



# Deuterium retention in tungsten at fluences of up to $10^{26}$ D<sup>+</sup>/m<sup>2</sup> using D<sup>+</sup> ion beams

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## ABSTRACT

Deuterium retention in two types of polycrystalline tungsten (PCW) was studied as a function of incident ion fluence, ion energy, and specimen temperature. (i) D retention at 300 K, as a function of D<sup>+</sup> fluence, demonstrated a trend to saturation in both the Rembar hot-rolled thin foil and Plansee tungsten plate. At 500 K, new D retention results for the Plansee PCW showed an increasing trend with increasing incident D<sup>+</sup> fluence without any indication of saturation, in agreement with previous results for Rembar PCW [A.A. Haasz, J.W. Davis, M. Poon, R.G. Macaulay-Newcombe, *J. Nucl. Mater.* 258–263 (1998) 889–895]. Even when the incident D<sup>+</sup> fluence was increased to  $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup>, which is in the fluence range of plasma devices, there was still no sign of saturation. (ii) The temperature dependence results for the Plansee PCW show a decreasing trend in D retention as the temperature is increased from 300 to 500 K. These results differ from previous studies of Rembar PCW [A.A. Haasz, J.W. Davis, M. Poon, R.G. Macaulay-Newcombe, *J. Nucl. Mater.* 258–263 (1998) 889–895], but are similar to those seen for single crystal tungsten [M. Poon, A.A. Haasz, J.W. Davis, R.G. Macaulay-Newcombe, *J. Nucl. Mater.* 313–316 (2003) 199]; an explanation for the different behaviour is suggested. (iii) Varying the D<sup>+</sup> energy from 100 to 500 eV/D<sup>+</sup> plays a minor role in the amount of D retained, suggesting that D retention in W depends more on the W structure, incident ion fluence and specimen temperature, rather than on the incident ion energy when the energy is below the threshold for damage formation ( $\sim 960$  eV for D on W).

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## 1. Introduction

Tungsten is one of the plasma-facing materials planned for the International Thermonuclear Experimental Reactor (ITER), primarily due to its high melting temperature, low physical sputtering yield, high energy threshold for physical sputtering, and resistance to chemical sputtering [1]. Since sputtered W atoms could cause high radiative losses if they reach the core plasma, the use of tungsten is proposed for regions with low plasma temperatures, corresponding to energies below the sputtering threshold. In addition to plasma contamination concerns, hydrogen retention is also a critical issue for ITER, as it can affect fuelling efficiency, plasma density control, and tritium inventory and permeation through the wall or into coolant channels. Therefore, the underlying mechanism of hydrogen trapping in tungsten has been widely studied, but with inconsistent results, especially when it comes to ion fluence and specimen temperature dependence.

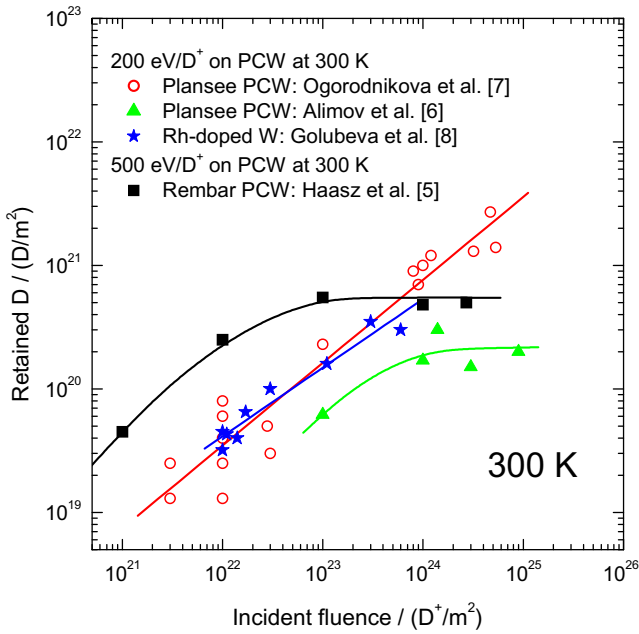
It is anticipated that the typical irradiation fluence in the ITER divertor will be  $\sim 10^{28}$ – $10^{29}$  DT/m<sup>2</sup> [1]. Linear plasma devices with high flux densities ( $\sim 10^{22}$  D<sup>+</sup>/m<sup>2</sup>s), such as PISCES [2] and TPE [3,4], may be able to reach or come close to such high fluences. By comparison, ion beams, good at producing well-characterized ions, are

limited to fluxes of  $10^{17}$ – $10^{20}$  ions/m<sup>2</sup>s; incident fluences of  $10^{17}$ – $10^{26}$  ions/m<sup>2</sup> can be obtained by varying both fluxes and irradiation times. While these fluences may be able to simulate wall fluences in tokamaks quite well, they fall way short of simulating fluences in the divertor. Therefore, estimation of hydrogen inventories in the ITER divertor from ion-beam data requires extrapolating the lower fluence results.

