

Saturation of deuterium retention in self-damaged tungsten exposed to high-flux plasmas.

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Abstract. Polycrystalline, annealed tungsten targets were bombarded with 12.3 MeV W⁴⁺ ions to various damage levels. Deuterium was implanted by high-flux plasmas in Pilot-PSI ($>10^{24}$ m⁻²s⁻¹) at a surface temperature below 525 K. Deuterium retention has been studied by Nuclear Reaction Analysis and by Thermal Desorption Spectroscopy. We found that deuterium retention is strongly enhanced by the tungsten bombardment and that saturation occurs at a W⁴⁺ fluence of about $3 \cdot 10^{17}$ m⁻². The maximum deuterium concentration in the damaged region was measured to be 1.4 at.%. This is in accordance with other experiments that were carried out at much lower fluxes. We therefore conclude that the saturation behaviour and the maximum retention are not affected by the high fluxes used in our experiments.

A simple geometric model is presented that assumes that the saturation solely originates in the tungsten irradiation and that explains it in terms of overlapping saturated volumes. The saturated volume per incident MeV ion amounts to $3 \cdot 10^4$ nm³. From our results, we are able to obtain an approximate value for the average occupation number of the vacancies.

1. Introduction

In future magnetic fusion devices as ITER, tungsten is foreseen as divertor material. The divertor has to withstand high density, low temperature plasma fluxes. Good thermal properties as its high melting point, high thermal conductivity and low erosion rate make tungsten favourable over other materials. However, there is concern that hydrogen isotopes are retained in tungsten. Firstly, for safety reasons, it has been decided for ITER that the tritium inventory should be kept below 700 g [1]. Secondly, for efficiency reasons, it is important that the fuel is not retained in the wall. Understanding the retention properties of W is important for ITER and for fusion reactors beyond ITER. Continuous bombardment with 14.1 MeV neutrons degrades material properties and introduces damage into the material. In this paper we focus on the influence of damage in tungsten on deuterium retention.

Several studies have shown that irradiation damage strongly enhances deuterium retention [2] [3] [4]. Tyburska et al [2] noticed that a linear increase of damage did not lead to a linear increase of deuterium stored in the sample and that the defects can be removed by annealing. Saturation of the deuterium retention was observed at 0.3 displacements per atom (dpa), independent of implantation fluence. Wright et al [3], who did similar experiments at a much higher flux, showed that the deuterium retained in damaged tungsten saturates below 0.22 dpa.

The main aim of this investigation is to determine the irradiation damage level where deuterium retention in tungsten saturates for high-flux plasmas, and to understand the saturation behaviour. A systematic study of deuterium retention in tungsten as a function of target temperature and irradiation damage level was performed. Analysis was done by Nuclear Reaction Analysis and Thermal Desorption Spectroscopy. The results are explained by a simple geometric model based on overlapping volumes.

2. Experiment

2.1 Target preparation

Polycrystalline tungsten targets (PLANSEE, 99.96 % purity) were annealed for 1 hour at 1273 K at a background pressure of $5 \cdot 10^{-4}$ Pa. The dimensions of the targets are 20 mm in diameter and 1 mm in thickness. In the present experiments, unpolished targets were used. The cleanliness of the targets was checked by XPS before and after plasma exposure. In both cases, the XPS signal was dominated by carbon (40-70 %) and oxygen (20-40 %). This can be ascribed to native carbon and oxide layers of a few nanometres thick, present on the targets due to their exposure to air. At ambient conditions hydrocarbons are deposited onto the tungsten target. During plasma exposure, the chemical erosion of carbon [5] is an efficient process and formation of water removes the oxide layer. It is therefore likely that during plasma exposures no carbon nor oxide was present. The tungsten signal was only about 10 %. This low signal is partly caused by the rough surface of the target; polished samples showed an increase of the signal by a factor 3. With XPS, trace amounts of copper and calcium were measured after Pilot-PSI [6] exposure. They were deposited on the targets during plasma exposure. Their contribution to the XPS signal was very limited and well below 1 %.

2.2 W^{4+} irradiation

Damage caused by neutron irradiation was simulated by heavy ion bombardment. Radiation damage by neutrons and by heavy ions is to some extent comparable [7]. For both neutrons and heavy ions, the ratio of low-energy to high-energy primary knock-on atoms is low, and they both create dense collision cascades [8]. However, one should realize that there are also differences between heavy ion and neutron irradiation. Damage created by heavy ions is concentrated in a narrow region below the surface, while neutron damage exhibits a homogeneous defect concentration very deep in the material. Furthermore, the detailed nature of the defects is unclear and may be different and neutron collisions will lead to transmutations whereas heavy ions will not.

