



Discharge Cleaning of Carbon Deposits

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ABSTRACT

Experimental results of discharge cleaning of carbon deposits are presented. Deposits were prepared by creating plasma in pure methane. The methane was cracked in RF discharge at the output power of 250 W. The resultant radicals were bonded to the wall of discharge vessel forming a thin film of hydrogenated black carbon with the thickness of about 200nm. The film was then cleaned in situ by oxygen plasma with the density of about $1 \times 10^{16} \text{ m}^{-3}$, electron temperature of 5 eV, neutral gas kinetic temperature of about 100°C and neutral atom density of $6 \times 10^{21} \text{ m}^{-3}$. The treatment time was 30 minutes. The efficiency of plasma cleaning was monitored by optical emission spectroscopy. As long as the wall was contaminated with carbon deposit, substantial emission of the CO molecules was detected. As the cleaning was in progress, the CO emission was decreasing and vanished after 30 minutes when the discharge vessel became free of any carbon. The results are explained by interaction of plasma radicals with carbon deposits.

1 INTRODUCTION

Plasmas are often generated in discharge vessels made of carbon. Although carbon in any form (especially carbon fiber composite) has excellent thermal, mechanical and chemical properties, it is not an ideal first-wall material for fusion reactors. Hydrogen plasma weakly interacts with the first wall material causing sputtering as well as chemical erosion. The resultant removed material is deposited at other parts of plasma reactor. The problem is severe for the case of ITER reactor, which will be characterized by a relatively strong erosion of carbon in the divertor. In order to assure proper operation of the device, it will be necessary to remove the carbon deposits occasionally. Probably the only feasible way of doing so is discharge cleaning using oxygen plasma.

2 EXPERIMENTAL

Experiments were performed in plasma system shown in Figure 1. The discharge vessel was a cylindrical tube with diameter of 40 mm and the length of 600 mm. The tube was made of a borosilicate glass (Schott 8250). At both ends it was joined to kovar rings, which were welded to standard KF 40 flanges. The glass-to-metal joint was bakeable up to 400°C. On one side, the tube was connected to the vacuum system. The system was pumped with a two-stage oil rotary pump with the pumping speed of 4.4 l s^{-1} and the base pressure of 0.1 Pa. All connections between the pump and the discharge vessel were made of components with the conductance of approximately two orders of magnitude higher than the pumping speed of the

pump, so that the effective pumping speed in the discharge vessel was nearly as high as that of the pump. The pressure in the system was measured with a Pirani gauge. Prior to the experiments the gauge was calibrated with a precise baratron. A recombinator for O atoms was placed in front of the gauge in order to prevent its degradation during plasma experiments. The discharge vessel was placed in a coil connected to a RF generator. The frequency of the generator was 27.12 MHz and the output power approximately 250 W. The other side of the discharge vessel was connected to a gas inlet system. Commercially available oxygen was leaked to the system through a leak valve. The discharge vessel was forced air-cooled.

Plasma parameters were measured with Langmuir and catalytic probes, as well as by optical emission spectroscopy. The electron density and temperature in plasma were about $1 \times 10^{16} \text{ m}^{-3}$ and 5 eV, respectively. The optical emission spectra were measured with the S2000- Ocean Optics spectrometer.

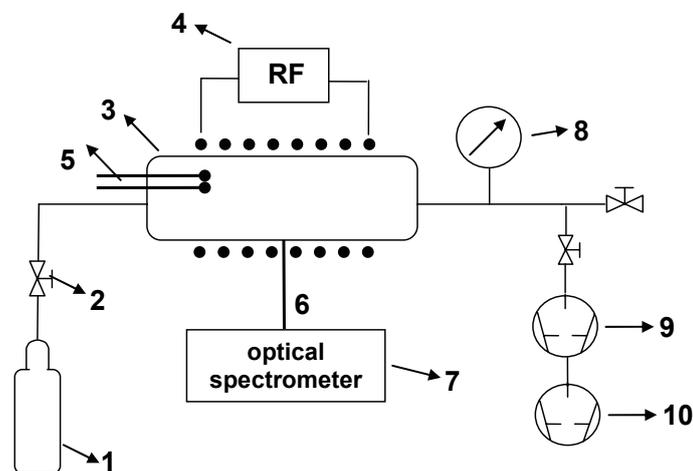


Figure 1: The experimental setup. 1 – gas flask, 2 – leak valve, 3 – discharge tube, 4 – RF generator with matching network, 5 – Langmuir and catalytic probes, 6 - optical fibre, 7 – optical spectrometer, 8 – vacuum gauge, 9 – roots pump, 10 – rotary pump.

