ELSEVIER

Journal of Nuclear Materials 266–269 (1999) 793–797

journal of nuclear materials

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 142092, Russian Federation*
[b] *Institute of Physical Chemistry, Russian Academy of Science, Moscow, Russian Federation*
[c] *General Atomics, P.O. Box 85608, San Diego CA 92186-5608, USA*
[d] *University of California, San Diego, CA, USA*
[e] *Sandia National Laboratories, Albuquerque, NM, USA*

Abstract

An ATJ graphite sample, arranged to receive parallel heat flux on a small region of the surface, was exposed to DIII-D divertor plasma using the DiMES (Divertor Material Evaluation System) mechanism. The sample was exposed to 600 ms of outer strike point plasma in a single discharge. Actual divertor tiles are subjected to such high heat flux due to misalignments, subjecting leading edges to the parallel flow of plasma, or during disruptions. The sample was constructed to collect the eroded material directed downward into a trapping zone onto a Si disk collector. The surface heat flux onto the divertor floor at the location of the sample during the exposure was 200 W/cm^2. Taking the local field pitch into account, the parallel heat flux was about 5 kW/cm^2. After exposure, the graphite sample and Si collector disk were analyzed using SEM, NRA, RBS, Auger spectroscopy, IR and Raman spectroscopy. Thermal desorption was studied also. The deposited coating on the graphite sample is an amorphous carbon layer. Just upstream of the high heat flux zone, the redeposition layer has a globular structure. The deposition layer on the 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 3.8×10^{18} cm^{-2}, maximum D areal density is about 3×10^{17} cm^{-2}. The thermal desorption spectrum had a peak at 1250 K.

Keywords: DiMES; Erosion/redeposition; Graphite; DIII-D

1. Introduction

Divertor tiles are subjected to very high heat flux conditions in a tokamak during both normal and transient events. Misalignments between neighboring tiles will exist in the divertor, and these misalignments will be subjected to the parallel heat flux, which is typically 50–100 times larger than the flux to the tile surface during normal operation. High heat flux onto the normal tile surfaces can also occur in transient events such as ELMs or disruptions. Here we describe an experiment using the DiMES apparatus [1] on DIII-D which is designed to subject a leading edge of a graphite sample to the divertor parallel heat flux (about 50 MW/m^2) in order to examine the erosion and redeposited layers produced under these extreme conditions.

Erosion during both normal operations due chemical and physical sputtering and abnormal events such as disruptions, arcs, and other types of plasma instabilities causes the formation of deposition layers on the surfaces

* Corresponding author. Tel.: +1 619 455 2863; fax: +1 619 455 5608; e-mail: west@gav.gat.com.

0022-3115/99/$ – see front matter
PII: S0022-3115(98)00604-7

of plasma-facing elements of fusion reactors [2]. These layers usually have different structure and physical-chemical properties compared to the original surfaces. The plasma interaction with such layers results in neutral recycling and impurity sources that have a direct influence on the tokamak plasma. In addition, radiation safety issues arise due to trapping and retention of H isotopes in the deposited layers. Detailed data on the formation and physical properties of films formed in today's tokamaks are needed for the assessment of the first wall erosion and tritium inventory and safety issues in future long pulse ignited devices.

2. Experimental procedure

2.1. Plasma exposure

A graphite sample was inserted into the divertor floor of DIII-D using the DiMES sample exchange system [1]. It was located at a major radius of 148.5 cm and arranged to receive parallel heat flux on a small leading edge region of surface area 0.7×6 mm^2. The sample was constructed to collect the ablated material directed downwards into a trapping zone and onto a Si disk collector located about 1 cm below the interaction region. The Si collector disk was protected from direct interaction with the plasma. The sputtered and eroded material directed upward into the incident plasma was ionized and redeposited onto neighboring graphite regions of the sample surfaces. The sample was exposed to 600 ms of outer strike point plasma. The divertor electron temperature, T_e, was about 60 eV, and the divertor electron density was 2.5×10^{20} m^{-3}, measured using both Langmuir probes and Thomson scattering. The average heat flux onto the graphite sample during the exposure, measured using an infrared camera, was about 200 W/cm^2, and the parallel heat flux was about 5 kW/cm^2.

