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Imitation of deuterium plasma interaction with the surface of carbon materials in gaseous divertor conditions

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Abstract

The experiments on simulation of gas divertor conditions were done in the LENTA facility under interaction of a plasma flow with neutral gas. The samples of carbon materials were exposed in a steady-state deuterium plasma (ion energy 5 eV, ion flux 5×10^{21} $m^{-2}\,s^{-1}$, fluence 10^{26} m^{-2}) at 1470 K (MPG-8) and at 1320 K (SEP NB31). Heavy deuterocarbon molecules (C_2D_2, C_2D_4, C_2D_6) were observed in mass spectra of the discharge. This fact and high erosion yields show the presence of chemical erosion. Deuterium accumulation in carbon materials was studied by elastic recoil detection analysis. The integral deuterium content is 6×10^{18} m^{-2} in SEP NB31 and 1.95×10^{19} m^{-2} in MPG-8. The profiles of C and Mo atom distributions in deposited layer on Mo collector is 'X'-like. Carbon atoms distribution in deposited layer on Si is uniform. The integral deuterium content in co-deposited layers is 1.4×10^{21} m^{-2} on Si and 4.8×10^{20} m^{-2} on Mo. A globular structure of co-deposited layer on Mo collector was found.

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Keywords: Carbon; Co-deposition; Deuterium; Divertor; Erosion

1. Introduction

In the ITER design, the carbon–fiber composites (CFC) are considered as the material suitable for the most heat-loaded units of the divertor. The graphite erosion by hydrogen isotope ions is well studied in the energy range >50 eV. Studies of the plasma confinement under enhanced gas pressures (with gaseous divertor) need the experimental data on the erosion of carbon materials, the erosion products and the accumulation of hydrogen isotopes in them at the ion energies <10 eV. The characteristic electron temperatures, T_e, in the near-wall plasma under conditions of a gaseous divertor are assumed to be of ~1 eV. At such values of T_e in the deuterium plasma the ions, bombarding the surface, acquire an energy of the potential jump, which corresponds to 5 eV. The researchers, who studied the chemical erosion of graphite at the plasma facilities with the energies of hydrogen isotope ions close to those expected in the ITER divertor, have found in recent years a considerable effect of a specific ion flux on the coefficient of the graphite chemical erosion [1–6]. At T_e of ~5 eV, an essential reduction in the chemical erosion coefficient of graphite is observed at ion fluxes $>10^{21}$ $m^{-2}\,s^{-1}$ [1,5]. At the same time, in the majority of the graphite chemical erosion studies, the ion flux was in the range from 10^{18} to 10^{20} $m^{-2}\,s^{-1}$ [1].

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This paper presents some results of the studies of deuterium plasma interaction with carbon materials at the deuteron energy of 5 eV.

2. Experimental technique

The gaseous divertor conditions were simulated at the LENTA facility with a plasma beam discharge under the deuterium molecular pressure in the 'gaseous target' equal to 7 mTorr. A device of $10 \times 30 \times 40$ mm, for irradiating the materials is shown in Fig. 1. Some erosion product collectors (Si and Mo plates of 12 class surface quality) were installed at the mounting attachment of the irradiated sample. The collector temperature was dependent on their location relative to the irradiated target, being equal to 870 K for a Mo collector and ~370 K for a Si collector. An analysis of the sputtered deuterocarbons was performed with magnetic mass-spectrometer MI 1201V-type that has a high resolving power (>500). The ion source of the mass spectrometer was directly connected with a discharge chamber of the facility by a stationary channel. Experiments have begun from the mass-spectra measurements of the gas leaking in (deuterium), then the mass spectrum of a gas under the discharge in deuterium at the working irradiation parameters, but without carbon materials, was measured; finally the measurements were done under the plasma interaction with carbon materials. The parameters in the deuterium plasma column were monitored with a single Langmuir probe located 3 cm distant from the axis. The measurements have shown that the electron temperature in the column was 1 eV. Hence, the energy acquired by the deuterons was 5 eV, on an average. At the specific ion flux from the plasma to the target surface 5×10^{21} $m^{-2}\,s^{-1}$, the irradiation fluences reached 10^{26} m^{-2}. A diameter of the irradiated spot upon the specimen was equal to 2.5 cm.

