Studies of deposition of boron films by PECVD of He/o-carborane mixtures in TJ-II.

F. Tabarés, D. Tafalla, E. Oyarzabal, B. López-Miranda and A. Baciero.

Fusion Department- CIEMAT. Av Complutense 40. 28040 Madrid. Spain

Fusion Department- CIEMAT. Av Complutense 40. 28040 Madrid. Spain

Abstract: Boronization of the first wall of the TJ-II Stellarator is accomplished by a DC Glow Discharge in a mixture of helium and o-carborane (C_2B_{10} H_{12}) at a total pressure of ~1 Pa and total currents from 1 to 2 A. Four ovens are used for the injection of carborane and four anodes and 4 turbomolecuar pumps in a symmetric configuration are run during the boronization process. Studies aimed at producing a homogeneous layer with low H content and strong O gettering activity are here described and modelled.

Keywords: Boronization, fusion devices, PECVD, glow discharge.

1. Introduction

Wall conditioning of fusion devices is a common practise required for the effective control of plasma impurities and particle recycling [1]. Although applying cold plasma discharges in the vacuum vessel in He or H_2 provides a way to significantly reduce oxygen and carbon in the plasma, sputtering of high Z elements (Fe, Ni, Cr, W, Mo.) from the first wall represents a source of highly radiative impurities affecting the power balance of the plasma and its purity. Therefore, coating techniques based on light elements (Li, B, C.) have become very popular in the fusion community [2]. Excellent results have been obtained so far and many techniques were developed for this purpose.

Among all possible options, including radiofrequency, microwave, low power plasmas, the simplest approach from the instrumental point of view remains the DC Glow discharge, with the vacuum vessel acting as a cathode, on which a film precursor is injected at low concentration. For the case of born films (with very good properties in terms of oxygen getter and H recycling) several film precursors have been used. Although diborane stands as the simplest molecular species, its toxicity and explosive characteristics makes it rather difficult to implement. On the contrary, tri-methyl boron (B $(CH_3)_3$), decaborane ($B_{10}H_{14}$) and orto-carborane ($C_2B_{10}H_{12}$) are preferred whenever the implementation of the safety protocol associated to handling diborane cannot be fulfilled. Even when some of these molecules have carbon in their formulae, their performance as B film precursors has proven comparable to B_2H_6 [3].

2. TJ-II boronization system

The systematic boronization of TJ-II (starting in 2001) was the chosen procedure for obtaining a low Z scenario during hot plasma generation [4]. Boronization was carried out by injecting o-carborane in a He DC-GD and keeping the vessel walls at room temperature. O-carborane (the less harmful B precursor) was selected since its high vapour pressure makes it possible to control the feeding rate into the plasma by simply heating its container at T<100ºC. An important point in a device with a complicated geometry like TJ-II is to obtain a good uniformity of the B/C film all over the exposed wall. For this purpose, four ovens were installed in symmetrical positions of TJ-II vacuum vessel. In addition, the four anodes and turbopumps available, also symmetrically spaced, were used during the process. A total amount of 4 g of carborane (1 g each oven) was injected during He GDC. This amount is enough to cover the inner wall exposed to plasma with a B/C film of 50 nm. All the process was monitored by a Residual Gas Analyser (RGA). Unfortunately, the continuous ramp-up of the temperature made it difficult to estimate the cracking efficiency from the RGA data during the deposition (see below). Peaks at amu 70 and 144 in the mass spectra were used as a reference for o-carborane, while the increase in amu 2 is the main signature of molecular cracking into the plasma. Figure 1 shows the RGA spectra for the plasma on-off conditions in a carborane-He mixture. A conspicuous depletion of all the peaks associated to the precursor molecule and a concomitant rise of the H2 peaks in the plasma-on conditions can be seen.

