Deuterium retention in tungsten damaged with W ions to various damage levels

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Highlights

- W samples were pre-irradiated with 4.8 and 20 MeV W ions to various displacements per atom (dpa).
- Under following exposure to D plasmas radiation-induced defects were occupied by diffusing D atoms.
- At ≥ 0.1 dpa, the D concentration in the damage zone demonstrated weak dependence on the damage level.

Abstract

W samples were irradiated at 300 and 573 K with 4.8 and 20 MeV W ions to displacement damage levels in the range from 0.022 to 50 displacements per atom at the damage peak. 50 μm thick W samples were exposed to high flux D plasma at 550 K on the side opposite to the damaged one, whereas 2 mm thick W samples were exposed to low flux D plasma at 403 K on the damaged side. Trapping of deuterium at displacement damage was examined by the D(^{3}He, p)^{4}He nuclear reaction with ^{3}He energies between 0.69 and 4.0 MeV allowing determination of the D concentration up to a depth of 6 lm. It was found that (i) at the damage level above 0.1 dpa, the concentration of the W-ion-induced defects responsible for trapping of diffusing D atoms depended very weakly on the numbers of displacements per atom, and (ii) the quasi-saturation concentration of the defects decreased by a factor of two as the W-ion irradiation temperature increased from 300 to 573 K.

1. Introduction

Due to its favorable physical properties, such as low erosion yield and high melting temperature, tungsten (W) is employed as a candidate material for plasma-facing high heat-flux structures in future fusion reactors. As plasma-facing material in the fusion reactors, W will be subject to intensive fluxes of energetic deuterium (D) and tritium (T) particles, as well as 14 MeV neutrons (n) from the D-T fusion reaction. Neutron irradiation generates displacements in the bulk of W and creates thus defects at which hydrogen isotopes can be trapped. Furthermore, irradiation of W with fusion neutrons creates several radioactive isotopes of tungsten and rhenium which might have effects on behavior of tritium in the n-irradiated W. Note that for W divertor elements in ITER, the level of neutron-induced displacement damage for whole campaign calculated with a displacement threshold energy of E_d = 38 eV[1] can reach 0.7 displacements per atom (dpa) [1], whereas in DEMO reactor the damage rate for W tiles is estimated to be 20–40 dpa per operational year [2]. Hydrogen retention in n-irradiated tungsten will occur throughout the bulk of the material and, therefore, there is the potential for large tritium inventories in fusion reactors [1,3]

The best way to investigate the influence of n-produced defects on hydrogen isotope inventory in W is to have a source of fusion neutrons which still does not exist. Another possibility is an irradiation of W samples with fast neutrons (E > 0.1 MeV) in nuclear fission reactors [4, 5, 6, 7]. However, because of low damaging rate and activation of samples, neutron irradiation to high damage levels (such as tens of dpa) is a difficult task.

One of the ways to investigate the influence of n-produced defects on the hydrogen isotope inventory is to simulate displacement damage in tungsten by irradiation with ions at energies in the range from several tenths to several tens of MeV. It has been experimentally shown [8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23] that irradiation of W materials with 0.2-20 MeV H, He, Si, and W ions leads to an enhancement of hydrogen isotope retention in comparison with undamaged materials. However, damage created by ions is concentrated in a narrow region below the surface (in the damaged zone), while neutron-induced damage extends to larger depths.

The dependence of D concentration trapped in the damage zone at the damage peak, ${}^{\mathrm{damage}}C_{\mathrm{D}}$, on the damage level in W was studied in several works [10, 13, 14, 16, 18, 21, 22]. For W pre-irradiated with 12 MeV Si ions and then exposed to low-energy, high flux D plasmas at 473 and 773 K, Wampler et al. [10] observed an increase of the concentration ${}^{\mathrm{damage}}C_{\mathrm{D}}$ as the damage level at the damage peak increased from 0.006 to 0.6 dpa (in this work the damage level

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[1] An uncertainty in the displacement threshold energy E_d affects only the level of predicted displacements per atom (dpa) and not the distribution of this damage.

