Tritium retention in displacement-damaged tungsten exposed to deuterium-tritium gas mixture at elevated temperatures

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Abstract

W samples, previously irradiated with 20 MeV W ions at room temperature to a displacement-damage level of 0.23 displacements per atom (dpa) at the peak of displacements, were exposed to a deuterium-tritium (D_2 – DT – T_2) gas mixture with a tritium content of 6% at a total pressure of 1.2 kPa and temperatures of 773 K and 973 K for 3 h. The concentration of tritium retained in the displacement-damage zone of these W samples was determined by a method combining chemical etching and subsequent analysis of tritium in the etching solution using liquid scintillation counting (CE-LSC).

Keywords: Tungsten, Ion-induced defects, Deuterium-tritium gas mixture, Liquid scintillation counting.

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1. Introduction

Due to its favorable physical properties, such as low erosion yield and high melting point, tungsten (W) is employed or is a candidate material for plasma-facing high heat-flux components in operating and developing fusion reactors such as ASDEX-Upgrade [1], ITER [2], QUEST [3], and DEMO [4]. As a plasma-facing material in future fusion reactors, tungsten will be subjected to intense fluxes of deuterium (D) and tritium (T) energetic particles, as well as 14 MeV neutrons arising from the D-T fusion reaction. Irradiation with fusion neutrons leads to a change in the microstructure of the W material due to the generation of displacements in the bulk [5, 6]. The appearance of displacement-damage-induced defects leads to concerns about the tritium inventory in the W components under exposure to the D-T plasma.

Increased deuterium retention by displacement damage-induced defects was demonstrated in laboratory experiments in which W samples irradiated with MeV-range W ions to displacement-damage levels in the range from 4.6×10^{-3} to 4.6 dpa[1] were subsequently exposed to D plasmas and D_2 gas [7-11]. The first results on deuterium retention in neutron-irradiated W after exposure to D plasmas demonstrates that neutron-induced defects created at relatively low displacement-damage levels in the range of 4.3×10^{-4} to 0.3 dpa significantly increase deuterium retention [12-20].

An important parameter for assessing the accumulation of hydrogen isotopes in displacement-damaged tungsten is the concentration of hydrogen isotopes retained in the damage zone. Deuterium concentration is usually determined using the $D(^3He,p)^4He$ nuclear reaction (NRA) [7-11]. However, when the concentration of deuterium is less than 10^{-2} at.%, the error in measuring a low concentration becomes significant and can reach 50-80 percent.

[1] In Refs. [7-11, 15, 16], displacement-damage levels in W samples irradiated with 20 MeV W and 6.4 MeV Fe ions were calculated using the program SRIM 2008.03 with the "Full Cascade" calculation option. Using the "Quick Kinchin Pease" calculation option for irradiation with MeV-range metal ions specified in [7-11, 15, 16] gives values of displacement-damage levels 2.2 times lower than indicated in the original works. In this paper, considering the results of experiments in which W samples were irradiated with MeV-range metal ions, previously published displacement-damage levels were recalculated using the program SRIM 2008.04 with the "Quick Kinchin Pease" calculation option. For a more detailed explanation, please see the Section "Experimental".

It seems that the use of radioactive tritium as a hydrogen isotope in studies of accumulation to low concentrations can increase the reliability of the results obtained [21].

The aim of this work was to study the retention of tritium in displacement-damaged W exposed to a $D_2 - DT - T_2$ gas mixture[2] at elevated temperatures. In this study, we used the tritium imaging plate technique [22-24] in combination with chemical etching and subsequent analysis of tritium in the etched layer using liquid scintillation counting (CE-LSC).

2. Experimental

Plates of polycrystalline tungsten (A.L.M.T. Co., Japan), 10×10×2 mm^3 in size, recrystallized fully at 2073 K by dry-hydrogen annealing after cutting and double-sided polishing, with a purity of 99.99 wt% and a grain size of 20-200 μm [25] were used in this work.

