

Hydrogen trapping in graphite materials in various conditions

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Abstract. The paper presents a short revue of the study of not been investigated specific features of hydrogen trapping in graphite occurring as the results of inelastic interaction of impinging ions with carbon materials (potential trapping). The influence of potential mechanism on different regularities of hydrogen trapping are considered. Among them there is trapping dependence on irradiating ion current density, ion energy, irradiation fluence, temperature of the investigated samples and oxygen addition in the plasma.

1. Introduction

The problem of hydrogen trapping in the graphites, graphite materials and carbon films was the subject of interest during last decades. The main reason of it was an application of graphites and graphite material as the plasma facing elements for number of fusion devices [1-3]. The main sides of carbon material behavior under ion and plasma irradiation were evaluated due to investigations made in fusion devices and in experimental stands. In particular, serious attention was paid on study of features of hydrogen trapping and retention in the graphite materials [4-7]. The authors of the paper studded of not been investigated specific features of hydrogen trapping in graphite occurring as the result of potential interaction of impinging ions with carbon materials and carbon films [8-11]. The paper presents a short revue of the main results of these works.

2. Deuterium trapping in carbon materials exposed to D plasma

2.1. Dependence of trapping on irradiating ion energy

The experiments were performed in a device [8] intended for irradiation of samples with plasma ions and for measurement of the amount of retained gases by the method of Thermal Desorption Spectrometry (TDS). The temperature ramp up during TDS investigation was equal to 5 K/s. Deuterium (D) plasma was ignited in a three electrode system composed of heated cathode, preliminary anode and anode. The specimens of carbon fiber composite (CFC) and pyrolytic graphite (PG) were investigated. The temperature of the samples during irradiation was 470 ± 10 K and fluence was 5×10^{23} at/m². The results were basically similar for both materials. The paper presents results for CFC and notice the differences between two materials, if it is needed.

The energy dependence of the amount of deuterium released as D₂ molecules from the CFC (carbon fiber composite) irradiated with D ions at various energies (Fig. 1) demonstrates that the deuterium retention decreases when the ion energy reduces from 1000 to 200 eV/D. The main peaks in D₂ thermal desorption spectra for the samples irradiated with deuterium ions at relatively high energies



(from 500 to 1000 eV/D) locate in the temperature ranges 900–1000 K and 1050–1150 K with shoulders between 1200 and 1300 K.

The authors of the paper [9] basing on the analysis of the papers dealing with measurements of TDS spectra concluded that temperature of TDS peaks is relevant to the irradiation conditions and reflects to some extent the structure of the near surface layer of irradiated material. In particular, they associate TDS peaks at 900–1000 K with traps created by keV-ions in the stopping zone, i.e. with the near surface layer damaged by implanted ions. It was also shown that the high temperature peaks and/or shoulders relate to hydrogen release from traps in non-damaged layers behind the stopping zone. It can be seen (fig. 2 b and c) that both parts of TDS spectra of high-energy irradiated samples (HEIS) increase with the ion energy. Thus, one can say that the deuterium trapping in HEIS occurs both in and behind the stopping zone and depends on the ion kinetic energy. This type of trapping can be named as “kinetic trapping”.

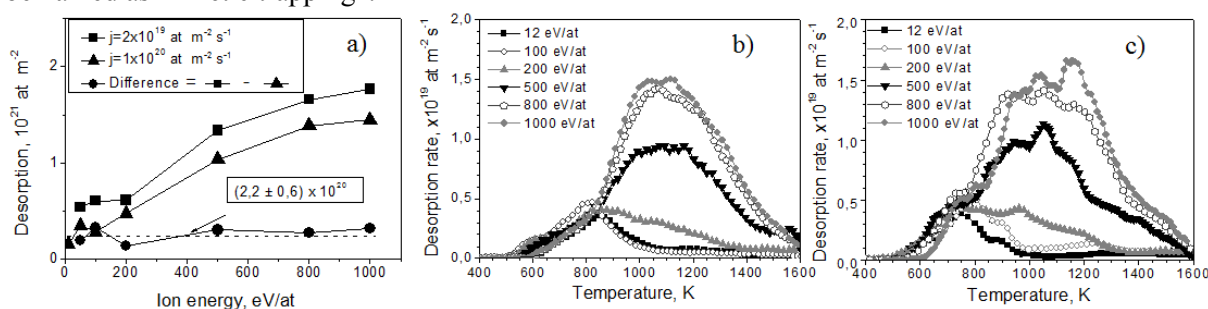


Figure 1. Total deuterium desorption from CFC (a), spectra of thermal desorption of PG (b) and CFC (c) irradiated with ion flux density 10^{20} at/m²s.

At ion energies below 200 eV/D, the decrease of the deuterium retention becomes inessential. A noticeable desorption from the CFC samples is observed even from the samples irradiated under floating potential [8]. Low energy ions cannot create traps through knock out collision with carbon atoms. Conclusion is made that low energy ions are captured at active centers created due to inelastic interaction with the surface carbon atoms. The active centers can also initiate trapping of D atoms from the layer of surface sorption. This type of trapping can be called as “potential trapping”.