was calculated with the displacement energy of E_d = 40 eV). In W damaged with 5.5, 12.3 and 20 MeV W ions and then irradiated with low energy (≤ 200 eV) D ions at 470 – 480 K, the concentration ${}^{\text{damage}}C_D$ saturated at 0.4 – 0.5 dpa calculated with E_d = 68 eV [14], E_d = 90 eV [16] and E_d = 40 eV [18]. On the contrary, using W pre-damaged with 300 keV H ions and then irradiated with 1 keV D ions at 473 K, Ueda et al. [13] showed that the concentration ${}^{\text{damage}}C_D$ increased linearly with the damage level up to ~0.2 dpa (E_d = 40 eV) and then, up to 1 dpa, the concentration ${}^{\text{damage}}C_D$ demonstrated insignificant growth. Takagi et al. [22] reported that for W damaged with 1.5 and 5 MeV W ions to 1.6, 11 and 110 dpa at the damage peak (E_d = 90 eV) and then exposed to low-energy deuterium RF plasmas, the average concentration of D in the damaged zone increased continuously with the damage level. Saturation of the hydrogen isotope retention is an important phenomenon because it gives an upper limit of the retention even for heavily damaged tungsten in fusion reactors.

This paper describes experiments to determine the damage level in W where the concentration of defects responsible for trapping of D atoms in the damage zone saturates. The displacement damage was created by irradiation with W ions at energies of several MeV. For neutrons and energetic heavy ions, the energy distribution of primary knock-on atoms peaks at high energies, and they both create dense collision cascades [24, 25]. Therefore, damages created by irradiation with energetic heavy ions can be good proxies for those induced by neutron irradiation.

To simulate properly the effects of displacement damage on the bulk retention of diffusing D atoms, the damaged W samples should be loaded with deuterium without formation of additional defects, which could interfere with W-ion-induced defects. In this work 50 μm thick W foils damaged at 300 K to various damage levels were exposed to high flux D plasma on the side opposite to the damaged one. In the second set of experiments, polycrystalline W samples damaged at 300 and 573 K up to 50 dpa were exposed on the damaged side to low-flux D plasma such that additional plasma-induced defects were not generated.

2. Experimental

Two types W materials were used in this work: (i) polycrystalline hot-rolled W foil; and (ii) mechanically-deformed polycrystalline W.

(i) 50 μm thick polycrystalline hot-rolled W foil from Goodfellow Cambridge Limited, England, has a purity of 99.95 wt.%. The microstructure of the W foil consists of two-dimensional flat grains, 2-5 μm in size and around 1 μm in thickness, positioned in parallel to the surface. No polishing and no annealing were performed after cutting of the foil into samples of 10 × 10 mm^2 in size. According to elastic recoil detection analysis [26], concentration of hydrogen in the hot-rolled W foils was below 10^{-2} at.%.

(ii) Mechanically-deformed W from A.L.M.T. Corp., Japan, has a purity of 99.99 wt.%. The microstructure of this W consists of anisotropically elongated grains along the deformation axis. The grain size is 1-3 µm in section and up to 5 µm in length. Individual elongated cracks observed between grains are due to the deformation treatment [27]. Two types of samples were prepared from mechanically-deformed polycrystalline W rods annealed at 1173 K for 30 min in a hydrogen atmosphere to relieve internal stresses occurred in the manufacturing process: square-shaped samples, 10 × 10 mm^2 in size and 2 mm in thickness, and disc-shaped samples were 6 mm in diameter and 0.2 mm in thickness. The sample were prepared such that the irradiated surfaces were perpendicular to deformation axis (i.e., to the heat transfer direction), which corresponds to the ITER specification [28]. The samples were mechanically polished, clean in the acetone ultrasonic bath, and then annealed in vacuum at 1173 K for 30 min to relieve stresses occurred in the polishing process.

The 50 µm thick hot-rolled W foils and square-shaped mechanically-deformed W samples were irradiated with 20 MeV W ions at 300 K to damage levels in the range from 0.022 to 0.5 dpa at the damage peak using the facility at IPP Garching. At Kyoto University, the mechanically-deformed W discs were irradiated with 4.8 MeV W ions at temperatures of 300 and 573 K to damage levels of 0.65 and 50 dpa at the damage peak. The numbers of displacement collisions[2], as a function of a depth, were calculated using the program SRIM 2008.03 [29], “full cascade option”, with a displacement energy of E_d = 90 eV [30, 31]. Note that for irradiation with 4.8 and 20 MeV W ions, the damage peak situated at a depth of 0.22 and 1.35 µm, respectively. In what follows the W-ion-irradiated W samples will be designated as “damaged” W samples, and a value of the damage level will be indicated as a number of dpa at the damage peak.

