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Interaction of a high density neon plasma with graphite

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Abstract

The interaction of a high density ($n_e = 2.5 \times 10^{18}$ m^{-3}), low temperature ($T_e = 20$ eV) neon plasma with a polycrystalline graphite sample has been studied by thermodesorption spectrometry. The results are shown to be qualitatively and quantitatively different from previous studies of graphite exposed to neon ion bombardment or to mixed hydrogen–neon plasma and exhibit some unusual features. On the other hand, the results exhibit striking similarities with those of other authors who have studied the helium ion implantation in graphite. The results are shown to be in accordance with the model used to explain the helium ion experiment taking into account the redeposited carbon layer.

Keywords: Plasma–wall interaction simulator; Desorption; Low Z wall material

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1. Introduction

The implantation of noble gases in materials has been extensively studied for bombarding ion energies greater than 1 keV. The experiments in the low energy range, below 500 eV, are rather scarce. The recent introduction of neon in the tokamak plasma to enhance the radiative properties of the scrape-off layer (SOL) [1–4] leads to more studies on the interaction of this gas with the graphite which is the main component of the tokamak inner wall.

Choi et al. [5] have studied the thermodesorption (TDS) of single crystal graphite after neon ion bombardment in the range (10–600 eV). They obtained simple TDS spectra with only one peak which they explained by a first order desorption mechanism. In a previous paper [6] we described the results of TDS from polycrystalline graphite exposed to a plasma obtained from hydrogen–neon gas mixtures similar to tokamak edge plasma with neon seeding. The results, qualitatively in accordance with those of [5], showed the desorption of implanted neon by hydrogen during the plasma exposure. To ascertain this interpretation of our results, we have exposed the graphite samples to pure neon plasmas and proceeded TDS with and without supplementary exposure to pure hydrogen plasmas. The results are described in the present paper. The TDS spectra obtained without hydrogen plasma exposure are shown to be qualitatively and quantitatively different from those of Choi et al [5] as well as those of our previous paper [6] with mixed plasmas. They exhibit a more complex structure with two peaks, similar to those obtained by Alimov et al. [7] for helium desorption. The subsequent hydrogen plasma exposure gives a rather surprising result, as the neon yielding the high temperature peak of the TDS is desorbed by the plasma while the low temperature peak remains nearly untouched.

2. Experimental

The experiments were performed on the surface plasma interaction simulation apparatus (SPISA) which has been described elsewhere [8]. The samples were polycrystalline graphite Le Carbone Lorraine 5890PT (grain size about 5 μm) $11 \times 11 \times 2$ mm^3 which is used for the inner wall coating of Tore Supra. A bias voltage can be applied to the sample, allowing a range of neon ion bombarding energy between the floating potential and 250 eV. The duopigatron plasma source was used essentially with pure neon. The plasma parameters were measured with a double Langmuir probe. The electronic temperature was about 20 eV and the ionic density was in the 10^{18} m^{-3} range. The plasma content was additionally checked by optical visible spectrometry and by quadrupole mass spectrometry [6]. The sample is exposed to the plasma via a hole (9 mm in

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diameter) drilled in the anticathode of the duopigatron source. The neon ion fluences were between 2 and 12×10^{18} ions cm^{-2} for a flux of 1.8×10^{18} ions cm^{-2} s^{-1} assuming that the saturation current is obtained essentially from single charged ions. This assumption could not be sustained with argon plasma where measurements by Gilles [9] exhibit an ArIII/ArII ratio greater than one with a duopigatron source operating with plasma parameters similar to ours. But the NeIII/NeII ratio obtained by Gilles for a neon plasma is about 0.05 with the same experimental conditions. Moreover, the QMS recordings of the neon and hydrogen pressures during the plasma shots exhibit a strong pumping effect of the hydrogen gas by the plasma, according to the good ionization efficiency of the hydrogen. But such a pumping effect is nearly absent for the neon gas. So, the ionization efficiency of the neon is weak and the NeIII production from the NeII ions can be considered negligible. The total exposure is done by 0.5 s plasma shots to prevent excessive heating of the sample by the ionic current.

It is impossible to prevent a slight amount of hydrogen being desorbed from the metallic walls of the source by the neon plasma. The ratio of the partial pressures of hydrogen and neon is about ten percent at the end of the plasma shot depending on the previous history of the apparatus. This hydrogen is ionized in the plasma and desorbs a small amount of the implanted neon.

In some experiments, the neon exposed sample was subsequently exposed to a pure hydrogen plasma to check our previous assumption [6] of the hydrogen ion induced desorption of the implanted neon. The parameters of these plasmas were $T_e = 15$ eV and $n_e = 5 \times 10^{18}$ m^{-3}. The exposures were also fractionated in 0.5 s shots.

