Suppression of vacancy formation and hydrogen isotope retention in irradiated tungsten by addition of chromium

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Abstract

To study the effect of the content of chromium (Cr) in the tungsten (W) matrix on the vacancy formation and retention of hydrogen isotope, the samples of the W-0.3 at.% Cr alloy were irradiated with 6.4 MeV Fe ions at a temperature range of 523–1273 K to a damage level of 0.26 displacement per atom (dpa). These displacement-damaged samples were exposed to D_2 gas at a temperature of 673 K and a pressure of 100 kPa to decorate ion-induced defects with D. The addition of 0.3 at.% Cr to the W matrix resulted in a significant decrease in the retention of D compared to pure W after irradiation especially at high temperature (≥773 K). Positron lifetime for W-0.3 at.% Cr alloy irradiated at 1073 K was almost similar to that for non-irradiated one. This indicates the suppression of the formation of vacancy-type defects (monovacancies and vacancy clusters) by 0.3 at.% Cr addition, which leads to the significant reduction on D retention in W-0.3 at.% Cr alloy.

Keywords: tungsten; chromium; deuterium retention; ion irradiation

1. Introduction

Tungsten (W) and its alloys are the most promising candidate materials for plasma-facing components, due to its high melting temperature, high sputtering threshold energy and low hydrogen solubility [1, 2]. In a future fusion device, W will be exposed to high fluxes of 14 MeV neutrons as well as high fluxes of energetic deuterium (D) and tritium (T) existing in the form of ions, atoms and molecules. The collision cascades caused by neutrons will produce various types of defects in the W lattice, which can trap hydrogen isotopes. The hydrogen isotope retention in neutron-irradiated W is orders of magnitude greater than in the undamaged one [3, 4, 5, 6]. A significant increase in the hydrogen isotope retention was also observed after irradiation with high energy ions carried out as a surrogate for neutron irradiation to generate displacement damage [7, 8, 9, 10]. Therefore, understanding the hydrogen isotopes retention in irradiated W is a key issue for safe operation of future fusion devices. Positron annihilation spectroscopy study performed by Toyama et al. showed the trapping of D at vacancy-type defects (vacancies and their clusters) in neutron-irradiated W [11].

Hasegawa et al. examined the microstructure development in W and W- Re alloys under neutron irradiation using transmission electron microscopy and found that Re in W suppresses void formation under neutron irradiation [12]. Hatano et al. [13] studied the D retention and the positron lifetime in W and W-5 at.% Re alloy irradiated with 6.4 MeV Fe ions at elevated temperatures. They observed a significant suppression effects of Re on the formation of vacancy-type defects and the D retention at temperatures ≥773 K. On the other hand, Wang et al. compared the D retention in the W-2.5 at.% Mo and W-5 at.% Ta alloys with the retention in pure W after irradiation with 6.4 MeV Fe ions at 1073 K and found no significant alloying effects [14]. The observations of Wang et al. [14] are consistent with the results reported by Suzudo et al. [15].

According to the results of first principles calculations by Suzudo et al. [16], the Re atom, as well as the Os atom, reduces the effective mobility of the W self-interstitial atom (SIA) by forming a dumbbell cluster with the SIA and enhances recombination with a vacancy. They also evaluated the interactions of other solute atoms with a W-SIA [15]. Among the additive elements examined, Cr had the largest binding energy with a W-SIA. The binding energy of the Mo atom with the SIA was clearly lower than that of Re, while Zr, Nb, Hf, and Ta do not form stable mixed-dumbbells. However, the influence of Cr addition and high temperature irradiation have not been examined by experiment.

In this study, the effect of Cr addition on D retention were studied by using 6.4 MeV Fe ion irradiation to W and W-0.3 at.% Cr alloy at a temperature range of 523–1273 K followed by exposure to D_2 gas at 673 K for 10 h for further confirmation of the consistency with the model based on a solute atom-W-SIA interactions. The effect of post-irradiation annealing was also examined by using the sample irradiated at 523 K and then annealed in vacuum at 1073 K.

