Scanning Electron Microscopy characterisation of carbon deposited layers in Tore Supra

E. Delchambre\textsuperscript{1}, C. Brosset\textsuperscript{1}, R. Reichle\textsuperscript{1}, P. Devynck\textsuperscript{1}, R. Guirlet\textsuperscript{1}, E. Tspirone\textsuperscript{1}, P. Roubin\textsuperscript{2}, C. Arnas\textsuperscript{2}, W. Saikali\textsuperscript{3}, C. Dominici\textsuperscript{3}, A. Charaï\textsuperscript{3} and Tore Supra team

\textsuperscript{1}CEA/DSM/DRFC, CE Cadarache, F13108 Saint-Paul-lez-Durance, France
\textsuperscript{2}Laboratoire PIIM, Centre Saint-Jérôme, F-13397 Marseille cedex 20, \textsuperscript{3}CP2M, Centre Saint-Jérôme, F-13397 Marseille cedex 20 France

Introduction

For long discharges in Tore Supra, an infrared safety system has been installed to survey surface temperature of the target plates located below the Toroidal Pump Limiter (TPL). During the 2002 experimental campaign, the surface temperature of the leading edge of the TPL neutraliser was seen to increase for the same plasma conditions. This is attributed to the growth of a carbon layer at the surface of the neutraliser (~ 10 cm\(^2\)) located at 4–8 cm outside the Last Closed Flux Surface (LCFS), leading to a temperature increase of about 400°C between virgin and layered surfaces [1]. A difference of surface temperature of about 200 °C was also measured between the top and the bottom of the neutraliser leading edge. Fig.1 displays the temperature on the top of the neutraliser versus the cumulated plasma discharge duration for two neutralisers respectively with 0 and 180 µm thickness carbon layers during ohmic plasma discharges. According to temperature measurements on the top and on the bottom of the neutraliser, the layer thickness is thicker on the top of the neutraliser, ~ 4 cm apart from the LCMFS, than on the bottom. This has been confirmed by visual inspection. For temperature safety analysis, target plates have been cleaned and carbon layers were sampled for Scanning Electronic Microscopy (SEM) study. SEM analyses, initially aimed at measuring the thickness and finding a correlation between the surface morphology and infrared emission (see Ref. [1]), were completed with Energy Dispersive X-Ray Spectroscopy and Time-of-Flight Secondary Ions Mass Spectrometry (ToF-SIMS). Thus, the relative concentration of selected impurities and their profiles through the carbon-deposited layers and indication about the growth mechanism were obtained. Correlating particular events featuring in the
last campaign at specific times with particular depth-locations of some impurities made growth rate calculations possible.

Experimental procedures

Medium and Ultra High Resolution SEM micrographs were taken using a field emission gun XL30 SFEG Philips apparatus allowing also to perform EDS analyses. The electron beam energy was 10 kV. ToF-SIMS, achieved in order to measure impurity profiles through the carbon-deposited layers, used 25 keV Ga⁺ ion beam. In this paper, we discuss results for selected impurities only which correspond to positive ions. The displayed profiles are normalised to the sum of all the recorded profiles. As ionisation efficiency is depending on the sputtered chemical element and as we do not performed any calibration, Tof-SIMS provides qualitative results and only the trend of the profile variations have to be analyse.

Results

The TS samples have been scraped from the leading edge of some of the 12 neutralisers which are located under the TPL, first on August 2002 after 7850 s of cumulated plasma discharge duration (sample 1) and second on December 2002 after 19600 s corresponding to the whole campaign (sample 2). On sample 1, the thickness was ~ 180 µm and ovoid-shaped structures with 40 – 100 µm of diameter and 50 –150 µm of height were observed (Fig 2a). A well-defined stratification of a few micrometers and following the neutraliser surface is observed on the cross section of these structures (Fig.2b). The sample 2 was ~ 800 µm thick and is characterised by a columnar structure (plasma-facing side) growing on a lamellar carbon-deposited layer of ~ 80 µm (Fig.3). SEM micrographs give an overall fractal shape of the plasma-exposed surface. We have measured self-similarity on 3...
orders of magnitude, from 0.1 to 100 µm (Fig 4). By using the box-counting method, we have measured the fractal dimension of the surface which is obtained by calculating the slope of log(N(r)) versus log(1/r), N(r) being the density of the columns of radius r. From these measurements, the fractal dimension is 2.15 which can be considered as a measurement of the self similar granularity of the surface.