The 50 µm thick W foils, damaged with 20 MeV W ions at 300 K, were exposed to low-energy, high-flux deuterium plasma at a temperature of 550 K on the side opposite to the damaged one. This was done to avoid an interference of plasma-induced defects [27] with

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[2] The number of displacement collisions indicates how many target atoms were set in motion in the cascade with energies above their displacement energy. A vacancy is the hole left behind when a recoil atom moves from its original site. If a moving atom strikes a stationary target atom and transfers more than its displacement energy to it, and the initial atom, after the collision, does not have enough energy to move onwards, and it is the same element as the atom it struck, then it just replaces that atom in the target and there is no vacancy created. The number of displacements is therefore determined as the sum of the numbers of vacancies and replacement collisions [24].

displacement damage created by energetic W ions, and to realize conditions where diffusing D atoms interact with W-ion-induced traps. In addition, an undamaged W foil was also exposed to the high flux D plasma. The linear plasma generator used for delivering plasma beams is described in Ref. [32]. To generate high flux D plasma, the D_2 working pressure was kept at about 1 Pa. As a result, a plasma beam with species of D_2^+ (over 80%) and D^+ (less than 20%) was obtained. A bias voltage of -80 V was applied to the W sample, resulting in incident energy of 76 eV, taking into account the plasma potential of about -4 V as measured by a Langmuir probe. The incident deuterium ion flux was fixed at 10^{22} D/m^{2}s. The samples were exposed to ion fluence of 3×10^{26} D/m^2. The exposure temperature of 550 K was set by the thermal contact between the sample and the cooled holder. The temperature was monitored using a type K thermocouple tightly pressing the rear of the sample.

The damaged sides of the mechanically-deformed square-shaped and disc-shaped W samples were exposed to D particles at temperature of 403 K to low flux D plasma formed by DC glow discharge [20]. The sample on a holder equipped with an ohmic heater and a thermocouple, and this holder served as anode. A tungsten disc located at a distance of about 10 cm from the sample holder was used as a cathode. Deuterium pressure in the chamber was maintained at 1 Pa, DC discharge voltage was 400 V, and discharge current averaged about 0.18 A. Because the sample was set on an anode, the main impinging particles were D atoms and molecules (D neutrals) in addition to electrons. The energies of atoms were in the range from few eV (atoms originated in the glow discharge) to ~150 eV (atoms reflected from the W cathode). The flux of implanted deuterium was estimated to be about 2×10^{18} D/m^{2}s by measuring D uptake in a Ti sample with thermal desorption spectroscopy after exposure in the DC glow discharge at 300 K for 10 and 60 min [20]

The deuterium profiles were determined by nuclear reaction analysis (NRA) at IPP, Garching. The D concentration within the near-surface layer (at depths up to about 0.5 μm) was measured by means of the D(^{3}He, α)H reaction at a ^{3}He energy of 0.69 MeV with total 3He ion charge of 10 μC acquired on a spot size of 1mm2. The α particles were energy-analyzed with a small-angle surface barrier detector at the laboratory scattering angle of 102º. The α spectrum was transformed into a D depth profile using the program SIMNRA [33].

To determine the D concentration at larger depths, an analyzing beam of 3He ions with energies varied from 0.8 to 4.0 MeV was used, and at these energies the total 3He ion charge was 5 μC. The protons from the D(^{3}He, p)^{4}He nuclear reaction were counted using a wide-angle proton detector placed at a scattered angle of 135º [34]. In order to extend the D concentration to depths of 6 μm, the program SIMNRA was used for the deconvolution of the proton yields measured at different ^{3}He ion energies. A deuterium depth distribution was assumed taking into account the near-surface depth profile obtained from the a particle spectrum, and the proton yield as a function of incident ^{3}He energy was calculated. The shape of the D depth profile was then varied using an iterative technique until the calculated curve matched the measured proton yields [35].

