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Erosion of carbon-based materials in a steady-state deuterium plasma

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Abstract

Experimental results on plasma induced erosion of graphites and CFC materials are presented. Experiments were performed in the LENTA linear simulator allowing the ion fluence corresponding to the ITER divertor conditions for one operation cycle ($\sim 10^{22}$ D^+/cm^2) to be achieved in a few hours. RG–Ti–91 titanium doped and RG–Ti–91 with 0.1% boron addition graphites (Russia), American POCO-AXF-5Q and European composites SEP N112-A and DUNLOP-exp B were taken for the study to compare their erosion properties under similar conditions. Erosion yield has been evaluated using weight loss measurements. Plasma contamination is monitored by optical spectroscopy and gas composition is analyzed by mass-spectrometric method during exposure. Both methods have shown intensive deuterocarbon formation for the ITER relevant working temperatures of the samples, 750°C and 1150°C.

1. Introduction

The selection of materials for plasma facing components of a fusion reactor is not an easy task particularly in the work on the ITER project [1]. The first wall and the divertor are subjected to extremely high particle and heat fluxes. On the basis of recent experimental data and calculations the following quantities are taken for characteristics of plasma attack on the divertor target in the ITER: ion flux density more than 10^{19} ion/cm^2 s, ion energy – down to 100 eV or less, heat load – about 5 MW/m^2 in normal operation mode, with short excursions to higher levels [1]. High erosion resistance combined with high thermomechanical performance is required for materials under these stringent conditions. The basic ITER design concept adopts a mix of materials for plasma facing components (beryllium, tungsten and carbon-based materials-CFCs) with CFCs for high-heat-flux areas of the divertor target, taking into account their advantages in thermal conductivity and thermomechanical properties. As to erosion behaviour of carbon-based materials and prediction of carbon armour lifetime, real plasma experiments are needed with parameters relevant to reactor divertor conditions. Linear devices with steady-state plasmas offer good possibilities for investigations of plasma induced erosion and for comparison of materials under relevant conditions which can be well defined and controlled. A series of experiments have been made in the LENTA linear device [2] to study the erosion of carbon materials under deuterium plasma impact in steady-state conditions.

2. Experimental

Continuous plasma in the LENTA facility is generated in a discharge zone driven by a high density electron beam passing through a gas under influence of crossed electric and magnetic fields. The homogeneous linear magnetic field induction is 0.1–0.2 T. An electron beam ($d = 1$ cm) is injected along the axis of cylindrical chamber ($D = 16$ cm) and a plasma is generated and sustained in the whole space of the chamber when a radial potential ($\sim$100 V) is applied between the beam and the chamber wall. Different diagnostic techniques are used for measurement of plasma parameters and

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composition control: e.g. Langmuir probes, optical spectrum analysis, mass-spectrometric gas analysis, with calorimeters and pyrometers for temperature and heat load measurements. More information about LENTA facility is available in Refs. [2,3].

General deuterium plasma performances are: power in plasma – up to 15 kW, electron density, $n_e = 10^{11}$–10^{13} cm^{-3}, electron temperature, T_e = 10–30 eV, heat flux on the wall, 1–500 W/cm^2, ion bombarding flux, 10^{17} – 10^{19} ion/cm^2 s.

Materials were investigated using the following procedure. Samples to be studied were put into the plasma column mounted on a holder moving radially and fastened to the end plate to limit the discharge in axial direction. The ion bombarding energy was determined by a bias potential that was applied to samples (from 0 to 1000 V). The holder's design allowed this to be done but in addition it was cooled by water to remove the excess induced plasma heating above that needed to maintain the required temperature. The exposure time depended on the experimental task and corresponded generally to simulation of the ITER normal operation cycle in which ion fluence accumulated at the divertor target is about 10^{22} ion/cm^2.

Plasma parameters were maintained at a given level in each experiment. Optical radiation from plasma column in the vicinity of irradiated samples ($L \sim 10$ cm) was monitored while gas composition in this region was analyzed with a mass spectrometer.

Weight loss of the samples was measured after irradiation in the plasma. The surface microstructure was analyzed by SEM after which EPMA, SIMS and TDS methods were used to study the various surface composition effects resulting from deuterium implantation.