The samples were bombarded at normal incidence with 12.3 MeV W ions. The W^{4+} ions, accelerated through a potential difference of 3.075 MV, were implanted with the 3 MV tandem ion accelerator at IPP-Garching. In order to obtain homogeneous irradiation, the beam ($\varnothing \sim 3$ mm) was raster scanned across the surface. The area damaged by the tungsten bombardment was 12 mm in diameter. The damage rate at which the targets were damaged was $(3 \pm 1) \cdot 10^{-4}$ dpa s^{-1} . During the tungsten irradiation, the targets were kept at room temperature. To predict the irradiation damage, calculations were done with the binary collision transport code SRIM^a. SRIM profiles were calculated for 0 K. The absolute level of displacement damage decreases approximately linearly with an increase of the displacement threshold energy (E_d). In literature, a wide range of values has been used: Sakamoto et al [9] and Tokunaga et al [10] used 40 eV

^a www.srim.org

determined by Lucasson et al [11]. Maury et al [12] measured by electron irradiation a value of 42 ± 1 eV in the $\langle 100 \rangle$ direction and 44 ± 1 eV for $\langle 111 \rangle$ orientation. Xu et al [13] showed with molecular dynamics simulations that the displacement threshold energy is indeed strongly dependent on the crystal orientation and ranges from 68 eV up to 250 eV. Others, like Ogorodnikova et al [14], use an average displacement energy of 90 eV as reported by [15]. In this article the average value of 90 eV is used, unless stated otherwise.

The SRIM results are shown in figure 1. Damage profiles extend to 1.5 μm below the surface and have a maximum damage level at about 0.8 μm depth. The targets were subjected to W^{4+} ion fluences up to $6.5 \cdot 10^{17} \text{ m}^{-2}$, which corresponds to peak damage levels of up to 0.45 dpa. These peak damage levels are used as reference for our experiments. For comparison to the results of Tyburska et al, the 0.4 dpa damage profile created by 5.5 MeV W^+ ions is also depicted. It is clear that 12.3 MeV ions penetrate deeper in the material and that the maximum damage level occurs at larger depth.

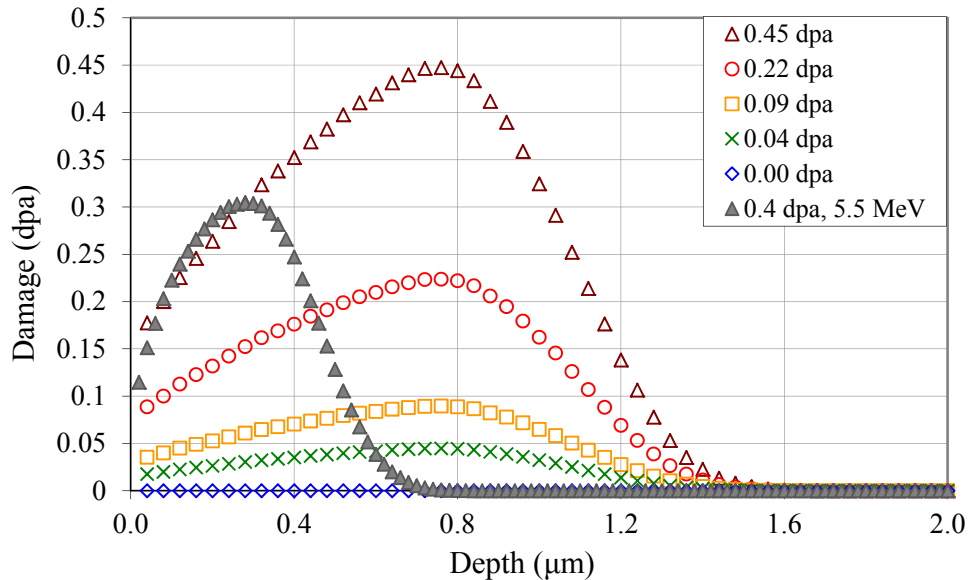


Figure 1: SRIM results of damage distribution for tungsten irradiated by 12.3 MeV W^{4+} ions. For comparison to the results of Tyburska et al [2], the 0.3 dpa damage profile created by 5.5 MeV W^+ ions is depicted as well (reconstructed from [2] and scaled to $E_d = 90$ eV).

2.3 Deuterium implantation by plasma exposure

Pilot-PSI is a linear plasma generator for plasma surface interaction (PSI) studies in support of the international fusion experiment ITER [6] [16]. Deuterium plasma is produced by a cascaded arc, which facilitates the production of unique plasma conditions. Particle fluxes of up to $10^{24} \text{ m}^{-2}\text{s}^{-1}$ are obtained with energy fluxes up to 10 MW/m^2 and electron temperatures of 0.5–5 eV. The damaged targets have been exposed for 100 s in Pilot-PSI in an axial magnetic field of 0.4 T.

