Deuterium retention after deuterium plasma implantation in tungsten pre-damaged by fast C\(^+\) ions

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Abstract. Thermal desorption of deuterium from W was investigated. Virgin samples and samples damaged by 10 MeV C\(^{3+}\) ions were implanted from plasma in the LENTA facility at 370 K and 773 K. In comparison with the undamaged sample, deuterium retention in the damaged sample slightly increased in the case of deuterium implantation at RT, but decreased in the case of deuterium implantation at 773 K. At 773 K, deuterium was concluded to diffuse far behind the D ion range in the virgin sample, while C implantation region was concluded to be a barrier for D diffusion in the damaged sample.

1. Introduction
Tungsten is considered as a plasma facing material in future fusion reactors, where it will be exposed to extreme heat and particle fluxes from plasma and to high energy neutrons and \(\alpha\) particles generated in D-T reaction. Radiation damage in tungsten will lead to degradation of mechanical properties, and also it may increase hydrogen isotope retention. Irradiation by fast ions is often used for simulation of neutron impact on materials. In a series of works \cite{1,2}, 10 MeV C\(^{3+}\) ions were used for damage production and serious changes in material were observed after irradiation. In particular, swelling of the damaged layer was reported in \cite{1}. Deuterium accumulation in damaged tungsten after D plasma exposure was analyzed in those experiments only in a very thin surface layer using ERD (Elastic Recoil Detection) technique.

In this work, deuterium retention after plasma exposure was measured by thermal desorption spectroscopy (TDS) in the samples identical to those used previously in \cite{1}. TDS provides information about the total deuterium retention in sample.

2. Experimental
Polycrystalline tungsten samples with the purity of 99.95\% were used in experiments. Some samples were preliminary damaged by 10 MeV C\(^{3+}\) ions with the fluence of \((1.5-2)\times10^{21} \text{ m}^{-2}\). According to SRIM calculations, this irradiation produces the average damage of 6.3-8.4 dpa with the maximum of 30-40 dpa at 3.5 \(\mu\)m \cite{1}. The average concentration of carbon atoms near the surface was less than 0.01 at.\%, while maximal concentration of carbon atoms reached several at.\% at 3.5 \(\mu\)m \cite{1}.

Two damaged and two virgin undamaged tungsten samples were exposed to deuterium plasma in the LENTA facility (Kurchatov Institute) in pair at room temperature (< 370 K) and at 773 K. The ion...
flux in the continued operation regime was \((1.3-2) \times 10^{21}\) D/m²s, and the fluence was of about \(10^{25}-10^{26}\) D/m². The energy of incident ions was 250 eV.

Deuterium retention in the samples after plasma exposure was investigated by means of thermal desorption spectroscopy (TDS) using the UHV TDS stand (MEPhI). The base pressure in the TDS chamber was less than \(5 \times 10^{-9}\) mbar. The samples were introduced into TDS chamber through the lock chamber with a separate pumping system that gives a low background signal during TDS runs. The sample temperature was measured by the W–Re thermocouple directly welded to the edge of the sample. The heating rate was 2 K/s, and the maximal temperature was 1450 K. Desorption of deuterium containing molecules (D₂, HD, HDO, D₂O) was measured by a quadrupole mass spectrometer (QMS). Calibration of QMS measurements was done after each experiment. Description of the TDS-stand and experimental procedure are given in [3] in more details. Table 1 summarizes the information about the investigated samples. Letters v and d denote virgin and damaged samples, respectively, while the numbers are the temperatures during deuterium implantation.

| Sample | C¹⁴ ions fluence, m² | Primary defect concentration (av. in 3.5 µm), dpa | Temperature, K | Plasma exposure fluence, D/m² | Erosion depth, µm |
|--------|----------------------|-----------------------------------------------|----------------|-----------------------------|-----------------|
| v370   | 0                    | 0                                             | 737 (RT)       | 5.8\times10^{25}            | 2.3             |
| v773   | 0                    | 0                                             | 773            | 1\times10^{26}              | 2.2             |
| d370   | \(2\times10^{21}\)  | 8.4                                           | 737 (RT)       | 3.0\times10^{25}            | 1.3             |
| d773   | \(1.5\times10^{21}\) | 6.3                                           | 773            | 1.2\times10^{25}            | 0.8             |

3. Experimental results and discussion

Deuterium thermal desorption spectra for the four samples are given in figure 1. The total deuterium retention for all samples is compared in figure 2. The data for the damaged sample are multiplied by 10 to be plotted in figure 1.

![Figure 1](image)

**Figure 1.** Deuterium thermal desorption spectra (D₂) for damaged and virgin W samples irradiated in deuterium plasma.

In the case of implantation at room temperature, deuterium desorption takes place in a wide temperature range from 450 K to 1400 K with several peaks. The maximum desorption flux is at 800 K for both (damaged and virgin) samples, but the number of deuterium is slightly higher in the damaged sample. In the case of implantation at 773 K, the desorption starts at the temperature of
implantation. For the virgin sample, the spectrum has one pronounced desorption peak with the maximum at 1100 K. For the damaged sample, the spectrum has a maximum at about the same temperature of 1050 K and an additional maximum at 1200 K. The striking effect is that the number of deuterium in the damaged sample is significantly less than that in the virgin sample.