In an implantation chamber connected to a 3 MV tandem accelerator located at Max-Planck-Institut für Plasmaphysik, Garching, two W samples were irradiated at room temperature with 20 MeV W ions to a fluence of 8×10^{17} W/m^2. As a result, the W samples were damaged to 0.23 displacements per atom (dpa) at the displacement-damage peak located at a depth of 1.35 μm. The displacement-damage profile was calculated using the program SRIM 2008.04 [26] with the "Quick Kinchin Pease" calculation option [27] and with a displacement threshold energy of E_{th} = 90 eV, as recommended by the American Society for Testing and Materials [28]. As noted in [27], when using the program SRIM 2008.04 with the same parameters but different calculation options ("Quick Kinchin Pease" or "Full Cascade") or evaluating different output files ("vacancy.txt" or "e2recoils.txt"), there might be a difference up to a factor of two in determining the number of displacements depending on the procedure applied, as stressed by Stoller et al. and Nordlund et al. [29, 30]. These two studies recommend using the "Quick Kinchin Pease" calculation option.

Irradiation with W ions was performed by scanning the W ion beam over a circular aperture with a diameter of 8 mm installed in front of the sample. Thus, about half of the surface area was uniformly irradiated with 20 MeV W ions, while the rest was not affected. This circumstance allows a comparative analysis of the accumulation of tritium in displacement-damaged and undamaged near-surface layers of the W sample.

The displacement-damaged W samples were exposed to a $D_2 - DT - T_2$ gas mixture with a tritium content of 6% at a total pressure of 1.2 kPa and temperatures of 773 K and

[2] Note that the concentration of DT is far higher than that of T_2.

973 K for 3 h. As previously shown in experiments with tungsten previously damaged with 20 MeV W ions and then exposed to D_2 gas at a pressure of 1.2 kPa, a exposure time of 3 h is sufficient to fill the displacement-damage zone with deuterium at 773 K [10].

Each W sample was placed in a quarts tube, heated to the required temperature, exposed to a $D_2 - DT - T_2$ gas mixture for required time, and quenched within 10 seconds after termination of the gas exposure by dropping the quartz tube into liquid nitrogen. After two hours, the quartz tube was removed from liquid nitrogen and warmed to room temperature. When this happened, hydrogen isotopes released from the sample were continuously evacuated by an ion getter pump.

A qualitative assessment of the amount of tritium retained in the undamaged and displacement-damaged areas of the W samples was evaluated using the tritium imaging plate technique in combination with chemical etching. As a standard for tritium autoradiography, a tritium-labeled polymer parallelepiped of $5 \times 7 \times 0.006$ mm^3 in size and with an activity of 3.87×10^4 Bq was used. The activity areal density of the standard was 1.1×10^9 Bq/m^2 (1 Bq = 5.6×10^8 T-atoms).

An imaging plate (IP) is a flexible image sensor based on photostimulated luminescence (PSL), realized by crystals of photostimulable BaFBr doped with Eu^{2+} as a luminescence center. The IP used in our experiments was BAS-TR2025 (Fuji Film Co. Ltd.) which consists of a 50 μm thickness photostimulable phosphor mounted on a plastic support. The IP surface was contacted simultaneously to the W surface and tritium standard and exposed to tritium beta electrons[3], which generates excited electrons in the phosphor crystal leading to F-center formation. In order to avoid the contamination of the IP with tritium, a thin (1.2 μm) polyphenylene sulfide film was inserted between the IP and the W sample and T standard. After exposure to tritium beta electrons, the IP was processed by an IP reader (FLA-7000, Fuji Film Co. Ltd.) to obtain a digital image of tritium. Tritium activity is expressed as the photostimulated luminescence (PSL) intensity (Fig. 1), which is proportional to the absorbed radiation energy.

An attempt was made to determine the depth profile of tritium concentration in the displacement-damaged W samples by removing surface layers from the damaged side by chemical etching in a mixture of an aqueous solution of NaOH and hydrogen peroxide H_2O_2. The opposite side and the edges of each W sample were protected by adhesive tape. Before and after each etching procedure, the sample was weighed on an analytical balance and the

[3] The escape depth of tritium beta electron in W is less than 0.3 μm [31].

