

# Deuterium retention in tungsten and tungsten-tantalum alloys exposed to high-flux deuterium plasmas

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Direct comparison of deuterium retention in the samples of tungsten and two grades of tungsten-tantalum alloys – W-1%Ta and W-5%Ta, exposed to deuterium plasma (ion flux  $\sim 10^{24} \text{ m}^{-2}\text{s}^{-1}$ , ion energy at biased target  $\sim 50 \text{ eV}$ ) at the plasma generator Pilot-PSI was performed using thermal desorption spectroscopy (TDS). No systematic difference in terms of total retention in tungsten and tungsten-tantalum was identified. The measured retention value for each grade did not deviate more than 24% from the value averaged over the 3 grades exposed to the same conditions. No additional desorption peaks appear in the TDS spectra of the W-Ta samples as compared to the W target, indicating that no additional kinds of traps are introduced by the alloying of W with Ta. In the course of the experiment the same samples were exposed to the same plasma conditions several times, and it is demonstrated that samples with the history of prior exposures yield an increase of deuterium retention of up to 130 % under the investigated conditions compared to the samples that were not exposed before. We consider this as evidence that exposure of the considered materials to ions with energy below the displacement threshold generates additional traps for deuterium. The positions of the release peaks caused by these traps are similar for W and W-Ta, which indicates that the corresponding traps are of the same kind.

Keywords: tungsten, tungsten-tantalum alloys, deuterium retention

## 1. Introduction

Currently tungsten and carbon fiber composite are considered as candidate materials for the ITER divertor [1]. The practical use of tungsten is hindered by its high ductile-to-brittle transition temperature (DBTT). Therefore, tungsten alloys with better machinability are considered to be used instead [2]. However, the retention of hydrogen isotopes (including radioactive tritium) in binary tungsten alloys has not been extensively studied up to now [3, 4].

In this work we examined the deuterium retention in W-Ta alloys using thermal desorption spectroscopy (TDS). The main goal of our experiments was to perform a direct comparison of deuterium retention in tungsten and in tungsten-tantalum alloys exposed to high flux deuterium plasma (keeping the exposure conditions the same for different grades). We also aimed at clarifying whether or not the presence of tantalum introduced new types of traps. It was therefore investigated whether additional desorption peaks appear in TDS spectra of W-Ta samples as compared to those of W. In addition

we aimed at clarifying the effect of subsequent plasma exposures on deuterium retention, the so-called history effect, and its dependence on the alloying of W with Ta.

In our study we used polycrystalline samples. The samples were exposed to high-flux deuterium plasmas ( $\sim 10^{24} \text{ ions m}^{-2}\text{s}^{-1}$ ) in the linear plasma simulator Pilot-PSI.

## 2. Experiment

The experimental part of the work was performed at the linear plasma generator Pilot-PSI at the FOM Institute DIFFER [5]. The plasma source in Pilot-PSI is based on a cascaded arc discharge, which generates high electron density ( $\leq 10^{20} \text{ m}^{-3}$ ), low electron temperature ( $\leq 5 \text{ eV}$ ) plasmas and operates in the pulsed regime. The high density is provided by focusing of the plasma beam by electromagnetic coils surrounding the plasma chamber. The plasma beam is not uniform – electron temperature and density profiles (and therefore also particle and heat flux profiles) are roughly Gaussian with FWHM being  $\sim 1 \text{ cm}$ . To characterize the beam profile, the maximum

density and surface temperature, corresponding to the center of the beam, are indicated in the text.

Electron temperature and density are measured by Thomson scattering. The ion flux is then calculated (under the assumption that ion temperature is equal to the electron temperature) as ([5])

$$\Phi = \frac{n_e}{2} \sqrt{\frac{2k_B T}{m_D}} \quad (1)$$

with  $m_D$  the mass of the deuterium ion,  $k_B$  – Boltzmann constant,  $T$  – electron temperature,  $n_e$  – electron density.

The surface temperature of a specimen is monitored by an IR camera.

In order to avoid creation of defects by displacement damage, the energy of the ions was kept well below the displacement damage threshold for  $D^+$  ions in tungsten ( $\sim 940$  eV [6]), namely, close to 50 eV.

