Study of the effect of the glow power on the growth of the boron coating obtained with boronizations in RFX-mod

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1. Introduction

Fusion devices are often operated using wall-coating methods involving the deposition of thin (~100 nm), low-Z layers instead of using as-installed, bare PFCs. These layers are found to enhance plasma performance (energy confinement) through impurity suppression, lower radiative losses and changes in recycling. The primary method used is termed "boronization" (see Ref. 1 and references therein). Boronization has been shown to effectively getter oxygen, thereby suppressing it in the plasma, which reduces core plasma radiation and sputtering of PFC surfaces. Boronizations in RFX-mod (reversed field pinch experiment, a/R = 0.46/2, I_{max}=2 MA) have been carried out by glow discharge plasmas using a gas mixture of He (90%) and B_2H_6 (10%). The thickness of the coating film after boronizations has been estimated by means of post mortem analyses of graphite samples. SIMS (Secondary Ions Mass Spectrometry) has been performed in order to obtain the depth profiles of the species present on the surface of the samples. During the boronization the gas exhaust has been monitored by a RGA (Residual Gas Analyzer) in order to assess the real time boron exhaust from RFX-mod vacuum vessel and hence assess the deposition of boron on the graphite first wall. Boronizations achieved at high glow discharge plasma power in RFX-mod showed a higher deposition rate of boron at the first wall and a lower boron exhaust combined with a decrease in the oxygen partial pressure. The deposition rate has been estimated from the boron depth retrieved from SIMS and normalized to the boronization duration. In Komatsu's papers [2,3,4] where the growth of boron films in PECVD (plasma-enhanced chemical vapor deposition) from (B_2H_6 + He) was studied, it was found that the reaction order makes a transition at a power threshold with an increase of the plasma power. Deciphering the physical chemistry between hydrogen, oxygen, boron and the carbon substrate is of great importance to improve the boronization favorable effect in RFX-mod. To this aim graphite samples that have been exposed during two boronizations achieved at a glow power range (100W-600W) have been studied by X-ray Photoelectron Spectroscopy and Auger Electron Spectroscopy).

2. RFX-mod experiment and the boronization plant

In fig.1 a schematic view of RFX-mod experiment and the boronization wall conditioning system is shown as well as the position of the exposed graphite samples. The boronization procedure in RFX-mod is documented in [5] and the SIMS measurements and considerations were published in [6]. The SIMS profile of boron gives the measure of the thickness of the boron film deposited during the wall treatment and the instant boron deposition efficiency is derived from to the measure of the boron partial pressure by the RGA. In order to investigate the chemical processes involved in boronizations achieved in RFX-mod, a ~250nm thick boron coating produced after a boronization achieved at room temperature and 450W glow discharge plasma power has been analyzed by XPS and AES spectroscopy combined with depth argon etching (Ar^+ ions sputtered at 1keV, 10^{-5}-10^{-4}mbar, and 10μA/cm^2). The XPS analysis was carried out with incident X-ray of Al Kα and the spectra of the O 1s, B 1s and C 1s were mainly measured.

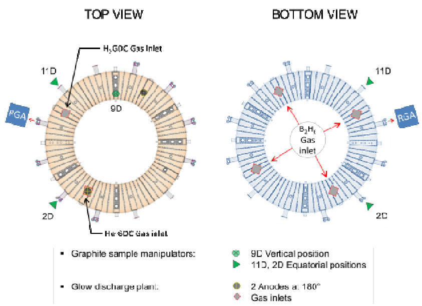

Fig. 1. Schematic view of RFX-mod torus, sample positions selected for the SIMS measurements, the GDC anodes, the RGA and the boronization gas inlet.

2. Experimental results

In fig.2.a the film thicknesses of the boron film estimated from SIMS depth profiles as a function of the duration of the boronizations at different toroidal angles are shown. By film “thickness” here it is understood the depth of the maximum of the SIMS amu=11 profile corresponding to the $^{11}B^+$ ions. Besides the boron film toroidal non-uniformity that has been documented elsewhere [7], it has been seen that the thickness of the boron film is not consistent with the boronization duration and different thicknesses have been observed for the same duration. The deposition rates of boron calculated by normalizing the estimated thickness obtained from SIMS

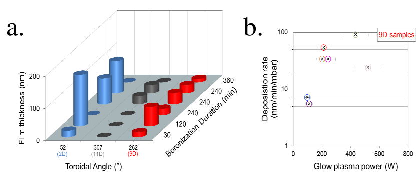

Fig. 2. Boron film thickness measured by SIMS on graphite samples exposed at different toroidal positions and boronizations of different durations (a), deposition rate of boron as a function of the glow plasma power (b).