averaged etch layer thickness was estimated from weight loss with an accuracy of 0.01 mg. In total, an average of 17 μm was eroded. In each etching process, a tritium-loaded W sample was immersed in a small flask containing 0.5 ml of a 20 wt% NaOH aqueous solution and 0.5 ml of hydrogen peroxide H_2O_2. The flask was shaken regularly to ensure homogeneous contact between the sample and the etching solution. After an appropriated period of etching, the sample was removed from the etching solution and repeatedly rinsed from the etched side with 4 ml deionized water. Then the rinsed water was poured into a flask containing an etching solution to collect all the tritium contained in the etched layer [32]. Finally, the amount of tritium in each flask was determined by the method of low background liquid scintillation counting (LSC) using an instrument LB-5 (Aloka Co., Japan). Using the amount of tritium detected in the flask after each etching procedure, the thickness of the etched layer, the percentage of tritium in the $D_2 – HD - T_2$ gas mixture, and the surface area of the W sample irradiated with 20 MeV W ions, it was possible to determine the total concentration of D and T atoms in each etched W layer averaged over the etched thickness. Note that the etched layers consisted of displacement-damaged and undamaged regions (recall that irradiation with W ions was performed through a circular aperture with a diameter of 8 mm, and the ratio of the areas of the irradiated surface to the unirradiated surface was 1 to 1). Obviously, that the tritium content in the undamaged region was significantly lower than the tritium content in the region damaged by irradiation with 20 MeV W ions [11].

The uniformity of chemical etching was verified by scanning electron microscopy (SEM) after the final etch step. It was found that chemical etching led to a significant development of surface roughness due to heterogeneous etching of crystallites with different face orientations (Fig. 2). Consequently, the use of a method combining chemical etching and subsequent analysis of tritium in an etching solution by LSC (CE-LSC) does not make it possible to correctly determine the tritium depth profiles in recrystallized W samples when etching layers with an average integral thickness exceeding the thickness of the sub-surface displacement-damage zone[4]. However, to correctly determine the concentration of tritium in the displacement-damage zone using etching of rather thin layers, a combination of conditions is required, which will be discussed below.

3. Results

[4] It can be expected that in the case of chemical etching of fine-grained polycrystalline tungsten, the development of roughness will not be as significant as in the case of recrystallized tungsten.

Figure 1 shows a false colour image of the tritium intensity for a displacement-damaged recrystallized W sample exposed to a $D_2 – DT – T_2$ gas mixture at a total gas pressure of 1.2 kPa and a temperature of 773 K for 3 h. The increased tritium intensity is clearly visible on the central rounded part of the W surface, which was irradiated with W ions. A significant increase in the tritium retention in the displacement-damaged subsurface W layer indicates the trapping of tritium at displacement-induced defects created due to irradiation with 20 MeV W ions.

A relative comparison of the intensities of photostimulated luminescence (PSL) (or, in other words, tritium activity) for undamaged and damaged areas of the W samples exposed to the $D_2 – DT – T_2$ gas mixture at 773 K and 973 K are shown in Fig. 3 (left ordinate scale, in arbitrary units, symbols). In addition, the same figure shows the change in the total concentration of deuterium and tritium in depth (expressed in units of the (D + T)/W atomic ratio) in the displacement-damaged zone of each W sample (right ordinate scale, histogram). Recall that the tritium concentration in each etched W layer was determined by liquid scintillation counting (LSC).

However, the depth profiles of hydrogen isotopes determined by the CE-LSC method do not correspond to the calculated displacement-damage profile. Depth profiling of tritium using chemical etching leads to the appearance of a smoothly falling tritium distribution reaching a depth of more than ten micrometers (Fig. 3). However, as reported in Ref. [10], deuterium depth profiles determined by a nuclear reaction analysis (NRA) showed that in recrystallized W samples damaged with 20 MeV W ions and then exposed to D_2 gas at temperatures in the range from 673 K to 973 K, deuterium concentration in the displacement-damage zone is almost constant at a depth of 2 μm and decreases by about two orders of magnitude already at a depth of 3 μm.

There are two possible explanations for the appearance of this smoothly falling tritium distribution. The first explanation is associated with the occurrence of roughness created as a result of uneven chemical etching of differently oriented faces of W crystallites (Fig. 2). But assuming that tritium is retained only in the displacement-damaged zone, it is difficult to explain such a relatively gentle decrease in the concentration of tritium with an increase in the total thickness of the etched layer.