Two grades of powder metallurgy W-Ta alloy were examined – one containing 1 mass % of Ta and the other 5 mass % of Ta. The actual Ta content in both grades was measured by energy-dispersive X-ray spectroscopy (EDX) and was found to be equal to the specified values of 1% and 5%. It was also found that Ta and W are uniformly mixed, no Ta precipitates were observed. In addition, we examined also samples of powder metallurgy W. All investigated samples were polycrystalline and were provided by Plansee. The samples were discs of 20 mm in diameter and had thickness of 1 mm. All samples were mechanically polished. Unless otherwise stated, they were subsequently annealed in vacuum at 1300 K for 1 hour.

Retention measurements were performed with TDS. The maximum temperature was 1273 K, the temperature ramp was 0.5 K/s, and the holding time at the maximum temperature was 5 minutes. The release flux of molecular HD ( $\Gamma_{HD}$ ) and  $D_2$  ( $\Gamma_{D_2}$ ) was measured by a Quadrupole Mass Spectrometer, with the total deuterium release flux ( $\Gamma_D$ ) being calculated as

$$\Gamma_D = \Gamma_{HD} + 2\Gamma_{D_2} \quad (2)$$

Quantification of the mass 4 signal, corresponding to the release of  $D_2$ , was performed using a calibrated  $D_2$  leak. Quantification of the mass 3 signal (HD) was performed using the averaged value of the signals of two calibrated leaks –  $H_2$  and  $D_2$ .

Signals of the other deuterium-containing molecular compounds, such as  $D_2O$  and HDO

were also detected, but because of the absence of the corresponding calibrated leaks, those could not be quantified. However, we believe that their contribution to the total retention is negligible. The reason is the following: we have the measurements of signals of masses 3, 4, 19 and 20 for the sample that was not exposed to plasma – that is, the measurement of background. It can be seen that the signal of mass 3 (as well as mass 4), measured for the deuterium-exposed sample is orders of magnitude higher than corresponding values of background. On the other hand, corresponding signals of masses 19 and 20 are fluctuating around the background value. Therefore, we conclude that the release of HDO and  $D_2O$  does not contribute any significant fraction to the total deuterium release and can be neglected, so that measurements of release of HD and  $D_2$  only adequately characterize total deuterium retention, and therefore the analysis of retention was performed based on the contributions of HD and  $D_2$ .

The experimental sequence was the following. Two samples of each grade (i.e., W, W-1%Ta, W-5%Ta) were used. One of the samples of each grade was exposed in Pilot-PSI once, with TDS measurements performed a week after the exposure. In the following we will refer to this set of samples as to the “samples without history”. Another sample was exposed once, with TDS performed as fast as was technically possible (1 hour 50 min  $\pm$  4 min) after the exposure. In the following these measurements will be referred to as “fast TDS”. After that, the same sample was exposed again, and again TDS was performed 1 h 50 min after the exposure. After that, the same sample was exposed again, but now TDS was performed a week after exposure. The samples from this set are referred to as “the samples with history”. The week lag time between exposure and TDS measurements ensured that only trapped inventories, properly characterizing the trapping properties of the investigated materials, were measured.

All the exposures were performed at as identical exposure conditions as technically achievable. The maximum flux in the center of the beam on all targets was in the range of  $8 \cdot 10^{23}$  –  $1 \cdot 10^{24}$   $m^{-2}s^{-1}$ , the maximum surface temperature in the range of 460 – 510 K, with the exception of the W-5%Ta sample without history, for which the flux was  $5 \cdot 10^{23}$   $m^{-2}s^{-1}$  and the maximum temperature 400 K. The exposure time in all cases was 70 s, all exposures were performed in single pulse. Exposure conditions for each exposure of each sample are summarized in Table 1. The temperature during exposure ensured that

vacancies were not mobile (in W, vacancies become mobile at 550 – 600 K [7, 8]).

Table 1

In all cases, the samples were stored in air between the end of exposure and the beginning of the TDS measurement.

#### 4. Experimental results

##### 3.1 TDS of the samples of tungsten and tungsten-tantalum alloys

Fig. 1 presents the TDS spectra of the investigated samples of W, W-1%Ta and W-5%Ta. It is evident that: (i) the TDS spectra of W-Ta samples do not have any additional desorption peaks as compared to samples of W; (ii) TDS spectra of all the investigated samples contain the low temperature release peak which has the same temperature position at ~500 K; (iii) the spectra of the W samples appear to consist of two unresolved peaks with a high-temperature peak at 600-650 K while the spectra of the W-Ta samples have only a long desorption tail stretching into the high-temperature region without any noticeable high-temperature peak.