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. Optical methods (infrared and Raman spectroscopy) were used to measure the thickness and structural properties of the deposited layer on the Si disk. Raman spectra were studied by means of a multichannel spectrometer with triple monochromatization and 514.5 nm excitation with a power density about 50 W/cm^2. Infrared spectra were detected with BOMEM DA3.002 spectrometer in the 500–20 000 cm^{-1} region. Rutherford backscattering (RBS) and Auger electron spectroscopy (AES) were used to evaluate the changes in chemical composition of the surface after exposure. The retention of deuterium in the coating was measured by counting protons from the d(^{3}He, p) α nuclear reaction with 700 keV ^{3}He beam (NRA). This analysis gives the areal density of deuterium within 1 μm of the surface. The beam spot size for RBS and NRA was 1×1 mm^2. The deuterium retention in the deposited layer on Si collector was studied by Thermal desorption spectroscopy (TDS). The samples, about $1 \times 1 \times 1$ mm^3, were mechanically cut from a central part of the deposited spot. The samples were placed in the UHV setup and ohmically heated from 300 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 1×10^{-6} Pa. Desorbed D_2 and HD molecules were analyzed by a calibrated quadrupole mass spectrometer (QMS). The deuterium amount was calculated with account of D_2 as 2D atoms and HD as 1 D atom. The deposition layer was also studied by Auger electron spectroscopy. Depth profiling was carried out with ion etching by Ar^+ with an energy of 1 keV during more than 12 h. The hardness of the deposition layer on Si was measured using the NanoScan measurement system based on the principles of scanning force microscopy.

3. Experimental results and discussion

3.1. Analysis of a carbon part of the sample

The surface morphology in different regions of the sample is very different. A dominant feature is deep localized pits (Fig. 1(a)). The erosion depth in these pits is rather large, from 40 to 134 μm and readily measured using microscopy.

While the outer strike point was located on the DiMES sample, a dramatic increase of CI light was observed using a spectrometer viewing directly onto the sample. Such an increase is indicative of the significant erosion of the graphite sample surface, resulting in the 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 [3]. This globular structure was also previously reported on redeposition layers observed on a fast plunging probe in the TEXTOR tokamak [4]. The deposition layer here is composed from pure amorphous carbon, as determined by RSA. However, it is not easy to study carbon layers on graphite surfaces. To date our focus has been the investigation of the deposition layer formed on the Si collector disk.

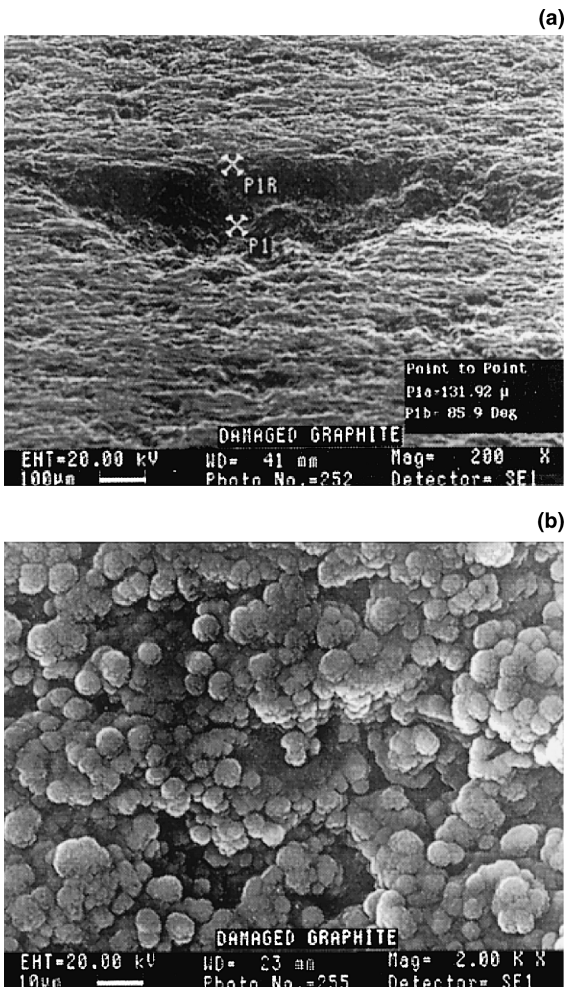

Fig. 1. The damaged surface (a) and globular formations (b) on the graphite sample.