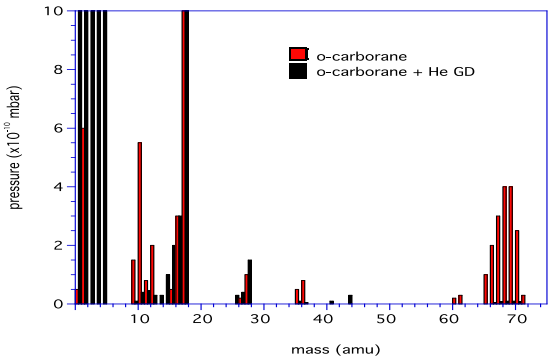

Figure 1. RGA spectrum in plasma on-off conditions during the boronization of TJ-II [4].

After boronization a period of 30 min of He GDC is required for depletion of the hydrogen retained in the film. The B/C film has an immediately gettering effect on the oxygen containing molecules (O_2, H_2O, CO, CO_2) and these species almost disappear from the residual gas spectrum. The effect diminishes after 1 day of ECRH plasmas, but it can be regenerated with a 30 min He GDC every day during several weeks (even several months depending of the plasma discharges)

3. Film Homogeneity

By analysing the continuity equation for the injected species, the cracking fraction of the precursor $(P_0\text{-}P)/P_0$ can be expressed by the simple equation:

$$(P_0\text{-}P)/P_0 = K_D^{eff}(T_e)\, n_e.\tau_r / (1 + K_D^{eff}(T_e)\, n_e.\tau_r) \quad 1)$$

where K_D^{eff} stands for the, temperature dependent, effective dissociation rate constants of the precursor in the plasma, n_e its electronic density and τ_r is the residence time of the vacuum system $\tau_r = V/S_{eff}$. Therefore, the microscopic plasma parameters, ne, Te, related to the GD characteristics, the dissociation constant of the precursor molecule and the pumping time determine the degree of cracking by the plasma. For high cracking factors, prompt deposition of the film near the inlet location is expected, thus leading to a highly inhomogeneous coating.

The behaviour predicted by eq.1 was tested in laboratory experiments. Table I shows some results. As seen, the deduced values for $K_D^{eff}(T_e)\, n_e.\tau_r$ show a linear dependence on plasma current, as expected for a simple proportionality between this parameter and the electronic density in the GD.

Table 1
Values of the *o*-carborane cracking ratio and the total cracking rate ($K_D^{eff}(T_e)n_e$) for different plasma currents (I_p)

I_P (mA)	Cracking ratio (%)	$K_D^{eff}(T_e)n_e$ (s^{-1})
20	80	80
50	92	220
200	99	1000

In this work, a systematic study of the relationship between the cracking fraction and the film spatial distribution in the vacuum vessel in TJ-II has been undertaken. A quartz microbalance was inserted at several locations toroidally apart from the injection point of decaborane. The plasma current and the system effective pumping speed were varied and a Langmuir probe was used for the characterization of the microscopic parameters of the discharge. As an example, preliminary results of the effect of varying the plasma current on the cracking and H release of carborane are displayed in Figure 2.

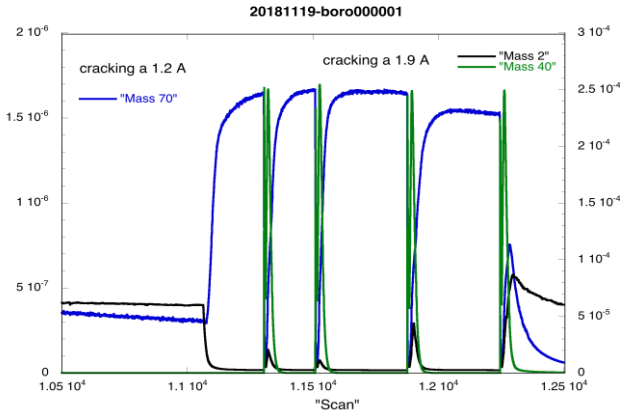

Figure 2. Evolution of amu 70 (o-carborane) and amu 2 (H_2) during a change in plasma current from 1.2 to 1.9 A. Also shown is amu 40 corresponding to the injection of Argon used for discharge initiation.