A second possible additional explanation of this gentle decrease in tritium concentration, as measured by the CE-LSC method, is as follows. It can be assumed that upon irradiation of recrystallized W samples with 20 MeV W ions, due to the appearance of

Frenkel pairs and the long-distance migration of self-interstitial atoms or due to other mechanism, dislocation-type low binding energy traps are created at depths exceeding the calculated depths of the dispacement-damage zone. At exposure temperatures ≥ 773 K, these traps are not able to retain hydrogen isotopes. But when displacement-damaged W sample is cooled within 10 seconds to the temperature of liquid nitrogen after termination of the gas exposure, the dissolved hydrogen isotope atoms can be captured by these traps. However, in the experiments described in Ref. [10], after termination of D_2 exposure, displacement-damaged W samples were cooled to room temperature for approximately 15 minutes. In this case, dissolved deuterium could be released from the sample without being trapped by the low binding energy traps. It should be emphasized that the second possible explanation is only an assumption. To verify the truth of this assumption, it is necessary to conduct additional experiments with fast and slow cooling of displacement-damaged tungsten samples after exposure to gaseous deuterium or tritium at elevated temperatures.

Despite the aforementioned drawback of uneven chemical etching as a profiling method, it is worth noting that measuring the tritium content in etched near-surface tungsten layers with an average thickness of up to 1.5 μm using the CE-LSC method (Fig. 3) allows one to estimate the tritium concentration (and, therefore, the total concentration of tritium and deuterium) in the displacement-damage zone. However, if, after chemical etching of a layer with an average thickness of 1.5 μm, the roughness is sufficiently high, then the concentration of hydrogen isotopes will be slightly underestimated.

In a displacement-damaged W sample exposed to a $D_2 – DT – T_2$ gas mixture at a total gas pressure of 1.2 kPa and a temperature of 773 K for 3 h, the relatively high total concentration of hydrogen isotopes (approximately 2×10^{-3} (D + T)/W) in the first etched near-surface layer with a thickness of 0.05 μm can be explained by the adsorption of hydrogen isotopes on the W sample surface. The total concentration of hydrogen isotopes in the two subsequent etched layers with thicknesses of 0.5 μm and 1.1 μm were determined to be 5.5×10^{-4} (D + T)/W and 4.6×10^{-4} (D + T)/W, respectively (Fig. 3, a). Given the complexity of preparing a solution for measuring tritium content by the CE-LSC method and the accuracy of measuring weight loss to determine the thickness of etched layers, we can say that the obtained values of the total concentration of hydrogen isotopes in the second and third etched layers were in good agreement with each other.

In a displacement-damaged W sample exposed to a $D_2 – DT – T_2$ gas mixture at 973 K, the total concentration of hydrogen isotopes in the first etched layer with a thickness 1.5 μm was determined to be 1.9×10^{-4} (D + T)/W (Fig. 3, b).

Data on the total concentration of hydrogen isotopes (in units of (D + T)/W atomic ratio) trapped in the displacement-damage zone of W samples after exposure to a $D_2 – DT – T_2$ gas mixture at a total pressure of 1.2 kPa and at temperatures of 773 K and 973 K for 3 h are shown in Fig. 4. Note that the accuracy in determination of tritium content in the $D_2 – DT – T_2$ gas mixture is not very high and, accordingly, the error in determining the total concentration of deuterium and tritium can reach 30 percent.

To verify the obtained values of the total concentration of deuterium and tritium, it is necessary to compare them with the previously obtained data on the D concentration in recrystallized W samples damaged at room temperature with 20 MeV W ions to 0.23 dpa and then exposed to D_2 gas at a pressure of 1.2 kPa and temperatures in the range from 773 K to 1073 K for 1-3 h [10]. In these experiments described in Ref. [10], the duration of D_2 gas exposure for each exposure temperature was chosen so that the entire displacement-damage zone was saturated with deuterium, and the D concentration was determined by nuclear reaction analysis (NRA). These previously published data on the D concentration (in units of the D/W atomic ratio) are additionally shown in Fig. 4.

Comparison of the data obtained in this work with previously published data [10] shows that the total concentration of hydrogen isotopes (D + T) in the displacement-damage zone determined by the CE-LSC method is in good agreement with the concentration of deuterium measured by NRA (Fig. 4). Obviously, the level of roughness after chemical etching of the W layer with an average thickness of 1.5 μm was not significant, which made it possible to fairly accurately determine the concentration of tritium in the displacement-damage zone.

