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The investigation of structure, chemical composition, hydrogen isotope trapping and release processes in deposition layers on surfaces exposed to DIII-D divertor plasma.

O.I.Buzhinskij^a, I.V. Opimach^a, V.A.Barsuk^a, I.I.Arkhipov^b, W.P.West^c, D. Whyte^d
C.P.C.Wong^c, W.R.Wampler^e

a-TRINITI, Troitsk, Russia, 142092.

b-Institute of Physical Chemistry, Russian Academy of Science, Moscow, Russia.

c-General Atomics, San Diego, CA, USA.

d- University of California, San Diego, USA.

e- Sandia National Laboratories, Albuquerque, NM, USA.

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ABSTRACT

The exposure of ATG graphite sample to DIII-D divertor plasma was provided by the DiMES (Divertor Material Evaluation System) mechanism. The graphite sample arranged to receive the parallel heat flux on a small region of the surface was exposed to 600ms of outer strike point plasma. The sample was constructed to collect the eroded material directed downward into a trapping zone onto a Si disk collector. The average heat flux onto the graphite sample during the exposure was about 200W/cm², and the parallel heat flux was about 10 KW/cm². After the exposure the graphite sample and Si collector disk were analyzed using SEM, NRA, RBS, Auger spectroscopy, IR and Raman spectroscopy. The thermal desorption was studied also. The deposited coating on graphite sample is amorphous carbon layer. Just upstream of the high heat flux zone the redeposition layer has a globular structure. The deposition layer on Si disk is composed also from carbon but has a diamond-like structure. The areal density of C and D in the deposited layer on Si disk varied in poloidal and toroidal directions. The maximum D/C areal density ratio is about 0.23, maximum carbon density is about 3.8*10¹⁸cm⁻², maximum D areal density is about 3*10¹⁷cm⁻². The thermal desorption spectrum had a peak at 1250K.

1. INTRODUCTION.

Erosion during both normal operations accompanied with chemical and physical sputtering and abnormal discharges with disruptions, arcs and other events of plasma instabilities causes the formation of deposition layers on the surfaces of plasma facing elements of fusion reactors. These layers usually have different structure and physical-chemical properties comparing to original surfaces. The plasma interaction with such layers can define both many processes occurring in tokamak plasma and radiation safety issues due to trapping and retention of H isotopes in deposited matter. That's why it is so important to study the structure and other properties of deposition layers.

This study is devoted to the investigation of carbon films deposited during plasma exposure onto graphite DiMES sample and Si disk used as a collector of ablated material. The exposure was provided by the DiMES mechanism [1] that allows installation of samples in the DIII-D divertor plasma region during a few discharges or even only one shot. Our experiment included the exposure during one shot. The various plasma diagnostics - Langmuir probes, Thomson scattering, IR cameras and so on - provided the parameters and conditions of the exposure - electron temperature, electron density, average and peak heat fluxes and so on. After exposure the samples (carbon sample and Si disk) were analyzed using SEM, NRA, RBS, Auger spectroscopy, IR and Raman spectroscopy. The results of this study are compared with our previous investigations of deposited layers, obtained in DIII-D.

2. EXPERIMENTAL PROCEDURE.

2.1 Plasma exposure.

A DiMES graphite sample inserted into the divertor floor of DIII-D using the DiMES sample exchange system. It was located at a major radius of 148.5 cm and arranged to receive the parallel heat flux on a small region of the surface. The sample was constructed to collect the ablated material directed downwards into trapping zone and onto Si disk collector, because it is more reasonable to study carbon layers on not carbon substrate. The sputtered and eroded material directed upward into the incident plasma were ionized and redeposited onto neighbouring graphite regions of the sample surfaces. The sample was exposed to 600 ms of the outer strike point plasma. The divertor electron temperature T_e was about 60 eV, and the divertor electron density was 2.5*10²⁰ m⁻³. The average heat flux onto the graphite sample during the exposure was about 200W/cm², and the parallel heat flux was about 10 KW/cm².

2.2 Methods of the post-exposure sample analysis.

After removal the sample surface (graphite part and Si disc) was analyzed by Scanning Electron Microscopy (SEM) and X-ray crystallography (RSA) to study the morphology and phase composition. The optical methods (infrared and Raman spectroscopy) were used to measure the thickness and structural properties of the deposited layer on Si disc. Raman spectrum were studied by means of a homemade multichannel spectrometer with tripple monochromatization and the 514.5 nm excitation with power density about

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50 W/cm². The infrared spectrum were detected with BOMEM DA3.002 spectrometer in the 500-20000cm⁻¹ region. Rutherford Backscattering (RBS) was used to evaluate the changes in chemical composition of the surface after exposure and finally, the retention of deuterium in the coating was measured by counting proton from the d(He,³p) α nuclear reaction with 700 KeV He beam (NRA). This analysis give the areal density of deuterium within 1 mkm of the surface. The beam spot size for RBS and NRA was 1*1mm. The deuterium retention in deposited layer on Si collector was studied by Thermal Desorption Spectroscopy (TDS) analysis. The samples about 1*1*1mm³ were mechanically cut off from a central part of the deposited spot. The samples were placed in the UHV setup and ohmically heated from RT to 1650 K at about 6 K/s linear ramp rate. The temperature was measured with an optical pyrometer. The pressure during the TD measurement was 1x10⁻⁶Pa. Desorbed D₂ and HD molecules were analyzed by calibrated quadrupole mass spectrometer (QMS). The deuterium amount was calculated with account of D₂ as 2D atoms and HD as 1 D atom. The deposition layer as also studied by Auger electron spectroscopy. The depth profiling was carried out with ion etching by Ar ions with energy of 1 KeV during more then 12 hours. The hardness of the deposition layer on Si was measured using the NanoScan measurment system, based on the principles of scanning force microscopy.