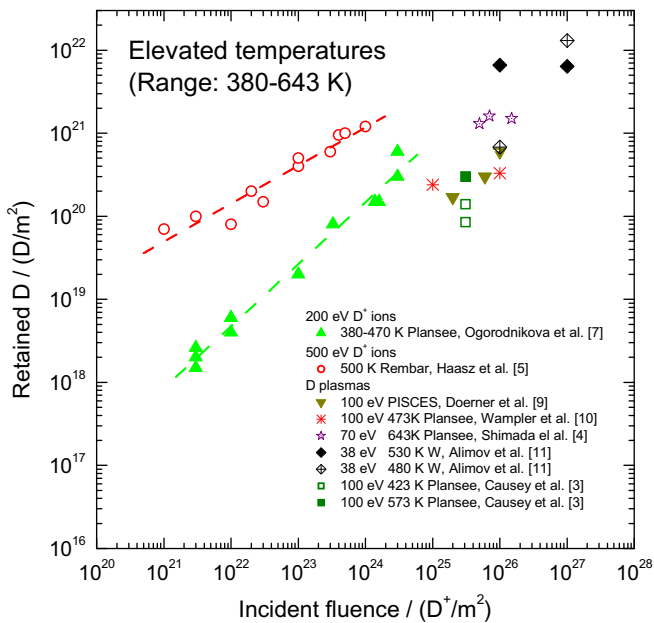
Previously published results on the fluence dependence of D retention in W with ion beams cover for the most part the fluence range below  $\sim 10^{25}$  D<sup>+</sup>/m<sup>2</sup>; by comparison, retention data from plasma devices reach fluences of about  $10^{25}$ – $10^{27}$  D/m<sup>2</sup>. Some of the previously reported results on D retention at 300 K and elevated temperatures (range: 380–643 K, mostly around 500 K) are plotted in Figs. 1 and 2, respectively. Here are some key findings of previous studies.

- (i) From ion-beam experiments, for irradiations of W at 300 K and incident D<sup>+</sup> energies of 200 and 500 eV, D retention is seen to saturate for incident D<sup>+</sup> fluences above  $\sim 10^{24}$  D<sup>+</sup>/m<sup>2</sup> at retention levels of about  $(2\text{--}6) \times 10^{20}$  D/m<sup>2</sup> [5,6]; see Fig. 1. Exceptions are the results of Ogorodnikova et al. [7] and Golubeva et al. (for rhenium-doped tungsten) [8] where saturation is not evident. The results of Alimov for 200 eV irradiation [6] show a clear trend to saturation for incident fluences above  $2 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup>, which is similar to the

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**Fig. 1.** Fluence dependence of D retention in PCW irradiated with 200 and 500 eV/ $D^+$  at 300 K. (Data obtained with ion beams [5–8]).



**Fig. 2.** Fluence dependence of D retention in PCW irradiated with  $D^+$  at energies in the range 38–500 eV and temperatures in the range 380–643 K. (Data obtained with ion beams [5,7] and plasmas [3,4,9–11]).

results of Haasz et al. [5]; however, Alimov's saturation level is  $\sim 2 \times 10^{20} D/m^2$ , which is about three times lower than the level in [5]. A possible explanation might be the use of different techniques for trapped D measurement – thermal desorption spectroscopy (TDS) in [5] and nuclear reaction analysis (NRA) in [6].

- (ii) Again, with ion beams, for irradiations at 500 K, with similar incident ion energies, all studies report an increasing trend in D retention with increasing  $D^+$  fluence, although with different slopes:  $\Gamma_{\text{Retained}} \propto I_{\text{Incident}}^{0.5 \text{ to } 0.7}$ . For example, for irradiation with 500 eV/ $D^+$  at 500 K the slope is 0.5 [5] and for 200 eV/ $D^+$  at 380–470 K the slope is 0.7 [7]; see Fig. 2.

**Table 1**

Impurity content of the Rembar and Plansee polycrystalline tungsten used in this study: Rembar (>99.96 wt.% pure W); Plansee (>99.97 wt.% pure W; metallic purity without Mo).

Impurity	Rembar (appm)	Plansee (appm)	Impurity	Rembar (appm)	Plansee (appm)
Hydrogen (H)	<900	<900	Molybdenum (Mo)	<200	<200
Carbon (C)	<500	<500	Silver (Ag)	<10	<20
Nitrogen (N)	<150	<70	Iron (Fe)	<100	<100
Oxygen (O)	<350	<250	Aluminum (Al)	<100	<100

- (iii) Results obtained in plasma devices and tokamaks at around 500 K, and ion energies <100 eV, over the fluence range  $10^{25}$ – $10^{27} D/m^2$ , show a large spread in D retention levels with no consistent trend; see Fig. 2. Some data show an increasing trend with fluence (e.g., PISCES [9]), while others show little or no change with increasing fluence (e.g., Shimada and Kolasinski [4], Wampler and Doerner [10], Alimov et al. [11]). At around an incident fluence of  $10^{26} D^+/m^2$ , the retained D levels from plasma devices (not including tokamaks) span about two orders of magnitude ( $10^{20}$ – $10^{22} D/m^2$ ).