Electron density and temperature of the plasma beam were determined by the Thomson scattering technique [17]. The plasma beam has a Gaussian profile with an electron density of about $3.0 \cdot 10^{20} \text{ m}^{-3}$ in the centre and a full width half maximum of ~ 1 cm. The maximum electron temperature was measured to be 0.7 eV.

During plasma exposure, the targets were electrically floating. Assuming that the ion temperature equals the electron temperature and using the Bohm criterion [18], the peak deuterium ion plasma flux can be determined to be $1.2 \cdot 10^{24} \text{ m}^{-2} \text{ s}^{-1}$. The flux on and the surface temperature of the target are shown in figure 2. The deuterium implantation area (\varnothing 16 mm) covered the pre-irradiated area. The surface temperature is determined by the heat flux of the plasma and by cooling of the target. In all experiments, temperature profiles of the surface of the target have been measured with a fast infrared camera (FLIR SC7500-MB). The maximum surface temperature was 525 K \pm 10 K. The emissivity of tungsten at these temperatures is very low and dependent of the surface structure. It is therefore possible that a systematic error of maximum 30 K was introduced by an error in emissivity coefficient. The cooling of the target was very effective; after switching off the plasma, the temperature of the targets was back to room temperature within typically 1-2 seconds.

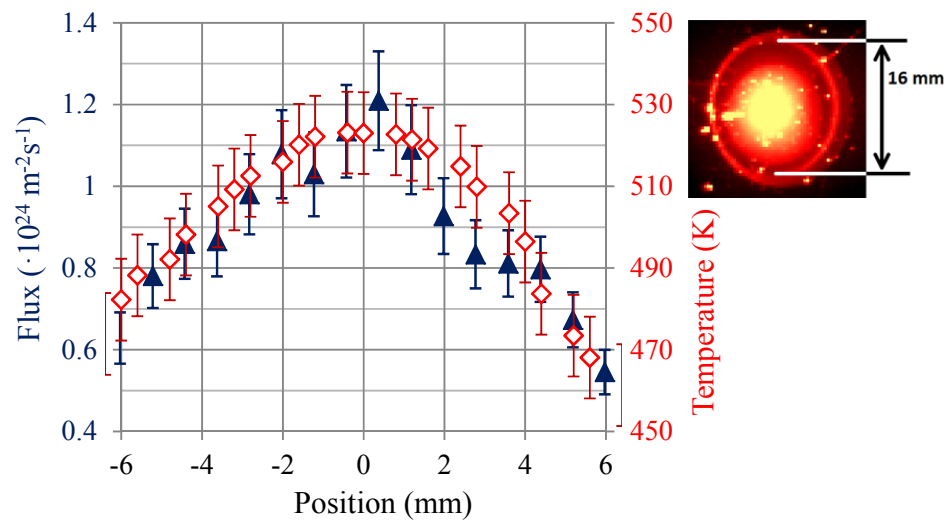


Figure 2: Radial dependence of the plasma flux (closed triangles), and surface temperature (open diamonds). The insert shows a typical infrared image.

2.4 Nuclear Reaction Analysis

Nuclear Reaction Analysis (NRA) of the targets was performed at IPP-Garching four weeks after plasma exposure. The nuclear reaction $D(^3\text{He}, p)^4\text{He}$ was used to obtain a local measurement of the deuterium concentration as function of depth [19] [20]. The ^3He beam spot of 1 mm in diameter was positioned at four spots on the target to extract a radial scan. At each position, the energy was scanned from 690 keV to 4.0 MeV to determine the deuterium concentration with a resolution of $\sim 0.5 \text{ } \mu\text{m}$ down to $6 \text{ } \mu\text{m}$ depth. The depth profiles of the retained deuterium were calculated from the measured proton energy distributions by use of the SimNRA program [21]. The surface roughness of the targets did not allow us to extract information from the alpha spectrum to obtain a near surface D concentration with a better resolution. The total amount of deuterium present in the top $6 \text{ } \mu\text{m}$ of the target was obtained by integrating the depth profiles.

2.5 Thermal Desorption Spectroscopy

The targets were analysed with Thermal Desorption Spectroscopy (TDS) at FOM-Rijnhuizen seven weeks after plasma exposure. The tungsten target was clamped to a ceramic heater and heated with a linear

temperature ramp of 1 K/s to 1273 K. A Balzers QMA125 Quadrupole Mass Spectrometer (QMS) monitored the mass 4 (D_2) and mass 3 (HD) signals in the residual gas in the chamber, to determine the total amount of deuterium released from the target during the temperature ramp. The absolute sensitivity was determined using calibrated leaks of H_2 and D_2 . For the sensitivity of the mass 3 signal, the average of the sensitivities of mass 2 and mass 4 was taken.