2. Experimental procedure

2.1 Materials

W and W-0.3 at.% Cr alloy samples with 10 × 10 × 0.5 mm^3 in size were prepared from the plates manufactured by A. L. M. T. Co., Japan by powder metallurgy (PM) followed by warm rolling. The fraction of Cr was limited to be 0.3 at.%. For larger Cr fraction, plates of W-Cr alloys broke during rolling processes before getting sufficiently high mass density, i.e. sufficiently low porosity, and were hence not considered useful for this work. Hereafter, the alloy samples are denoted as W-*x*M where M is the additive element and *x* is the fraction of it in atomic percent. All these samples were mechanically polished to mirror-like finish, cleaned in alcohol ultrasonic bath, and then outgassed at 1273 K for 1 h in a vacuum with a background pressure of ~10^{-5} Pa. This temperature was sufficiently high to relieve strain potentially induced during fabrication as well as decrease the content of hydrogen present in the samples as an impurity.

2.2 Ion irradiations

The conditions of ion irradiation are summarized in Table 1. The irradiation of W and W-0.3Cr alloy samples was performed with 6.4 MeV Fe ions to a fluence of 3.2 × 10^{18} Fe/m^2 at 523, 773, 1073 and 1273 K using the Dual-Beam Facility for Energy Science and Technology (DuET) at Kyoto University; those samples were irradiated under the same conditions as W and W-5Re alloy samples reported in [13, 14]. During irradiation, the background pressure in the vacuum chamber of the facility was about 10^{-5} Pa. According to the recommendation from [17, 18], the calculation by the program SRIM 2008.03 [19] with the "Quick Kinchin Pease" option was applied by setting the displacement threshold energy to be 90 eV [20]. For this condition SRIM results in a maximum damage level of 0.26 displacement per atom (dpa) at the damage peak which is situated at a depth of 1.2 μm. It should be noted that the SRIM results were calculated for W in this study and the value of 0.26 dpa obtained by "Quick Kinchin Pease" calculation option corresponds to 0.5 dpa calculated by "Full cascade option" in [13, 14]. Some of the W-0.3Cr alloy samples irradiated at 523 K were annealed in a vacuum at 1073 K for 1 h to examine the effects of post-irradiation annealing.

2.3 D loading

In order to decorate the created defects with D under equilibrium conditions without introducing additional displacement damage, D_2 gas exposure technique was applied for D loading. The W-0.3Cr alloy samples were exposed to D_2 gas at 100 kPa and 673 K for 10 h. The D loading conditions for all samples used in this study are summarized in Table. 1. For D_2 gas exposure, the samples were placed into a quartz tube connected to a high-vacuum pumping system and heated with the use of an external ohmic heater. The temperature was monitored by using a type-K thermocouple outside the tube. At the end of exposure, the D_2 gas was evacuated to a pressure below 1 Pa within several seconds and then the furnace was immediately removed to rapidly cool the samples.

2.4 Thermal desorption spectroscopy (TDS)

TDS measurements were performed to evaluate the D retention at the Hydrogen Isotope Research Center, University of Toyama. The sample was placed on a quartz glass stage installed in a quartz glass tube which was connected to a turbomolecular pump to evacuate to a high vacuum of 10^{-6} Pa. The samples were heated up to 1273 K with an oven at a heating rate of 0.5K/s. A type-K thermocouple inserted into the sample stage was used to monitor the sample temperature. Signals of mass-to-charge ratio channels 2, 3 and 4 of a quadrupole mass spectrometer (QMS) were measured and attributed to desorption of H_2, HD and D_2 gases. For the quantitative analysis, the QMS signals of channels 2 and 4 were calibrated by using standard leaks for H_2 and D_2 with an inaccuracy smaller than 10%, and the calibration constant for HD was assumed to be the average of that for H_2 and D_2. Signals of other deuterium-containing molecular compounds, such as D_2O and HDO, were also monitored. However, the partial pressures of those gas species showed no significant increase from the background level during TDS measurements.

