

## In-Situ Surface Modification by ECH Plasmas in Heliotron E

FUJITA Norihito\*, NISHIMURA Kazuhito, MIZUUCHI Tohru, KITO Toshiharu, KONDO Katsumi<sup>1</sup>, NAGASAKI Kazunobu, OKADA Hiroyuki, SANNO Fumimichi, OBIKI Tokuhiko, KOKURA Hikaru<sup>2</sup>, TOYODA Hirotaka<sup>2</sup> and SUGAI Hideo<sup>2</sup>

*Institute of Advanced Energy, Kyoto Univ., Uji 611-0011, Japan*

<sup>1</sup>*Graduate School for Energy Science, Kyoto Univ., Uji 611-0011, Japan*

<sup>2</sup>*Department of Electrical Engineering, Nagoya Univ., Nagoya 464-8603, Japan*

(Received: 30 September 1997/Accepted: 12 January 1998)

### Abstract

This paper describes in-situ surface modification methods by using ECH plasmas. A localized deposition of a B-film was observed for the ECH boronization in Heliotron E. The reason of the local deposition and how to improve the uniformity of a B-film are discussed. On the other hand, a unique evaporation technique of lithium by a "plasma-assisted heating" was examined in a small linear device for the ECH "lithiumization".

### Keywords:

wall-conditioning, boronization, ECH plasma, heliotron, lithium-coating

### 1. Introduction

The surface modification of plasma facing materials is one of the important issues to obtain a good plasma performance in present-day fusion devices. An *in-situ* coating technique is attractive as a surface modification method, since it is expected that oxygen impurity, hydrogen recycling and/or the erosion of the first wall are controlled by choosing a proper coating material. Instead of the conventional glow discharge method, we have developed the *in-situ* coating method using ECH plasmas. Such a coating technique under the existence of a magnetic field has some advantages. First, there is no need for shutoff of a field, which is a great advantage in future devices with superconductor coil systems. Secondly, we can expect the improvement of the toroidal uniformity of the deposited film owing to the fast parallel diffusion of reactive materials ionized by plasmas.

The boronization is effective in a reduction of light impurities and H-retention[1]. After the ECH boroni-

zation was performed in Heliotron E, a remarkable reduction of light impurities such as O and C was also observed[2]. However, metal impurities from the wall such as Fe and Ni were not reduced in contrast with the DC glow carbonization[3]. It is suspected that the uniformity of the B-film is not enough compared to the case of the carbonization. It is important to measure the film distribution and to improve the toroidal uniformity.

On the other hand, a Li-coating method is noticeable for the high gettering effect to O, CO and CH<sub>4</sub> and the possibility to control H-retention[4]. It has been reported that the wall conditioning with Li pellets leads to a great enhancement of the plasma performance in TFTR[5]. As a basic experiment to develop the optimum Li-coating technique, a "lithiumization" by an ECH plasma has been developed in a small linear device.

In this paper, we discuss the uniformity of the

\*Corresponding author's e-mail: fujita@center.iae.kyoto-u.ac.jp

B-film deposited by the ECH boronization in Heliotron E. Then we describe the basic experiments on the ECH Li-coating.

## 2. ECH Boronization

In Heliotron E, a boronization has been performed under the 2.45 GHz ECH discharge with a mixture gas of He and  $B_{10}H_{14}$ [6]. In order to study the uniformity of the deposited B-film, we measured the toroidal and poloidal profiles of the film thickness by placing sample plates (stainless steel) in the vacuum vessel (Fig. 1). The poloidal profile was measured by the samples on two vertical arrays (#11.5-O, #17.5-O) located at  $r/r_{\text{wall}} \sim 0.76$ . Each vertical array has three surfaces (front, left, right) so that we can get information on the direction from which reactive particles come.

The ECH boronization was performed twice with changing the gas-inlet positions. About 10 g of  $B_{10}H_{14}$  was consumed for each experiment. If the deposition is uniform, the average thickness of the B-film over the whole inner wall is expected to be  $\sim 200$  nm. Typical electron temperature, density and the neutral pressure are  $\sim 10$  eV,  $1-3 \times 10^{16} \text{ m}^{-3}$  and  $\sim 1 \times 10^{-2}$  Pa, respectively. The positions of the gas-inlets were #3.5, #18.0 in the first experiment and #14.5, #18.0 in the second one. The gas nozzles were set at the wall position in both cases. The film thickness on samples was analyzed with a step profiler. The results are shown in Fig. 2 and Fig. 3. The film thickness at #27.5-I was less than the detection limit ( $\sim 10$  nm) in both experiments. These results indicate that the thickness of the B-film strongly depends on the distance from the gas-inlets. The deposition of the B-film was localized at the