3. Results

3.1 Nuclear Reaction Analysis

NRA was carried out at several positions on the target to obtain deuterium depth distributions as function of temperature and flux. The results are shown in figure 3a-d. Also the retention for an undamaged target is shown. From figure 3, it is clear that W^{4+} pre-irradiation strongly enhances deuterium retention. The deuterium retention decreases as function of depth. The deuterium retention beyond 3 μm was below the detection limit of 10 ppm.

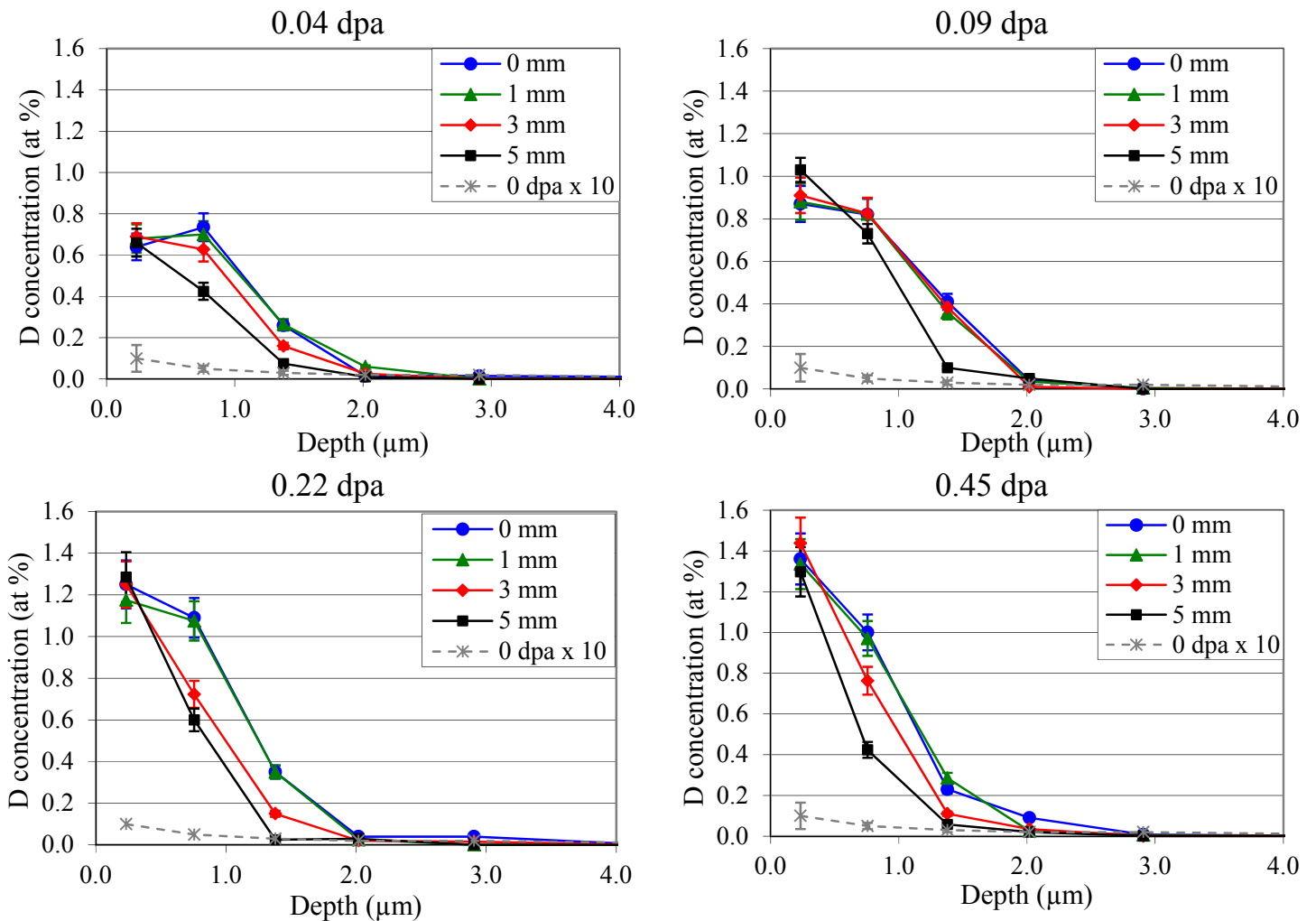


Figure 3: Depth distributions of retained deuterium in damaged tungsten targets, pre-irradiated to respectively (a) 0.045, (b) 0.09, (c) 0.22 and (d) 0.45 dpa. The shown D depth profiles were measured at 4

spots on the target: in the centre (~525 K); 1 mm off centre (~520 K); 3 mm off centre (~510 K); 5 mm off centre (~480 K). The grey data points and dotted line indicates the deuterium retention of an undamaged tungsten target (multiplied by ten).