The main TDS peak for the low-energy irradiated samples (LEIS) locates in the temperature range 700–850 K. The difference in the TDS peak positions for the LEIS and HEIS indicates that traps which are responsible for deuterium accumulation in the LEIS differ from those in the HEIS. Efficiency of low energy trap formation and filling seems to be at its maximum, when impinging ions stop in the near surface region, and decreases, as the ion penetration depth increases. Potential trapping constitutes presumable part of entire deuterium trapping at energies below 200 eV/D.

TDS peak and shoulders at the temperatures 500–600 K exist in the TDS spectra of both LEIS and HEIS. The same peak was found in TDS spectra of unirradiated CFC exposed to the air. Thus, one can assume that relevant traps are also formed by the mechanism of “potential trapping”.

The TDS peaks and shoulders in the temperature ranges 1050–1150 K and 1200–1400 exist in TDS spectra of hydrogen retained in CFC not subjected to any plasma irradiation, i.e. they relevant to the traps in the not damaged CFC structure. Thus, we believe that some part of implanted D atoms diffuses and are trapped in the not damaged area behind the ion range. This mechanism is not active for the dense PG structure. As a result, a peak in the temperature range 1200–1400 exists in TDS spectra for the CFC and absent in similar spectra for the PG.

Irradiation of the CFC samples with D ions at low ion flux of 2×10^{19} D/m²s to a fluence of 5×10^{23} D/m² leads to higher D retention as compared to the irradiation at high ion flux of 1×10^{20} D/m²s to the same fluence. This fact is observed for ion energies in the range from 10 to 1000 eV/D. The time/ion flux dependence of D trapping (fig. 1) shows that the increase of irradiation time results in growth of all the parts of TDS spectra. Trapping of impinging fast ions by kinetic mechanism does not depend on duration of the ion irradiation, whereas the probability of the surface penetration and

trapping is time dependent process. These finding shows that 1) potential trapping constitutes the main part of deuterium trapping at energies below 200 eV/D; 2) the fast ions provide potential trapping of D atoms from the layer of surface sorption layer, it seems to happen at expense of the energy of their in elastic collisions with the surface carbon; 3) the atoms penetrating surface by potential mechanism then can diffuse and be trapped in the traps created by fast ions in the ion stopping zone or in the not damaged area behind it.

Moreover, it is obviously, that number of deuterium traps created in the ion stopping zone of HEIS increases with increase of ion energy. But the amount of deuterium additionally trapped in longer experiments is practically the same for ions with different energies. Thus, one can believe, that dissociation of deuterium molecules and penetration of deuterium atoms into graphite is the limiting stage for particle diffusion and filling the traps in the stopping zone and in the bulk.

2.2. Dependence of trapping on the temperature of irradiated sample

It is seen (fig.2) that the trapping in LEIS steadily decreases with the elevation of the temperature during the sample irradiation. Contrary, the trapping in HEIS drops sharply between 500 and 650 K [10]. Fig. 2 c shows that in this temperature range the trapping is decreased in both low and high temperature traps, i.e. in the traps filled by both potential and kinetic mechanisms. This fact can also be explain by assumption that some fraction of particles penetrating the surface by potential mechanism is trapped in the kinetic traps.

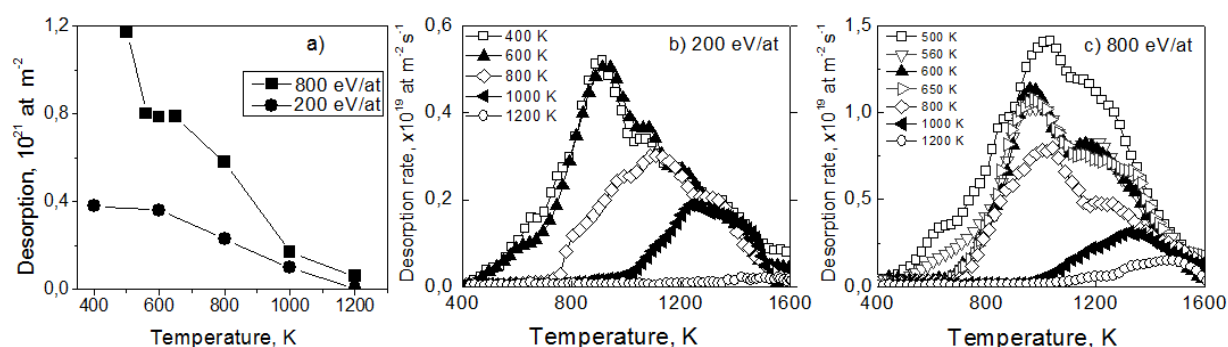


Figure 2. Temperature dependence of D_2 thermal desorption from CFC (a), spectra of thermal desorption of CFC irradiated by ions with energy 200 eV/at. (b), and 800 eV/at. (c).