2.5 Positron lifetime measurements

The positron lifetime of W and W-0.3Cr alloy samples irradiated with 6.4 MeV Fe ions at 1073 K were measured using a fast digital oscilloscope and BaF_2 scintillators with a time resolution of ~180 ps at full-width at half-maximum. Positron source was $^{22}NaCl$ of ~2 MBq, sealed with Kapton films. For comparison, the positron lifetime of non-irradiated W and W-0.3Cr alloy samples were also measured. The total number of events collected in each lifetime spectrum was 5×10^6. The PALSfit software was used to analyze the lifetime spectra [21].

3. Results

3.1 D retention in W-0.3Cr alloy

Figure 1 shows TDS spectra of D release from W and W-0.3Cr alloy samples irradiated with Fe ions at a temperature range of 523–1273 K to a damage level of 0.26 dpa and then exposed to D_2 gas with a pressure of 100 kPa at 673 K. For comparison, the TDS spectra of non-irradiated W and W-0.3Cr alloy samples are also plotted. The main desorption peaks appeared at 700–1000 K for both W and W-0.3Cr alloy samples, as observed for W and other binary W alloys in [13, 14]. The desorption peaks were largest for the samples irradiated at 523 K and became smaller as the irradiation temperature increased. The peaks for W-0.3Cr alloy samples were smaller than those for W samples at the same irradiation temperature, and the extent of difference between two materials increased as irradiation temperature increased. In the temperature region above 1000 K, the desorption rate of D from W sample irradiated at 1273 K was larger than that from W samples irradiated at lower temperatures. However, W-0.3Cr alloy sample irradiated at 1273 K showed smaller desorption rate than other samples in this temperature region.

Figure 2 shows correlation between irradiation temperatures and D retention in the damaged zones of W and W-0.3Cr alloy samples evaluated from TDS spectra. According to the calculation with the SRIM program, the thickness of damaged zone was less than 2 μm, while the thickness of the sample was 500 μm. Both D in damaged zones and non-irradiated volumes contributed to the D retention in the irradiated samples. Hence, the D retention in the damaged zones was calculated as the difference in D retention between irradiated and non-irradiated samples. For comparison, the D retention in the damaged zones of W and W-5Re alloy samples measured by means of nuclear reaction analysis (NRA) in [13] are also plotted. Both NRA and TDS showed a monotonical reduction in D retention in the damaged zones with increase in irradiation temperature. The D retention in the damaged zone of W-0.3Cr alloy was smaller than that in W at all examined temperatures, and the difference between the former and the latter increased with increase in irradiation temperature; at 1273 K, the D retention in W-0.3Cr alloy was smaller than that in W by an order of magnitude. This figure also shows the value of D retention in the W-0.3Cr alloy sample irradiated at 523 K and then subjected to the post-irradiation annealing at 1073 K. The value of D retention in W-0.3Cr alloy after post-irradiation annealing at 1073 K was far larger than that after irradiation at 1073 K and comparable with the W irradiated at this temperature. These results indicate that dynamic annealing effects under irradiation play an important role in reduction in D retention in W-0.3Cr alloy after the irradiation.

The D retention in W and W-0.3Cr alloy samples with and without irradiation at 1073 K is shown in Figure 3 together with that in W-2.5Mo and W-5Ta alloy samples examined in the same manner in [14]. The D retention in the non-irradiated and irradiated W-5Re alloy samples examined by means of TDS in the same manner is also plotted. The D retention in W, W-2.5Mo and W-5Ta alloys markedly increased by Fe ion irradiation due to trapping effects by radiation-induced defects, as reported in [13] and [14]. A far smaller extent of increase in D retention by the irradiation was observed for W-0.3Cr and W-5Re alloys, and the values of D retention in irradiated samples were comparable with those in the non-irradiated sample. The addition of 0.3 at.% Cr effectively suppressed the formation of trapping sites under Fe ion irradiation.