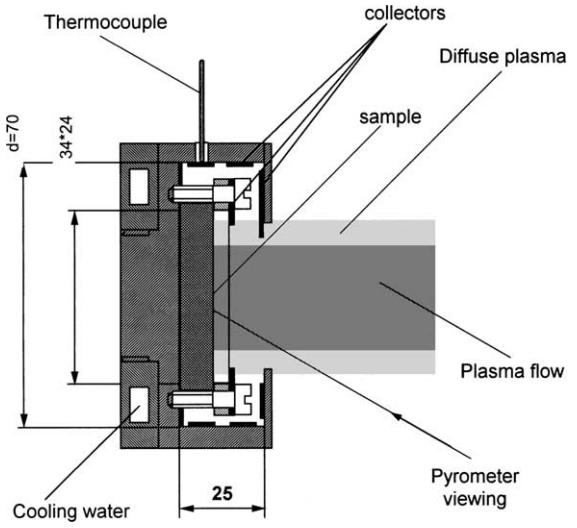

Fig. 1. A device for materials irradiation at LENTA facility in experiments on simulation of the gaseous divertor conditions.

The impurity-free graphite MPG-8 and the CFC SEP NB31 were used as targets. The temperature of the irradiated target in the spot was dependent on a power coming to it from the plasma and by a heat removal through a water cooled copper substrate pressed to its rear surface. This temperature was monitored with an optical pyrometer, being equal to 1320 K for SEP NB31 and 1470 K for MPG-8.

After the exposure, a change in the mass of specimens was determined by a weight technique.

The surface microstructures of the targets and the deposited layers on collectors were studied with the scanning electron microscope, JEOL. The chemical composition of target and collector surface layers was measured by the Rutherford backscattering (RBS) technique in the Van de Graaff accelerator. The He^+-ions scattered under the angle of 170° with the energy of 1.6 MeV were recorded using a surface barrier detector. The phase structure of the films deposited on the collectors was determined by the X-ray diffraction analysis technique in DRON-4 diffractometer.

To determine the deuterium depth distribution in the irradiated targets and in the co-deposited layers the elastic recoil detection analysis was used. For this purpose, the He^+-ion beam with the energy of 2.2 MeV bombarded the specimen under study at the angle of 15° to its surface. The recoil atoms were registered at the angle of 30° to the initial incidence direction of He^+-ions. In order to determine the absolute deuterium atom concentration, the energy spectra of the standard calibration specimens were measured.

3. Experimental results

Under the graphite exposure in the deuterium plasma, the lines for $M/e = 20$ (CD_4) and 28 (C_2D_2) were detected; an increase of intensities of the lines for $M/e = 18$, 16 and 14, bound up with CD_3^+, CD_2^+ and CD^+ ions, was observed; as well as the emergence of the more complicated heavy deuterocarbons with $M/e = 32$ and 36, probably $C_2D_4^+$, $C_2D_6^+$, C_3^+ ions, was brought out.

The chemical erosion yields of MPG-8 and CFC under 5-eV D^+-ion bombardment, calculated from the weight losses, are equal to 0.0170 atoms/ion (at 1470 K) and 0.0186 atoms/ion (at 1320 K) respectively. The high values of these coefficients and the presence of the deuterocarbon molecules in the mass spectrum confirm the fact that the chemical erosion of the carbon materials still takes place at the temperatures under study.

At the same time, according to the thermodynamical equilibrium of the H/C system, at temperatures >630 °C,

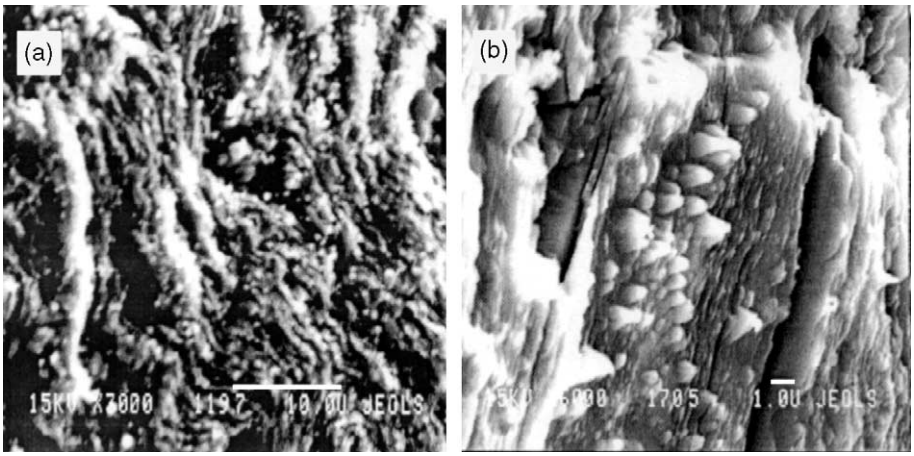

Fig. 2. Surface microstructures of MPG-8 graphite (a) and SEP NB31 composite (b) after deuterium plasma irradiation (5 eV, 10^{26} m^{-2}) at 1470 K (MPG-8) and at 1320 K (SEP NB31).

the transition from the CD_4-molecule formation to the D_2-molecule formation and emission takes place. As a result of the chemical erosion, a smooth polished graphite surface structure becomes rough (Fig. 2(a)). Along with an increase in the roughness, a great number of microparticles, <1 μm in size, emerges upon it. Probably, they are nuclei of the new structures forming under the back diffusion of deuterocarbon molecules to the target under high pressure of the neutral gas. This statement is confirmed by the composite microstructure, on separate fibres of which the cones have grown up (Fig. 2(b)).