3. EXPERIMENTAL RESULTS AND DISCUSSION.

3.1 The analysis of a carbon part of the sample.

The surface morphology in different regions of the sample is very different. It is seen the localized erosion spots. The erosion depth in these spots is rather large and varies from 40 mkm to 134 mkm (fig.1,a).

Fig.1 The damaged surface (a) and the globular formations (b) on the graphite sample.

While the outer strike point was located on DiMES sample, an increase of CI light was observed, that was caused by such a significant erosion of graphite sample surface, resulted in formation of damage spots. Just upstream of the high heat flux zone the redeposition layer has a globular structure (fig.1,b) and is very similar to the deposition layers that were observed on graphite tiles coated with boron carbide after a long term exposure (16 operational weeks) to DIII-D divertor plasma [2]. The deposition layer here is composed from pure amorphous carbon. However, it is not easy to study carbon layers on graphite surfaces, so the main attention was paid to investigation of the deposition layer formed on Si collector disc.

3.2 The study of the deposition layer formed on Si collector disc.

The deposited layer formed on Si collector contents only carbon and small additions of oxygen. The thickness of the layer varied from 400 to 3000Å, that is compared to the thickness of layers obtained after the longterm exposure of graphite tiles [2].

The IR, Raman spectroscopy study and hardness measurements showed that the layer has diamond like structure and hardness typical for rather hard surfaces, for example, like sapphire and is equal to 20 GPa (more detailed these results are discussed in [3]). Thus, Raman spectra has a broad band at 1515cm⁻¹ that is assigned to a presence of both sp² and sp³ bonds, IR spectra show a triplet around 2900cm⁻¹ which is assigned to the CH stretching modes.

Auger spectra of the film show also only carbon and some of oxygen. Very small additions of Cl and Ca disappeared after 2 hours of ion etching, so they are located in a very top surface and are connected with impurities from plasma or from air. carbon peak reached the maximum in an hour of etching and stayed in this level till the appearance of Si peak, that happened after 11 hours of etching. The shape of Auger carbon peak also proved the diamond-like structure of the film.

The areal density of carbon and deuterium in the deposited film varied in poloidal and toroidal directions (fig.2,a,b,c). The maximum areal density of D is about 3*10⁻¹⁷ cm⁻², maximum carbon density is

about $3.8 \cdot 10^{18} \text{cm}^{-2}$. The maximum D/C areal density ratio is about 0.23, consistent with a previous experiment of study deposition layers formed on boron carbon coated graphite sample [4]. Thus we can suppose that the main mechanisms of deposition layer formation are pretty similar for different plasma operations.

Fig. 2. D and C areal density in the deposited layer on Si collector (a, b) and the D/C areal density ratio (c).

The TD spectrum of deuterium from the sample (fig.3) was obtained in the temperature range of 830-1650 K and characterized by a main broad maximum at 1250 K. In general, the obtained spectra has the similarity with the TD spectra of thin films formed during the carbonization of TEXTOR [5]. It is important to note, that the films obtained in DIII-D did not change their gold-like colour during the heating in vacuum till 1650K, that points on the fact that diamond-like film structure is pretty stable and did not transform into graphite structure (in this case the film would be have black colour).

Fig. 3. Thermal desorption of D_2 and HD molecules from a central area of Si disk collector. Heating rate was about 6 K/s.

Thus, discussing results of our experiments, we can make some estimations.

We have got a very high D retention in the deposited layer only in 600 ms of the exposure, and after 1000 sec exposure (under linear extrapolation), the areal concentration will be about $5 \cdot 10^{20} \text{D}/\text{cm}^2$, that is a very significant amount, so, H isotope uptake is a real problem in tokamaks.

Diamond-like structure of deposited layer has high thermal desorption temperature, that indicate that in -situ cleanup of this type of coating may be very difficult. However, the nature of this coating could be change depending on the type of substrate material and the surface temperature at the time of deposition.

4.CONCLUSION.

The exposure of ATG graphite sample to DIII-D divertor plasma was provided by the DiMES (Divertor Material Evaluation System) mechanism. The graphite sample arranged to receive the parallel heat flux on a small region of the surface was exposed to 600 ms of outer strike point plasma. The sample was constructed to collect the eroded material directed downward into a trapping zone onto a Si disk collector. The average heat flux onto the graphite sample during the exposure was about 200 W/cm², and the parallel heat flux was about 10 KW/cm². The graphite part of the sample was seriously eroded only after one shot exposure and the damage depth was till 134 mkm. The deposited coating on graphite sample is amorphous carbon layer. Just upstream of the high heat flux zone the redeposition layer has a globular structure. The deposition layer in Si disk is composed also from carbon but has a diamond-like structure. The thickness of this layer is pretty big- about 3000 A, that demonstrates very high sputtering of carbon. The hardness of the film is rather high, similar to such hard surfaces like sapphire. The areal density of C and D in the deposited layer on Si disk varied in poloidal and toroidal directions. The maximum D/C areal density ratio is about 0.23, maximum carbon density is about $3.8 \cdot 10^{16} \text{cm}^{-2}$, maximum D areal density is about $3 \cdot 10^{17} \text{cm}^{-2}$. The thermal desorption spectrum had a peak at 1250K and it was shown that the film did not change its the colour under the heating till 1650K.

It is reasonable to continue the study of such layers obtained in tokamak conditions, perhaps increasing the number of exposure discharges and the temperature of substrate during the deposition and using protection coatings like boron carbide, W and diamond-like layers on carbon substrates.

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