The fraction of deuterium released as HD varied from run to run and contributed from 16 to 28% of the deuterium retention.

Fig. 1

##### 3.2 The first exposure: fast TDS and TDS after a week

Fig. 2 presents the comparison of the desorption spectra obtained for the exposures of the samples without history and for the first exposure of samples with history. This is, in essence, the comparison between desorption spectra of the equivalent samples (since samples with history haven't yet been exposed before), measured about 1 hour 50 minutes and a week respectively after the plasma exposure. The presented data demonstrate several important details: (i) Deuterium release in the TDS spectra measured an 1 hour 50 min after the exposure starts at considerably lower, and stops at somewhat higher, temperatures compared to those measured a week after the exposure; (ii) the total amount retained in the samples shortly after the exposure is considerably higher than a week after;

Fig. 2

##### 3.3 The samples with and without history: TDS after a week

Fig. 3 presents the comparison of the spectra measured after a week lag between the exposure and TDS, for samples with and without history. It demonstrates that in all investigated grades history of prior exposures influences the amount of retained deuterium. For all grades the total amount of deuterium remaining after a week in the sample with the previous history of exposures is higher than for the sample which was exposed only once. In general the shape of the spectrum is different for samples with and without history while the position of the release peaks remains virtually unchanged.

Fig. 3

##### 3.4 The annealed and unannealed samples

An additional sample of W was exposed (only once) to similar plasma conditions as all the other samples (maximum flux  $8 \cdot 10^{23} \text{ m}^{-2} \text{ s}^{-1}$ , maximum temperature 463 K), but this one was polished and not annealed before the exposure. TDS on this sample was performed a week after the exposure. Fig. 4 presents the comparison of the desorption spectrum of this sample with the spectra of the annealed samples with and without history, measured a week after exposure.

It is evident that the spectra of the annealed sample with history and unannealed sample without history are very similar. Also, the total retention for these samples is higher as compared to the annealed sample without history.

Fig. 4

#### 4. Discussion

A general overview of the total amounts of deuterium retained after each performed exposure is presented in Table 2.

Table 2

From Table 2 it can be seen that generally in terms of total retention there is no systematic difference between W and W-Ta, nor between the two grades of W-Ta, i.e., W-1%Ta and W-5%Ta. The measured values of total retention for each grade did not deviate from the average value for each exposure step by more than 24%. This suggests that the implementation of W-Ta as plasma-facing material will not lead to a significant increase of tritium retention in the

corresponding plasma-facing components. It should however be noted that this is based on experiments in a limited temperature range and that for a decisive answer it would be necessary to perform comparable measurements in a wider range of surface temperature and ion flux.

The fact that the total amount of retained deuterium is considerably lower for samples that were stored for a week after the exposure prior to TDS measurement, comparing to the samples that were measured shortly after the exposure, combined with the fact that release as measured in fast TDS starts at considerably lower temperatures than that measured in TDS after a week, indicates that 1 hour 50 minutes after the exposure there is still large amount of deuterium atoms in the weakly bound state. Careful consideration of the fast TDS spectra of W sample suggests that it features the small unresolved peak at  $\sim 420$  K. Therefore we suggest that the difference of the measured retention between fast TDS and TDS after a week is caused by the traps with small trapping energy which still retain some deuterium 1 hour 50 minutes after the exposure, possibly together with the small contribution of solute deuterium, but which is largely released during waiting time of one week at room temperature. It is known that in tungsten dislocations exhibit the lowest trapping energy among all the defects, so this low-temperature peak can be attributed to the trapping on dislocations. Since these atoms are weakly bound to the crystal lattice of the material, they are quickly released even at room temperature. Therefore, TDS performed a week after the exposure reveals only strongly trapped deuterium. Similar observations of the decrease of retained deuterium amount in tungsten over the waiting (storage) time were reported in [9 – 11].

As was already mentioned, for all grades the total amount of deuterium remaining after a week in the sample with the history of prior exposures is higher than for the sample which was exposed only once. Fig. 1a, 2b and Table 2, indicate that this holds true for the total amount of deuterium remaining after 1 hour 50 minutes in the samples of both W-Ta grades. At the same time, for W the second exposure shows a reduction in D retention compared to the first exposure. Taking into account the previous statement and comparing the results of fast TDS and those measured a week later, this indicates that the impact of the pre-exposure on the dynamics of solute deuterium release is different for W and W-Ta.