3. Results

3.1. 50 µm thick W foils pre-irradiated at 300 K with 20 MeV W ions to various damage levels and exposed on the opposite side to high flux D plasma at 550 K

For W foils irradiated with 20 MeV W ions at 300 K to the damaged level of 0.022 dpa and then exposed on the opposite side to low-energy, high flux D plasma at 550 K to incident D ion fluence of 3×10^{26} D/m^2, deuterium depth profile on the damaged side coincides with the calculated damage profiles (Fig. 1a) indicating that radiation-induced defects are decorated by D atoms diffusing from the plasma-exposed side. Additionally, D atoms can migrate from the damage side surface due to exposure to D_2 gas at a pressure of ~1 Pa (i.e., at gas pressure maintained in the linear plasma generator during plasma exposure). As the damage level increases above 0.022 dpa, the D concentration at the damage peak increases roughly in proportion to the damage level, and then, at the peak damage levels over 0.1 dpa, the gradient of the increase becomes shallow (Fig. 1b).

It should be noted that lowest D concentration at the damage peak observed for the lowest damage level of 0.022 dpa is about 4×10^{-2} at.% (Fig. 1a), whereas the H concentration in the unannealed W foils is below 10^{-2} at.%. There is no data on redistribution of the inherent hydrogen from intrinsic traps to ion-irradiated defects; however, the influence of the inherent hydrogen on the D retention in the damage zone is thought to be negligible.

For the undamaged W foil exposed on one side to the high flux D plasma, the D depth profile on the opposite side demonstrates a concentration of about 10   2 at.% on the surface and about 5×10^{-4} at.% in the bulk, i.e., at depths of 46–49 µm (Fig. 1a).

On the plasma exposed side, deuterium depth profiles in the damaged W foils are characterized by sharp near-surface concentration maxima of (4–7) × 10^{-2} at.%, and, at depths of 2–5 µm, by a concentration of about (0.8–1.5) × 10^{-2} at.% (Fig. 1a). Such scatter of the concentration data can be explained by large error in determination of the D concentration due to low statistics of the proton counts. Concentration minima at depths around 1 µm is thought to be due to thermal migration and release of D atoms after termination of the exposure.

In the plasma-exposed undamaged W foil the D concentration at depths of 3–5 μm is approximately half compared to that in the damaged W foils. Note that the same result was observed previously for 25 μm thick W foils, undamaged and damaged with W ions on the one side and exposed to the D plasma on the opposite side [15]. It has been proposed by Tyburska et al. [15] that irradiation of W foils with MeV W ions generates not only radiation-induced defects but also stresses the matrix lattice until plastic deformation occurs to alleviate the tensions. This deformation is assumed to be responsible for generation of vacancies, vacancy clusters and dislocation loops [36] at depths far beyond the displacement damage zone, i.e., at distance of several tens of micrometers. Hereupon, the D concentration at these depths increases due to trapping at vacancy-types defects.

3.2. Mechanically-deformed polycrystalline W pre-irradiated at 300 K with 20 MeV W ions to 0.5 dpa and exposed on the same side to low flux D neutrals at 403 K

In undamaged mechanically-deformed W exposed at 403 K to low flux D plasma[3] for 3 h, deuterium is detected at depths less than 0.03 μm, and the D concentration in deeper layers (at depths ≥ 0.03 μm) is below 10^{-2} at.% (Fig. 2). Generation of 20 MeV W-ion-induced displacement damage at 300 K and subsequent exposure of the damaged side to the low flux D plasma for the same exposure time of 3 h significantly increases the D concentration at depths up to 1 μm, and full width at half maximum (FWHM) of the plateau-like D depth profile is about 0.75 μm (Fig. 2). After exposure to the low flux D plasma for 12 h, the FWHM increases twice, whereas the D concentration at the plateau reaching 1.3 μm remains unaltered (Fig. 2). Thus, we can say that the D depth profiles are consistent with saturable strong traps[4] being filled by D atoms diffusing from the surface.

It should be noted that in the mechanically-deformed W damaged to 0.5 dpa, the damage level increases from ~0.2 to 0.5 dpa with increasing depth from the surface up to 1.35 μm (see damage profile in Fig. 2). However, after exposure to the low flux D plasma for 12 h, the D concentration is almost uniform up to 1.2 μm (Fig. 2). This is also evidence that the concentration of defects responsible for trapping of diffusing D atoms increases insignificantly at the damage levels above 0.2 dpa.