It is pertinent to note that the CE-LSC method can also be used to determine the concentration of tritium in tungsen materials exposed to deuterium-tritium plasmas.

6. Summary

The concentration of tritium retained in the recrystallized W samples pre-irradiated with 20 MeV W ions at room temperature to 0.23 dpa at the displacement-damage peak and then exposed to a $D_2 – DT – T_2$ gas mixture with a tritium content of 6% at a total gas pressure of 1.2 kPa and temperatures of 773 K and 973 K for 3 h was determined by a method combining chemical etching (CE) and subsequent analysis of tritium in the etched layer by liquid scintillation counting (LSC). Comparison of the data obtained in this work with previously published data [10] shows that the total concentration of hydrogen isotopes (D + T) in the

displacement-damage zone determined by the CE-LSC method is in good agreement with the concentration of deuterium measured by NRA.

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Figures

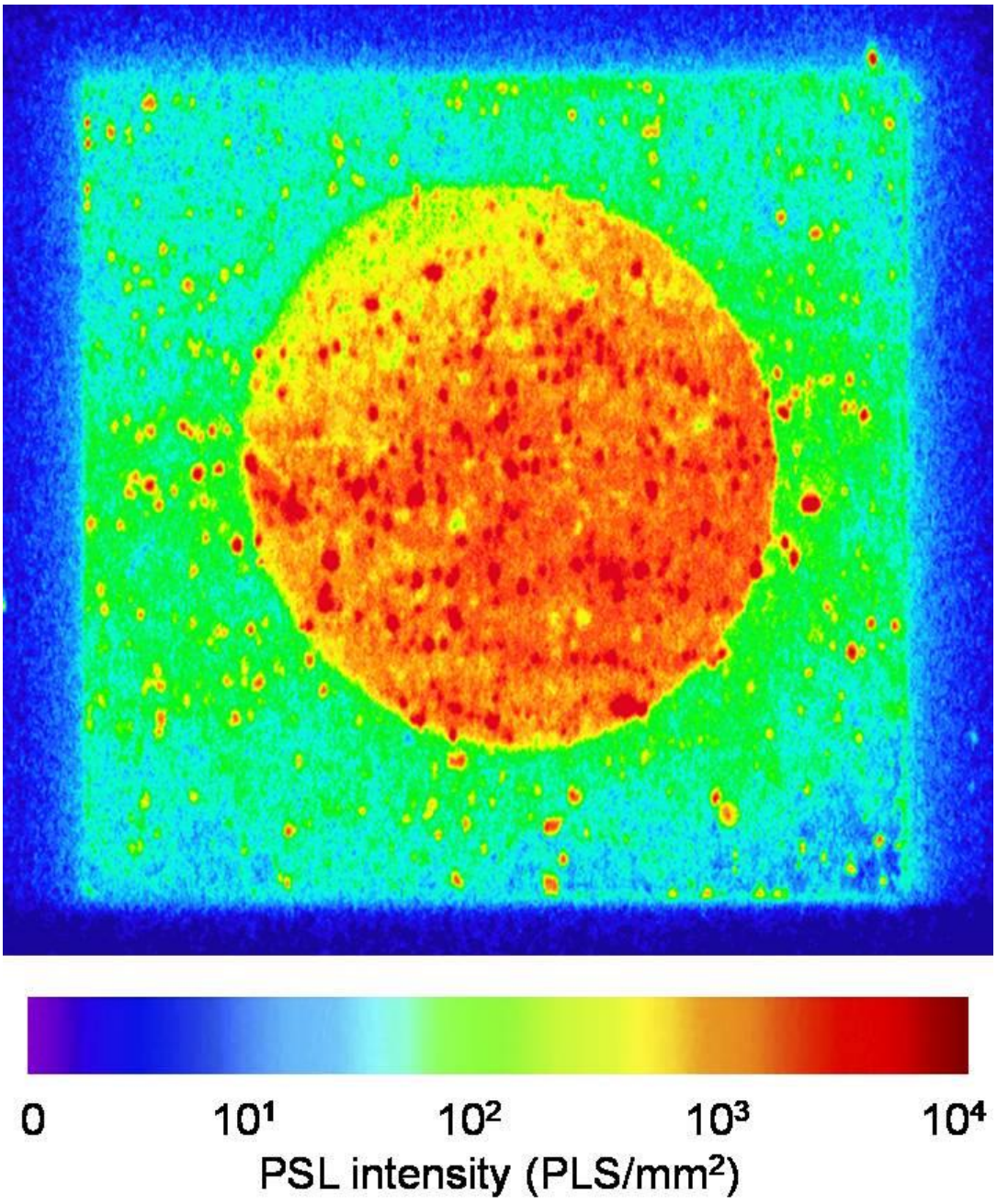

Figure 1. Tritium image of a recrystallized W sample damaged with 20 MeV W ions to 0.23 dpa at the depth of the damage peak and then exposed to a D_2 – DT – T_2 gas mixture with a tritium content of 6% at a total gas pressure of 1.2 kPa and a temperature of 773 K for 3 h. The image was taken about 3 h after tritium loading. Tritium level is higher in the red area and less in the blue area.