3. Experimental results

3.1. Erosion evaluation

The following carbon-based materials of Russian and foreign origin were taken for the study: RG–Ti-91 (recrystallized graphite containing 7.5% mass. Ti), RG–Ti–B(I) (RG–Ti-91 with 0.1% mass. B, boron replacing carbon in the lattice), RG–Ti–B(II) (RG–Ti-91 with 0.1% mass. B, amorphous boron introduced mechanically in graphite), isotropic graphite POCO AXF-5Q (USA), carbon fiber composites Dunlop, SEP N112-A (EU). A comparative study of these dense graphites containing titanium and boron as dopants for improvement of erosion properties, pure POCO porous graphite and CFCs was made in deuterium plasma.

Experiments were carried out at sample temperatures of carbon samples corresponding to characteristic values of divertor operation range, i.e. from 750°C to 1200°C.

Table 1 shows the weight losses of the various fabricated carbon samples (1 cm in diameter) after plasma bombardment at 750°C. In this case, the exposure time was 1.11×10^4 s and the particle flux was $j = 0.34 \times 10^{18}$ ion/cm^2 s. Samples were exposed at floating potential giving an average ion bombarding energy of about 50 eV. The total ion fluence in this experimental run reached 0.4×10^{22} ion/cm^2. Our evaluation of the erosion yield for these materials is given in Fig. 1 which shows that the erosion yield of porous graphite POCO and of composites Dunlop and SEP are considerably higher (by a factor of 2–2.5) than those of dense graphites RG–Ti and RG–Ti–B.

The erosion yield of POCO and RG–Ti graphites for different temperatures (770°C and 1150°C) is given in Fig. 2. These data were obtained under similar conditions as the results described in the above paragraph. The most important difference concerns the ion bombardment energy which, in this case, was in the range 200–220 eV. The total ion fluence was 1.1×10^{22} ion/cm^2 while the ion flux density was 3.3×10^{17} ion/cm^2 s for the sample temperature of 770°C and 6×10^{17} ion/cm^2 s for 1150°C. As in Fig. 1, a larger erosion yield of porous POCO graphite is seen relative to the RG–Ti and RG–Ti–B graphites at temperatures near 750°C. Comparing the data of Figs. 1 and 2 we may conclude that at 750–770°C the dependence of erosion yield on ion energy was not very strong when the energy was decreased from 200 to 50 eV. We see also that erosion yields of the dense graphites do not have a strong dependence on temperature value.

3.2. Analysis of gaseous products

Analysis of gaseous products in the plasma in the vicinity of the exposed samples has been carried out to investigate the chemical erosion processes. This analysis has been conducted periodically by means of a high resolution ($R > 500$) mass spectrometer (MI 1201), its beam source being connected to the vacuum chamber of the LENTA device.

Two typical mass spectra are shown in Fig. 3. The first of them (dashed lines) demonstrates the composition of the feed gas being injected into the chamber

Table 1
Weight loss of carbon samples. Irradiation conditions: exposure time 3 h, ion energy 50 eV, ion fluence 0.4×10^{22} ion/cm^2, ion flux 0.34×10^{18} ion/cm^2 s, sample temperature 750°C

Material	Weight loss after bombardment, ΔM (g)
RG–Ti–B (I)	0.0054
RG–Ti–B (II)	0.0049
RG–Ti-91	0.0052
Dunlop	0.0121
SEP N112-A	0.0194
POCO AXF-5Q	0.0178

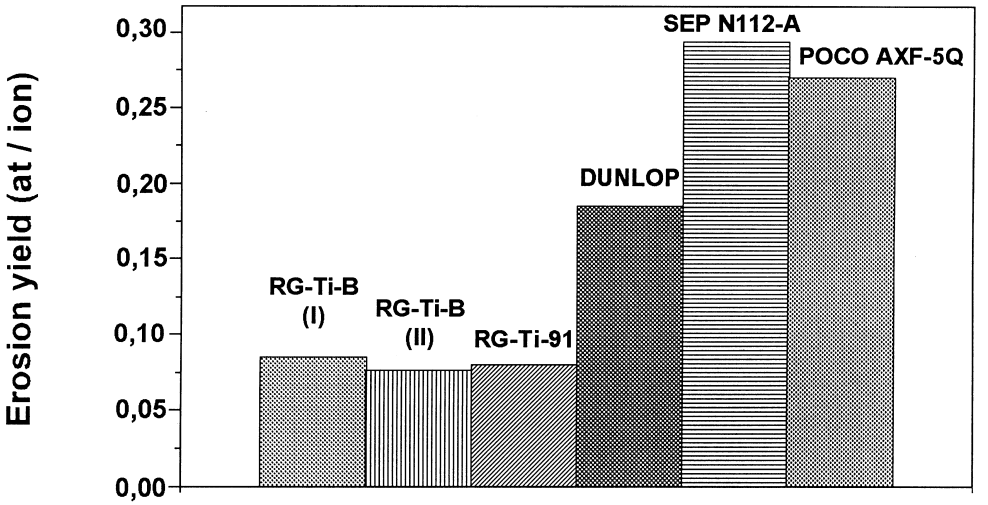

Fig. 1. Erosion of carbon-based materials bombarded by deuterium plasma. Exposure time 3 h, ion energy 50 eV, ion fluence 0.4×10^{22} ion/cm^2, ion flux 0.34×10^{18} ion/cm^2 s, sample temperature 750°C.

