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Section 9. Other topics

Basic experiments on in-situ magnetized boronization by electron cyclotron resonance discharges

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Abstract

Boronization based on electron cyclotron resonance (ECR) discharges is studied in a linear laboratory device under a parallel magnetic field of ~0.1 T. A 2.45 GHz ECR discharge in a low-pressure helium/decaborane mixture gives thin boron films localized on the walls near the gas inlet. In order to understand such film distributions, artificial deposition on plasma-exposed substrates is carried out. Two types of deposition processes are successfully discriminated in the magnetized plasma. One is neutral-induced deposition near the gas inlet and the other is ion-induced deposition in a downstream region. The latter plays a significant role in high-field high-density discharges. A simple scaling of deposition profile for each type of deposition is given along with its implication for achieving a deposition uniformity.

Keywords: Wall conditioning; Wall coating; Low Z wall material; Plasma–wall interaction simulator

1. Introduction

Plasma-assisted boron thin film deposition [1,2] i.e. boronization, has been used as a powerful technique for wall conditioning. To date, dc glow discharges in the absence of magnetic fields have widely been used for boronization to remove impurity and reduce hydrogen recycling very effectively. Such *non-magnetized* boronization will be of little use in future large devices such as LHD, W7X and ITER, since a steady-state magnetic field permanently exists after start-up of super conducting coils. There is no established method for periodically conditioning surfaces in the presence of high fields. One of the most promising candidates is the use of ECR (electron cyclotron resonance) discharges in a range of microwave frequencies.

The ECR-based boronization has been performed in Heliotron-E [3,4] and Alcator C-Mod using a 2.45 GHz discharge under a magnetic field of ~0.1 T. Boron thin film distributions along a toroidal magnetic field may be considerably different in comparison with the standard boronization. However, only little information on the spatial distribution of boron thin films deposited onto toroidal vessel walls has been obtained due to many restrictions on such measurements in the actual fusion devices. In general, there are two groups of precursors of boron film growth; neutral radicals (B_xH_y: x, y = integer) and ionic radicals ($B_xH_y^+$). It has been an open question which radical species are dominant precursors. The presence of a magnetic field may enable discrimination of ionic deposition from neutral deposition owing to a large difference of cross field diffusion between ions and neutrals.

In this paper, we report a basic study of ECR-based boronization where a linear machine is used for the sake of detailed measurements. The spatial distributions of boron films deposited on the vessel wall and substrates inserted in the plasma are measured and discussed from a viewpoint of the ion and radical transport in the magnetized plasma.

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2. Experimental

The laboratory experiment of ECR-based boronization [5] was performed in a linear machine of a stainless steel cylindrical vessel of 14 cm in diameter and ~1 m in length at room temperature, as shown in Fig. 1(a). A 2.45 GHz, 600 W microwave guided in TE_{10} mode is axially launched through a quartz window from a high field region to give the ECR condition. A helium plasma is produced at low pressures of 0.05–0.5 Pa, diffusing along a uniform magnetic field $B_0 = 0.075$ T along the z axis. A diameter of plasma column is adjusted by a grounded metal limiter of different diameters ($D = 4$, 10 cm), while the plasma is terminated by a movable grounded end plate of 14 cm in diameter. Boronization is carried out by injecting decaborane $B_{10}H_{14}$ [6] at a partial pressure of ~0.01 Pa with helium from a radially movable gas inlet (open stainless steel pipe). Henceforth, the axial position $z = 0$ refers to the position of the gas inlet and the positive x direction is defined to be the radial direction along the gas injection (see Fig. 1(a)).

A single Langmuir probe was used to measure the electron temperature T_e, the electron density n_e and the plasma potential V_p. In order to prevent the probe surface from boron film deposition, a plasma exposure time of the probe was minimized and the probe was cleaned by heating before the measurements. Typical plasma parameters at the 400 W discharge are $T_e \sim 6$ eV, $n_e \sim 3 \times 10^{17}$ m^{-3} and $V_p \sim 20$ V and they are almost uniform along magnetic lines of force. The radial distributions of probe ion current at the negative bias of -50 V with respect to the grounded vessel are shown for different powers in Fig. 1(b). Here the ion current is proportional to n_e since T_e is nearly independent of the radial position. The electron density is found to be rather uniform over the radius determined by the limiter ($D = 10$ cm in the present case). The plasma density was observed to linearly increase with the power and weakly increase with the pressure. As is well known, an influence of magnetic field on the probe may lead to serious errors in estimating the absolute electron density.

