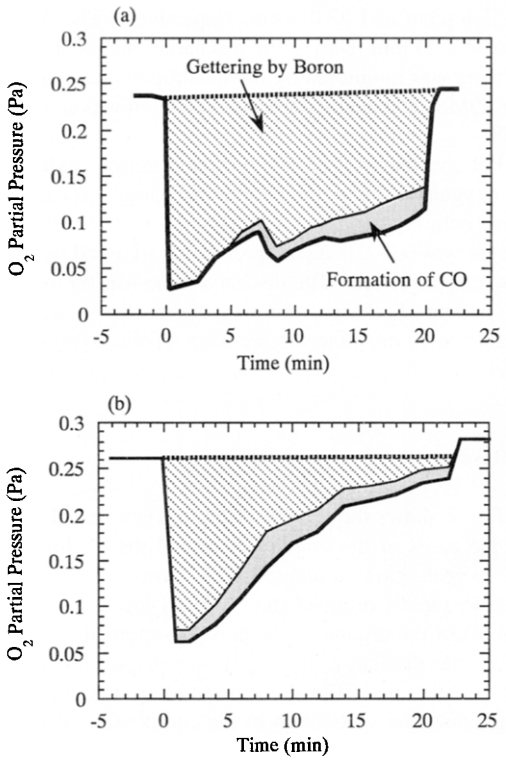


Fig. 2. Change of oxygen partial pressure during oxygen discharge in cases of graphite liner (a) and SS liner (b).

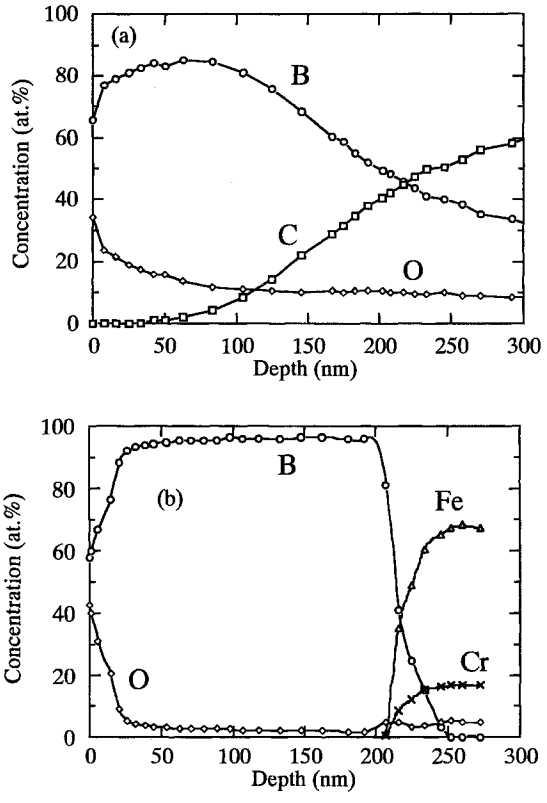


Fig. 3. Depth profile of the atomic composition of boron film after oxygen discharge in cases of graphite (a) and SS (b).

the depth up to 20–30 nm. The oxygen concentration at the surface was 40 at.%. In the case of the graphite, the oxygen was trapped in depth up to 100 nm. Since the graphite structure is very porous, it can be assumed that the boron film is deposited also in the pores. Thus, the oxygen shall be trapped in the deeper region, compared with the case of the SS liner. The oxygen concentration at the surface was 35 at.%. From the patterns of AES differential spectra for boron and oxygen, it was seen that the oxygen was chemically bonded with the boron in the form of boron oxide, B–O.