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[3] Note that the flux of implanted deuterium was estimated to be about 2×10^{18} D/m^{2}s.

[4] Saturable traps are traps having finite capacity. Traps where D in solution becomes trapped when it encounters unoccupied traps are denoted as strong traps [10].

3.3. Mechanically-deformed polycrystalline W pre-irradiated at 300 and 573 K with 4.8 and 20 MeV W ions to various damage levels and exposed on the same side to low flux D plasma at 403 K

In mechanically-deformedWdamaged at 300 K with 20 MeV W ions to 0.3 and 0.5 dpa and then exposed to the low flux D plasma for 9 h, a plateau in the D concentration profiles reaches about 1.3 μm, whereas the sub-surface region damaged to ≥ 0.1 dpa extends up to a depth of about 2 μm (Fig. 3a). Obviously, D plasma exposure for 9 h does not provide sufficient amount of diffusing D atoms to occupy all W-ion-induced defects.

After irradiation at 300 K with 4.8 MeV W ions to 50 dpa and following low flux D plasma exposure, the D depth profile demonstrates a plateau with the same D concentration as for the W sampled damaged with 20 MeV W ions to 0.3 and 0.5 dpa. However, the 50-dpa-concentration plateau and tail extend beyond the 4.8 MeV W damage profile (Fig. 3a).

In mechanically-deformed W irradiated with 4.8 MeV Wions at 573 K to a damage level of 0.65 dpa, deuterium is retained practically within the damage zone. However, for higher damage level of 50 dpa, the D profile demonstrates trapping of deuterium at depths far beyond the calculated damage profile (Fig. 3a).

After irradiation with MeV-range W ions at temperatures of 300 and 573 K to the damage levels in the range from 0.5 to 50 dpa and following exposure to the low flux D plasma, the D concentration at the profile plateau demonstrates very weak dependence on the number of displacements per atom. However, after damaging at 573 K the concentration at the plateau is lower by a factor of ~2 compared to that for damaging at 300 K (Fig. 3b).

4. Discussion

Analyzing data on the D concentration observed after irradiations of damaged W to D plasmas or D ions at different conditions, it is necessary to keep in mind that at the same concentration of the ion-induced defects, the saturation value of the D concentration in the damage zone depends both on the W target temperature and on the concentration of D atoms in solute state maintained under D plasma/ions irradiation [20]. Therefore, we cannot compare directly values of the D concentration measured after different conditions of D loading.

It has been found that (i) the D concentration at the damage peak increases in proportion to the damage level roughly, and then, at the damage levels above 0.1 dpa, the gradient of the increase becomes shallow, and (ii) at the damage levels in the range from 0.5 to 50 dpa, the concentration of the W-ion-induced defects responsible for trapping of diffusing D atoms demonstrates very weak dependence on the number of displacements per atom. These results are in line with data on the damage level dependence of the D concentration reported in Refs. [10,13,14,16,18] for damage levels below 1 dpa. On the other hand, Takagi et al. [22] have reported that in W pre-irradiated with 1.5–5 MeV W ions at 300 K and then exposed to D plasma at elevated temperatures (373–663 K), average concentration of D retained in the damage zone for one sample damaged with 5 MeV W ions to 110 dpa is higher by a factor of about 1.6 than that for W damaged with 1.5 MeV to 1.6 dpa.

Deuterium profiles shown in Fig. 3a demonstrate that irradiation with MeV-range W ions to higher damage level of 50 dpa does not increase the defect concentration in the damage zone but extends the depth where defects are generated up to the saturation concentration. Above-mentioned mechanism of stress-induced generation of defects responsible for trapping of D atoms can explain the D depth profile extension with increasing damage level. Thus, the increase of the average D concentration in the work by Takagi et al. [22] is thought to be partly due to an extension of the primary damage zone with growing damage level, as mentioned above, and an increase of the D amount retained in the extended damage zone[5].