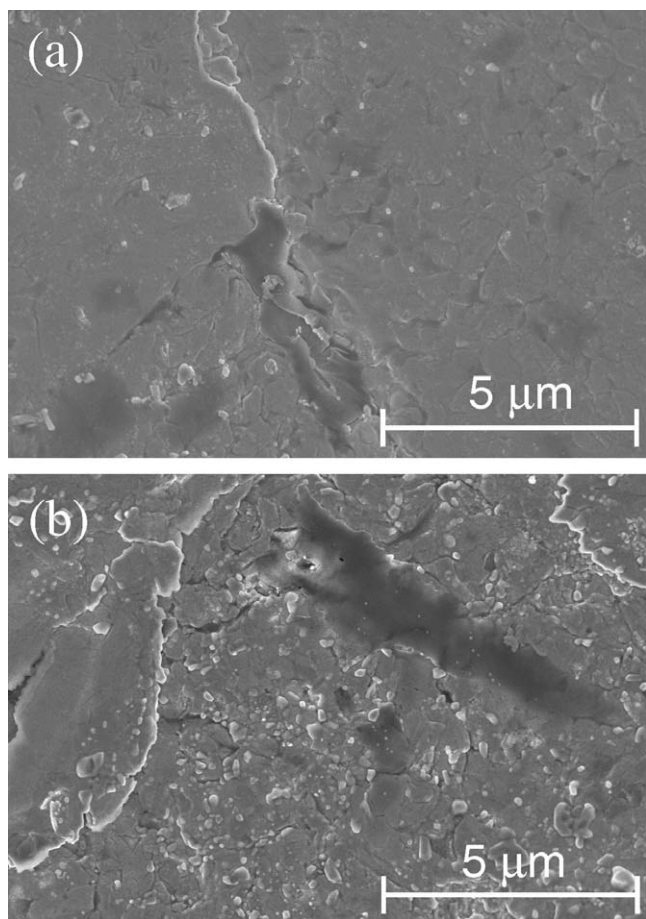
It is expected that the observed differences in the D retention results obtained by both ion beams and plasma devices are largely due to different material structures, specimen preparation procedures and irradiation conditions. From the available data for 500 K irradiations (see Fig. 2), it is not evident whether the amount of retained D will level off or continue to increase as ITER-relevant fluences are approached, i.e.,  $>10^{26} D^+/m^2$ . The present study was undertaken with the primary objective to extend the incident ion-beam fluence range (typically  $<10^{25} D^+/m^2$ ) to the high fluences achieved in plasma devices ( $\geq 10^{26} D^+/m^2$ ) to ascertain whether the increasing trend seen at lower fluences prevails. Also, with an attempt to resolve some of the differences in the existing D-retention database obtained with ion beams, we have undertaken to study the effects of material structure (Plansee 'plate' vs. Rembar 'foil'), incident ion energy, and specimen temperature on D retention in polycrystalline tungsten (PCW).

## 2. Experiment

### 2.1. Specimens

Two types of polycrystalline tungsten were used in this study: hot-rolled tungsten (25  $\mu\text{m}$  thick and 99.96 wt.% pure) produced by the Rembar Corporation and tungsten plates (1 mm thick and 99.97 wt.% pure) made by Plansee. The Rembar foil specimens were cut to  $5 \times 10 \text{ mm}^2 \times 25 \mu\text{m}$  strips and the Plansee specimens to  $5 \times 5 \times 1 \text{ mm}^3$  plates. The impurity content as provided by the manufacturers for both materials is shown in Table 1.

Prior to  $D^+$  irradiation, the specimens were first annealed at 850–950 K for 30 min, followed by cooling down over 2 h to avoid quenching. Specimens were then installed in the implantation chamber without any further chemical or mechanical treatment. For each implantation a new specimen was used to avoid any cumulative effect of repeated irradiations on the same spot [12]. We note that the 950 K maximum temperature reached in this study is much lower than that obtained in previous D retention experiments performed by our group at the University of Toronto, which was typically  $\sim 1500$  K. This happened inadvertently by using a chromel–alumel thermocouple (TC) instead of the high temperature W–Re TC (W–5% Re/W–26% Re) used in previous experiments. Assuming that we were using the W–Re TC, we stopped the heating at what we thought was  $\sim 1500$  K, which in fact turned out to be  $\sim 950$  K according to the chromel–alumel



**Fig. 3.** SEM images of a Plansee PCW plate (after TDS heating to 1200 K): (a) on the irradiated-spot, and (b) off-spot area. Irradiation at 500 K with 200 eV/D<sup>+</sup> to a fluence of  $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup>.

voltage–temperature calibration table. All temperatures obtained with the chromel–alumel TC have been re-derived based on the chromel–alumel calibration. We further note that some of the TDS was performed with the W–Re TC and these cases are explicitly identified in the text and the figure captions.

In order to examine the effects of surface structure, scanning electron microscopy (SEM) images were obtained for some of the specimens irradiated with 200 eV/D<sup>+</sup>,  $1 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup> at 300 K, subsequent to performing TDS. Comparison of the Plansee irradiated-spot and off-spot areas revealed no apparent difference in surface structure. In the case of the Rembar specimen, the irradiated area revealed some surface ‘smoothing’ caused by D<sup>+</sup> irradiation. SEM images were also obtained for a Plansee specimen irradiated at 500 K with 200 eV/D<sup>+</sup> to a fluence of  $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup> (our highest fluence). In this case, the irradiated spot (Fig. 3a) appears to be smoother than the off-spot area (Fig. 3b). Otherwise, no significant differences in surface structure are evident. This contrasts previous Rembar foil results for 500 eV/D<sup>+</sup> irradiation at 500 K where blister formation was seen already at  $1 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup> [12].

## 2.2. D<sup>+</sup> irradiation

All D<sup>+</sup> irradiations were performed in the ultra-high vacuum single-beam ion accelerator facility at the University of Toronto Institute for Aerospace Studies (UTIAS). D<sub>3</sub><sup>+</sup> ions bombarded the test specimens at normal incidence through a 1.5-mm diameter aperture with a flux density of  $4\text{--}7 \times 10^{19}$  D<sup>+</sup>/m<sup>2</sup>s. Final beam energies of 300–1500 eV D<sub>3</sub><sup>+</sup> (100–500 eV/D<sup>+</sup>) were achieved by decel-

erating a 3000 eV D<sub>3</sub><sup>+</sup> beam at the target by the application of a 1500–2700 V bias voltage. Beam currents were measured directly on the specimens. For irradiations performed at temperatures above 300 K, a ceramic heater was used to heat the specimens and the temperature was measured with a chromel–alumel thermocouple placed between the specimen and the mica insulator which separated the specimen from the aperture plate. Ion fluences  $>3 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup> required accelerator run times of more than ~12 h. Therefore, the high-fluence runs were performed over several days with periodic interruptions. The longest implantation,  $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup>, required 30 days with the accelerator operating about 8–10 h per day. During the long-duration irradiations at elevated temperatures, with periodic interruptions, the specimen temperature was kept constant at the test temperature only when the ion beam was on. When the beam was turned off the temperature was allowed to cool to room temperature.