For all grades the trapped deuterium amount as measured by TDS after a week is higher for the samples that were exposed before than for the samples that were not. This indicates that plasma

exposure creates additional trapping sites within the material of the sample. The possibility of modification of plasma-exposed tungsten leading to creation of additional damage has been demonstrated in earlier work ([12, 13]).

Enhanced retention in the pre-exposed samples has been observed before [14, 15]. However, there exists a significant difference between the presented data and the ones available from the literature. The impact of the pre-exposure of the samples on the retention in subsequent exposures was reported to arise due to the high-energy ( $\sim 1$  keV, while in our experiments it was only  $\sim 50$  eV), low-flux ion exposure and manifested itself as an increase of a high-temperature desorption peak. In our experiments, however, pre-exposure leads to an increase of the low-temperature desorption peak. For the W-Ta samples we did not observe any profound high-temperature peak at all.

As was mentioned in Section 3.1, samples of W-Ta do not have additional desorption peaks as compared to W. This means that the alloying of tungsten with tantalum does not introduce additional kinds of traps. The fact that deuterium retention is not systematically higher for W-Ta indicates that this alloying does not increase the concentration of trapping sites either. On the other hand, the position of one of the release peaks of W and of the only release peak of W-Ta is similar for all investigated grades. Since the TDS conditions were identical, this means that the corresponding trapping energy is the same. Therefore, it seems likely that there exists a common kind of trapping sites for W and for W-Ta, while W possesses additional kinds of traps, absent (or present in considerably lower concentrations) in W-Ta.

It should be noted that surface temperature profile, as mentioned in Section 2, is non-uniform during the exposure. Therefore the shape of the measured TDS spectrum might be affected by the fact that it contains contributions from different positions on the sample, which were in fact exposed at different temperatures. However, this effect, if present, is compensated for by the fact that we perform the comparison of spectra from samples exposed at the same conditions.

In order to identify the trapping sites generated by the plasma exposure in the investigated samples under the given exposure conditions we used the comparison of the TDS spectra of annealed and unannealed samples without history, (Fig. 4). It is evident that the history of prior exposures has the same effect on the shape of TDS spectrum and total retention as does the absence of annealing – it enhances the

low-temperature release peak. This suggests that this low-temperature peak is associated with a specific kind of traps, the concentration of which decreases upon annealing and increases by plasma exposure. It is known that annealing decreases the amount of thermodynamically non-equilibrium defects, such as dislocations, and non-equilibrium concentration of vacancies. This might be an explanation for the decrease of the low-temperature peak by annealing. On the other hand, vacancies can be generated by the high flux plasma exposure as a result of plastic deformation due to high mechanical stresses created by the deuterium concentration strongly exceeding the solubility limit [16 – 18]. Dislocations themselves are probably too weak traps to retain deuterium at room temperature, as was discussed above. This suggests that low-temperature release peak on the TDS spectra of the investigated samples corresponds to the trapping on vacancies. This agrees with our previous results [4], as well as with the results published in [19] where it has been discussed that in unpolished tungsten damaged by MeV-range W ions the low-temperature D<sub>2</sub> desorption peak might contain contributions from release of deuterium trapped on vacancies. Consequently this implies that the high-flux low-energy plasma exposure of tungsten and tungsten alloys results in a modification of these materials which manifests itself as an increase of density of vacancies, which are subsequently responsible for the enhanced trapping – i.e., the history effect.

## 5. Conclusions

The deuterium retention in W-Ta alloys, exposed to high flux plasmas, was experimentally studied. For the first time, the retention in these alloys was compared to that in pure W – an issue necessary to be clarified in order to evaluate the possible use of W-Ta instead of tungsten as a material for plasma-facing components. The main conclusion is that for the conditions used in the experiments – ion flux  $\sim 10^{24}$  m<sup>-2</sup>s<sup>-1</sup>, ion fluence  $\sim 5 \cdot 10^{25}$  m<sup>-2</sup>, ion energy  $\sim 50$  eV, surface temperature  $\sim 480$  K – neither W nor any of the investigated W-Ta grades exhibit systematically higher retention as compared to the others.