At the measurement points in the centre of the target and 1 mm off center, which were exposed to the highest particle flux and which had the highest surface temperature, the deuterium was found deepest in the material. This agrees with the observations of Wright et al [3], who found that in this temperature regime, deuterium retention is primarily determined by diffusion: at the highest surface temperature, deuterium diffuses deepest into the tungsten material (figure 3).

For the measurements nearest to the surface (0.25 μm), deuterium retention is independent of position and thus of surface temperature and particle flux. Deuterium retention does clearly increase with damage level, which indicates that the retention at the surface is mostly determined by the defect concentration. The maximum in deuterium retention is about 1.4 at.%, which agrees well with the maximum deuterium concentration of 1.5 at.% found by others [2] [3] [22]. Both Tyburska et al [2] and Fukumoto et al [22] found an additional surface effect, where the retained deuterium is much higher in a very thin layer at the surface. By investigation of the surface morphology, it became clear that these high values originated from cracks and blister formation. This surface effect can also be observed in our experiments, as is clear from the experiments on the undamaged target, but only gives a minor contribution to the retention of the damaged targets (caused by the limited depth resolution of 0.5 μm).

The measured deuterium retention profiles do not match the calculated damage profiles as shown in figure 1, with the measured retention deeper in the material being significantly lower than expected from the damage profiles. This also holds when the effect of saturation on the damage profiles is considered, leading to flatter profiles. The mismatch is probably caused by the limited diffusion. This is supported by the fact that for a specific target, the retention at the surface is the same for all positions whereas deeper in the material retention is larger in the center (where flux and temperature are higher).

The integrated NRA data, i.e. the deuterium retained in the first 6 μm of the target, is shown in figure 4. Clearly, the retention is close to its saturation value at a W^{4+} pre-irradiation fluence of $3 \cdot 10^{17} \text{ m}^{-2}$. Using a value of 90 eV for the displacement energy, this corresponds to 0.2 dpa.