(without discharge) and the second one gives the mass-composition of the gas in the discharge during exposure of the samples at 770°C. The essential difference between these two spectra is seen in the high rise of the peak with mass to charge ratio $M/e = 20$ in the second case relating to the ions of deuterized methane CD_4^+. This peak is complex and the high resolution of our mass spectrometer makes possible to determine the contribution of each ion species into its intensity. The contribution of D_2O^+ ion is negligible compared with CD_4^+; then the CD_4^+ intensity in the CD_4^+–ND_3^+ doublet (see Fig. 3) is several times higher than ND_3^+ one. The presence of

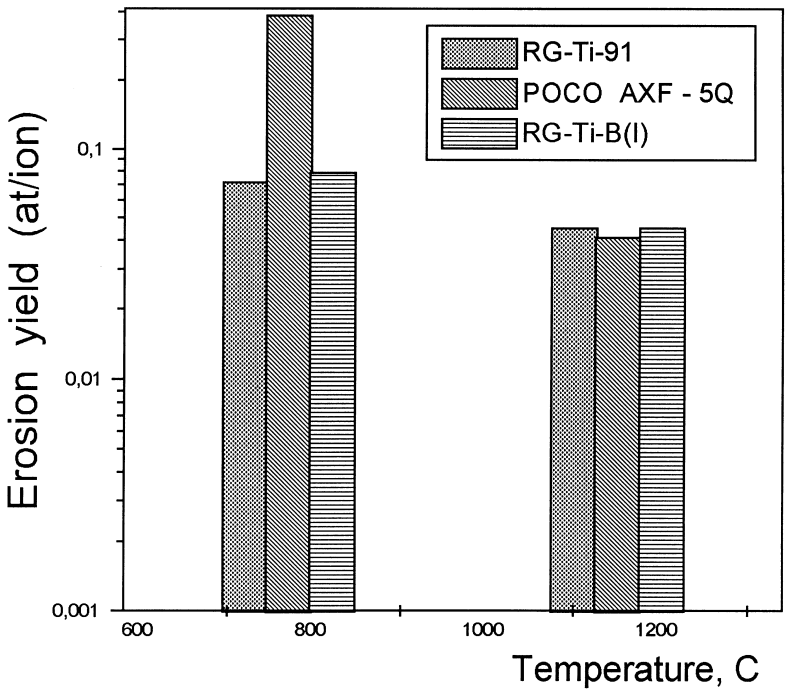

Fig. 2. Comparison of plasma induced erosion for two temperatures of carbon samples 770°C and 1150°C. Ion fluence 1.0×10^{22} ion/cm^2, ion energy 200–220 eV, ion flux $(0.3–0.6) \times 10^{17}$ ion/cm^2 s.