The discharge vessel was covered by a thin film of amorphous hydrogenated carbon by creating plasma in pure methane at pressure of 50 Pa for 10 minutes. The thickness of the carbon film was about 200 nm. The scanning electron microscope image of the carbon film is shown in Figure 2. The gas methane flask was then replaced with the oxygen flask and plasma was created at the pressure of 70 Pa, where the O density, as determined by the catalytic probe was about $6 \times 10^{21} \text{ m}^{-3}$. The radiation from oxygen plasma during removal of carbon film was monitored by optical emission spectroscopy. Figure 3 represents some typical spectra, while Figure 4 and 5 represent the relative intensity of some peaks during cleaning procedure.

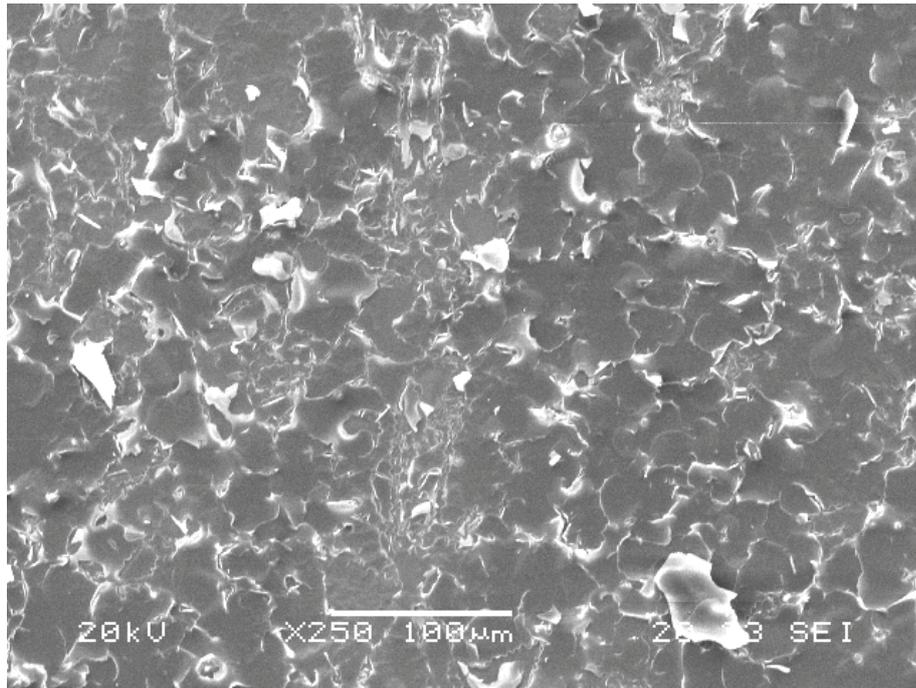


Figure 2. Scanning electron image of the carbon deposit.

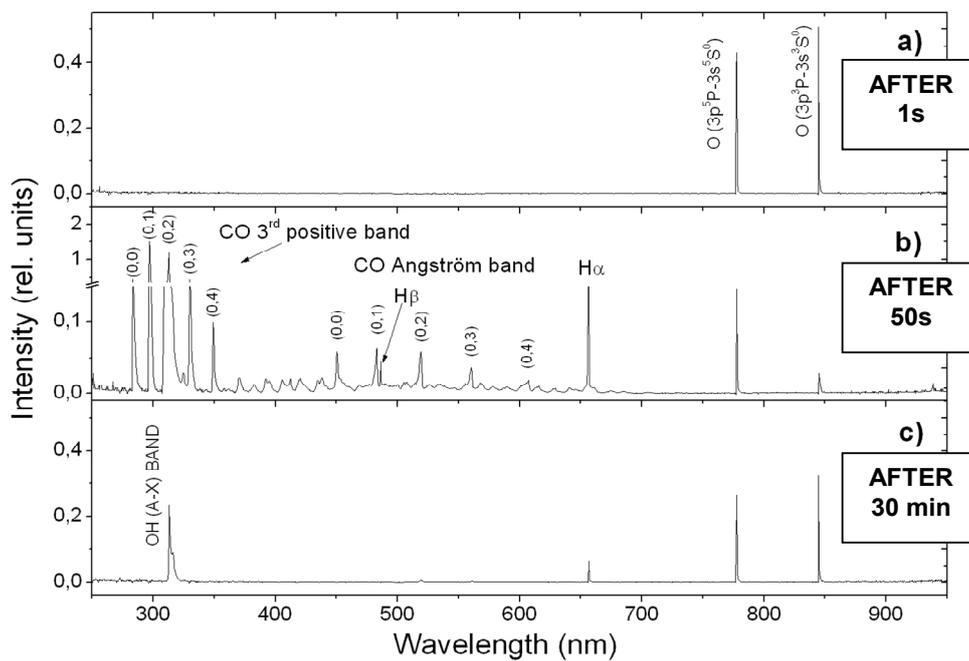


Figure 3. Some OES spectra during cleaning of carbon deposit with oxygen plasma.