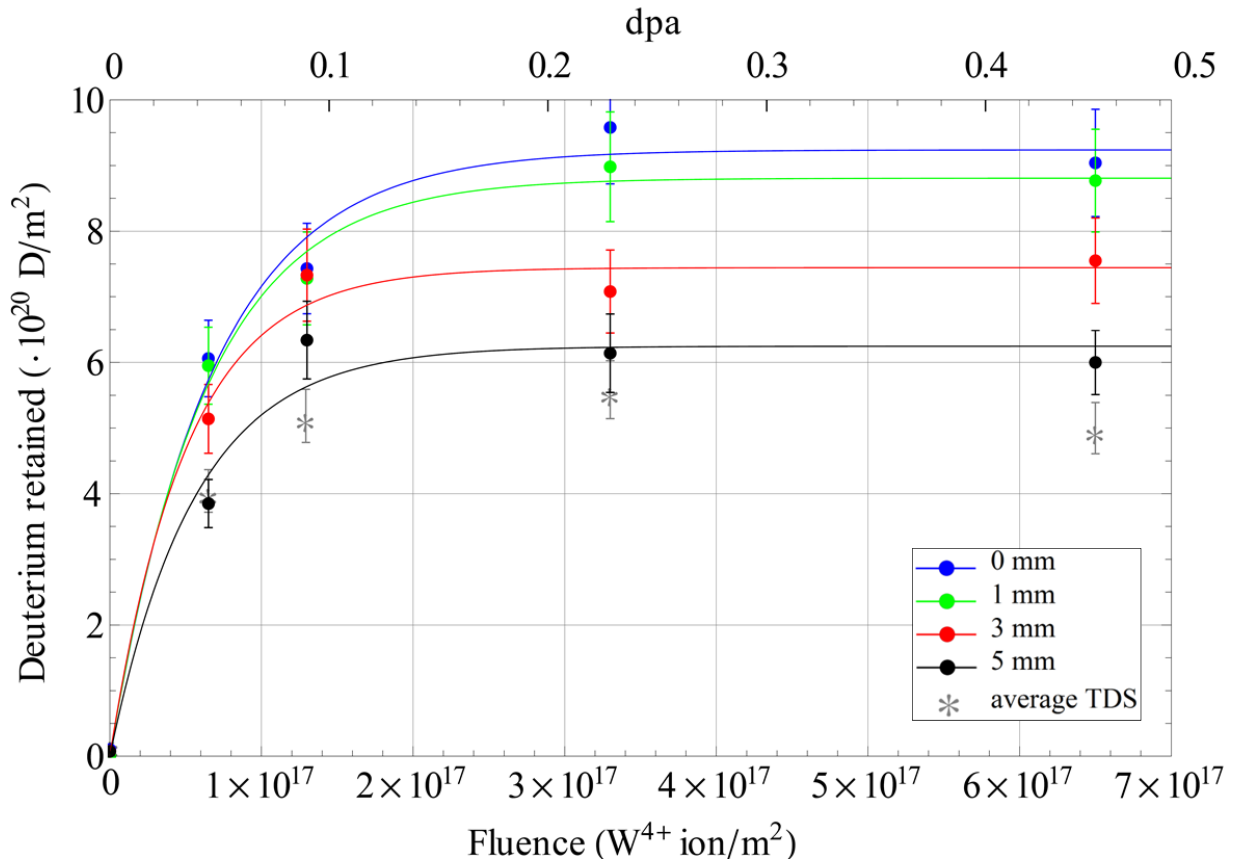


Figure 4: Amount of deuterium retained in the first 6 μ m as a function of pre-irradiation W ion fluence. Deuterium retention is significantly enhanced by W^{4+} pre-irradiation and increases with pre-irradiation damage. Saturation of deuterium retention is reached around 0.2 dpa. A comparison with the TDS data is made by dividing the total amount of deuterium desorbed during TDS by the surface area.

3.2 Thermal Desorption Spectroscopy

We investigated our targets with TDS to get information on the defect types in which the deuterium is trapped. The TDS results for the D_2 signal are shown in figure 5. The undamaged target shows a small low temperature peak at around 550 K, caused by deuterium trapped in intrinsic defects usually associated with dislocations and grain boundaries [3]. It is likely that these naturally exist in the material, as the annealing temperature of 1273 K is still below the recrystallization temperature of tungsten. Since the deuterium ion energy is much lower than the displacement threshold of tungsten, it does not seem likely that these defects are created by plasma exposure. However, several mechanisms have been suggested for defect creation by deuterium ion implantation below this threshold, e.g.: plastic deformation by deuterium super-saturation [23] and creation of vacancies by recoil implantation of carbon and oxygen impurities arriving at the tungsten surface [24].

Pre-irradiation of the targets enhances the intensity of the low temperature peak and introduces a high temperature peak at about 850 K. The enhancement and broadening of the low temperature peak towards higher temperatures can be explained by defect creation. In literature, various defect types have been identified in the temperature range 400-650K, i.e., dislocations and grain boundaries [25] [26], and

single vacancies (with one [26] [27] or two D atoms trapped [28]). The high temperature peak is usually associated with deuterium trapped in vacancy clusters [22] [25] [29]. The high temperature peak measured by Wright et al [3] is significantly less pronounced than the one in our experiments. This might be caused by the lower surface temperature during plasma exposure as used in their experiments (480 K), which limits the clustering of vacancies.

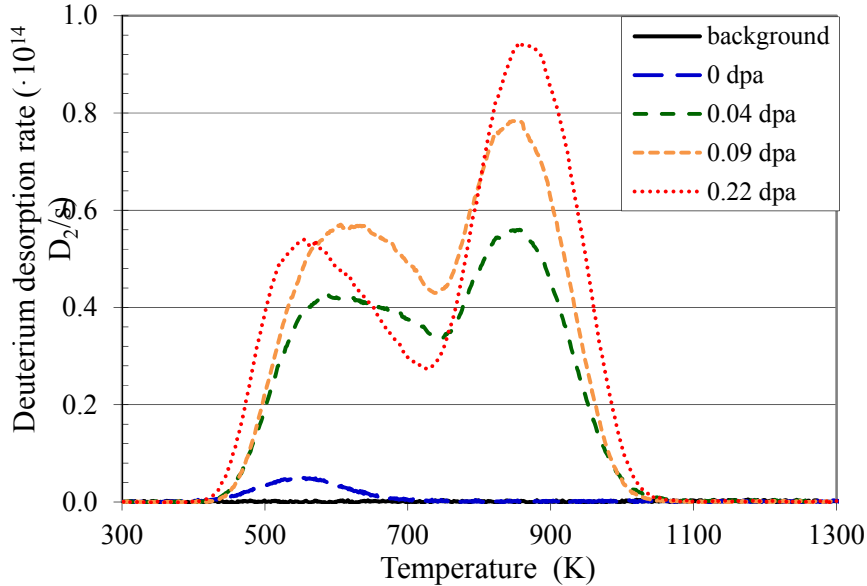


Figure 5: Thermal desorption spectra (D_2 signal) of the targets, temperature ramp was 1K/s. The background signal was measured at a target that was not irradiated by tungsten ions, nor exposed to deuterium plasma, and is virtually 0 at all temperatures. Unfortunately, the measurement at 0.45 dpa was not successful due to technical problems in the heating rate. However, the integrated TDS profile could be used to calculate the total deuterium retention.