The desorption spectra of both investigated W-Ta grades consist of one major release peak at  $\sim 500$  K with desorption tails stretching into the high-temperature region. Desorption spectra of the investigated W targets also have a release peak at  $\sim 500$  K; in some cases they have in addition an unresolved high-temperature peak at 600-650 K.

The absence of the additional release peaks on the desorption spectra of W-Ta samples as compared to those of W indicates that alloying with Ta does not introduce the additional kinds of traps. The proximity of the values of the total retained amount for all investigated grades shows that the presence of Ta does not significantly influence the concentration of trapping sites either.

A clear influence of exposure of a target to plasma has been observed on the deuterium retention for a subsequent exposure, i.e., the history effect. It was observed that the history effect leads to an increase of deuterium retention by up to 130%. This indicates that significant generation of trapping sites for deuterium occurs as a result of low-energy, high-flux plasma exposure.

## Acknowledgments

This work, supported by the European Communities under the contract of Associations between EURATOM and SCK•CEN, was carried out within the framework of the European Fusion Development Agreement. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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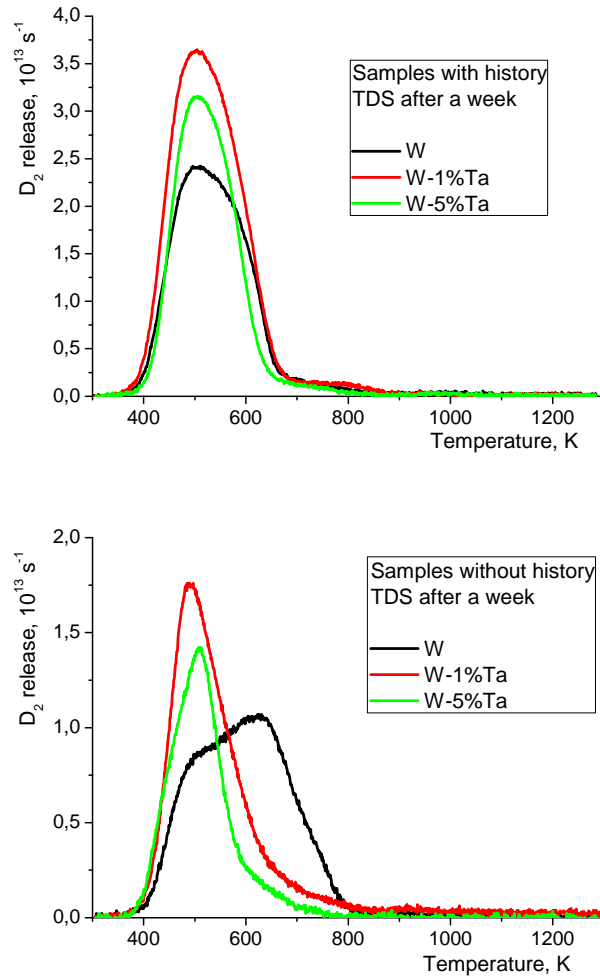


Fig. 1 TDS spectra of  $D_2$  release from the samples of W, W-1%Ta and W-5%Ta with history and without history of previous exposures.