measurements to the working pressure and the boronization duration are plotted in fig. 2.b, and the partial pressure of m/e=26 characteristic of the boron concentration in the gas exhaust during the boronization and the oxygen partial pressure have been plotted as a function of the instant power of the glow discharge, computed as the product of the measured current and voltage (IxV) of the anodes and shown in fig. 3. The XPS measurements were performed with a total energy resolution of 1eV allowing the determination of core level shifts with an accuracy of 0.1 eV. The C 1s and B 1s core lines are shown in fig. 4.

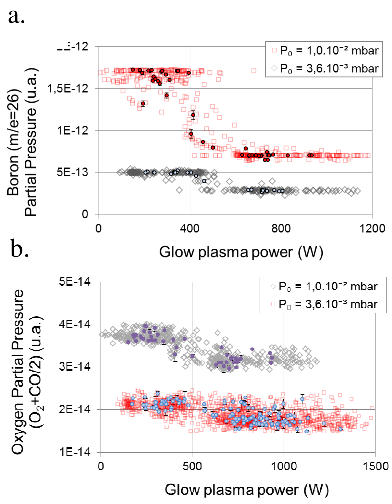

Fig. 3. Instant partial pressures of m/e=26 characteristic of the boron in-vessel concentration (a) and oxygen (b) as a function of the instant acquisition of the glow power (IxV.

3. Discussion of the results

According to SIMS measured profiles, boronizations in RFX-mod achieved at glow plasma power lower than 200W produced a film with low boron thickness while boronizations achieved at a glow power higher than 200W have shown a significantly higher (~10x) boron deposition rate. Similarly, the partial pressure of all the masses associated to the B_2H_6 molecule in the exhaust gas measured by the RGA show a dramatic decrease when the glow plasma power is increased over 400W while the working gas pressure is held constant. This can be interpreted as a significant increase in the boron deposition rate on the first wall at GDC power higher than 400W, in qualitative agreement with the results obtained by SIMS, though at higher power threshold. It is also clearly seen that the oxygen partial pressure decreases at higher glow power, suggesting the capability of boron to getter oxygen also during the deposition process. The depth XPS analysis of the boron coating for a boronization achieved at room temperature and high glow plasma power has shown a poor surface oxidation and a high in-depth concentration of oxygen. The reason of such feature is speculated to be a combined high glow plasma power and high oxygen residual pressure effect. The predominant chemical

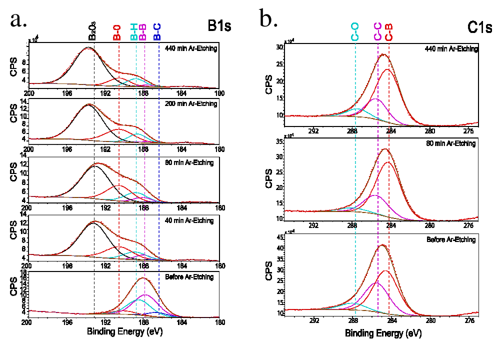

Fig. 4. XPS deconvolution of the B 1s (a) and C 1s (b) core lines.

bond in the deconvolution of boron line corresponds to the stoichiometric boron oxide B_2O_3, with much smaller contributions of B-B, B-H and B-C lines. The absence of B_2O_3 interaction on the surface is presently unknown and investigations of this feature will be the subject of future work. The chemistry of samples exposed to boronizations achieved at low glow power is still missing and is scheduled this year.

4. Summary and conclusions

The efficiency of boron wall-coating of the graphite first wall in RFX-mod for boronizations achieved at different glow plasma power and wall temperature has been performed. It was found that for boronizations achieved at higher glow plasma power >200W the deposition rate of boron measured by SIMS spectroscopy is significantly higher with respect to boronizations achieved at low power. It has also been observed a reduction in the instant partial pressures of boron and oxygen measured by a RGA in correspondence to GDC power higher than about 400W. The two methods gave the same qualitative result though at a different power threshold value. The discrepancy is still not clear and will be investigated in future analysis. XPS depth profile of the boron coating has revealed a considerable in-depth oxygen concentration combined with dominant stoichiometric boron oxide B2O3 complexes while very small boron oxide was observed on the surface. In the context of correlating these features to the boronization experimental conditions in terms of plasma power, wall temperature and oxygen residual pressure, further XPS depth analyses are needed.

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