3.2 Positron lifetime in W-0.3Cr alloy

The results of positron lifetime measurements are summarized in Table 2. For comparison, the positron lifetimes of W and W-5Re alloy samples irradiated at 1273 K [13] are also listed. The value of positron lifetime in non-irradiated W was 134.8 ± 0.5 ps, which was comparable with the value of 133.9 ± 0.5 ps reported in [13]. Obviously, the irradiation of W with 6.4 MeV Fe ions to 0.26 dpa at 1073 K resulted in significant increase in positron lifetime (163.7 ± 0.5 ps). In contrast, no significant increase in positron lifetime was observed for W-0.3Cr alloy after the Fe irradiation under the same conditions; the positron lifetime in the W-0.3Cr alloy sample irradiated at 1073 K was evaluated to be 140.8 ± 0.5 ps, while that in the non-irradiated sample was 141.0 ± 0.5 ps. The negligible increase in positron lifetime suggests that Cr addition significantly suppressed the formation of vacancy-type defects (monovacancies and vacancy clusters) during high temperature irradiation. This suppression of defect formation was the cause of clear mitigation of irradiation effects on D retention described in 3.1. The addition of 5 at.% Re also resulted in the suppression of formation of vacancy-type defects as shown in Table 2 and [13]. In conclusion, Cr provided a strong suppression effects on the formation of vacancy-type defects as Re did but at far lower fraction (0.3 at.% for Cr vs. 5 at.% for Re).

4. Discussion

4.1 The effect of Cr as an alloying element

As mentioned earlier, the increase in D retention by Fe ion irradiation was significantly suppressed in the W-0.3Cr alloy samples in comparison with pure W. The desorption peaks of D from W-0.3Cr alloy samples appeared at the same temperature region as W samples (Figure 1). This means that the binding energy between radiation-induced defects and D atoms was comparable in these two materials. Hence, the significant reduction in D retention in W-0.3Cr alloy was not due to weaker trapping in W-0.3Cr alloy but it originated with lower defect density in W-0.3Cr alloy samples than W samples. The results of position lifetime measurements given in Table 2 also showed that Cr enhanced annihilation of vacancy-type defects.

Suzudo et al. [15] evaluated the binding energy of various solute elements at the substitutional site to a SIA of W by first principles calculations. They found that among all the solute elements examined a Cr substitutional atom has the largest binding energy (3.02 eV) with a W-SIA; this interaction produces a W-Cr mixed dumbbell that has a low-symmetrical direction of <11h> (h~0.3) and has the low energy barrier of migration (0.12 eV) and rotation (0.39 or 0.12 eV, depending on the rotating direction) [15].

Accordingly, this mixed dumbbell easily migrates with three-dimensional (3D) motion at high temperature such as 1073 K, instead of extremely fast one-dimensional (1D) motion, as they observed for SIAs. Because of the large binding energy of the mixed dumbbell, it rarely splits back into a SIA and a Cr substitutional atom. Kinetic Monte-Carlo simulations conducted by Suzudo et al. [16] indicated that mixed dumbbells in W crystals with the 3D motion have substantially larger probability of recombination with a nearby vacancy than that for SIAs. Thus, the density of surviving vacancies and their clusters, being the most effective trapping sites of D, should be reduced. This should be the primary cause of the reduced D retention in the W-Cr samples after Fe ion irradiation. In addition, each migrating W-Cr mixed dumbbell interacts also to a substitutional Cr atom causing a Cr-Cr dumbbell, which is immobile unless it splits back into a W-Cr mixed dumbbell and a Cr substitutional. This splitting reaction has a large activation energy (3.06 eV) [15]; thus, this interstitial defect is significantly stable and becomes an effective annihilating site for migrating vacancies. Therefore, the formation of Cr-Cr dumbbells is likely to be the secondary cause of the reduced D retention. In both scenarios, the stability of mixed dumbbells is a key parameter.