## 2.3. Thermal desorption spectroscopy

The amount of retained D was measured by TDS in a separate vacuum system. Prior to performing TDS, the TDS chamber was baked at about 330–350 K for 2 h to improve the vacuum to below  $5 \times 10^{-8}$  Torr. Such a mild baking also results in the release of deuterium from ‘shallow traps’ [13], leaving only deeply trapped D to be released during the higher-temperature TDS heating.

A chromel–alumel thermocouple was spot-welded to the implanted specimen. The specimen was then placed in the TDS chamber on a heating cradle, heated at 1.5–2 K/s (linear ramp) up to 950–1050 K, and held there for 2–3 min. The released species at masses 2 (H<sub>2</sub>), 3 (HD), and 4 (D<sub>2</sub>) were monitored by a Hiden quadrupole mass spectrometer (QMS) during the heating process. The QMS signals were calibrated prior to each TDS run using calibrated leak bottles of H<sub>2</sub> and D<sub>2</sub>. The sensitivity of HD was assumed to be the average of the H<sub>2</sub> and D<sub>2</sub> sensitivities.

After realizing the thermocouple mix-up noted in Section 2.1, we were interested in finding out whether all of the trapped D was released by heating to 950 K. Although heating to 950 K should be sufficient to release the trapped D from vacancies and voids [5,14], further TDS to 1200 K was performed on one of the specimens that has already undergone TDS to 1050 K; this time the W–Re thermocouple was used.

## 3. Results and discussion

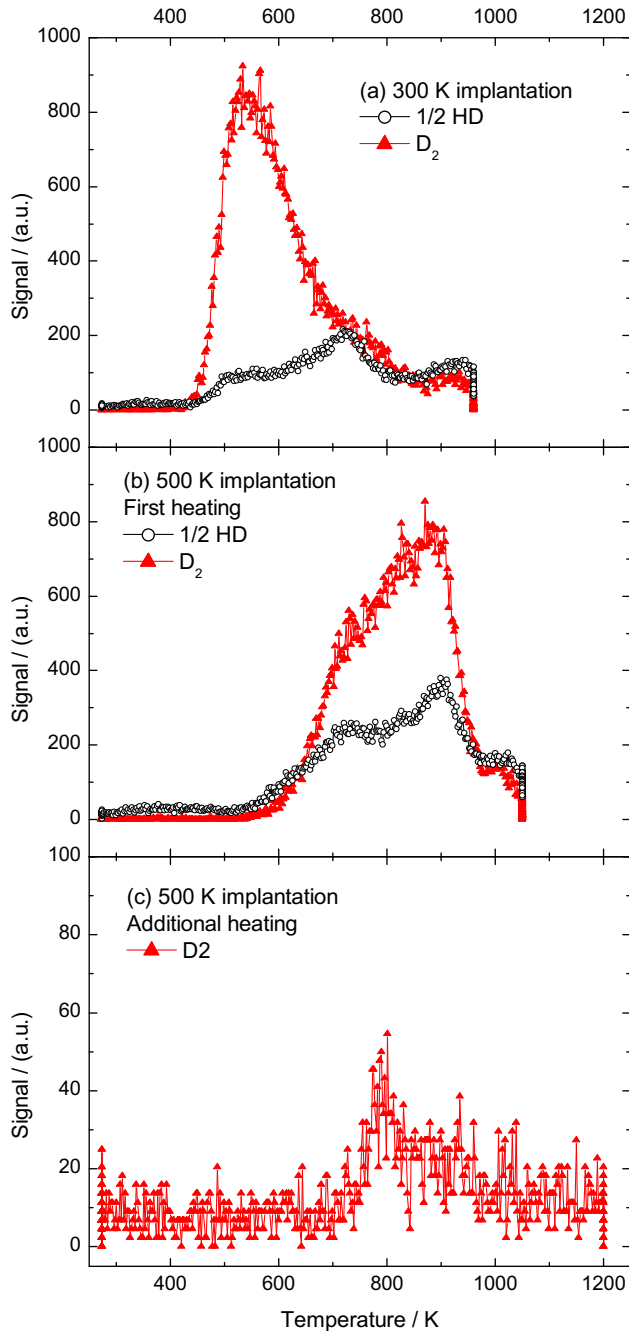
### 3.1. Thermal desorption spectra

#### 3.1.1. TDS of Plansee irradiated at 300 K

A typical desorption spectrum is shown in Fig. 4a for a specimen irradiated with 200 eV/D<sup>+</sup> to a fluence of  $3 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup>. The specimen was heated to 950 K with a ramping rate of 1.6 K/s. The release of deuterium begins at 400 K, and the primary release peak is at ~500 K with a high temperature shoulder extending to ~900 K.

#### 3.1.2. TDS of Plansee irradiated at 500 K

A typical desorption spectrum is shown in Fig. 4b for a specimen irradiated with 200 eV/D<sup>+</sup> to a fluence of  $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup>. The specimen was heated to 1050 K with a ramping rate of 1.7 K/s. Deuterium release starts at 600 K, with the majority of deuterium being released in a broad peak between 700 and 900 K. In order to ensure that all deuterium had been released from this Plansee W specimen, an additional heating at a ramp rate of 1.6 K/s to 1200 K was conducted; this time the W–Re thermocouple was used. The resulting spectrum is shown in Fig. 4c; only a small desorption peak at ~800 K is observed, containing  $\sim 2.4 \times 10^{18}$  D/m<sup>2</sup> which is <1% of the D released during the first



**Fig. 4.** QMS signals of  $H_2$ , HD, and  $D_2$  obtained during TDS of Plansee specimen for 200 eV/ $D^+$  irradiations at (a) 300 K and fluence of  $3 \times 10^{24} D^+/m^2$ ; (b) 500 K and fluence of  $8 \times 10^{25} D^+/m^2$ ; (c) same conditions as in (b) but with additional TDS heating with a ramp rate of 1.6 K/s to 1200 K and using the W-Re TC. Note: The Scale in (c) is 10 $\times$  more sensitive than in (a) and (b).