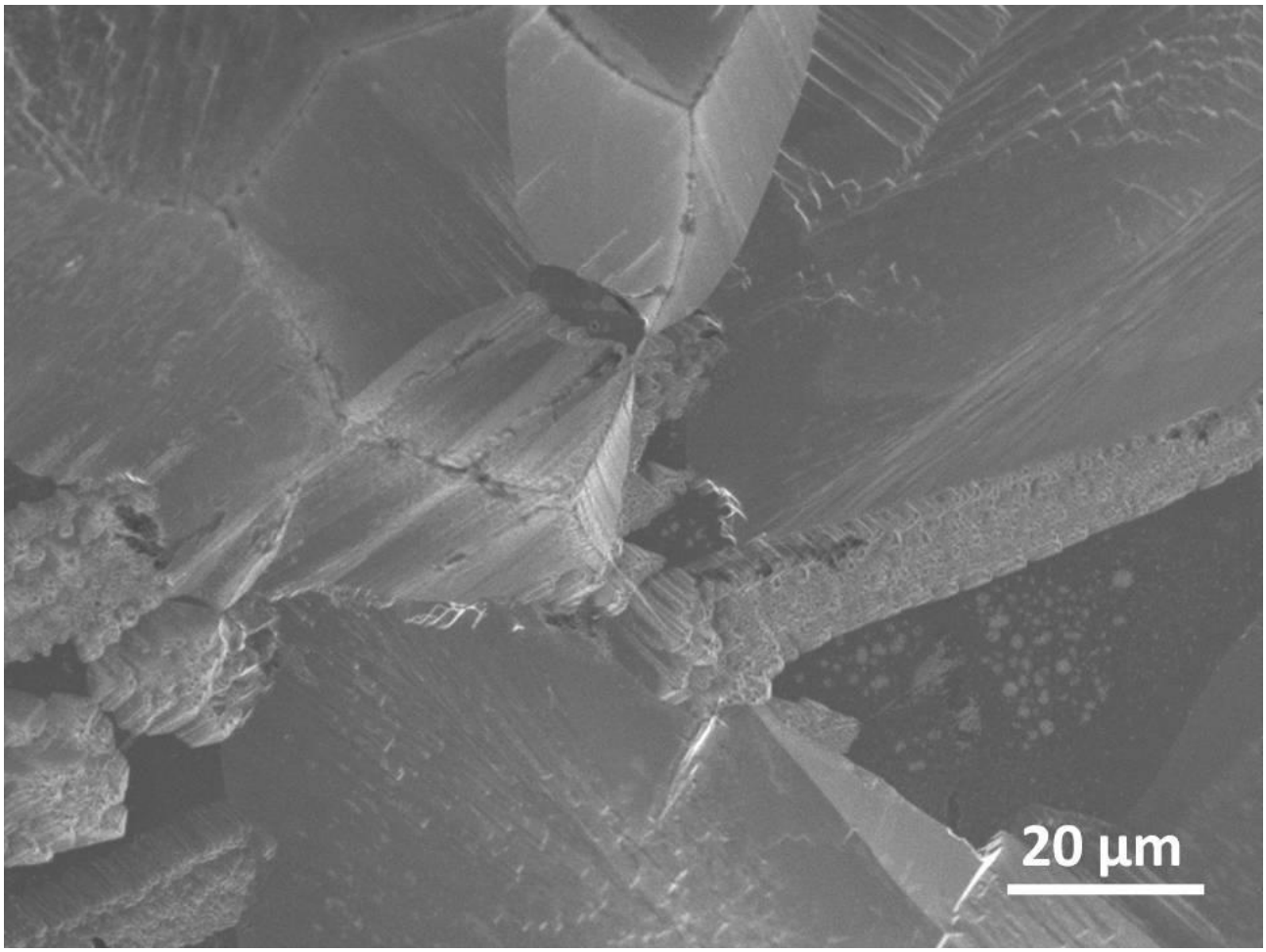

Figure 2. SEM image of the recrystallized W sample surface after chemical etching of a layer with an average thickness of 17 μm.

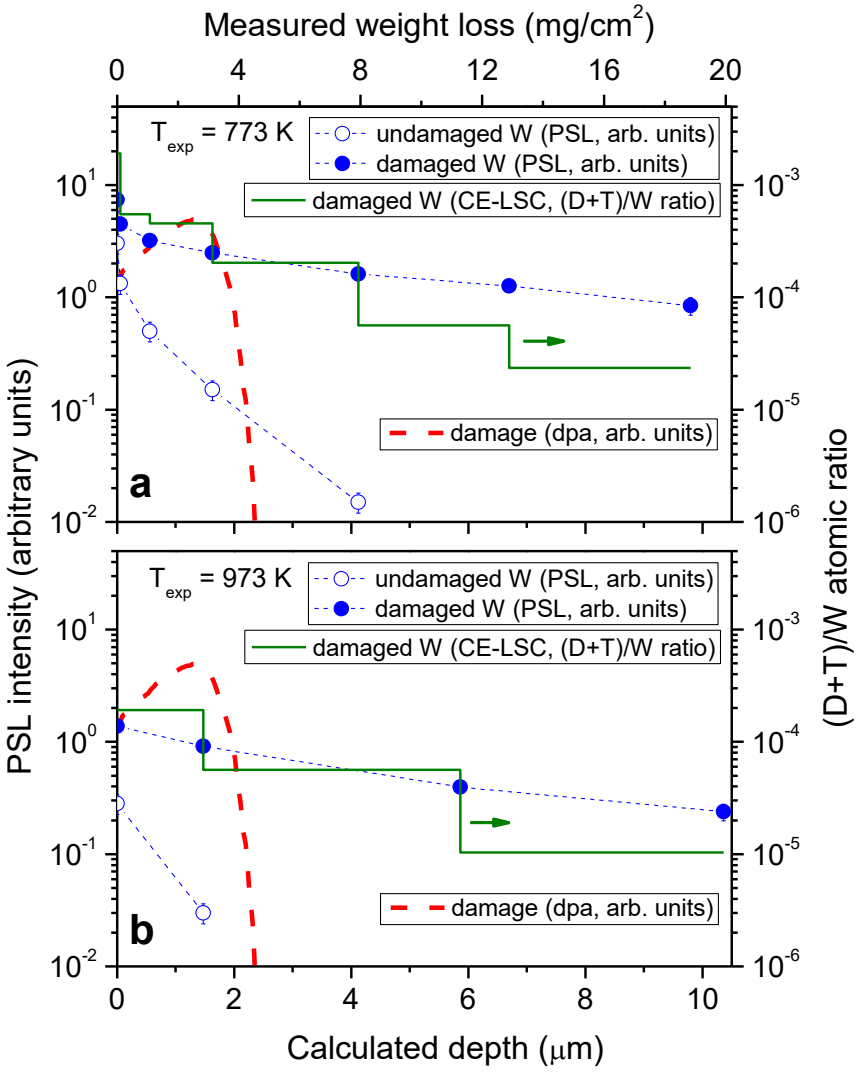

Figure 3. Left ordinate scale, symbols: - Depth dependence of tritium activity, expressed as the intensity of photostimulated luminescence (PSL) (in arbitrary units), in undamaged and displacement-damaged zones of recrystallized W samples exposed to a $D_2 - DT - T_2$ gas mixture with a tritium content of 6% at a total gas pressure of 1.2 kPa and temperatures of 773 K (a), and 973 K (b) for 3 h. Note that the PSL intensity reflects the tritium content in a thin surface layer, since the escape depth of tritium beta electron in W is less than 0.3 μm. Uncertainty in determining the escape depth of tritium beta electrons does not allow us to use the PSL intensity to determine the tritium concentration in near-surface W layers.