After the plasma exposure, the sample was transferred into an ultrahigh vacuum chamber without breaking of the vacuum. It was heated from its initial temperature of about 320 K (due to the ion bombardment exposure) to 1120 K. The temperature rise was electronically driven and perfectly rectilinear. The TDS were performed at 0.5, 1.0 and 2.0 K s^{-1}. The sample heating was obtained by a combination of thermal radiation and electron bombardment on the unexposed surface of the sample. During the temperature rise, the partial pressure of neon was recorded with a quadrupole mass spectrometer. A low neon signal was registered on the QMS before the beginning of the heating, but the transfer duration was about 300 s and the calculated amount of desorbed neon before the heating was negligible.

Auger microanalysis depth profiling was tentatively worked out, but the neon signal was too small to be measured, as in Ref. [5].

3. Results

Typical TDS spectra after sample exposure to pure neon plasma are shown in Fig. 1. They are qualitatively and quantitatively different from those obtained after exposure to mixed hydrogen–neon plasmas, as can be seen in Fig. 2 and Ref. [6]. For the pure neon plasma, the low temperature peak is at the same location and has approximately the same amplitude as the mixed plasma result, but it is followed by a completely new and broader peak at 600 K.

Contrary to our previous result, the main peak height and the integral of the spectra, which corresponds to the total amount of adsorbed neon, are proportional to the total fluence of the neon ions. They are also dependent on the bombarding energy. As in Ref. [6], the lower temperature peak nearly does not depend on these parameters.

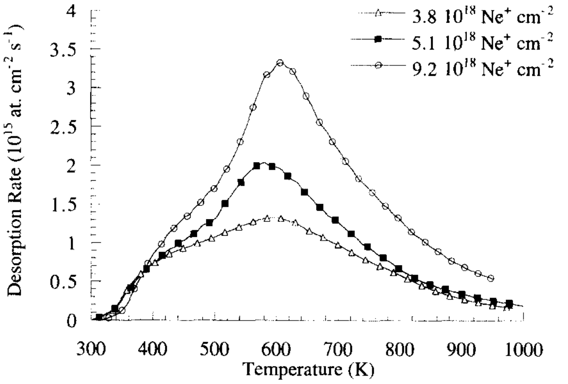

Fig. 1. Thermodesorption spectra of neon from graphite exposed to neon plasma. The location of the main peak does not depend on the fluence. One can observe, on the filled-squared curve, a slight shift of the main peak to lower temperature due to the parasitic hydrogen ion induced desorption of the implanted neon atoms during the plasma. A high temperature tail does exist, indicating the presence of a codeposited layer of neon and carbon. Heating rate 1 K s^{-1}.

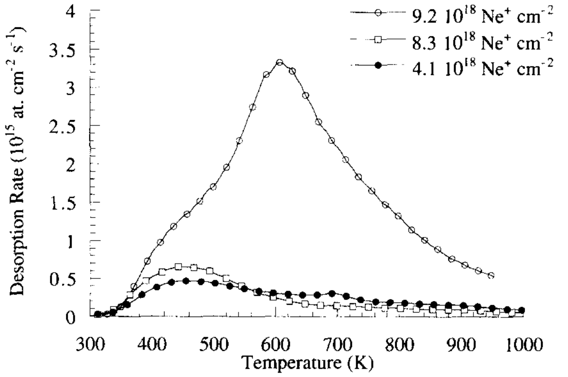

Fig. 2. TDS spectra, heating rate 1 K s^{-1} Open circles: after exposure to a pure neon plasma; squares: after exposure to a mixed hydrogen neon plasma with a similar neon fluence; full circles: the pure neon plasma exposure was followed by an hydrogen plasma exposure. The low temperature peak remains nearly unaffected by the hydrogen plasma.

Table 1
Some values obtained for thermodesorption spectra with similar hydrogen partial pressures

Fluence (10^{18} ions cm^{-2})	Heating rate ($\mathrm{K\ s}^{-1}$)	Desorbed neon amount (10^{17} atoms cm^{-2})	Peak temperature (K)
5.9	0.5	8.7	580
2.5	1.0	3.3	600
9.2	1.0	10.0	600
5.2	2.0	6.3	615

One can see the small shift of the main peak location with the heating rate, the independence of the peak location and the dependence of the amount of implanted neon on the fluence.

The location of the high temperature peak depends on the heating rate of the sample and is, as expected, displaced to the high temperature side when this rate is increased. Another shift of the location of the main peak is produced by the presence of parasitic hydrogen in the plasma, the peak temperature being lowered by the bombarding hydrogen ions. The measurement of the peak shift as a function of the heating rate is however possible by comparing only the spectra obtained with nearly identical hydrogen partial pressures. This shift, being at the limit of the uncertainty of the temperature measurements, is not sufficient to allow activation energy calculations.

The peak location is independent of the amount of implanted neon.