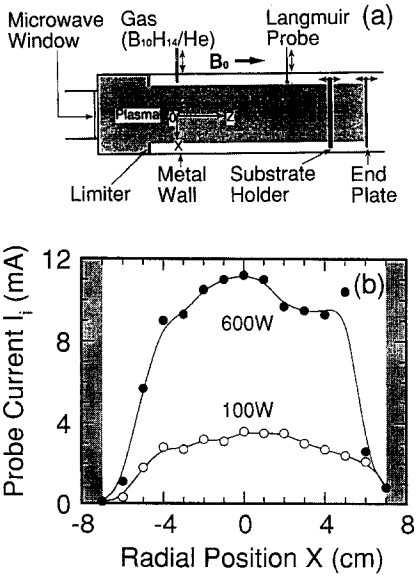

Fig. 1. (a) Schematic of experimental apparatus. (b) Radial profile of probe ion current for different discharge powers. $D = 10$ cm and 0.44 Pa helium/0.015 Pa decaborane.

Many silicon substrates of 1 cm^2 in area are placed on the vessel wall to observe a profile of deposited boron. The thickness of the boron film after ECR-deposition on each substrates is measured ex-situ by a step-profiler. In addition, the following deposition experiments were carried out to trace boron-containing ions and neutrals in the plasma. A linear array of substrates on a grounded holder (2 × 14 cm) along the x axis across the plasma column is immersed in the plasma as shown in Fig. 1(a). This sample holder is placed at several axial positions to obtain the deposition profile on the x–z plane. Moreover, a full three-dimensional profile is also obtained by attaching a number of substrates to the end plate which is moved along the z axis.

3. Results and discussion

3.1. Plasma with gas injection inside, diameter of plasma column 4 cm

A thin plasma column was formed by a 4 cm diameter limiter where decaborane was injected from the point of $x = 0$ and $z = 0$ inside the plasma. Fig. 2 shows examples of areal distributions of boron films deposited by a 450 W, 90 min discharge in 0.07 Pa helium (flow rate 0.05 sccm) and 0.01 Pa decaborane. The boron films deposited on the vessel wall ($x = 7$ cm) facing the gas inlet pipe are sharply localized near $z = 0$ and they are much thicker than the films on the wall ($x = -7$ cm) behind the gas inlet, probably because neutral radicals have their initial velocities toward the positive x direction.

The radial distributions of film thickness at $z = 7.5$ cm have large humps outside the 4 cm diameter plasma column. At $z = 32$ cm, however, no film is recognized in the scrape-off layer ($|x| > 2$ cm). These results are qualitatively understood in terms of ionic deposition and neutral deposition as follows. Boron hydride ions e.g. mass number $M = 12$ (BH^+) and the temperature $T_i = 0.15$ eV, gyrate around the magnetic field line as $\Omega_i \tau_i \gg 1$ and the ion Larmor radius = 5.5 mm $\ll$ the plasma diameter where $B_0 = 0.075$ T, $p = 0.1$ Pa, and Ω_i and τ_i denote the ion cyclotron frequency and the ion collision time, respectively. Thus, the ions are transported along B_0 so that the films observed at $z = 32$ cm are mainly deposited by ions. The films on the vessel wall facing the gas inlet ($x = 7$

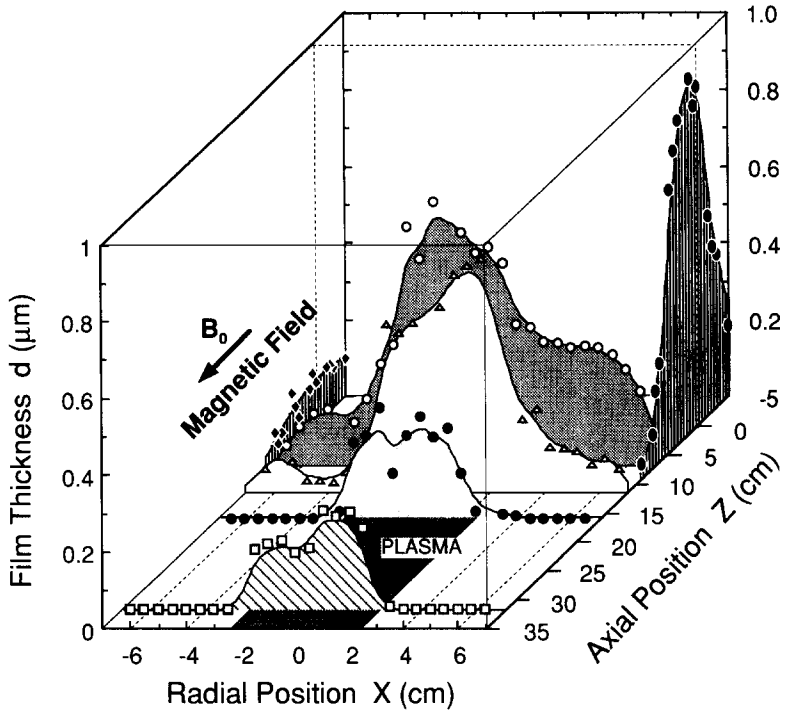

Fig. 2. Radial distribution of boron film thickness for different axial positions, together with the z dependence of film thickness of boron deposited on the side walls, $x = -7$ cm and 7 cm. $D = 4$ cm, 450 W and 0.07 Pa helium/0.01 Pa decaborane.

cm) or behind the gas inlet ($x = -7$ cm) are considered to be deposited by neutral radicals. Moreover, the humps observed outside the plasma column ($|x| > 2$ cm) and near the gas inlet ($z < 15$ cm) are also attributed to the neutral deposition.