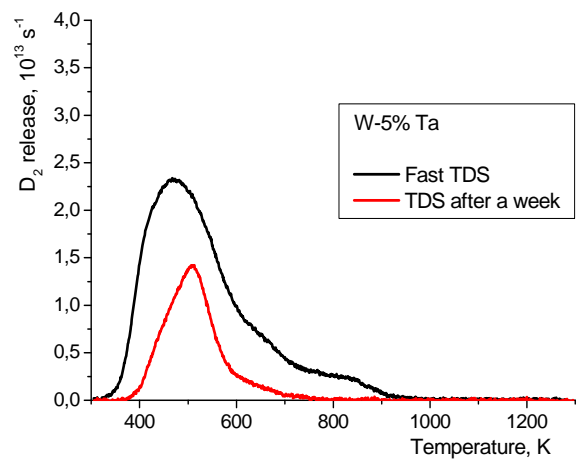
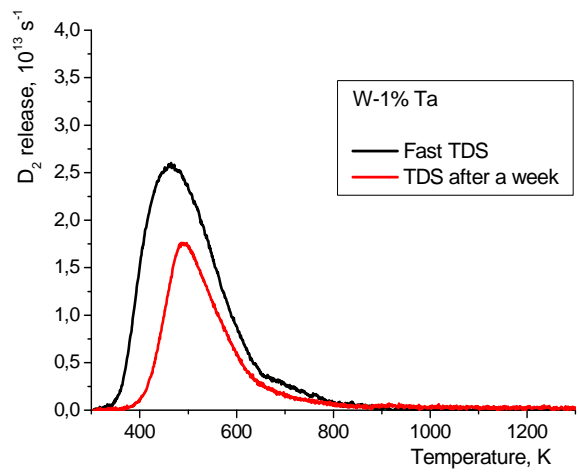
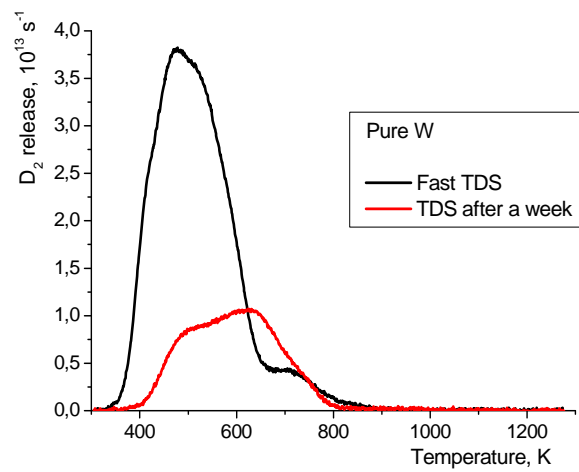


Fig. 2 Comparison of desorption spectra, recorded shortly after the first exposure of the sample with history (“fast TDS”) and a week after the exposure (for the samples without history).



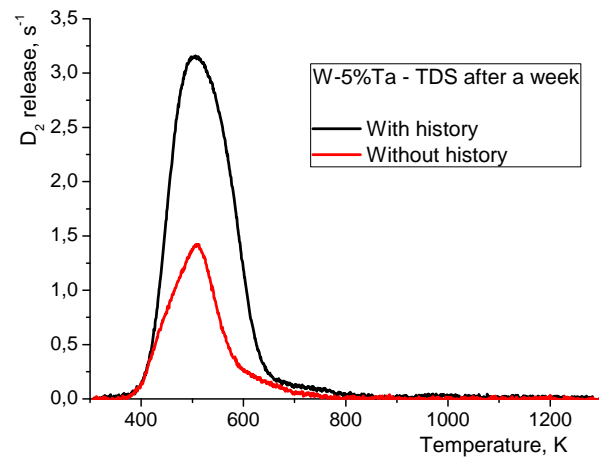
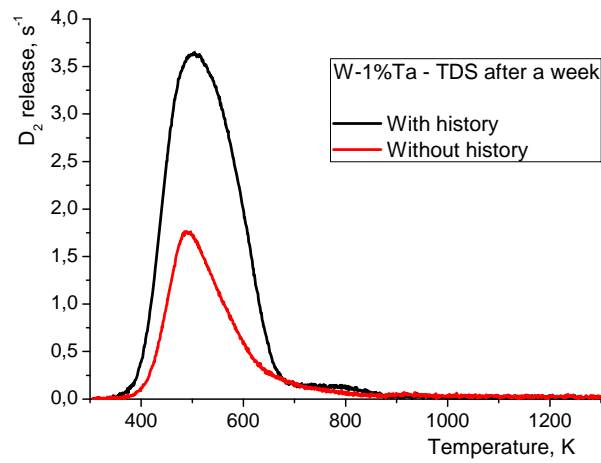
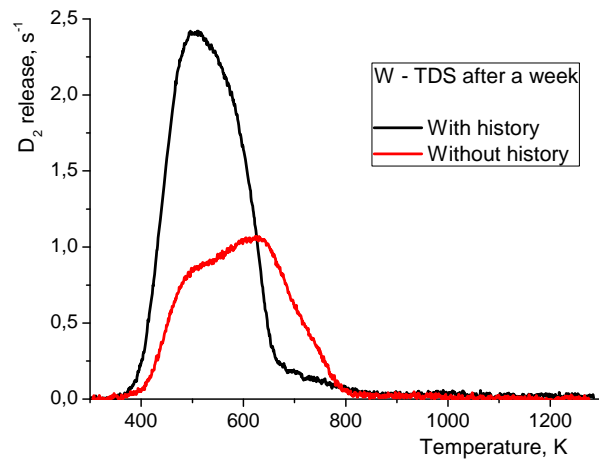


Fig. 3 Comparison of the spectra of D<sub>2</sub> release, recorded a week after the exposure, for the samples with and without history.