The total deuterium retained by TDS divided by the damaged surface area (\varnothing 12 mm) is shown in figure 4. The average value obtained by TDS is somewhat lower than the local D retention (NRA) at 5 mm off-center. This may be caused by a misalignment between the center of the plasma beam and the center of the self-implantation area of 1 mm.

4. Model

The deuterium retention in the first 6 μ m underneath the surface as function of pre-irradiation fluence is shown in figure 4. The retention clearly saturates at a W^{4+} fluence of about $3 \cdot 10^{17} \text{ m}^{-2}$. We developed a simple geometric model that assumes that the saturation solely originates in the tungsten pre-irradiation and that explains this saturation in terms of overlapping volumes.

The model is an extension of a 2D model, where a surface with area A is randomly covered with objects with surface area a . The expectation value of the fraction $f(n)$ of surface A covered with n such objects is given by the following differential equation:

$$\frac{df}{dn} = \frac{a}{A} [1 - f(n)] \quad (1)$$

The solution of this equation is given by:

$$f(n) = 1 - \exp(-n a/A) \quad (2)$$

MeV ions bombarding a material create Frenkel pairs. At sufficiently high pre-irradiation fluence, the material becomes saturated with Frenkel pairs: a new vacancy created will immediately recombine with an interstitial present in the material. This spontaneous recombination leads to a maximum concentration of vacancies. Each single vacancy occupies an average spontaneous recombination volume, V^{vac} .

Each individual MeV ion creates a unique, very complex, 3D distribution of vacancies. SRIM simulates these individual distributions and calculates, among others, the number of vacancies as function of depth averaged over many MeV ions. The shape of this average vacancy distribution as function of depth d , $vac_{\text{SRIM}}(d)$, resembles the graphs shown in figure 1. In our model we assume that, instead of a unique distribution, each individual MeV ion creates this average vacancy distribution. The average effective damage volume of an ion, $V_{\text{eff}}^{\text{ion}}$, can be expressed in terms of V^{vac} in the following way:

$$V_{\text{eff}}^{\text{ion}} = V^{\text{vac}} \cdot \sum_{\text{layers}} vac_{\text{SRIM}}(d) \quad (3)$$

$V_{\text{eff}}^{\text{ion}}$ represents the damage volume created by a single MeV ion saturated with vacancies. With the MeV ions incident at random positions on the target, the damage volumes will start to overlap as soon as the fluence is sufficiently high, and saturation occurs.

We extended the 2D case (equation 2) to a 3D model by splitting up the target in N layers, parallel to the surface and of equal thickness. The surface area a in equation 2 has to be taken different for each layer. The relative values are determined by the shape of $vac_{\text{SRIM}}(d)$. We furthermore replace the fraction $f(n)$ by the deuterium retention. The model leads to the following function, where the summation is taken over the N contributing layers:

$$D_{\text{ret}}(F) = \frac{D_{\text{sat}}}{N} \sum_{\text{layers}} \{1 - \exp[-F \cdot vac_{\text{SRIM}}(d) \cdot V^{\text{vac}}/\Delta d]\} \quad (4)$$

Here, $D_{\text{ret}}(F)$ is the deuterium retention as function of the pre-irradiation fluence F and D_{sat} the maximum deuterium retention when the sample is saturated with vacancies. The thickness of the layers is given by Δd . The function (equation 4) was fitted to the four sets of data points shown in figure 4 with D_{sat} and V^{vac} as fit parameters. Note that, although $vac_{\text{SRIM}}(d)$ and V^{vac} are both directly affected by the choice of E_d , $V_{\text{eff}}^{\text{ion}}$ is not.

In figure 4, the data with the fits are shown. It is clear that the fit function describe the measurements quite well. The deuterium retention from a single MeV ion, incident on a non-irradiated target, amounts to $1.3\text{-}1.7 \cdot 10^4$ D/ion, which can be directly obtained from the slope at 0 fluence. The average effective damage volume of an MeV ion, $V_{\text{eff}}^{\text{ion}}$, was found to be $(3 \pm 1) \cdot 10^4$ nm³.