The distribution of deuterium in MPG-8 after its exposure for 14 h to the irradiation fluence of 10^{26} m^{-2} is given in Fig. 3. The extrapolation of this curve to the axis of abscissas shows that deuterium is contained in a 230 nm thick layer. Its integral concentration is 1.95×10^{19} m^{-2}. The 5-eV deuteron penetration deep into graphite was caused by the diffusion of deuterium (at 1470 K in the experiment). The D-diffusion coefficient, estimated from the distribution profile and equal to 1.05×10^{-18} m^2 s^{-1}, is in good agreement with the data by Tanabe [7].

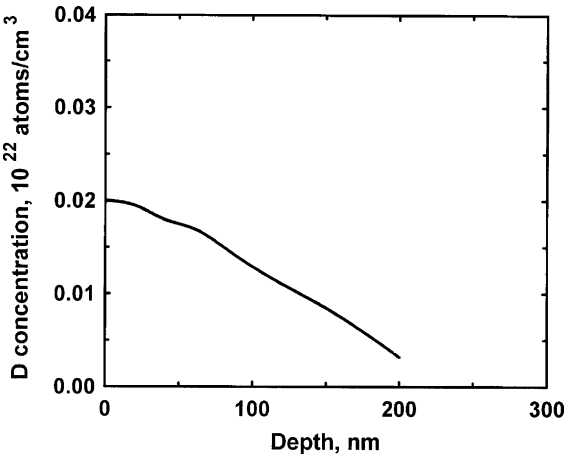

Fig. 3. Deuterium depth distribution in MPG-8 graphite after exposure in deuterium plasma (5 eV, 10^{26} m^{-2}, 1470 K).

The measurements by Wilson [7] have shown that the maximum of catching the 100-eV tritium implanted to the dose of 2×10^{24} m^{-2} into various kinds of graphite is observed at 1470 K. According to these data, the integral tritium content in MPG-8 is $\sim 5 \times 10^{20}$ m^{-2}, i.e. considerably more hydrogen isotopes are trapped in comparison with the results of our measurements. Apparently, under the bombardment of graphite by 5-eV D^+-ions an accelerated desorption of deuterium occurs, in particular as the chemically eroded deuterocarbon molecules are observed.

The content of D atoms in the 300 nm thick CFC layer is even less (6×10^{18} m^{-2}) under conditions of the experiment.

The spectra of the RBS from the initial silicon surface and from a surface of the carbon layer deposited on the silicon collector show that Si surface has been oxidized before the graphite was exposed to the plasma, it consists of 53 at.% Si and 47 at.% O. The carbon film deposited on Si, 230 nm thick, includes 13 at.% O, 0.05 at.% Si and small metallic impurities. A deuterium distribution profile in this carbon layer is shown in Fig. 4. The whole of deuterium is within the co-deposited layer, i.e. deuterium does not diffuse into the substrate. The integral D concentration in the layer is 1.4×10^{21} m^{-2} and essentially exceeds the amount of deuterium in MPG-8. That is apparently caused by a high concentration of the deuterium (atoms, molecules, deuterocarbons) reflected and

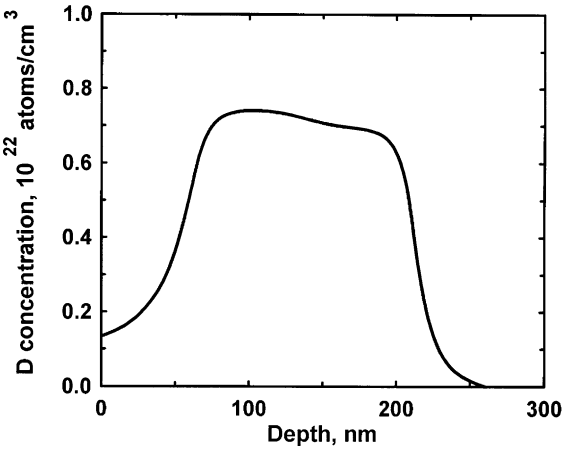

Fig. 4. Deuterium depth distribution in carbon layer deposited on silicon collector after MPG-8 graphite exposure in deuterium plasma (5 eV, 10^{26} m^{-2}, 1470 K).