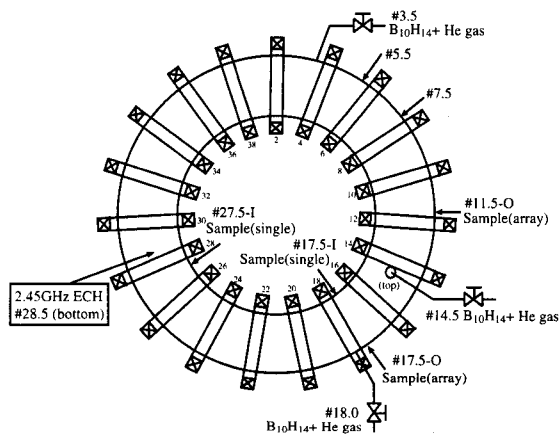


Fig. 1 The ECH boronization system and locations of the samples.

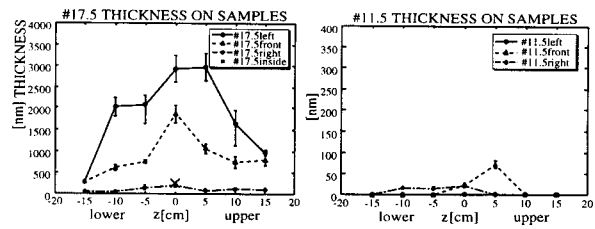


Fig. 2 The vertical profile of the B-film thickness (the gas-inlet #3.5, #18.0).

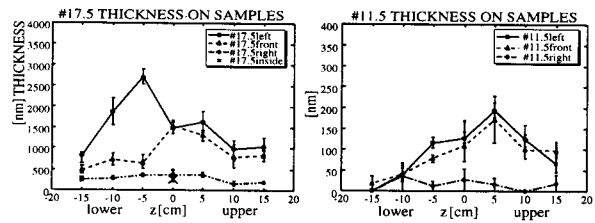


Fig. 3 The vertical profile of the B-film thickness (the gas-inlet #14.5, #18.0).

position close to the gas-inlet, particularly on the surface facing the gas-inlet. Generally in the boronization using  $B_2H_6$  and  $B_{10}H_{14}$ , a toroidally uniform film tends to be less formed due to the high sticking efficiency of the dissociated particles than in the carbonization using  $CH_4$ [7]. Since the mean-free path of  $B_{10}H_{14}$  in our experimental condition is not short enough compared with the dimension of the device, the injected neutral particles will mainly flow to the wall area which directly faces the gas-inlet. Even if they are ionized, they will immediately deposit to the wall close the gas-inlet owing to the parallel diffusion in the short  $L_c$  (the connection length of the edge magnetic field to the wall) region near the wall.

If the film thickness is given as an exponential function,  $d = d_0 \exp(-x/L)$ , as discussed in Ref.[8], we have  $L = 43$  cm from Fig. 3, where  $L$  is a characteristic decay length and  $x$  is a distance from the gas-inlet along the toroidal direction. Using this value, we can estimate the film thickness at #11.5-O samples for the gas injection from #3.5 and #18.0. The actual thickness (72 nm) shown in Fig. 2 is, however, about five times larger than the estimate (15 nm). The vertical profiles of the B-film thickness on the front side of the #11.5-O array have their peaks at the position  $z \sim 5$  cm, where the SOL plasma touches the array. These observations suggest that some fraction of the injected  $B_{10}H_{14}$  molecules is ionized in the long  $L_c$  region and flows along the field line. Such a process contributes to toroidally uniform

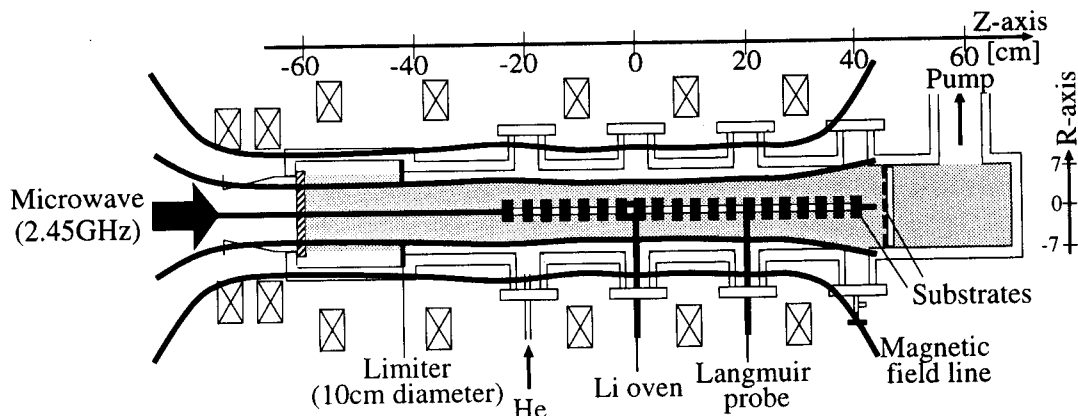


Fig. 4 A schematic of the ECH Li-coating system.