Right ordinate scale, histogram: - The total concentration of deuterium and tritium (in units of the (D + T)/W atomic ratio) in the displacement-damaged zone of recrystallized W samples, as determined by the CE-LSC method. The error in determining the total concentration of deuterium and tritium can reach 30 percent.

The calculated displacement-damage profile is indicated by dashed line (dpa, in arbitrary units).

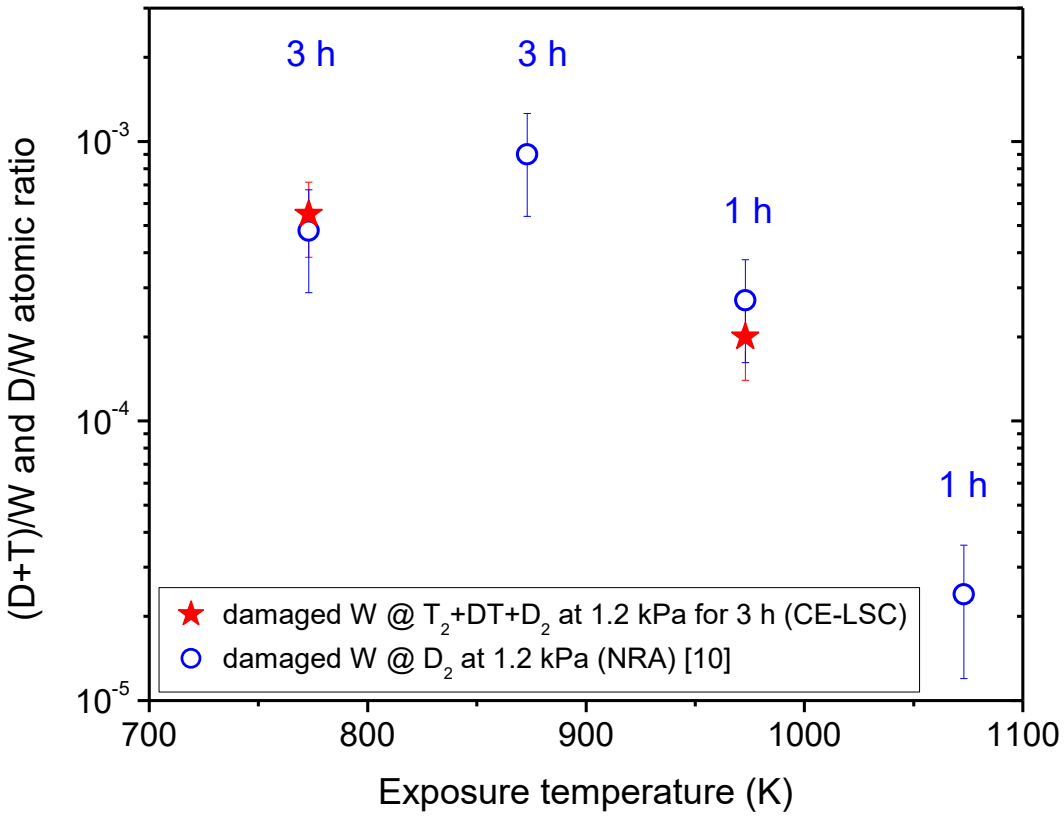

Figure 4. The total concentration of hydrogen isotopes and the D concentration (in units of the (D+T)/W and D/W ratios) in the displacement-damage zone of recrystallized W samples exposed to (i) a D_2 – DT – T_2 gas mixture with a tritium content of 6% at a total gas pressure of 1.2 kPa for 3 h, and (ii) D_2 gas at a pressures of 1.2 kPa for 1-3 h (the exposure time in the D_2 atmosphere for each exposure temperature is shown in Figure) [10], as a function of the sample temperature maintained during gas exposure.