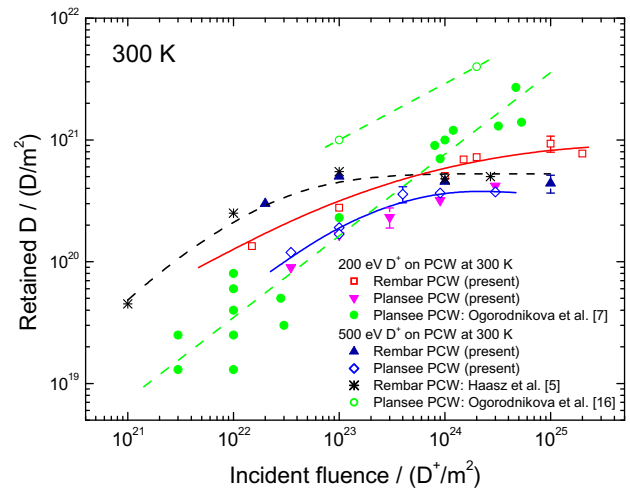
desorption at 1050 K ( $5 \times 10^{20} D/m^2$ ). This confirms that TDS heating to 950–1050 K was sufficient to release essentially all of the trapped D from the specimens.

The TDS profiles for both the 300 K and 500 K irradiations are consistent with previous measurements for polycrystalline [5] and single crystal tungsten [15].

### 3.2. Fluence dependence of D retention in tungsten at 300 K

#### 3.2.1. Irradiation with 500 eV/ $D^+$ at 300 K

New measurements of D retention in the Rembar foil due to irradiation by 500 eV/ $D^+$  at 300 K as a function of incident fluence



**Fig. 5.** Fluence dependence of D retention in Plansee and Rembar specimens irradiated with 200 and 500 eV/ $D^+$  at 300 K. Present results and published data [5,7,16]. (All present TDS retention data shown here were obtained with the chromel–alumel TC).

are plotted in Fig. 5. The incident ion flux was maintained at about  $5\text{--}7 \times 10^{19} D^+/m^2s$  and the fluence ranged from  $1 \times 10^{22}$  to  $1 \times 10^{25} D^+/m^2$ . For comparison, previously published D retention results for the Rembar [5] and Plansee materials [16] under the same irradiation conditions (500 eV  $D^+$ , 300 K) are included in Fig. 5. The present Rembar results agree well with the results of Haasz et al. [5]. For low fluences ( $<1 \times 10^{23} D^+/m^2$ ), D retention increases with increasing incident  $D^+$  fluence; for incident fluences above  $\sim 1 \times 10^{23} D^+/m^2$  the retention tends to saturation at a level of  $\sim 5 \times 10^{20} D/m^2$  compared with  $\sim 6 \times 10^{20} D/m^2$  in [5]. However, both of these results differ significantly from the Plansee results of Ogorodnikova et al. [16], where the D retention levels are higher, with no sign of saturation (albeit they only have two data points).

#### 3.2.2. Irradiation with 200 eV/ $D^+$ at 300 K

New measurement of 200 eV/ $D^+$  irradiation on the same type of Rembar foil as used above and in [5] at 300 K for the fluence range  $1 \times 10^{22}\text{--}2 \times 10^{25} D^+/m^2$  are also included in Fig. 5; here too, D retention shows a trend to saturation at  $\sim 8 \times 10^{20} D^+/m^2$  for incident fluences above  $2 \times 10^{24} D^+/m^2$ . For this 200 eV case, saturation occurs at an incident fluence 10 times higher than that for the 500 eV case, and the retention value is about a factor of two higher than the 500 eV case. Similar to the 500 eV case above, the D retention results of Ogorodnikova et al. [7] for a Plansee PCW irradiated with 200 eV  $D^+$  show an increasing trend without a clear sign of saturation; although, with scatter in the data above  $\sim 10^{24} D^+/m^2$  the start of saturation cannot be ruled out; see Fig. 5. The incident fluence in [7] was in the range  $3 \times 10^{21}\text{--}6 \times 10^{24} D^+/m^2$ , and for the highest fluence ( $6 \times 10^{24} D^+/m^2$ ), the retention level reached as high as  $3 \times 10^{21} D/m^2$ , almost five times higher than the saturation level observed in the present study.

The question arises whether such a disagreement could be caused by the different types of tungsten specimens used in the various studies, namely, Rembar hot-rolled thin foil used by Haasz et al. [5] and in the present study, and Plansee plates used by Ogorodnikova et al. [7,16].

#### 3.2.3. Comparison of Rembar and Plansee irradiated with 200 and 500 eV/ $D^+$ at 300 K

With an attempt to resolve the above discrepancies, a new set of  $D^+$  irradiation experiments with 200 and 500 eV/ $D^+$  ions were performed with both Rembar and Plansee specimens at 300 K using the same experimental facilities and procedures. The ion flux for

these runs was  $3\text{--}4 \times 10^{19} \text{ D}^+/\text{m}^2\text{s}$ . The new results are included in Fig. 5. The Plansee plate irradiated by  $500 \text{ eV}/\text{D}^+$  ions tends to saturate at  $\sim 4 \times 10^{20} \text{ D}^+/\text{m}^2$ , which is only slightly lower than the  $\sim 5 \times 10^{20} \text{ D}^+/\text{m}^2$  saturation level seen for the Rembar foil irradiated at the same conditions. However, a significant difference in D retention is seen between the Plansee plate and the Rembar foil at lower incident fluences ( $< 1 \times 10^{23} \text{ D}^+/\text{m}^2$ ), where retention values are about a factor of three lower in the plate than in the foil.