It is well known that in the course of collisions between high-energy particles and lattice atoms, vacancies and interstitials (Frenkel defects) are created [24]. The development of radiation-induced vacancy and interstitial concentrations occurs due to competing processes. These defects can be lost either through recombination of vacancies and interstitials or by reaction with defect sinks (voids, dislocations, dislocation loops, grain boundaries or precipitates) [24]. Deuterium atoms injected into damaged W settle into interstitial solution sites, diffuse through the metal lattice, and become trapped at vacancy-type defects, like single vacancies and vacancy clusters. Obviously, at damage levels of ≥ 0.5 dpa the rate of the vacancy-interstitial recombination in the primary damage zone becomes comparable with the rate of Frenkel defect generation, and the concentration of vacancy-type defects saturates. However, one cannot exclude migration of vacancies and interstitials beyond the primary damage zone.

According to collision-cascade simulation in the associated body-centered-cubic arrays of atoms, generation of collision cascades in W may result in direct formation of vacancy clusters [37]. As reported in Ref. [38], irradiation of W, at 300–310 K, with 100–150 keV heavy ions (Zn, Hg, and W) to fluences up to 10^{19} ions/m^2 or fast neutrons to fluences up to 5.4×10^{23} n/m^2 produces vacancy damage consisting predominantly of small clusters (each containing less than

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[5] Small depth of the damaged zone generated with 1.5–5 MeV W ions (up to ~0.2–0.5 lm) and an insufficient resolution of a proton detector utilized to determine D depth profiles with the use of the D(^{3}He,p)^{4}He nuclear reaction at ^{3}He energy of 1.7 MeV [22] does not allow accurate measurement of the D concentration at the depth of the damage peak. At these experimental conditions, the amount of D retained at depths lp to 2 lm influences strongly the shape of the proton energy spectrum.

ten vacancies). Formation of large vacancy clusters (or nano-sized voids) comprising 300–600 vacancies in W after irradiation with 50 keV W ions at 300 K to the damage levels from 0.01 to 5.5 dpa was observed with the use of field ion microscopy [39].

5. Summary

To determine the damage level at which a concentration of defects responsible for trapping of diffusing D atoms reaches saturation, W samples were irradiated at 300 and 573 K with 4.8 and 20 MeV W ions to damage levels in the range from 0.022 to 50 dpa at the damage peak. Under following exposure to low and high flux D plasmas correspondingly at 403 and 550 K, radiation-induced defects were occupied by diffusing D atoms. It has been found that (i) at the damage levels in the range from 0.1 to 50 dpa, the concentration of the W-ion-induced defects demonstrates very weak dependence on the number of displacements per atom, and (ii) the quasi-saturation concentration of the W ion-induced defects decreases twice as the W-ion irradiation temperature increases from 300 to 573 K.