The Table 1 summarizes the results obtained on the peak temperature and the total amount of desorbed neon with the different parameters.

The effect of hydrogen plasma exposure on the sample preimplanted with a plasma of pure neon is shown in Fig. 2. The result is somewhat surprising because the first peak, which is thermally desorbed more easily, remains nearly unaffected by the energetic bombardment of the hydrogen ions, and it is the neon desorbing at higher temperature which is desorbed by the hydrogen plasma. This result is consistent with the result described above on the shift to the low temperature of the main peak involved by the presence of hydrogen in the neon plasma.

4. Discussion

The main results of the experiments described above are:

(i) The presence of the high temperature peak in the TDS spectra obtained after the exposure of the sample to pure neon plasma. This result evidences a neon implantation process different from our previous results on mixed hydrogen–neon plasmas.

(ii) The striking similarities between our TDS spectra and those from Alimov et al. [7] for edge oriented pyrolitic graphite and EK98 polycrystalline graphite exposed to 40 keV helium ions concerning as well the overall shape as the peak temperatures.

(iii) The preferential desorption of the neon which gives the higher TDS peak temperature by the hydrogen plasma.

The literature on implanted neon in graphite is rather scarce [5,6,10] and does not correspond to the fluences and bombarding energy used in our experiments. On the contrary, helium ion implantation has been extensively studied and the mechanisms of implantation and thermal desorption are increasingly being understood. The helium studies can give some insight in the thermal desorption mechanisms of neon because the van der Waals radius of helium, 0.18 nm, compares well to the radius of neon, 0.16 nm, and the diffusion phenomena are likely similar. The atomic mass difference is more important for the implantation process because of the better efficiency of the energy transfer of the neon incoming ions to the carbon lattice. As the graphite used in the previously described experiments is polycrystalline and isotropic, relevant comparisons can only be made with studies on similar graphites or edge oriented pyrolitic graphite.

Alimov et al. [7] have extensively studied base oriented, edge oriented, and fine grain polycrystalline graphite exposed to 40 keV helium ions in a range of fluences from 0.5 up to 6.9×10^{18} He^{+} cm^{-2}. Except for BO HOPG, they obtained thermodesorption spectra very similar to those of the Fig. 1 both in the overall shape and in the main peak temperatures: 640 K (EK98), 750 K (EO HOPG) to be compared to those of Table 1. Our slower heating rates easily explains the slight difference in the peak temperatures. As in our preceding paper [6] for mixed hydrogen–neon plasmas, they explain the first thermodesorption peak by a diffusion limited process. The value of 0.35 eV that we obtained for the activation energy of neon diffusion is to be compared to the values between 0.7 eV and 1.1 eV obtained by other authors [11–13] for the helium diffusion. This difference can be tentatively attributed to the difference in the van der Waals diameters which are slightly larger than the distance between the basal planes for helium and nearly equal for the neon atoms. The authors of Ref. [7] explain their main peak by the escape of helium from bubbles that they have seen by transmission electron microscopy, followed by a rapid diffusion along the basal planes. Such bubbles were observed by Chernikov et al. [14] in similar experiments and by Almqvist et al. [15] using AFM characterization after