High incident fluences of  $200 \text{ eV}/\text{D}^+$  ions implanted into the Plansee plate at 300 K also lead to a saturation level of  $\sim 4 \times 10^{20} \text{ D}^+/\text{m}^2$  – very similar to the 500 eV case above. The fluence dependence curve for  $200 \text{ eV}/\text{D}^+$  tracks the 500 eV curve very well, showing that every 200 eV data point is close to the corresponding 500 eV point at the same incident fluence. A trend to saturation is also seen in the Rembar foil; however, the saturation level is  $\sim 8 \times 10^{20} \text{ D}^+/\text{m}^2$  – about a factor of two higher than in the Plansee plate. It is possible that the higher D retention in the Rembar foil is associated with a higher trap concentration induced in the foil during the hot-rolling manufacturing process. Although we would anticipate different surface structures for the Rembar and Plansee materials, the SEM images taken on and off the irradiation areas were not very helpful in identifying major surface differences.

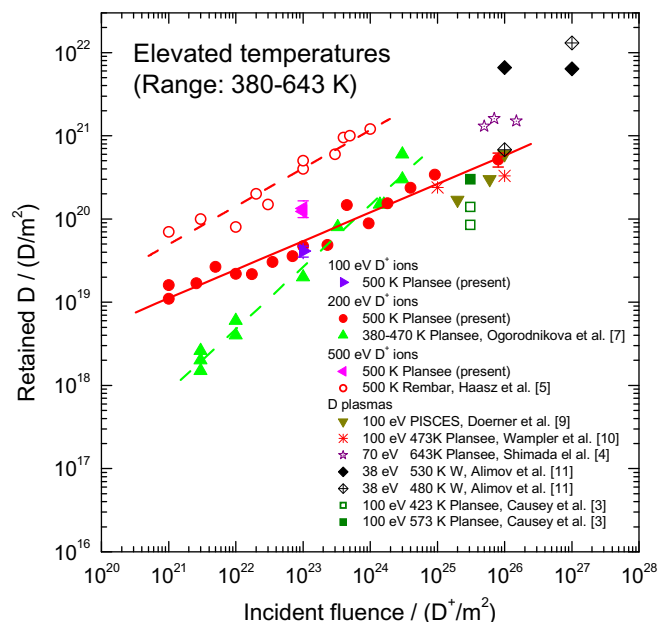
A possible explanation for the saturation trend at 300 K was suggested by Haasz et al. [5] based on NRA depth profiles measured on both the front and back surfaces of a Rembar tungsten foil – the same material as used in the present study. The depth profile of the trapped D on the front surface of the irradiated specimen showed that the trapped D extended well beyond the implantation zone ( $\sim 40 \text{ nm}$ ) to  $\sim 500 \text{ nm}$ , while on the back surface no D was detected, implying that diffusion may be impeded in the near-surface preventing trapping sites deep in the bulk to be reached. By comparison, for irradiation at 500 K, D is able to diffuse to the back surface as detected by NRA [5].

### 3.3. Fluence dependence of D retention in tungsten at 500 K

In contrast to the 300 K cases, where D retention in tungsten tends to saturate, independent of the incident ion energy and type of tungsten, deuterium trapping at 500 K, as measured in ion-beam experiments, consistently shows an increasing trend with incident fluence, e.g., [5,7]. In the present study, we have extended the Plansee D-retention database for  $200 \text{ eV}/\text{D}^+$  irradiations at 500 K to a fluence of  $8 \times 10^{25} \text{ D}^+/\text{m}^2$  and also obtained new Plansee retention results for 100 and  $500 \text{ eV}/\text{D}^+$  irradiations at 500 K; see Fig. 6. Also included in Fig. 6 are previously published ion-beam data by Ogorodnikova et al. [7] (Plansee,  $200 \text{ eV}/\text{D}^+$ , 380–470 K), Haasz et al. [5] (Rembar,  $500 \text{ eV}/\text{D}^+$ , 500 K), and several D retention results for plasma devices [3,4,9–11].

The present  $200 \text{ eV}/\text{D}^+$  irradiation results at 500 K show an increasing trend in D retention in the Plansee tungsten plate with increasing incident fluence with a slope of 0.35–0.4, with no indication of saturation; see Fig. 6. Irradiation studies, also on Plansee tungsten, under similar irradiation conditions ( $200 \text{ eV}/\text{D}^+$ , 380–470 K) by Ogorodnikova et al. [7] also showed an increasing trend in D retention, but with a slope  $> 0.5$ ; see Fig. 6. A slope of 0.5 would correspond to a diffusion-limited trapping process, while a slope of  $< 0.5$  may indicate additional loss mechanisms; e.g., surface roughness may offer alternate escape pathways for diffusing D atoms. A slope greater than 0.5, as observed by Ogorodnikova [7] suggests a retention mechanism operating in addition to diffusion; possibly some form of trap creation.

The comparison between Plansee (present study) and Rembar tungsten [5] for  $500 \text{ eV}/\text{D}^+$  irradiation at 500 K shows that the retained amount of D in the Plansee tungsten plate at a fluence of  $\sim 10^{23} \text{ D}^+/\text{m}^2$  is about 3–4 times lower than in the Rembar foil. As was noted above for irradiation cases at 300 K, the differences



**Fig. 6.** Present results and published data from [5,7] on the fluence dependence of D retention in Plansee and Rembar W irradiated with ion beams of 100, 200 and  $500 \text{ eV}/\text{D}^+$  at 500 K. (All present TDS retention data shown here were obtained with the chromel–alumel TC). Retention data obtained in plasma devices [3,4,9–11] are also shown for comparison.