3.2. Plasma with gas injection outside, diameter of plasma column 10 cm

Using a large limiter of $D = 10$ cm, a thicker plasma column is produced where decaborane is injected from the outside of plasma i.e. the point of $z = 0$ and $x = -6$ cm near the wall. The radial distribution of boron film thickness was measured inserting the sample holder at various z positions. The deposition rates obtained are displayed on the x–z plane in Fig. 3. For the low power case of 100 W, a considerable amount of deposition is found in a region near the gas source ($z < 25$ cm), mainly induced by neutral radical sticking. The plasma decomposition of decaborane near the gas inlet gives rise to abundant neutral radicals which can cross magnetic field line, diffuse isotropically and stick onto the nearby walls.

For the higher power of 600 W, the deposition rate becomes lower near the gas inlet and higher in a downstream region ($z > 30$ cm) compared with the data for 100 W. The ECR plasma has relatively high densities (several times 10^{16} m^{-3}) even at 100 W where most of decaborane gas is decomposed. The present result shown in Figs. 2 and 3(a) suggest that electron-impact dissociation of decaborane yields much more neutral radicals than ions, possibly due to lower thresholds. At high powers such as 600 W, more primary ionization takes place together with secondary processes of radical ionization. Accordingly, more neutral radicals are converted at the higher power to ions which are preferentially transported along B_0. As a consequence, the deposition rate at the position far from the gas inlet is increased at the high power.

3.3. Boron film deposition by neutral radicals

As discussed above, the low-power boron deposition near the gas source is primarily caused by neutral species. Fig. 4 shows a semi-logarithmic plot of the deposition rate obtained at the position $z = 2$ cm extremely close to the gas inlet. Here the decaborane gas is radially injected from $z = 0$ and $x = -6$ cm into the relatively thick plasma whose density is uniform for $|x| < 5$ cm. One-dimensional crude arguments [7] based on diffusion and dissociation processes predict the exponential decay of deposition rate as $\gamma(x) \propto \exp(-x/L_n)$ where the characteristic length L_n is given by:

$$L_n = (D_n/\nu_d)^{1/2} = C(pn_e)^{-1/2} \quad (1)$$

where D_n and ν_d denote the diffusion constant of decaborane colliding with helium (pressure p) and the electron-impact dissociation frequency, respectively, with the proportional constant C. In fact, the experimental points for

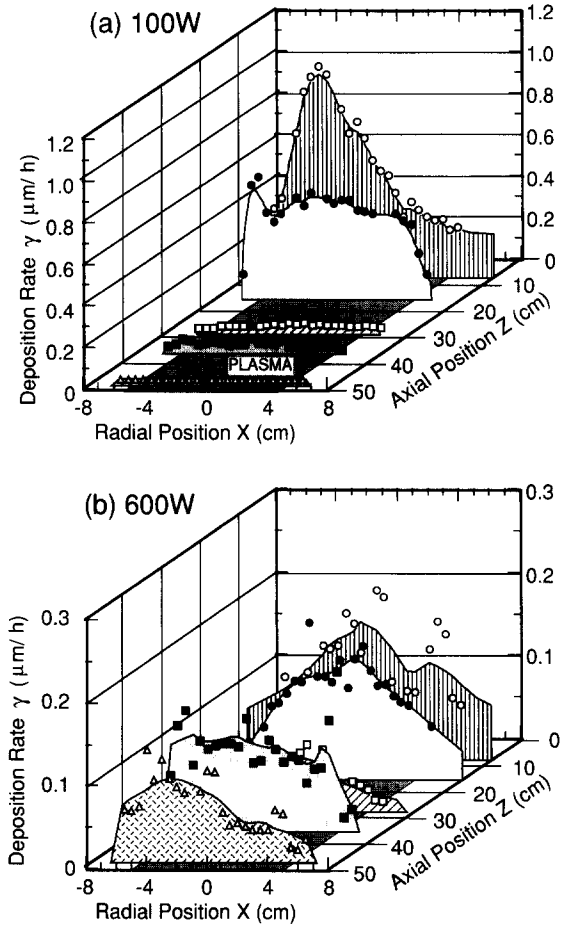

Fig. 3. Radial and axial distributions of boron deposition rate for (a) 100 W and (b) 600 W discharges. $D = 10$ cm and 0.21 Pa helium/0.015 Pa decaborane.