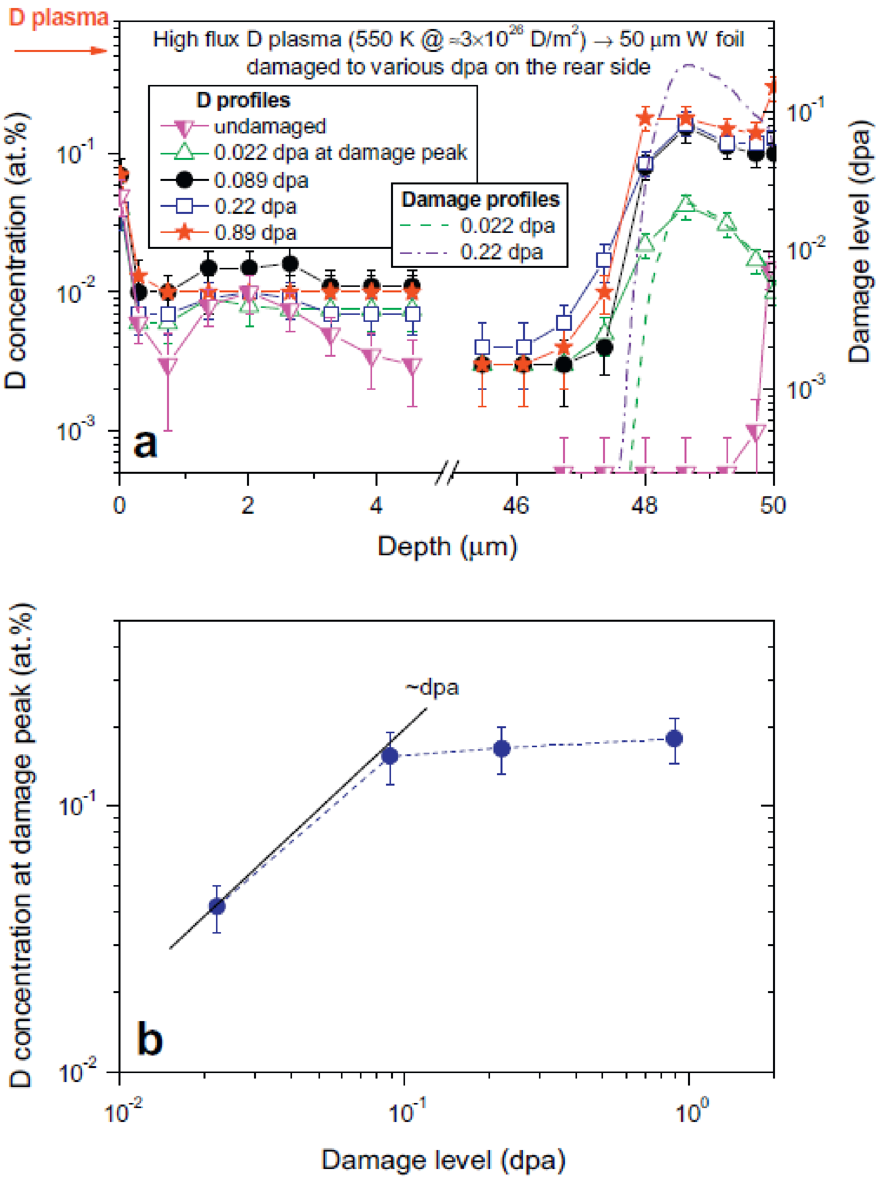

Figure 1. (a) Depth profiles of deuterium retained in 50 lm thick polycrystalline hot-rolled W foils, undamaged and damaged by irradiation at 300 K with 20 MeV W ions on the rear side to various damage levels, after exposure on the front side to high flux D plasma with a D ion fluence of about 3 × 10^{26} D/m^2 at 550 K. Damage profiles are plotted with the use of right ordinate axis. (b) The D concentration at a depth of the damage peak, as a function of the damage level. The solid line indicates linear proportionality between the damage level and the D concentration.

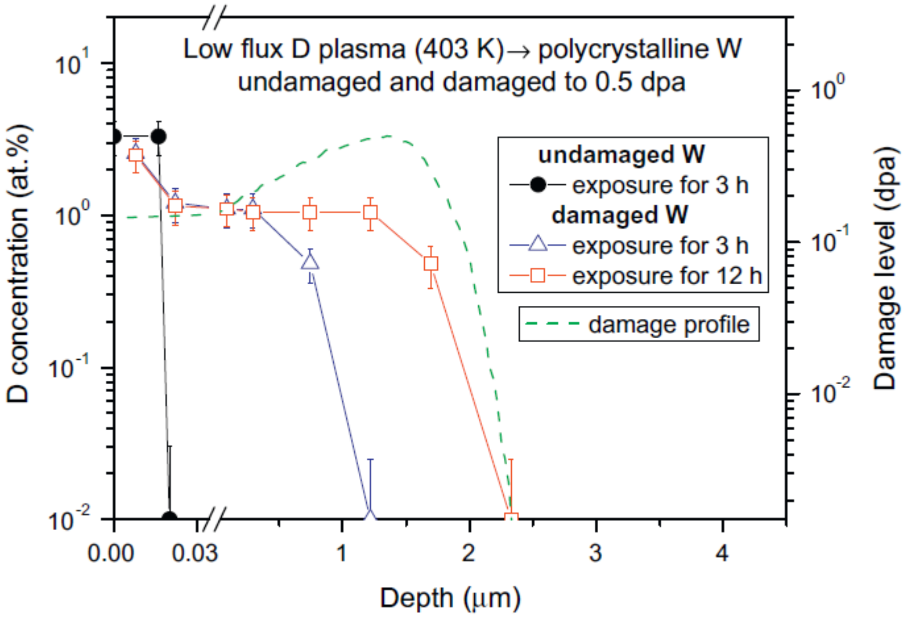

Figure 2. Depth profiles of deuterium retained in mechanically-deformed polycrystalline W samples, undamaged and damaged at 300 K by irradiation with 20 MeV W ions to 0.5 dpa, after exposure at 403 K to low flux D plasma for 3 and 12 h. The flux of implanted deuterium is estimated to be about 2 × 10^{18} D/m^2 s. Damage profile is plotted with the use of right ordinate axis.