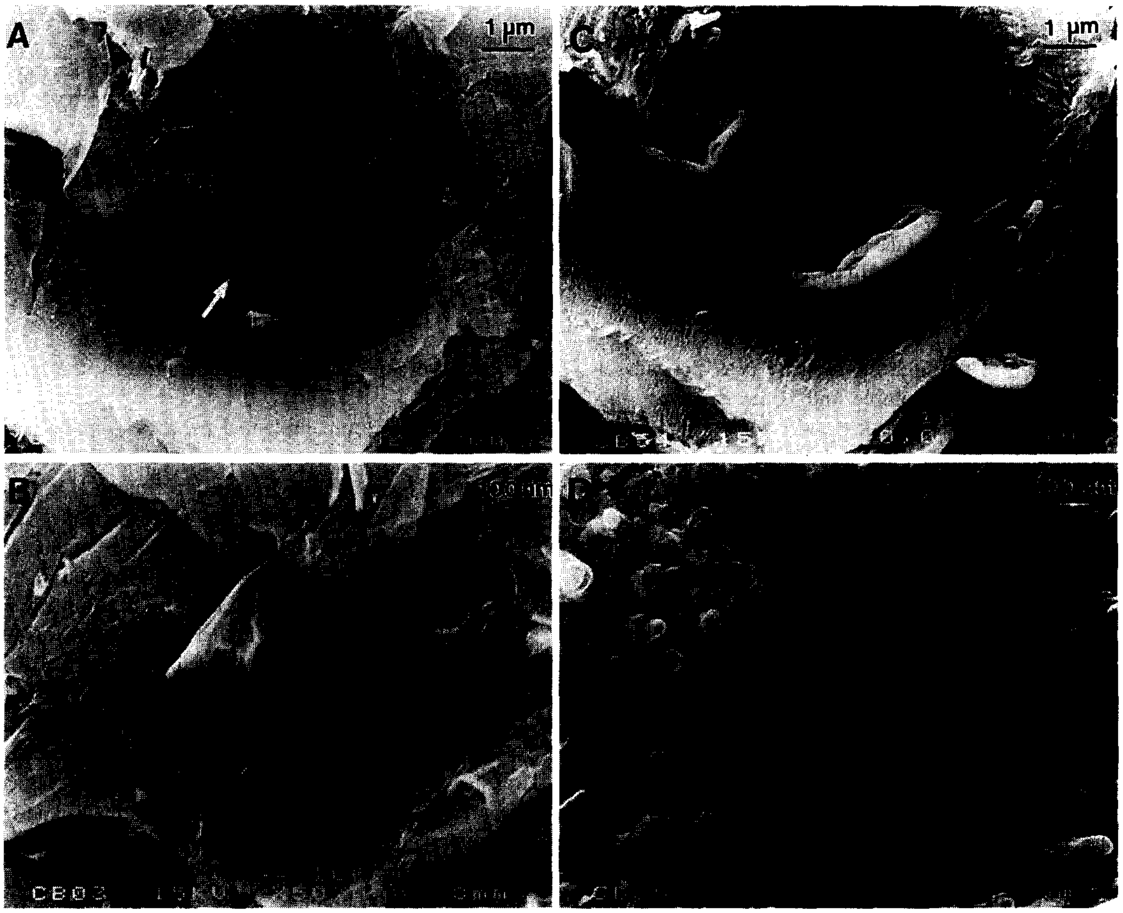

Fig. 3. SEM micrographs of the same location of a graphite sample before, (a) and (b), and after, (c) and (d), neon plasma exposure up to a fluence of 2×10^{19} Ne^{+} cm^{-2} for different magnifications, (a) and (c): $\times 10^{4}$, (b) and (d): $\times 5 \times 10^{4}$. One can see the surface modifications caused by the plasma exposure: the roundness of all the sharp edges of the graphite polycrystals and the numerous nearly circular structures on the initially flat large terraces parallel to the basal planes. These structures are similar in shape and size to the TEM micrographs of Ref. [7] and the AFM images of Ref. [15].

ionic deuterium bombardment. The small thickness of the implantation layer in our experiments prevented us from performing TEM observations, but high resolution scanning electron microscopy (Fig. 3) exhibits circular structures very similar in shape and dimension to those of Refs. [7,15].

The resemblances of the thermodesorption spectra and the electron micrographs are such that we can suppose that the bubble assumption of Ref. [7] reasonably holds also for our neon experiments. If it is the case, the apparently surprising desorption of the more strongly bonded neon (in the bubbles) by ionic hydrogen prior to the neon diffusion between the basal planes can be explained by the damages made to the bubbles by the hydrogen and the opening of easy diffusion paths.

Our experiment differs from those of Alimov et al. [7] in some ways. The plasma implantation energy is two orders of magnitude weaker than in the ion bombarding experiments. Therefore the range of implantation obtained by TRIM calculation is in the one nanometer range compared to the hundred nanometers range of the high bombarding energy experiments. The fluxes are two orders of magnitude larger.

The plasma parameters in our experiments are very similar to those of the MAP experiments [16,17]. In these papers, a strong redeposition rate is obtained with graphite exposed to an argon plasma. As the atomic mass of neon is nearer to the atomic mass of carbon than argon, the efficiency of the sputtering is better and the ionization efficiency of the sputtered carbon ion by the plasma is roughly the same as in the MAP argon experiments. So we can assume that there exists a redeposited carbon layer on the surface of the graphite with a thickness proportional to the fluence in the used range. A small amount of neon is likely to be embedded in this layer as seen in the TFTR experiment [3], where such a redeposited layer is important. This codeposition of neon with carbon explains the high temperature tail of our main peak which is going too slowly down to be accounted for by the first order mechanism which holds in the bubble explanation.

5. Conclusion

When exposed to a dense medium-temperature neon plasma, the polycrystalline graphite adsorbs the incoming neon ions via three processes: (i) between the basal planes of graphite (low temperature peak), (ii) inside bubble like cavities (main peak), (iii) in a coredeposited layer (high temperature tail). The thermal desorption of the neon occurs in the (i), (ii), (iii) order when the temperature is increased. If the neon plasma is mixed with hydrogen as in the radiating SOL of the tokamaks, the process (ii) does not occur because of the opening of the graphite cavities by the hydrogen ions. If the pure neon implanted graphite is exposed to a hydrogen plasma, the (ii) corresponding neon is desorbed before the (i) and (iii).

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