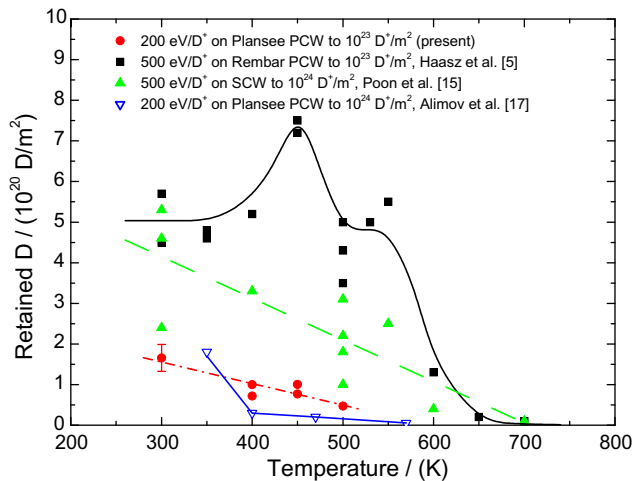
might be explained by different surface structures produced during manufacturing – more trapping sites created in the Rembar foil during hot rolling. Also plotted in Fig. 6 is a single point for  $100 \text{ eV}/\text{D}^+$  irradiation of Plansee at 500 K and  $1 \times 10^{23} \text{ D}^+/\text{m}^2$ ; this point falls just slightly below the 200 eV retention curve, indicating only a weak energy dependence; more on this in Section 3.5.

### 3.4. Irradiation temperature dependence of D retention in tungsten

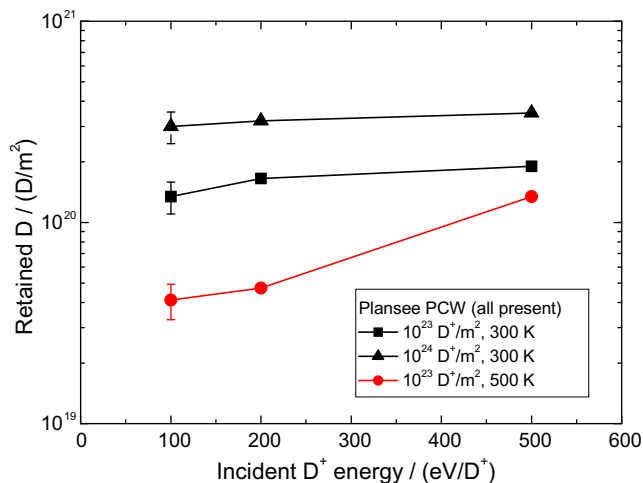
In the present study, we have also examined the dependence of D retention on specimen temperature during irradiations using  $200 \text{ eV}/\text{D}^+$  at fluxes of  $4\text{--}6 \times 10^{19} \text{ D}^+/\text{m}^2\text{s}$  and a fixed fluence of  $1 \times 10^{23} \text{ D}^+/\text{m}^2$ . The measured D retention, as a function of temperature (300–500 K), is shown in Fig. 7. Results for Rembar PCW ( $500 \text{ eV}/\text{D}^+$ ,  $1 \times 10^{23} \text{ D}^+/\text{m}^2$ ) by Haasz et al. [5], Plansee PCW ( $200 \text{ eV}/\text{D}^+$ ,  $1 \times 10^{24} \text{ D}^+/\text{m}^2$ ) by Alimov and Roth [17], and single crystal tungsten ( $500 \text{ eV}/\text{D}^+$ ,  $1 \times 10^{24} \text{ D}^+/\text{m}^2$ ) by Poon et al. [15] are also included here for comparison. For the present results, the highest D retention was found at 300 K ( $\sim 1.7 \times 10^{20} \text{ D}^+/\text{m}^2$ ), followed by a decreasing trend with increasing temperature, reaching  $\sim 5 \times 10^{19} \text{ D}^+/\text{m}^2$  at 500 K. We note that the different fluence dependencies observed at different temperatures – e.g., saturation at 300 K (Fig. 5) and no saturation at 500 K (Fig. 6) – imply that any temperature dependence profile will strongly depend on incident fluence. A temperature dependence curve at a higher incident fluence ( $> 10^{24} \text{ D}^+/\text{m}^2$ ) is likely to show a peak in retention at 400–500 K, as the amount of D retained at 300 K will not increase significantly over the  $10^{23} \text{ D}^+/\text{m}^2$  incident fluence value, while the amount retained at 500 K will increase continuously with fluence.

### 3.5. Ion energy dependence of D retention in tungsten

Although the maximum possible energy transfer from  $500 \text{ eV D}^+$  to a W atom (21 eV) is insufficient to create a displacement ( $\sim 38\text{--}44 \text{ eV}$  [18,19]), the maximum energy transferable from  $500 \text{ eV D}^+$  to oxygen atoms is 200 eV and to carbon atoms it is 250 eV, assuming two-body collisions. (Oxygen and carbon both



**Fig. 7.** Temperature dependence of D retention in different types of tungsten. For the present results, the lower data point at 200 eV/D<sup>+</sup> and 450 K was obtained for a Plansee specimen annealed (prior to irradiation) at 1500 K; the W–Re TC was used during both annealing and TDS. All other present results were obtained for specimens annealed at 950 K; with the chromel–alumel TC being used during both anneal and TDS. Data from [5,15,17] are also shown for comparison.