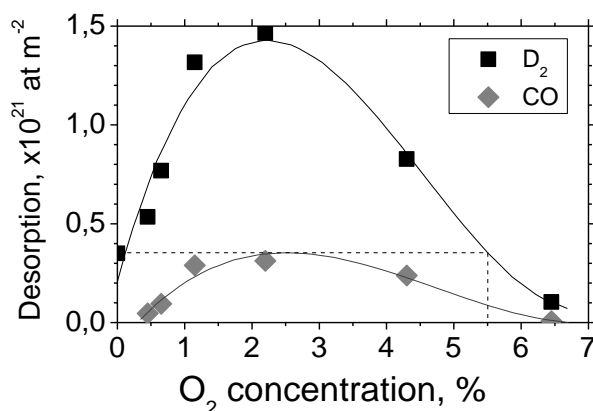


Figure 3. Deuterium and CO retention in CFC in dependence on oxygen concentration in plasma. (Ion energy is 50 eV/at., ion flux density is 10^{20} at/ m^2 s and fluence is 5×10^{23} at/ m^2)

2.3. Influence of oxygen addition to a deuterium plasma on D trapping

It is seen (fig.3 a) that trapping of both oxygen and deuterium increases with the increase of oxygen concentration in plasma. They reach maximums when oxygen concentration elevates up to $\approx 2\%$, decreases with further growth of oxygen concentration, and matches the trapping in CFC irradiated in D_2 plasma, when oxygen concentration equals 5.5%. These results could be a consequence of two opposite influences on deuterium trapping. On one hand, oxygen interaction with surface activates formation of active centers and deuterium trapping. On the other hand, oxygen sorbed on the surface more than deuterium, thus, hardening reaching the surface by deuterium. As the result, the increase of the oxygen concentration above certain limit leads to decrease of deuterium trapping.

Trapping of deuterium results in modification of graphite structure. It seems to be the reason of oxygen penetration and trapping and finally the reason of enhancement of oxygen retention. That is why change of deuterium trapping eventually leads to similar change of oxygen trapping.

3. Conclusion

The main results of the work can be summarized as follows. Deuterium trapping in- and desorption from CFC and PG samples irradiated with plasma ions was examined with thermal desorption spectrometry. It has been found that: 1) the deuterium trapping takes place even when energy of deuterium ions approaches zero; 2) for ion irradiation to equal fluences, the deuterium retention is higher at lower ion flux, i.e. the deuterium retention increases with the irradiation time.

Deuterium ions at energies above 200 eV/D are captured in the traps created due to their elastic collisions with carbon atoms. This process may be named as “kinetic trapping”.

The low energy ions are captured at active centers created due to inelastic interaction with the surface carbon atoms. The active centers created by both low- and high energy ions can also initiate trapping of D atoms from the layer of the surface sorption. The term “potential trapping” is proposed for this type of trapping. Potential trapping constitutes presumable part of entire deuterium trapping at energies below 200 eV/D. Under high energy irradiation the atoms penetrating surface through potential mechanism can fill the traps formed by fast ions in the ion stopping zone.

Time dependence of trapping under both low- and high energy irradiation is provided by trapping through potential mechanism.

Deuterium and oxygen trapping in the oxygen added plasma with the increase of oxygen concentration in plasma up to $\approx 2\%$ and decrease with further growth of oxygen concentration.

References

- [1] Y. Hirohata, T. Tanabe, Y. Oya et.al., JNM, 363–365 (2007), P.854-861
- [2] Y. Corre, R. Dejarnac, J. Gardarein, et al., JNM, 463 (2015), P. 832-836,
- [3] T. Eich, B. Sieglin, A. Scarabosio et al., JNM, 438 Supplement, (2013), P.S72-S77
- [4] V. Philipps, R.Neu, J.Rapp, U.Samm, M. Tokar, T.Tanabe, M.Rubel, (2000) Plasma Physics and Controlled Fusion, 42 (12 SUPPL. B), pp. B293-B310.
- [5] H. Atsumi, T.Tanabe, T. Shikama, (2015) Fusion Science and Technology, 67 (2), pp. 245-249.
- [6] J. Küppers, The hydrogen surface chemistry of carbon as a plasma facing material, Surface Science Reports, Volume 22, Issues 7–8, 1995, P. 249-321
- [7] L.L. Snead and M. Ferraris, 4.18 - Carbon as a Fusion Plasma-Facing Material, In Comprehensive Nuclear Materials, edited by Rudy J.M.Konings, Elsevier, 2012, Pages 583-620.
- [8] A. Airapetov, L. Begrambekov, C. Brosset, et al, JNM, 390–391 (2009), P.589-592
- [9] A. Airapetov, L. Begrambekov, S.Vergazov, et al. Journal of Surface Investigation: X-Ray, Synchrotron and Neutron Techniques. 2010. T. 4. № 4. P. 567-571.
- [10] A. Airapetov, L. Begrambekov, S.Vergazov, et al. (2010) Bulletin of the Russian Academy of Sciences: Physics, 74 (2), pp. 227-232.
- [11] L. Begrambekov, A. Ayrapetov, V. Ermakov. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 315 (2013), P. 110-116