deposition. If we can increase the ratio of the ionized particles in a long  $L_c$  region, the toroidal uniformity of a B-film should be much improved[6,9]. In order to do this, we are planning to insert a gas nozzle inside the outermost magnetic surface, where  $L_c$  is infinite. The higher  $T_e$  and  $n_e$  in the confinement region will promote the ionization of the injected gas more effectively.

### 3. Li Coating

The experiment of an ECH Li-coating has been performed in a linear device with a cylindrical vessel as shown in Fig. 4. An ECH plasma is produced by 2.45 GHz microwaves. The diameter of the plasma column is adjusted by a grounded metal limiter of 10 cm in the diameter, located at the downstream side of a resonance region. The Li container of 2.5 cm cubic, made of molybdenum, is inserted to the plasma center and biased with +50 ~ 200 V for inducing an electron current to flow into the container. By this current, the container is heated up to ~ 700 K, and Li evaporates. This method needs no heater, and makes the coating system simple.

A Langmuir probe was used to measure the electron temperature, density and the space potential. As shown in Fig. 5, the distribution of the electron density ( $1-6 \times 10^{16} \text{ m}^{-3}$ ) was limited from -5 cm to +5 cm in the R-direction by a limiter. The density was increased as the microwave power increasing. The electron temperature was  $3.5 \pm 0.5 \text{ eV}$ , and the space potential was  $9.5 \pm 1.0 \text{ V}$  under the 100 W discharge in He pressure of 2.7 Pa. These parameters were independent of microwave power in this experimental condition. The radial profiles were almost flat.

Stainless steel substrates were arranged axially along the wall and radially on the plate near the end of the chamber to observe the spatial distribution of film

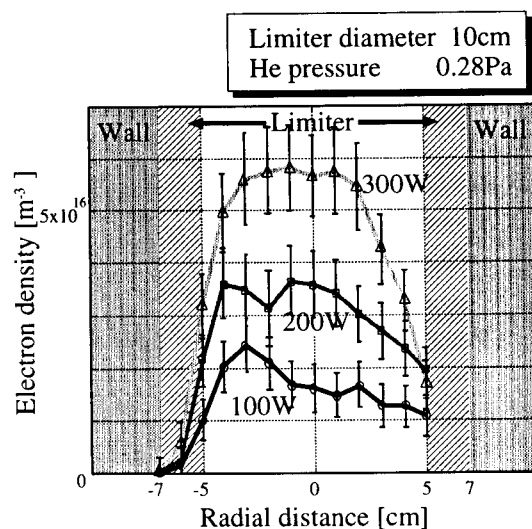


Fig. 5 The radial profile of the electron density.

thickness as shown in Fig. 4. To observe the *in-situ* spatial distribution of lithium in the chamber, the line emission from the plasma was measured at the four view ports.

The experiment was performed under the condition of  $P_{\text{He}} \sim 0.28 \text{ Pa}$ ,  $P_{\text{Li}} \sim 1 \times 10^{-6} \text{ Pa}$  (vapor pressure),  $P_{\text{ECH}} \sim 100 \text{ W}$ , and  $V_{\text{bias}} \sim +180 \text{ V}$ . The line radiation from  $\text{Li}^+$ ,  $\text{Li II}$  (548.5 nm), was hardly observed. In addition, the mean-free path for ionization of Li,  $\lambda_{\text{ion}}$ , is estimated at about 1 m under the experimental condition. Considering the plasma dimension, 1.2 m in the axial length, 0.10 m in the diameter, this suggests that ionization events rarely occur.