After the oxygen discharge, the helium discharge was done for 60 min, and then the oxygen discharge was again conducted for 20 min. Fig. 4 shows the change of the oxygen partial pressure during the oxygen discharge. The oxygen gettering amounts for the cases of the SS and the graphite liners were $(0.9–1.7)\times 10^{16}$ O/cm$^2$ and $(1.2–1.4)\times 10^{16}$ O/cm$^2$, respectively. The recovery of oxygen gettering is seen in the rapid decrease of $O_2$ pressure during the oxygen discharge conducted after the helium discharge. The recovery of the oxygen gettering ability in the case of the graphite was roughly the same as that in the case of the SS. The AES analysis showed that the oxygen concentration at the surface became low just after the helium discharge. The etching may contribute such recovery. The effective etching area due to helium ion bombard-

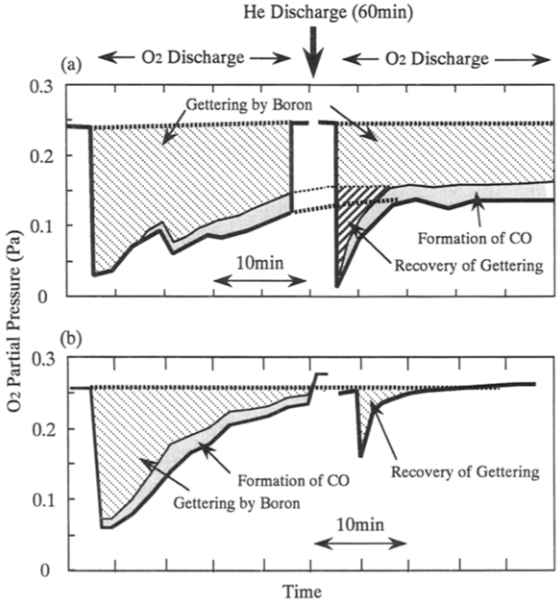


Fig. 4. Change of oxygen partial pressure during the oxygen discharge after the helium discharge in cases of graphite (a) and SS (b).

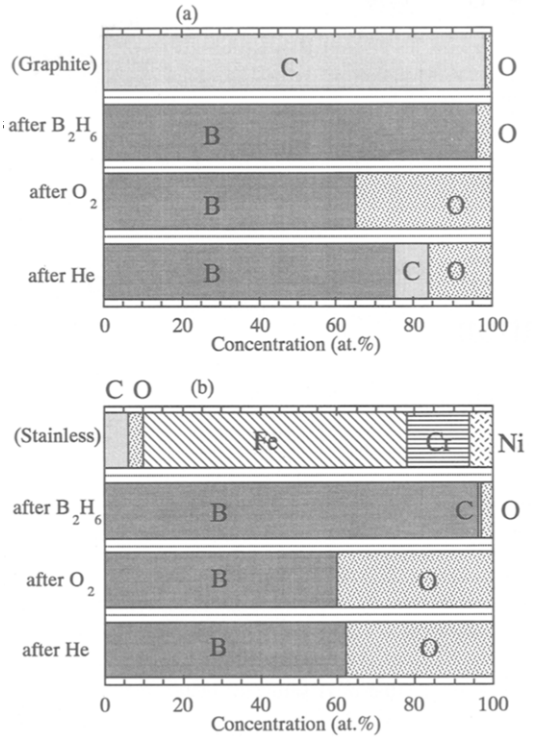


Fig. 5. Change of atomic composition at the surface after boronization, oxygen discharge and helium discharge in cases of graphite and SS.

ment shall not depend on the surface porosity. Thus, the recovery for the gettering becomes comparable between them. It is noted that, in the case of the SS liner, the emission of CO was not observed after the helium discharge. It is presumed that the carbon impurity on the wall was removed by the first oxygen discharge.

Fig. 5 shows the change of the atomic composition at the surface in the case of the graphite and the SS liner. By the boronization, the boron concentration at the surface became $\sim 95\%$ in both cases. After the oxygen discharge, the oxygen concentration became up to $\sim 40\%$. After the helium discharge, the oxygen concentration somewhat decreased. These changes are qualitatively consistent with the previous RGA data.

## 4. Summary

The boronization and the oxygen gettering experiments were carried out both for the cases of the graphite and the SS liners. After the boronization, the boron concentration at the surface became $\sim 95\%$ in both cases. During the oxygen discharge, the gettered oxygen amount was measured by the RGA. The oxygen gettering capability of the boron film deposited on the graphite was twice larger than that on the SS, because of the large effective surface area of the boron film or graphite. The AES analysis showed that the oxygen concentration at the surface became 35–40 at.% after the oxygen discharge. The helium discharge was observed to be effective to recover the oxygen gettering ability. This recovery may be due to the etching of the helium ion on the oxide layer.

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