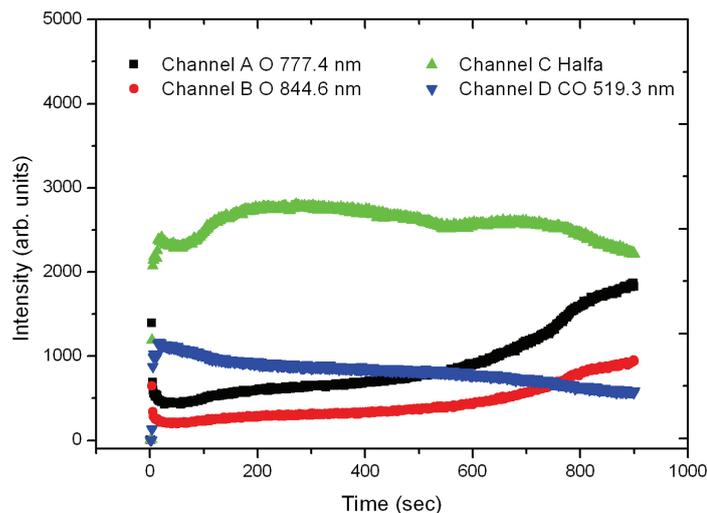


Figure 4. Intensity of different spectral features during first 15 minutes of oxygen plasma cleaning.

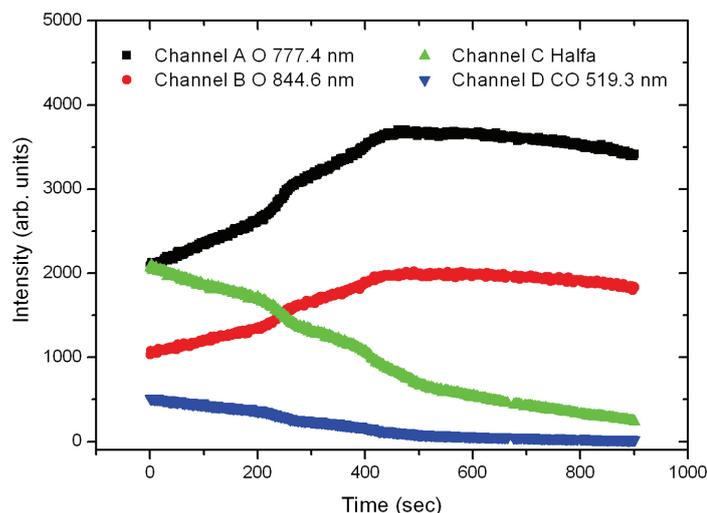


Figure 5. Intensity of different spectral features during next 15 minutes of oxygen plasma cleaning.

3 DISCUSSION

The evolution of different peaks as measured by optical emission spectroscopy during discharge cleaning of the carbon deposit on the surface of the discharge vessel is explained by slow etching of the carbon film with radicals present in oxygen plasma. As oxygen plasma is ignited, the atomic oxygen peaks at 777.4 and 844.6 nm are the highest. In the next few seconds, however, the O peaks are decreased on the expense of other peaks. Both H α and CO peaks became dominant indicating strong interaction of oxygen plasma with the deposit. The H α is particularly high indicating a rather high concentration of hydrogen in the deposit. After several seconds of plasma treatment, the CO and hydrogen peaks start decreasing slowly while the O peaks slowly recover. This effect cannot be explained by lower oxidation rate, since the temperature of the discharge wall has not yet reached the equilibrium value in few seconds. The slow decrease of the CO and H peaks is rather explained by cleaning of the

extremely thin deposits found near the end of the discharge vessel where oxygen is leaked. Namely, the thickness of the deposit is constant only in the central part of the discharge vessel. Close to the edges the thickness is much smaller. Actually, at the side where the gas is leaked, the discharge vessel is optically transparent even without cleaning. As the cleaning procedure is well on its way, more surface become free of the deposit and the CO and H peaks keep decreasing. After about half an hour of plasma cleaning, the entire vessel becomes visually clean and the CO peak vanishes. The hydrogen peak still persists what is explained by water adsorption on the walls. Namely, it is well known that the heat of adsorption of water on surfaces is much higher than that of CO. While the CO is rapidly pumped from the discharge system as soon as created, the water vapor still persists on the walls for a certain time.

4 CONCLUSIONS

A study on discharge cleaning of a thin film of amorphous hydrogenated carbon with oxygen plasma was presented. Optical emission spectroscopy proved to be a powerful method for monitoring the evolution of the cleaning process. As long as the discharge chamber was covered with the carbon deposit, strong emission originating from CO and hydrogen atom excited states was detected. As the cleaning process was well on its way, the CO and H emissions were slowly decreasing and the O emissions were increasing. The disappearance of the CO peak indicated the end of the cleaning procedure, while the H peak persisted for some more minutes, what was explained by adsorption of water molecules on the surfaces of the vacuum system.

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