The distribution of  $\text{Li I}$  (610.7 nm) intensity and the film thickness are shown in Fig. 6, where the horizontal axis is a distance on Z-axis. The  $\text{Li I}$  intensity in Fig. 6 is normalized by the intensity of  $\text{He I}$  (388.9 nm)

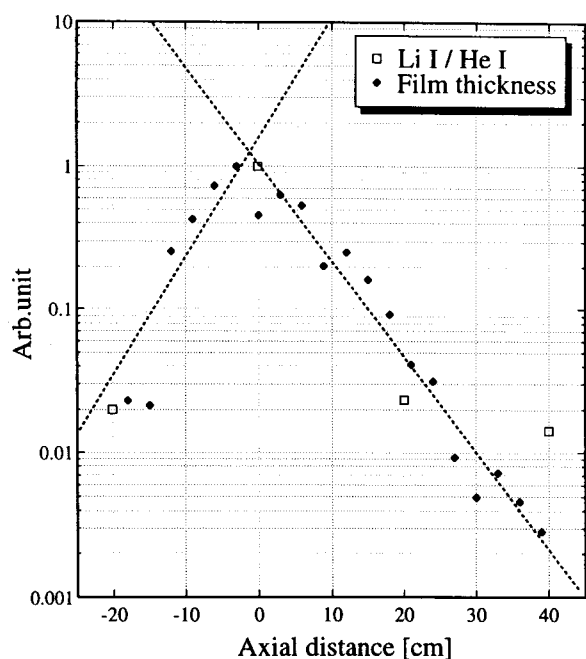


Fig. 6 The axial profile of Li spectrum and film thickness.

to remove influence of the change of the electron density. The distribution of the film thickness is normalized by the thickness of the thickest substrate. Both distributions show the exponential decay with a decay length of  $\sim 0.07$  m.

In order to understand the observed distribution, we considered a simple model where the neutral lithium diffuses to the Z-direction with a loss to the R-direction at the constant rate. The particle balance for neutrals is described by  $\partial n / \partial t = D \partial^2 n / \partial x^2 - \nu_a n$ , where  $\nu_a n$  is the loss rate to the R-direction, and  $D$  is diffusion coefficient. Supposing that plasma is in a steady state, we obtain  $n = n_0 \exp(-x/L)$ , where  $L = (D/\nu_a)^{1/2}$ . From Fig. 6, we have  $L^{\text{exp}} \sim 0.07$  m. Assuming that collisions between  $\text{Li}^0$  and  $\text{He}^0$  are dominant in this experimental condition,  $\nu_a^{\text{model}}$  and  $D^{\text{model}}$  are estimated at  $\sim 2 \times 10^4 \text{ s}^{-1}$  and  $\sim 9 \times 10^2 \text{ m}^2 \text{ s}^{-1}$ , respectively. Therefore  $L^{\text{model}}$  becomes  $\sim 0.06$  m, which is in good agreement with  $L^{\text{exp}}$ . This means that the neutral particles mainly contribute to the film distribution owing to a low ionization rate in this experiment. For 2.45 GHz ECH plasmas in Heliotron E, the ionization mean-free path is estimated at  $\sim 0.2$  m. Therefore, if we apply this Li-coating method to Heliotron E, we can expect toroidally uniform deposition by lithium ions due to a higher ionization rate.

#### 4. Summary

The distribution of B-film thickness along the torus by the ECH boronization was measured in Heliotron E. It is confirmed that B-film is localized near the gas-inlet and there is very thin film far from the gas-inlet as suspected from the less reduction of metal impurities after the boronization. It is considered that a rather short mean-free path of the injected gas and a short  $L_c$  of the field line near the wall cause such a localized deposition. Direct injection of the reactive gas into the confinement region will promote the ionization and improve the toroidal uniformity of the film deposition.

A simple Li-coating technique by using the ECH plasma was examined in a small linear device, and a Li-film could be produced by the heating of the container by inflow of the electron current from the ECH plasma. The film deposition was, however, dominated by Li neutrals since the ionization events were rarely occurred in this experimental condition. If this method is applied to Heliotron E, it is expected that a higher ionization rate will enhance toroidally uniform deposition.

#### References

- [1] J. Winter *et al.*, J. Nucl. Mater. **162-164**, 713 (1989).
- [2] K. Kondo *et al.*, J. Nucl. Mater. **220-222**, 1052 (1995).
- [3] N. Noda *et al.*, J. Nucl. Mater. **162-164**, 769 (1989).
- [4] H. Sugai *et al.*, J. Nucl. Mater. **220-222**, 254 (1995).
- [5] J.A. Snipes *et al.*, J. Nucl. Mater. **196-198**, 686 (1992).
- [6] T. Mizuuchi *et al.*, *Proc. of 10th Int. Conf. Stellarator*, Madrid, p.81 (1995).
- [7] G.L. Jackson *et al.*, J. Nucl. Mater. **196-198**, 236 (1992).
- [8] O.I. Buzhinskij and Y.M. Semenets, Fusion Technol. **32**, 1 (1997).
- [9] H. Kokura *et al.*, J. Nucl. Mater. **241-243**, 1217 (1997).