As it can be seen, a significant change in the depletion of amu 70 in the plasma takes place as Ip is increased to 1.9 A. Also, the ratio of amu 2 to amu 70 is higher at 1.9 (last cycle) although clear hints of running out of carborane are observed at the last cycle, after the last Ar peak. Accordingly, running the GD plasma at 1.2 A during boronization should provide a more uniformly distributed film and a better performance during hot plasma operation.

Another important issue is the control of hydrogen inventory associated to the deposition of the film. In the presence of hot deuterium plasmas, a dilution of the plasma species by its lighter isotope has a negative impact on the fusion product. In hydrogen plasmas, density control in the presence of a desorbing wall becomes

challenging. The most effective technique would be keeping the wall temperature at T>350 ºC, but at present this is only possible in very few fusion devices. Therefore, conditioning of the film by He DC GD plasmas becomes a standard technique after boronization.

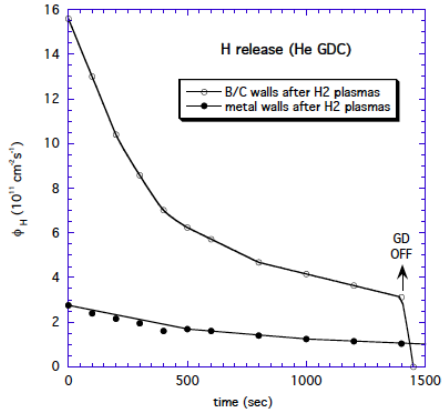

Figure 3. H_2 desorption by a He GD after hot plasma operation in H for two different wall states.

Figure 3 shows the release of H2 from the wall after hot plasma operation in H in TJ-II by a He GD. As seen, a much higher level of desorption takes place in boronized walls as compared to bare metal. Aside from wall temperature, the composition of the film precursor and the DC plasma characteristics make a difference on the H uptake by the film. High temperature plasmas, as those obtained by Electron Cyclotron discharges, promote the contribution of ionic fragments to the film growth, leading to a lower H content of the film [5]. For DC GD, the gas pressure and its throughput also have impact on the resulting film composition. In order to test this effect, the referred parameters were also varied aiming at a low H content of the film, which was measured by mass spectrometry in He GD and by TDS of samples exposed to the boronization at several locations.

Finally, the performance of the conditioning method was tested in hot plasmas in TJ-II. The recycling characteristics together with the plasma contamination by O and C were used as indicators of the film quality. Table 2 summarizes the main parameters achieved under metallic and boronized walls in ECRH plasmas in TJ-II [6]. A higher edge and central electron temperature, lower radiated power and Zeff value were systematically recorded after the B coating.

Table 2.

Characteristic plasma parameters for metallic and boronised first wall scenarios in TJ-II

Wall	$T_e(0)$ (keV)	T_{edge} (eV)	n_{edge} ($\times 10^{17}$ m^{-3})	T_i (eV)	P_{rad} (kW)	Z_{eff}
Metal	0.75	17	6.5	100	57	1.5
Boronised	0.95	50	6.5	80	15	~1

The $\langle n_e \rangle$ line value is 0.65×10^{19} m^{-3} in both cases.

Since bare metal surface is not available anymore in TJ-II, a comparative study of the effect of deposition parameters on the achieved hot plasmas will be presented here.

References

[1] K.H. Burell. Plasma Phys Control Fusion **36** (1994)
[2] J. Winter. Plasma Phys Control Fusion **36** B263 (1994)
[3] O. Buzhinskij and Y. Semenets. Fus. Technol**. 31**, 1, (1997)
[4] D. Tafalla and F. L. Tabarés. Vacuum 67 393 (2002)
[5] Y Ito et al. Plasma Sources Sci. Technol. 5, 305 (1996)
[6] F. L. Tabarés et al. J. Nucl. Mater **313-316** 839 (2003)