5. Discussion

We have studied deuterium retention for targets pre-irradiated with 12.3 MeV W^{4+} ions and subsequently exposed in Pilot-PSI to high-flux plasmas. From integrated NRA data, it is clear that saturation of deuterium retention takes place at a W^{4+} fluence of about $3 \cdot 10^{17}$ ions m^{-2} , which corresponds to ~ 0.2 dpa. Since the absolute value of the created damage is inversely proportional to the displacement threshold energy, we can rescale the saturation point found by Tyburska et al [2] to ~ 0.3 dpa ($E_d = 90$ eV), measured at fluxes of about 10^{22} $m^{-2} s^{-1}$. Since these values are very close and, in addition, since the maximum retention levels are quite similar, we conclude that the high flux of $>10^{24}$ $m^{-2} s^{-1}$ as used in the present experiments does not significantly affect deuterium retention. This agrees with our model that assumes that the saturation behaviour solely originates in the damage created before the D irradiation.

From the analysis of our measurements, we were able to obtain the average effective damage volume of the MeV ions used in our experiments and the number of deuterium atoms retained per MeV ion. With this information an estimate of the occupation number of the vacancies can be made.

We have estimated the number of vacancies created by an MeV ion in two different ways. The first method uses the number of Frenkel pairs derived from the Norgett-Robinson-Torrens (NRT) formula [30]. The integrated damage energy output files of SRIM were used to calculate the number of Frenkel pairs created by each MeV ion: on average $2.0 \cdot 10^4$ vacancies. Note that the calculated number of vacancies depends on the choice made for E_d . However, the actual number of vacancies per incident ion is lower, since the NRT formula does not take into account the recombination of defects produced in the collision cascade itself. Molecular Dynamics simulations (MD) have shown that the recombination drastically decreases the amount of vacancies [31] [32]. Troev et al [32] used molecular dynamics simulations of tungsten to determine the cascade efficiency, defined as the ratio between Frenkel pairs calculated by MD and the theoretical number of Frenkel pairs derived by the NRT formula. They found that 20-25% of the Frenkel pairs survive, the rest annihilates. The damaged volume of the MeV ion is then left with $4\text{-}5 \cdot 10^3$ vacancies. The second method uses the spontaneous recombination volume of Frenkel pairs. From our experiments we found an average effective damage volume of an MeV ion of about $3 \cdot 10^4$ nm^3 . In literature, the spontaneous recombination volume of Frenkel pairs in tungsten is reported to be in the range of 100-200 at. vol [33] [34] or $1.6\text{-}3.2$ nm^3 . Hence, the effective damage volume corresponds to $(0.9\text{-}1.9) \cdot 10^4$ vacancies. The results from the two methods are somewhat different, and further investigation is needed.

From our experiments, we deduced a number for the amount of retained deuterium: $(1.3\text{-}1.7) \cdot 10^4$ deuterium atoms per incident MeV ion. Dividing this experimental number by the number of vacancies as discussed above, yields a value for the average occupation number of a vacancy of 2.6-4.2 for method 1 and 0.7-1.8 for method 2. It seems that even though the fluence as used in our experiments was not sufficiently high to fully saturate all created vacancies (diffusion limited, see discussion figure 3), the average occupation number is at least close to 1. At longer exposure times, therefore, deuterium retention will be enhanced and the average occupation number is anticipated to increase further.

6. Conclusions

Polycrystalline, annealed and pre-damaged W targets were exposed to high-flux plasmas in Pilot-PSI at a surface temperature below 525 K. The deuterium retention has been investigated by NRA and TDS. We found that the deuterium retention is enhanced by pre-irradiation with 12.3 MeV W ions and that it

saturates at W ion fluence of $\sim 3 \cdot 10^{17} \text{ m}^{-2}$, which corresponds to $\sim 0.2 \text{ dpa}$ ($E_d = 90 \text{ eV}$). We found that the deuterium saturation retention in the damaged W is not significantly affected by the high fluxes of $\sim 10^{24} \text{ m}^{-2}\text{s}^{-1}$ used in our experiments, and that it is dominated by the pre-irradiation damage. This is in accordance with the fact that our data fit quite well to the simple geometric model, which assumes that the saturation of deuterium retention in the tungsten material solely originates in the W^{4+} self-implantation. We further found that the average occupation number of the vacancies is at least close to 1 and that it will increase for higher fluencies.

We used heavy ions (W^{4+}) to simulate the effect of neutron irradiation with heavy ion bombardment on tungsten as present in the divertor of fusion reactors like ITER. Assuming neutron damage to be comparable to damage from heavy ions, the hydrogenic retention would amount to a maximum value of 1.4 at.% for surface temperatures below 525 K. At higher surface temperatures the retention is expected to be less, because of annealing of the vacancies.

In the present study, we focussed on deuterium retention in the first few micrometers. Here, the deuterium retention is dominated by the pre-irradiation damage. Further investigations are needed on the effect of high flux plasmas on surface morphology, blistering etc. These studies will be carried out in the future.

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