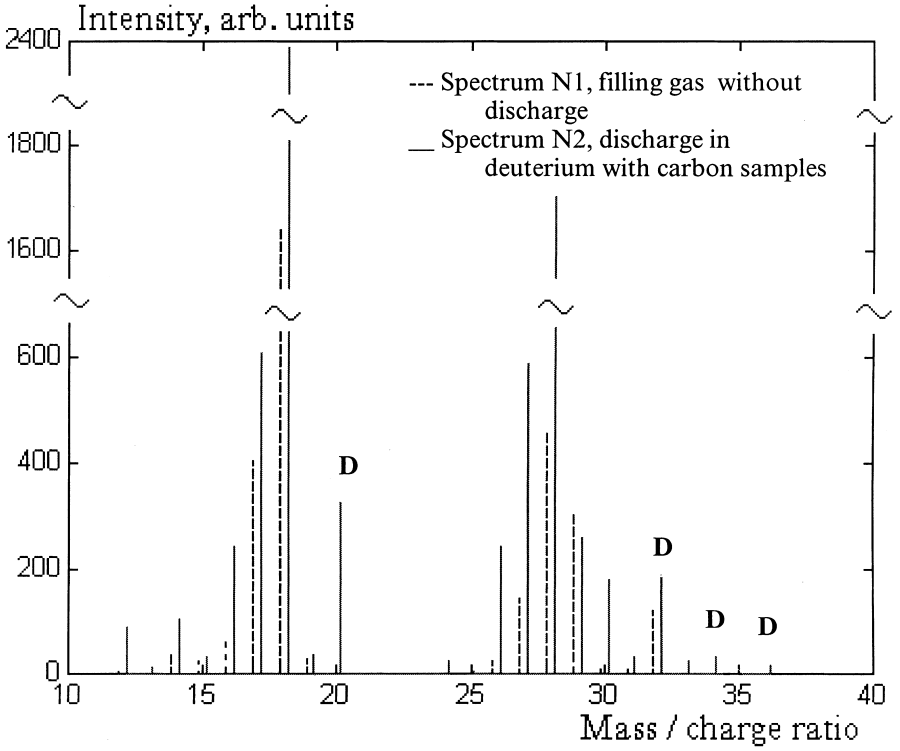

Fig. 3. Comparison of two typical mass spectra for initial gas (dashed peaks) and gas sampling during exposure of carbon materials in plasma (solid peaks). Doublet peaks corresponding to deuterocarbon generation during bombardment are labeled by a D-symbol.

ND_3^+ ions may be due to plasma interaction with a boron carbonitride insulator, and the mass spectrum of the deuterium discharge without carbon samples (not shown in Fig. 3) gives additional evidence of it.

The presence of the single-charged ions CD_3^+, CD_2^+, CD^+ and C^+ causes the sharp intensity increase of the peaks with the following M/e ratios: $M/e = 18$, 16, 14 and 12, respectively. Also, one can see from Fig. 3 that more complex deuterocarbons are being formed during the exposure, for example, C_2D_2, C_2D_4, C_2D_6, and doublets $M/e = 36$, 34 which are attributed to C_3^+, $C_2D_6^+$, $C_2D_5^+$ and CD_3O^+ ions. The $M/e = 32$ peak corresponding to molecular oxygen is transformed into a doublet revealing $C_2D_4^+$ ion production. The sharp growth of the $M/e = 30$, 28, 26 peaks and also the appearance of the peak $M/e = 24$ are explained by the generation of the ($C_2D_3^+$, NO^+), ($C_2D_2^+$, CO^+, N_2^+), C_2D^+ and C_2^+ ions, respectively.

A mass spectrum in the deuterium discharge without samples was also taken and it showed the complete absence of peaks with $M/e = 36$, 34, 24, 20, 12. This confirmed that the interaction of plasma with carbon samples gives rise to many chemical reactions and results in a large spectrum of deuterocarbons being observed.

The generation of deuterocarbons was also observed by optical techniques. A part of visible spectrum (4100–4400 Å) taken from the discharge is presented in Fig. 4. In (a) the case of the plasma without samples is seen while in (b) the spectrum corresponds to the case of the sample exposure ($T = 770$°C) under the same plasma conditions. From these figures one can see that the signal measured in the 4270–4312 Å wavelength interval between the D_δ and D_γ lines and identified as a signal of the CD band, significantly increases thus giving evidence for the presence of deuterocarbons in the plasma volume near the samples. A similar measurement was carried out for a sample temperature of about 1150°C showing an even more significant increase of the signal mentioned above and indicating a rising rate of deuterocarbon generation.

4. Discussion

Mechanisms of carbon materials erosion under ion bombardment have been investigated during the last years. However, the complex behaviour induced in fusion reactor divertor and first wall surfaces under operating conditions involving high temperatures and plasma interactions, has yet to be adequately understood [4–6].

Thus, the possibility of chemical erosion of carbon materials in plasma environment at high temperatures in