Deuterium after plasma implantations is retained mainly due to trapping in intrinsic defects and defects produced by deuterium implantation and radiation damage. Diffusion of deuterium into the W bulk in presence of both types of defects is low at low temperatures, so one can suggest that deuterium is trapped mainly in a thin near surface layer below the carbon ion range. TDS from the virgin and damaged samples for low temperature implantation take place nearly in the same temperature range. This can mean that intrinsic and radiation defects in W are of the same type, and the difference is only in the quantity of defects. The deuterium retention for low temperature plasma implantation is nearly the same for the virgin and damaged samples, and it is only slightly higher for the damaged sample. This means that the net concentration of radiation defects is less than that of intrinsic defects in the region of trapping at low temperature. One can conclude from this that the trapping takes place very close to the surface well below the C ion range, where the concentration of radiation defects is very small as it was discussed above. The obvious difference between the amplitudes of two peaks in the spectra for the damaged and virgin samples is due to difference in the number of defects of different binding energies in the virgin and damaged samples.

Figure 2. Total deuterium retention in four samples.

Defect trapping usually decreases with temperature, and one can see that this is the case also in these experiments both for the virgin and damaged samples. At the elevated temperature of implantation, defects with a low binding energy do not retain deuterium during implantation. This is why TDS after implantation at 773 K have only high temperature parts that start from the temperature of implantation.

The prominent effect for the virgin sample is that the high temperature part of TDS after irradiation at 773 is more intensive than after irradiation at RT. The high temperature peaks in TDS can be explained either by defects of higher binding energy or by trapping in and release from deeper layers of the material, which are far from the surface. If to accept that the defect structure in the virgin sample does not change significantly in the temperature range below 773 K, one may suggest that the high temperature peak for the virgin sample implanted at 773 K is due to intrinsic defects that are deeper in the sample bulk. These defects are populated due to deuterium diffusion. With increase of the temperature, diffusion rate increases, traps far from the surface populate, and the high temperature part of TDS increases. This is what one can see in figure 1 for the virgin sample. Moreover the total
amount of deuterium trapped in the virgin sample only slightly decreases with temperature. Diffusion into the bulk is suggested also in [4, 5] and some other experiments.

The high temperature peaks for the damaged sample seem to be also due to defects placed far from the surface outside the D implantation range, but increase of temperature leads to decrease of the high temperature TDS peaks and drastic decrease of the total number of the deuterium trapped in the damaged sample. This can be explained if deuterium diffusion into the bulk at high temperatures is blocked in some way after irradiation by carbon ions. The opposite and expectable result was observed in [4, 5]: retention in a W sample damaged by MeV tungsten ions increased, though the damage level in our experiment was remarkably higher than in [5]. The difference between [5] and this work could be due to effect connected to implanted carbon atoms, which may be the reason of the diffusion barrier for deuterium atoms. If this barrier is connected with C irradiation, it must be placed at the implantation range of carbon, which is behind the implantation range of deuterium. Due to this barrier, deuterium can not diffuse deep into the bulk even at 773 K, and both the high temperature peaks and total number of deuterium in TDS decrease due to irradiation by high energy carbon ions. According to calculations, the main part of carbon atoms was concentrated at the depth of 3.5 µm, while the concentration near the surface was less than 0.01 at.%.. The consideration given above leads to the conclusion that the high temperature peaks correspond to deuterium trapped at depths much larger than the carbon ion range. The retained fluences of deuterium in the virgin and damaged samples implanted at 773 are similar, but the depth distributions are very different. The virgin sample has a rather high D concentration at small depths close to the D implantation range, while the damaged sample has a very small D concentration with a wide concentration profile spreading well behind the range of C ions.

The barrier effect of carbon and nitrogen was proposed also for explanation of experiments on ion-driven deuterium permeation [6] and deuterium accumulation [7] in tungsten: it was observed that both permeation and accumulation increased if impurities (C, N) were added to the incident flux. This was explained by formation of a thin barrier layer on the surface, which prevented re-emission of implanted deuterium back in vacuum.

4. Summary
Deuterium trapping in tungsten damaged by 10 MeV C³⁺ ions was investigated by means of thermal desorption spectroscopy. The damage led to some increase of D trapping at 370 K but drastic decrease of deuterium trapping at 773 K in comparison with the virgin sample. The experimental data were interpreted in the following way:

Trapping is due to both intrinsic and radiation induced defects.
At 370 K deuterium diffusion is slow in the field of defects and deuterium is trapped to a high concentration in the near surface region not far from the D implantation range.
At 773 K deuterium diffusivity is high enough to transport D atoms into the W bulk. Therefore deuterium diffuses far from the surface, its concentration is very small but the diffusion length is high, so the integral trapping is about as high as at 370 K implantation.
Carbon irradiation produces a diffusion barrier behind the D implantation range that prevents D diffusion into the W bulk. Therefore in the case of D implantation at 773 K, the number of trapped deuterium drastically decreases in the sample pre-irradiated by C ions.

Acknowledgements
This work was supported in part by National Research Nuclear University MEPhI in the framework of the Russian Academic Excellence Project (contract No. 02.a03.21.0005, 27.08.2013).

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