EDS gave the relative mass concentration of impurities trapped in the carbon-deposited layer and, in particular, has allowed to identify a boron-rich carbon sub-layer. Specific measurements were done on both sides of the thin boron layer observed at ~100 µm (Fig. 5). Before the boronisation, carbon and iron concentration are respectively 78% and 13%, whereas they are respectively 91 % and 3 % after boronisation. These results have been confirmed by ToF-SIMS analysis performed on the same sample (Fig. 6). In Fig 6, boronisation can be seen as a boron peak located at 140 µm from the CFC/carbon layer interface. The impurity profiles show a decrease after this peak whereas hydrogen and carbon signals increase (the carbon profile will not be discussed here).

Discussion

Similar structures were also observed in the divertor region of JT-60U [3]. Columnar structures were observed for all the re-deposition on the inner target, while lamellar structures were only observed at the shallower depths in the thicker layers, as lamellar/columnar-layered structures. Those lamellar structures are interpreted to be due to the higher deposition temperature. In our case, the growth mechanism of the carbon-deposited layers observed on the neutraliser surfaces is a fractal growth (Fig. 4b). The columns follow a direction of growth which seems to be parallel to the magnetic field lines underneath the TPL. The similarities of our micrographs with SEM images of dust particles obtained in a helium plasma with graphite electrodes [2], led us to think that the mechanism at the origin of these layers is carbon ion ballistic deposition. The deposited layer is thicker on top of the neutraliser leading edge than on the bottom which is consistent with turbulent transport of the carbon ions in the scrape off layer.
EDS and ToF-SIMS analyses show a decrease of the metallic impurities (Fe, Cr, Ni) above the boron-rich carbon layer (Fig. 5 and 6). This is in accordance with Fe plasma spectroscopic measurements (Fig. 7) which show a decreased of the FeXV brightness in the plasma after boronisation [4]. Besides, the increase of hydrogen signal above the boron peak (Fig. 6) is consistent with the experimental increase of tokamak carbon wall H storage induced by boronisation [5,6] and with a study of H/boron-doped graphite interaction [7]. Two boronisations were realised during the 2002 campaign: the first after 6720 s of plasma discharge duration and the second after 11340 s. Two boron peaks were respectively observed on sample 2 at ~140 µm and ~ 190 µm from the layer surface. According to these results, we calculate an average growth rate of about ~ 20 nm/s. Three single and broad Fe, Cr, Ni peaks observed at ~ 50 µm below the boron peak (Fig 6), are believed to correspond to the melting of a stainless steel electronic ripple plate during the long discharges performed before the first boronisation. For these long discharges, the cumulated duration is ~ 1800 s, that gives a growth rate of about 30 nm/s.

Conclusion

SEM micrographs have allowed to measure the deposited layer thickness and to study the specific fractal and stratified structure. Energy Dispersive X-ray Spectroscopy analysis has permitted to distinguish carbon layers corresponding to boronisation and then to deduce an average growth rate of about 20 nm/s. The growth rate is not constant and is likely to depend on plasma operation parameters. These analyses completed by ToF-SIMS analysis have shown a beneficial effect of the boronisation on metallic contamination of the plasma, confirming the in situ optical spectroscopic measurements. These analyses have also shown an increase of hydrogen storage in carbon layer due to boronisation. Although the measurements performed on deposited layer are very local, the results reflect the history of TS 2002 campaign.

[1] JC Vallet et al., this meeting, P-1.137.
[4] Remy Guirlet, this meeting, P-1.139.