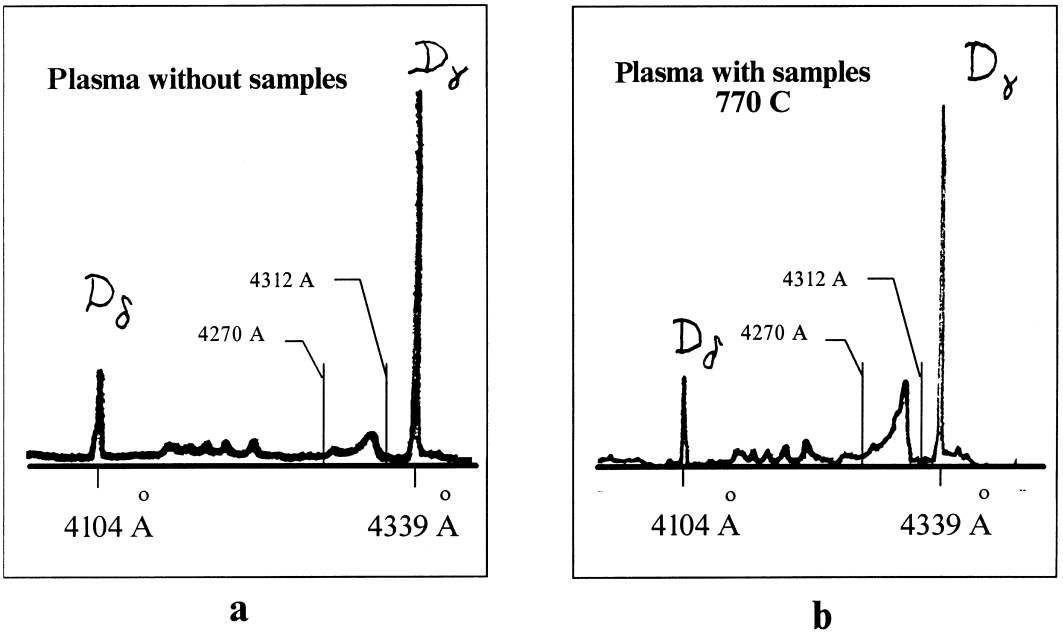

Fig. 4. Optical spectra from a plasma in the neighbourhood of the sample position: (a) discharge in deuterium without samples, (b) discharge with samples in the plasma (770°C). The CD-band signal between 4270 and 4312 Å increased in presence of carbon samples.

presence of co-deposition processes needs to be revealed in more detail. Our experiments were carried out for temperatures above the expected limit for the chemical erosion of carbon materials (500–600°C) so that the results were expected to be attributed to physical sputtering and to radiation enhanced sublimation. However, our measurements in the 750–1150°C range show that chemical processes on the surface of carbon samples cannot be neglected in erosion considerations at high temperatures. Direct detection of deuterium–carbon compounds in these plasma bombardment experiments on carbon samples by mass spectrometry and optical measurements gives evidence of chemical reactions. Moreover, we observed an increase of optical signal of deuterocarbons in spite of a certain decrease of total erosion rate for the studied materials at higher temperatures (1150°C). These facts encourage us to further investigations of chemical reactions in plasma interactions with carbon materials at high temperatures. Note the correspondence of our findings with the experimental results of chemical erosion obtained in the TEXTOR tokamak [6] were the erosion rate maximum was shifted to the value about 800°C.

Another important issue is plasma erosion at low ion energy (from a few tens to several eV and even lower) [7]. Our progress in reducing the average ion bombarding energy from 200 eV down to 50 eV is rather important in this respect. The obtained data show that the erosion decreases over this range for RG–Ti and POCO graphites at 750°C but the erosion yield dependence on energy does not seem to be very strong (see also [8]).

One more feature of the erosion process looks evident from our experiments: this is the much higher erosion of graphites with high porosity (POCO) and with highly developed surface (CFCs) at about 800°C. This may relate to a particular gas regime established on such a surface under a plasma flux, leading to the enhancement of chemical reactions.

As to graphite doping, then our study has not shown any pronounced reduction effect on RG–Ti graphite erosion in the considered conditions at the level of 0.1% wt boron addition.

5. Conclusion

The study may be summarized as follows. The erosion of carbon materials induced by deuterium plasmas under steady-state conditions at high temperatures (750–1150°C) has been studied in the LENTA facility. Experimental conditions corresponded to the simulation of one normal operation cycle ion dose of the ITER divertor ($\sim 10^{22}$ ion/cm^2). Porous graphite (POCO) and carbon composites (CFCs) exhibited much higher (by a factor of 2–3) erosion rates than dense titanium doped RG–Ti graphites at 750°C. This could shorten the lifetime of the divertor armour if made of these composites.

Direct measurements have shown the intensive formation of deuterocarbons CD_4, C_2D_2, C_2D_4, C_2D_6 and of their products under deuterium plasma impact on carbon materials. Mass analysis of gas composition and optical monitoring of the near sample plasma region during exposure give evidences of chemical reactions. This could result in formation of co-deposited layers and the uptake of corresponding hydrogen species on the reactor walls.

Taking into account that these measurements were made for surface temperatures exceeding the expected limits of the chemical erosion (which were established largely in research with ion beams having rather low flux density, and with generally a higher energy range), we suppose that more attentive investigations of plasma induced erosion at 800–1200°C is needed for better evaluation of carbon materials armour of the fusion reactor divertor and of the first wall.

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