**Fig. 8.** Energy dependence of D retention in Plansee specimens irradiated at 300 and 500 K. (All TDS retention data shown here were obtained with the chromel–alumel TC).

appear as bulk impurities, and perhaps more importantly as surface impurities.) Due to their larger masses, the energy transfer from O or C to W is more efficient than from D<sup>+</sup> to W and these energies are above the threshold for displacement damage (140 eV for O and 175 eV for C, assuming a displacement threshold of 41 eV, which is the average value from [18,19]). Thus, it is possible for 500 eV D<sup>+</sup> to create vacancies in W through intermediate recoil collisions with O and C impurities on the surface of W [14]. But 200 eV and 100 eV D<sup>+</sup> cannot induce such knock-on effects because the maximum energy transfer from D<sup>+</sup> to O (80 eV) or C (100 eV) is insufficient to create displacements in W [18,19].

Irradiations were performed on Plansee specimens at 100, 200 and 500 eV/D<sup>+</sup> energies and 300 and 500 K temperature; see Fig. 8. Three different fluence/temperature combinations are shown. In the first case, 300 K and incident fluence of  $1 \times 10^{23}$  D<sup>+</sup>/m<sup>2</sup>, the retained D shows only a slight increase as the energy is increased from 100 to 500 eV/D<sup>+</sup>. The second case, for an incident fluence of  $1 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup> at 300 K, similarly shows only a slight in-

crease with increasing energy. As expected from the fluence dependence curves in Fig. 5 for irradiations at 300 K, the amount of retained D at an incident fluence of  $\sim 10^{24}$  D<sup>+</sup>/m<sup>2</sup> is about a factor of two higher than at  $\sim 10^{23}$  D<sup>+</sup>/m<sup>2</sup>.

For the last case in Fig. 8, a fluence of  $\sim 10^{23}$  D<sup>+</sup>/m<sup>2</sup> was used and the irradiation temperature was raised to 500 K. At 500 K the retention levels for all energies are noticeably lower than for the two 300 K cases, but a factor of three increase in retention is noted as the energy increases from 200 to 500 eV/D<sup>+</sup>.

Given the scatter in the experimental results, it is only possible to conclude that for D<sup>+</sup> energies below 500 eV/D<sup>+</sup>, D retention in the Plansee tungsten at 300 K depends only weakly on the incident ion energy. The dependence is stronger at 500 K and it is possible that this is associated with recoil displacements due to O and C impurities, as noted above, coupled with increased deuterium diffusion.

## 4. Summary and conclusions

### 4.1. Fluence dependence

#### 4.1.1. Irradiation at 300 K

Fluence dependence plots of D retention in both Rembar and Plansee tungsten at 300 K show a trend to saturation. In the Rembar foil, for 500 and 200 eV/D<sup>+</sup> irradiations, similar saturation levels are seen ( $\sim 5 \times 10^{20}$  D/m<sup>2</sup> and  $\sim 8 \times 10^{20}$  D/m<sup>2</sup>, respectively). However, the onset of saturation with 500 eV/D<sup>+</sup> occurs at an incident fluence of  $\sim 1 \times 10^{23}$  D<sup>+</sup>/m<sup>2</sup>, while for 200 eV/D<sup>+</sup> it occurs at  $\sim 1 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup>, about an order of magnitude higher. For the Plansee tungsten plate, irradiations by 500 and 200 eV/D<sup>+</sup> ions demonstrated similar retention behaviour, with retention levels of  $\sim 4 \times 10^{20}$  D/m<sup>2</sup> for incident fluences above  $5 \times 10^{23}$  D<sup>+</sup>/m<sup>2</sup>.

#### 4.1.2. Irradiation at 500 K

In the present study, only the Plansee tungsten plate material was used for 500 K irradiations. The fluence dependence curve for 200 eV/D<sup>+</sup> irradiations shows an increasing trend – no sign of saturation – with increasing incident D<sup>+</sup> fluence up to the highest incident fluence used ( $8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup>). At this fluence the retained amount was  $\sim 5.2 \times 10^{20}$  D/m<sup>2</sup>. We note that at 500 K, the retained amount of D at an incident fluence of  $\sim 8 \times 10^{25}$  D<sup>+</sup>/m<sup>2</sup> is in the *ball park* of retention results obtained in plasma devices. The remaining question is whether the increasing trend continues above  $\sim 10^{26}$  D<sup>+</sup>/m<sup>2</sup> into the ITER fluence regime.

### 4.2. Temperature dependence

For 200 eV/D<sup>+</sup> irradiation of Plansee tungsten D retention was seen to decrease with increasing irradiation temperature in the 300–500 K range. The highest retention level was  $\sim 1.7 \times 10^{20}$  D/m<sup>2</sup> at 300 K, followed by a linear decreasing trend, reaching  $\sim 5 \times 10^{19}$  D/m<sup>2</sup> at 500 K. It is noted, however, that the temperature dependence will be strongly dependent on the incident fluence.

### 4.3. Ion energy dependence

The energy dependence curves of D retention in Plansee tungsten irradiated at 300 K to fluences of  $\sim 1 \times 10^{23}$  D<sup>+</sup>/m<sup>2</sup> and  $\sim 1 \times 10^{24}$  D<sup>+</sup>/m<sup>2</sup> show only a slight increase with incident energy from 100 to 500 eV/D<sup>+</sup>. For irradiations at 500 K to a fluence of  $\sim 1 \times 10^{23}$  D<sup>+</sup>/m<sup>2</sup>, the D retention increased noticeably (a factor of  $\sim 3$ ) as the energy increased from 100 to 500 eV/D<sup>+</sup>. These limited results indicate that the energy of incident D<sup>+</sup> plays a minor role in affecting D trapping in polycrystalline tungsten.

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