Experimental study of the erosion of Ar/H2 plasma facing carbon surfaces: optical emission spectroscopy, mass spectrometry and spectroscopic ellipsometry measurements

Experimental study of the erosion of Ar/H_2 plasma-facing carbon surfaces: optical emission spectroscopy, mass spectrometry and spectroscopic ellipsometry measurements.

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Carbon materials could be used for divertor plates in the ITER fusion device. Numerous studies (experimental or modeling) have been undertaken to better understand the chemical processes involved in carbon erosion by hydrogen atoms or ions. These works have been performed for several kinds of carbon layers and different hydrogen (or deuterium) ion fluxes. Results of an experimental study on carbon-material erosion under hydrogen bombardment will be presented. Optical emission spectroscopy and mass spectrometry have been employed to determine the presence of excited and stable molecules that are formed under these conditions. *Ex situ* spectroscopic ellipsometry has been used to calculate the erosion rate. In order to determine this erosion rate during plasma exposure and with a better precision, preliminary results on *in situ* spectroscopic ellipsometry will be presented.

1. Introduction

Graphite and carbon materials are used as plasma facing materials in today's thermonuclear fusion experiments due to their superior thermo-mechanical properties. Major disadvantage of carbon materials is the high chemical reactivity of carbon with hydrogen ions which leads to large erosion rates. During the experiments discussed here, we have performed optical emission spectroscopy and mass spectrometry measurements in order to detect which carbon-containing species are formed when plasma produced carbon layers are exposed to an Ar/H_2 plasma expansion. In order to detect unambiguously the species coming from the sample, a D_2 flow in addition to the standard C_2H_2 flow was used during some depositions of the carbon layers. Deuterated species have been detected by means of mass spectrometry. Erosion yields have been calculated for different hydrogen ion fluxes by several authors. The results have been combined in one graph by Roth *et al.* [1] (figure 1). Most of these results have been obtained using optical emission spectroscopy on CH or CD or mass spectrometry. Some authors used weight loss measurements to determine the erosion yield with a better precision [2] [3]. In a paper of Westerhout et al.[4] results are reported with ion fluxes exceeding 10^{24} $m^{-2}s^{-1}$. They are in agreement with the results shown by Roth et al. in [1].

We have determined the change of thickness of our carbon samples due to plasma exposure by spectroscopic ellipsometry.

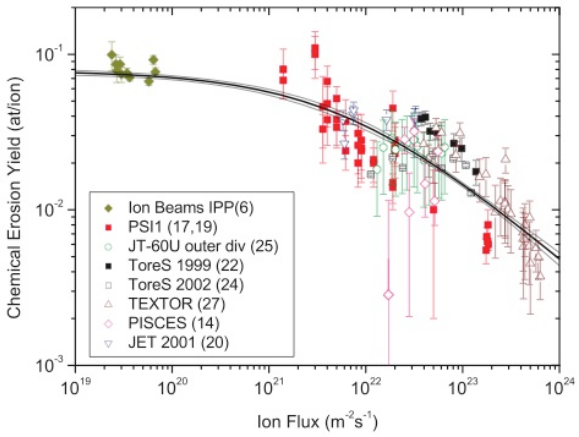

Figure 1: Chemical erosion yield as function of the H ion flux taken from Roth *et al* [1].

2. Experimental setup

We used an expanding thermal plasma to obtain a hydrogen flow to the carbon samples. The plasma source is a cascaded arc operating on mixtures of argon and hydrogen. The temperature of the samples could be controlled and was kept constant around 300 K. For the optical emission measurements an AvaSpec Avantes 2048-4-TD spectrometer was used. The mass spectroscopic measurements were performed with a Quadrupole Mass Spectrometer (Pfeiffer, QMS 200).