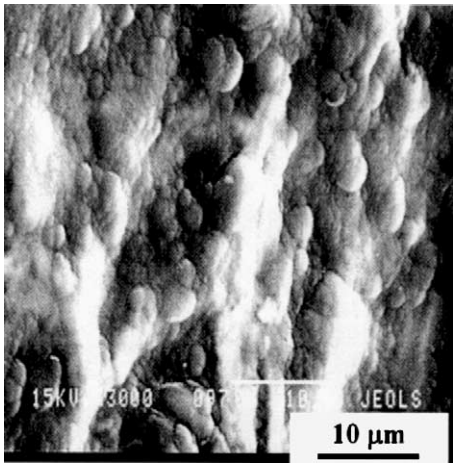

Fig. 5. Surface microstructure of the C–D co-deposited layer on Mo collector at 870 K in gaseous divertor simulation experiments at LENTA facility (steady-state deuterium plasma with ion energy 5 eV, ion flux 5×10^{21} m^{-2} s^{-1}, fluence 10^{26} m^{-2}).

desorbed from the graphite target. In the co-deposited layer atomic ratio D/C = 0.07.

The forming surface layer deposited on Mo collector is not uniform. Its microstructure is shown in Fig. 5. There are some zones, where the formation of globules, 1–5 μm in diameter, is seen. The film flaking-out is observed in some zones. The chemical composition of the film, measured with RBS, is given in Fig. 6. At the temperature of 870 K, a homogeneous mixing of carbon and metal atoms is observed on the collector surface. The mixed layer is 400 nm thick. The X-ray diffraction analysis of this layer shows that the deposited layer is a

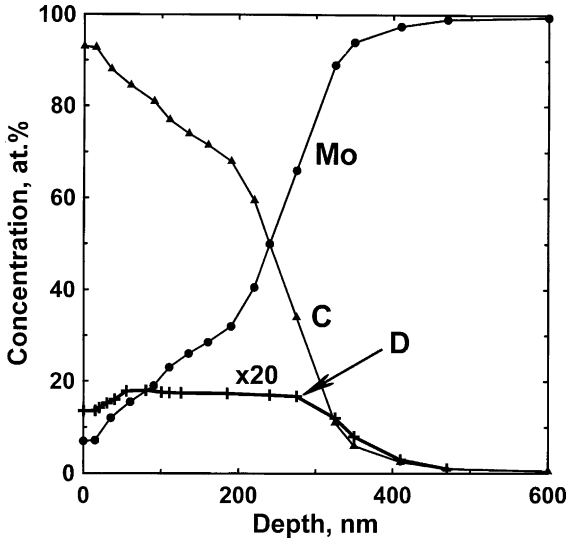

Fig. 6. Component depth distribution in C–D co-deposited layer on Mo collector at 870 K in gaseous divertor simulation experiments at LENTA facility (steady-state deuterium plasma with ion energy 5 eV, ion flux 5×10^{21} m^{-2} s^{-1}, fluence 10^{26} m^{-2}).

mixture of carbide and semicarbide phases, as well as of a solid carbon solution in molybdenum. The phase composition of the layer changes in depth. It is characteristic that deuterium is accumulated in co-deposited mixed layer only, similar to that on the Si collector. The integral deuterium concentration in the 400 nm thick layer is 4.8×10^{20} m^{-2}, the atomic ratio D/C = 0.02.

The analysis of the findings shows that the deuterium concentrations in the C–D co-deposited layers and the thickness of co-deposited layers will be different in various sections of the divertor. The temperature is a determinative factor.

4. Conclusion

Chemical erosion of MPG-8 graphite and CFC SEP NB31 with the formation of deuterocarbon molecules CD_4, C_2D_2, CD_3, CD_2, CD, C_2D_3 and C_2D_6 has been observed at 1320–1470 K. The yields of chemical erosion by 5-eV D^+-ions for MPG-8 and SEP NB31 are 0.0170 and 0.0186 atoms/ion, respectively.

As a result of the MPG-8 and CFC exposure in the deuterium plasma at the ion energy of 5 eV, irradiation temperature of 1320–1470 K and fluence of 10^{26} m^{-2}, a portion of the trapped deuterium is negligibly low being equal to 2×10^{-5}% of the number of bombarding D^+-ions.

The integral content of the trapped deuterium and the thickness of C–D co-deposited layers on collectors

are dependent on their location relative to irradiated target and temperature.

Mixed carbon–metal layer, consisting of carbide and semicarbide phases as well as of solid carbon solutions in metals, is formed on Mo collector at the temperature 870 K.

Deuterium atoms are within the co-deposited layers only. Their integral content in the layers is equal to 1.4×10^{21} m^{-2} on Si collector and 4.8×10^{20} m^{-2} on Mo collector. Deuterium does not diffuse into the substrates.

The atomic ratios D/C in co-deposited layers are 0.02 on Mo collector and 0.07 – on Si collector.

Formation of globules was observed in the layers deposited on Mo collector.

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