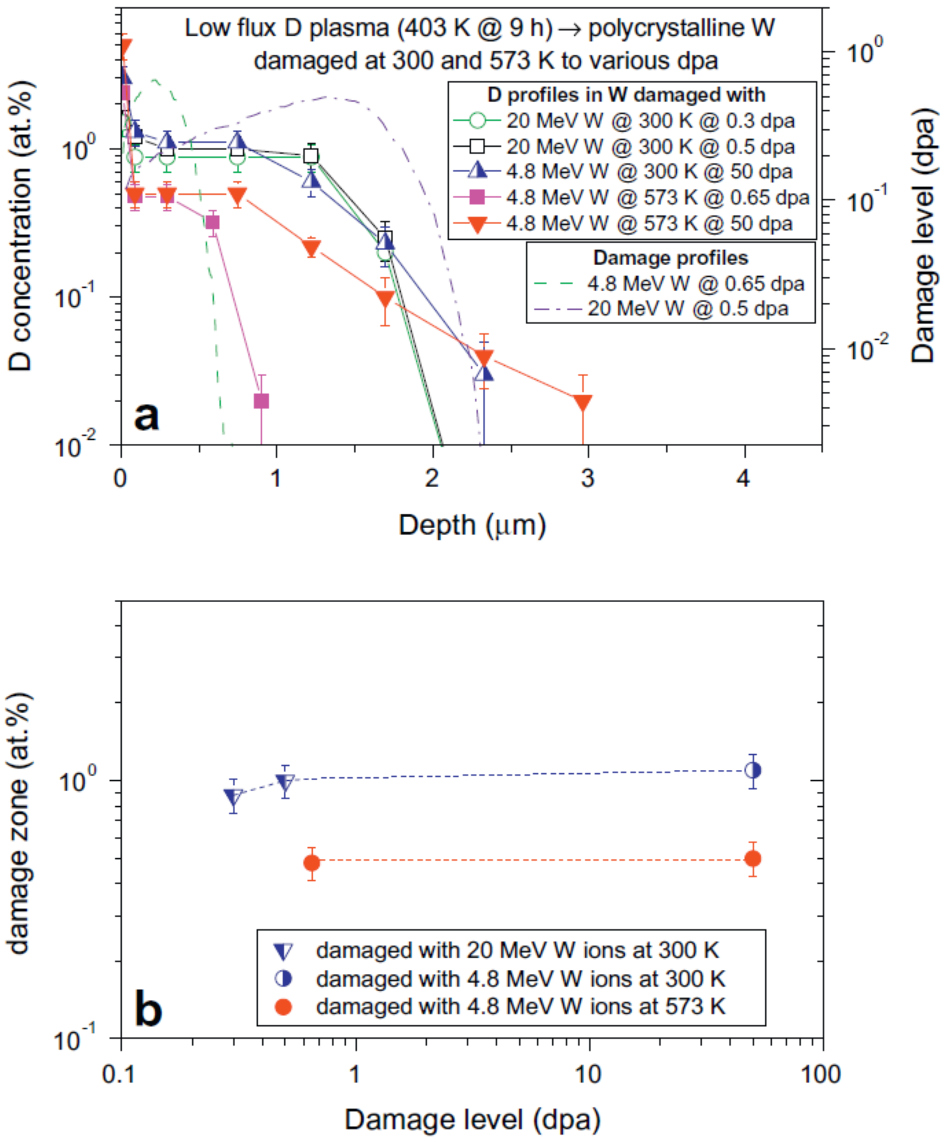

Figure 3. (a) Depth profiles of deuterium retained in mechanically-deformed polycrystalline W samples, damaged at temperatures of 300 and 573 K by irradiation with 4.8 and 20 MeV W ions to various damage levels, after exposure at 403 K to low flux D plasma for 9 h. The flux of implanted deuterium is estimated to be about 2×10^{18} D/m^2 s. Damage profiles are plotted with the use of right ordinate axis. (b) The D concentration in the damage zone, as a function of the damage level. Data shown in panel (b) were published earlier in Ref. [21].

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