The diamond like carbon samples were deposited – in our group – at room temperature by means of an Ar plasma with a flow of 100 sccs. A flow of 15 sccs C_2H_2 was injected into the expanding thermal plasma. This flow was kept constant for all depositions. Also added to the mixture was either 5 or 10 sccs of D_2 or H_2. The deposition time for all

samples was 25 seconds. The thickness of our diamond like carbon samples is around 2000 nm. This thickness was measured before and during experiments by means of *ex situ* spectroscopic ellipsometry (SE) measurements. *Ex situ* SE was performed using a J.A. Woollam, Inc. M2000U visible and near-infrared SE. The measurements and data analysis were performed using WVASE32 ellipsometry analysis software. This kind of measurement allowed us to calculate the erosion rate of our samples.

3. Results

All the measurements have been performed in argon/hydrogen plasma with argon injected through the arc and hydrogen in the background. We can control the hydrogen ion fluxes on the sample by changing the relative flow of argon and hydrogen. We obtained a high flux of hydrogen ions on the sample for a low concentration of hydrogen (2%) and no hydrogen ions for a higher concentration (20%). The typical arc current was 50A, the values of gas flow were 50 sccs for argon and between 1 and 10 sccs for hydrogen.

3.1 Optical emission measurements

Under our experimental conditions, the only carbon-atom containing molecule that was detected via light emission was the CH radical at $\lambda = 431$ nm (figure 2).

No emission peaks were detected for CH_4, C_2 or C_2H_2. In order to detect these small hydro-carbons and other higher mass hydrocarbons, we have performed mass spectrometry.

3.2 Mass spectrometry measurements

By means of mass spectrometry carbon-containing species like C, CH_4, C_2H_x (x= 2; 4; 5) and C_3H_y (y= 6; 8) have been detected. We did not detect species C_4H_z. Our spectra are in good agreement with those obtained by Mech *et al.* [5]. When experiments were performed with samples containing deuterium, we have detected HD molecules. The molecules were only detected at pressures above 1 mbar. The signal strength decreases in time as the sample erodes under plasma exposure.

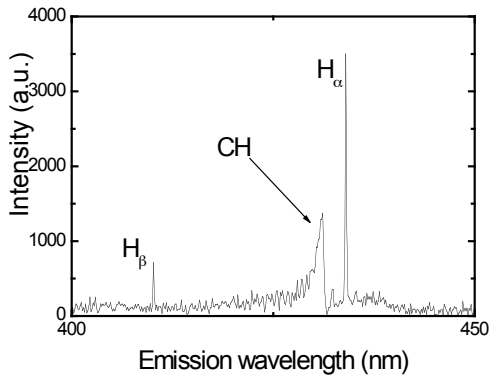

Figure 2: Part of the emission spectrum recorded during the argon/hydrogen plasma exposure of a thin plasma deposited carbon layer.

3.3 Spectroscopic ellipsometry measurements

From performed *ex situ* spectroscopic ellipsometry measurements on the samples before and after exposure to the plasma. Exposure times were ranging from 20 seconds to 3 minutes. These first measurements allowed us to calculate the erosion rate in pure argon plasma and argon-hydrogen mixtures (figure 3). The rate is 2.26 ± 0.21 nm/s.

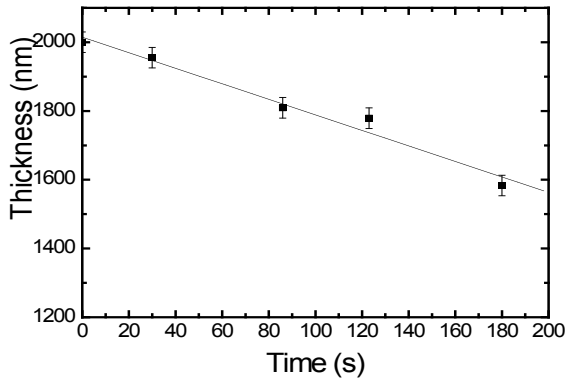

Figure 3: Thickness of the carbon sample versus exposure time in argon-hydrogen plasma.

These first results will be substantiated by *in situ* ellipsometry measurements, which should allow us to determine the change of thickness during exposure with a better precision.

4. Conclusion

We have characterized the kind of hydrocarbon species formed during Ar/H_2 plasma exposure of plasma deposited carbon layers by means of optical emission spectroscopy and mass spectrometry. The erosion rate during these experiments has been determined by spectroscopic ellipsometry measurements.

5. References

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