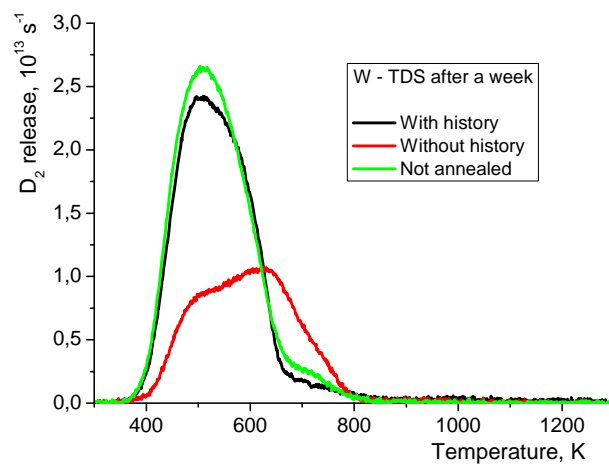


Fig. 4 TDS spectra of D<sub>2</sub> release from the annealed W sample with history, annealed and not annealed W samples without history, measured a week after the plasma exposure.

Samples	Exposure / TDS after	W	W-1%Ta	W-5%Ta
With history	First / 1 h 50 min	$8.9 \cdot 10^{23}$	$8,5 \cdot 10^{23}$	$8.2 \cdot 10^{23}$
		478	470	478
	Second / 1 h 50 min	$8.6 \cdot 10^{23}$	$1.0 \cdot 10^{24}$	$9.1 \cdot 10^{23}$
		463	498	488
	Third / a week	$8.5 \cdot 10^{23}$	$9.0 \cdot 10^{23}$	$8.7 \cdot 10^{23}$
		463	498	498
Without history	First / a week	$7.8 \cdot 10^{23}$	$9.9 \cdot 10^{23}$	$4.8 \cdot 10^{23}$
		508	483	396

Table 1 Exposure conditions: maximum exposure flux (upper sub-section,  $\text{m}^{-2}\text{s}^{-1}$ ) and maximum exposure temperature (lower sub-section, K).

Samples	Exposure / TDS after	W	W-1%Ta	W-5%Ta
With history	First / 1 h 50 min	$\frac{3,9*10^{16}}{+24 \%}$	$\frac{2,5*10^{16}}{-20 \%}$	$\frac{3,0*10^{16}}{-4 \%}$
	Second / 1 h 50 min	$\frac{3,5*10^{16}}{-6 \%}$	$\frac{3,9*10^{16}}{+4 \%}$	$\frac{3,8*10^{16}}{+2 \%}$
	Third / a week	$\frac{2,3*10^{16}}{-10 \%}$	$\frac{3,1*10^{16}}{+20 \%}$	$\frac{2,3*10^{16}}{-10 \%}$
Without history	First / a week	$\frac{1,4*10^{16}}{+10,5 \%}$	$\frac{1,4*10^{16}}{+10,5 \%}$	$\frac{1,0*10^{16}}{-21 \%}$

Table 2 Total deuterium amount retained in the investigated samples for the different conditions (the upper line) and its deviation from the value averaged over the 3 grades at the same conditions (the bottom line).

Fig. 1 TDS spectra of D<sub>2</sub> release from the samples of W, W-1%Ta and W-5%Ta with history and without history of previous exposures.

Fig. 2 Comparison of desorption spectra, recorded shortly after the first exposure of the sample with history (“fast TDS”) and a week after the exposure (for the samples without history).

Fig. 3 Comparison of the spectra of D<sub>2</sub> release, recorded a week after the exposure, for the samples with and without history.

Fig. 4 TDS spectra of D<sub>2</sub> release from the annealed W sample with history, annealed and not annealed W samples without history, measured a week after the plasma exposure.

Table 1 Exposure conditions: maximum exposure flux (upper sub-section, m<sup>-2</sup>s<sup>-1</sup>) and maximum exposure temperature (lower sub-section, K).

Table 2 Total deuterium amount retained in the investigated samples for the different conditions (the upper line) and its deviation from the value averaged over the 3 grades at the same conditions (the bottom line).