3.2. Analysis of the deposition layer formed on Si collector disc

AES of the film shows also only carbon and some oxygen. Very small additions of Cl and Ca disappeared after 2 h of ion etching, so they are located in a very top surface and are most likely connected with impurities from handling after exposure. The carbon peak reached a maximum after 1 h of etching and stayed at this level until the appearance of the Si peak after 11 h of etching. The thickness of the layer varied from 400 to 3000 Å, comparable to the thickness of layers obtained on plasma facing surfaces after long-term exposure (approximately 2000 s of high power plasma exposure) of graphite tiles [3].

The Raman spectrum has a broad band at 1515 cm^{-1} which is assigned to a presence of both sp^2 and sp^3 bonds. IR spectra show a triplet around 2900 cm^{-1} which is assigned to the CH stretching modes. Thus, the IR and Raman spectroscopy studies show that the layer has a diamond-like carbon (DLC) structure. The hardness is measured to be quite high, 20 GPa, typical for such DLC films (more detailed results are discussed in Ref. [5]). The shape of the Auger carbon peak also indicates a diamond-like structure.

The areal density of carbon and deuterium in the deposited film varied in the poloidal and toroidal directions (Fig. 2(a)–(c)). The toroidal direction corresponds to distance from the exposed graphite edge, with the edge located at about 20 mm on the abscissa and the expected trapping zone extending from 0 to 20 mm. The maximum areal density of D is about 3×10^{17} cm^{-2}, maximum carbon density is about 3.8×10^{18} cm^{-2}. The maximum D/C areal density ratio is about 0.23 consistent with a previous experimental study of deposition layers formed on boron carbide coated graphite samples [6].

The thermal desorption (TD) spectrum of deuterium from the sample was obtained in the temperature range of 300–1650 K and characterized by a broad peak centered at 1250 K (Fig. 3). In general, the spectra are similar to the TD spectra of thin films formed during the

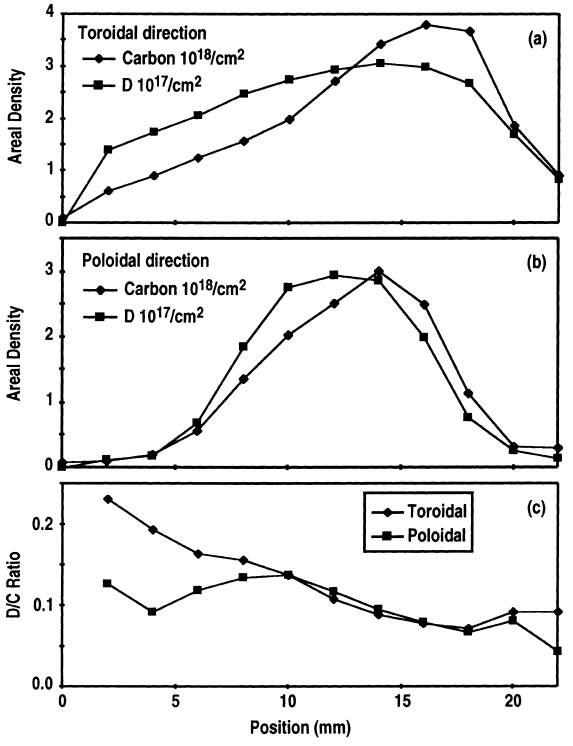

Fig. 2. D and C areal density in the deposited layer on the Si collector (a), (b) and the D/C areal density ratio (c).

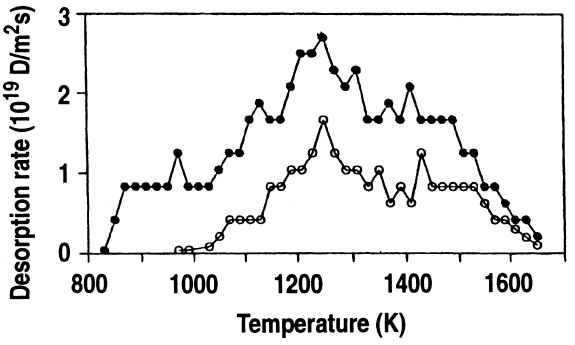

Fig. 3. Thermal desorption of D_2 (solid circles) and HD (open circles) molecules from a central area of Si disk collector. Heating rate was about 6 K/s.

carbonization of TEXTOR [7]. The films obtained in DIII-D had a gold-like color indicating an optically transparent film typical of DLC [8]. During heating in vacuum, the visual appearance of the film did not change up to the maximum temperature, 1650 K. These TD results indicate that the diamond-like film structure is stable and did not transform into a graphitic structure (in this case the film would have a black color). Integration of the TD desorption rate shows rough agreement with the measured D areal density using NRA.