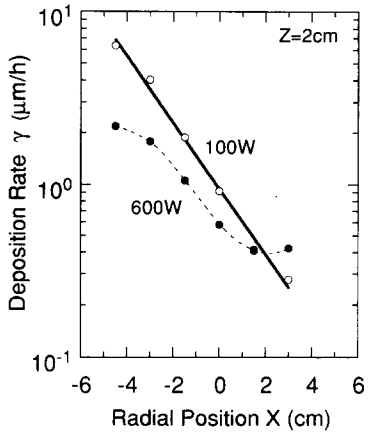

Fig. 4. Boron deposition rate γ at $z = 2$ cm as a function of the radial position x when decaborane is injected from $z = 0$ and $x = -6$ cm. $D = 10$ cm and 0.16 Pa helium/0.012 Pa decaborane.

100 W clearly show such exponential dependence across B_0 and the solid line in Fig. 4 in turn gives an empirical value of the constant as $C = 1.6 \times 10^6$ ($\mathrm{Pa}^{1/2}$ $\mathrm{m}^{-1/2}$). For higher powers such as 600 W, the deposition process becomes complicated due to ionic contribution and does not follow a simple exponential decay as seen in Fig. 4. The decrease in deposition rate near the gas inlet at high powers is again interpreted by the rapid ion loss along B_0.

3.4. Boron film deposition by ionic species

Fig. 5 shows the deposition rate along the z axis for the radial position fixed at $r = 0$. As seen in Figs. 2 and 4, the deposition caused by neutral radicals sharply localizes in a short scale L_n of ~ 5 cm near the gas source. Therefore a slow decrease in the deposition rate for $z > 10$ cm in Fig. 5 corresponds to the ion flow along B_0. Let the diffusion constant of boron containing ions across B_0 be $D_\perp = D_\parallel/(1 + \Omega_i^2\tau_i^2)$ for the parallel diffusion constant $D_\parallel$. Suppose the ion density to vary as $J_0(k_\perp r)\exp(-z/L_i)$ with a boundary condition of $k_\perp a = 2.40$ (the first root of the Bessel function J_0) at the plasma radius a. Then we obtain the characteristic length L_i as

$$L_i = \left(D_\parallel/D_\perp\right)^{1/2}/k_\perp = \left(1 + \Omega_i^2\tau_i^2\right)^{1/2}(a/2.40). \quad (2)$$

Since an axial decrease in ion density is caused by the ion diffusion across to B_0, boron films are axially deposited following the scaling of Eq. (2). Thus, the deposition uniformity along B_0 will be achieved for larger values of $\Omega_i\tau_i$ and a. In the present experiment, one may take the values as $a \sim 7$ cm and $\Omega_i\tau_i \sim 12$ for $M = 12$ (BH^+) and hence $L_i = 35$ cm as indicated by the solid line for 600 W in Fig. 5. At the low power of 100 W, the influence of neutral radicals appears for $z < 20$ cm as indicated by the dashed line in Fig. 5.

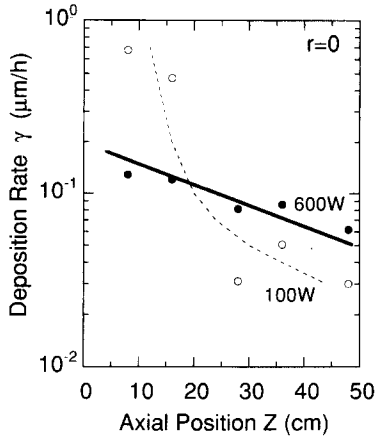

Fig. 5. Boron deposition rate γ at $r = 0$ as a function of the axial position z when decaborane is injected from $z = 0$ and $x = -6$ cm. $D = 10$ cm and 0.21 Pa helium/0.015 Pa decaborane. The solid line indicates the predicted slope for characteristic length $L_i = 35$ cm.

4. Conclusion

ECR-based boronization was performed in a linear laboratory device to get information on the boron deposition kinetics. Contribution of ionic radicals to deposition was clearly discriminated, for the first time, from neutral radical contribution under the magnetic field. The characteristic length for deposition profiles are derived for both ion- and neutral-induced depositions. The present results suggest that a toroidal uniformity of boronization in the next generation large devices can be achieved by ion-induced deposition more easily than by neutral-induced deposition, although the ion drift due to grad B and curvature in B could produce poloidal asymmetries in a simple toroidal magnetic configuration.

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