4. Discussion

Estimates of the net tritium retention in a tokamak reactor due to the leading edge effect would require a detailed examination of the divertor design and engineering tolerances. In this experiment, the rate of D retention per unit area of exposed edge was very high (about 1.6×10^{19} cm^{-2} s^{-1}). A rough estimate for DIII-D indicates that at least half of the total D retention in the divertor is due to the erosion at leading edges and redeposition deep in the tile gaps [9]. It is worthwhile to note that in addition to the normal tile gaps, there are multiple orientation misalignments that can result in significant plasma erosion of leading edges. Thus, using the "roof top" strike plate design does not entirely eliminate the leading edge problem.

The diamond-like structure of the deposition layer is known to provide a hard durable film. Such films have been used for many technological applications (e.g., Ref. [8]). The TD spectra indicate that the hydrogenic atoms are tightly bound in this film. This leads to the speculation that in situ cleanup of this type of coating may be very difficult. However, the nature of this coating could change depending on the type of substrate material and the surface temperature at the time of deposition. Further studies are warranted if graphite remains a candidate reactor divertor material.

5. Conclusion

An in situ experiment to directly measure the effect of very high heat and particle fluxes on a leading edge of a graphite sample has been carried out using the DiMES sample insertion mechanism in the DIII-D divertor. The sample was constructed to collect the eroded material directed downward into a trapping zone onto a Si disk collector. The graphite sample, arranged to receive the parallel heat flux (5 kW/cm^2) on a small region of the surface (0.7×6 mm), was exposed to 600 ms of outer strike point plasma. Such high flux conditions could be expected at tile gaps and due to tile misalignments. During ELMs and disruptions, such high heat fluxes are expected even on the normal surfaces. The graphite part of the sample was seriously eroded after one shot exposure. Localized erosion (pits) depth was as much as 134 μm.

Redeposition layers were observed on the low heat flux plasma facing surfaces of the graphite sample as well as on the Si collector. 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 the Si disk is composed also from carbon and deuterium but has a diamond-like structure. The thickness of this layer is about 3000 Å, indicating a very high sputtering rate of carbon from the exposed edge. The hardness of the film is rather high – comparable to 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 3.8×10^{18} cm^{-2}, maximum D areal density is about 3×10^{17} cm^{-2}. The thermal desorption spectrum had a peak at 1250 K, and it was shown that the film did not change its color under heating up to 1650 K. The measurements indicate that a deposition rate of tritium in a reactor per unit area of exposed leading edge would be quite high, 1.6×10^{19} cm^{-2} s^{-1}. These results indicate that estimates of retained T in a reactor due to this effect should be carried out if graphite is to be used as a plasma facing material. Such estimates require detailed studies of the divertor design and the engineering tolerances and are beyond the scope of this paper. Estimates for DIII-D indicate that about half of the retained deuterium is in the tile gaps and results from the leading edge effects [9].

References

[1] C.P.C. Wong et al., J. Nucl. Mater. 196–198 (1992) 871.
[2] G. Federici et al., Fusion Eng. Design 28 (1995) 136.
[3] O.I. Buzhinskij et al., J. Nucl. Mater. 233–237 (1996) 787.
[4] T. Scholz et al., J. Nucl. Mater. 241–243 (1997) 848.

[5] V.N. Denisov et al., Phys. Lett. A 239 (1998) 328.
[6] I.V. Opimach et al., 17th IEEE/NPS Symp. on Fusion Engineering, 6–11 October 1997, San Diego, CA, poster 2.17.
[7] J. Winter et al., Nucl. Instr. Meth. B 23 (1987) 538.
[8] V.G. Radchenco, S.M. Pimenov, V.G. Pereverzev et al., Diamond-based composites and related materials, NATO ASI Series 3, High Technol. 38 (1997) 39.
[9] D.G. Whyte, R. Bastasz, J.N. Brooks et al., these Proceedings.