Wang et al. [14], conducted similar experiments for different binary alloys, i.e., W-Ta, W-Mo and W-Re alloys. The binding energies of Ta, Mo, and Re substitutional atoms to a SIA are -0.72 eV, 0.35 eV, and 0.74 eV, respectively [15]. Based on the aforementioned mechanism, no suppression of D retention is expected for W-Ta alloys, and weak and substantial suppression effects are expected for W-Mo and W-Re alloys, respectively. These predictions roughly agree with the TDS results, as shown in Figure 3. The suppression effect by 0.3% Cr addition was almost equivalent to that by 5% Re addition, suggesting that the effect by Cr addition is larger as expected from the larger binding energy to a SIA. Note that attractive interactions of a Re substitutional atom both to a vacancy and a SIA causes radiation-induced precipitation, which is the origin of irradiation hardening. In this respect, Re addition also has a demerit as an alloying element for W under irradiation. This is also the case for W-Os alloys. To clarify the effect of Cr fraction on radiation-induced defects in W-Cr alloy materials, further investigation is necessary.

4.2 Effect of irradiation temperature

The D retention in W-0.3Cr alloy after irradiation at 1073 K was substantially smaller than that after irradiation at 523 K and subsequent post-irradiation annealing at 1073 K. This observation suggests that the enhanced annihilation of vacancy-type defects by Cr addition originated with the dynamic annealing effect during high temperature irradiation. Similar effects were also observed in W-5Re alloy as reported in [13]. It appears that the rate of interaction between vacancy-type defect and W-Cr mixed dumbbell under irradiation is higher than that under the absence of irradiation by orders-of-magnitude.

The TDS spectrum of D released from W irradiated at 1273 K (Figure 1) showed a shoulder in the higher temperature region, which indicates that a part of trapping sites in W formed at 1273 K have a stronger bond with the D atom(s) than the defects in W irradiated at lower temperatures. The positron lifetime in the W sample irradiated at 1273 K was slightly longer than that in the sample irradiated at 1073 K. It should be noted that the lifetime spectrum of W irradiated at 1273 K was fitted well using two components, 125 ± 1 ps and 470 ± 20 ps, and the average lifetime was evaluated to be 168.0 ± 0.5 ps as given in Table. 2. Troev et al. [22] have reported that the positron lifetime is 108 ps in W matrix, 200 ps in a monovacancy, and 437 ps in 37V cluster (V is a monovacancy). The value for the short-life component (125 ± 1 ps) was close to the positron lifetime in the non-irradiated samples, while that for long-life component (470 ± 20 ps) corresponded to relatively large vacancy clusters. Therefore, it is plausible that a part of trapping sites having a stronger bond with the D atom(s) was the relatively large vacancy clusters formed by the dynamic annealing at 1273 K during irradiation. In the previous study [14], similar and relatively large desorption shoulders in the high temperature regions were also observed for the W-*x*Re (*x*=1, 3 and 5 at.%) alloys after irradiation at 1073 K. The mechanisms underlying these high temperature shoulders are currently unclear because the irradiation of W-Re alloy at high temperature led to no significant change in positron lifetime (Table 2). Nevertheless, such shoulder was not observed for W-0.3Cr alloy even after the irradiation, suggesting that the addition of Cr does not induce formation of a defect with strong bond with hydrogen isotope atom(s).

5. Conclusions

D retention in W-0.3Cr alloy was investigated after irradiation with 6.4 MeV Fe ions to 0.26 dpa at the temperature range of 523–1273 K. The samples irradiated at 523 K and then annealed at 1073 K were also prepared to compare the effects of dynamic annealing during irradiation and post-irradiation annealing. The main findings are:

(1) The D retention in the W-0.3Cr alloy was substantially smaller than that in W after irradiation at high temperatures (≥773 K);

(2) The effect of post-irradiation annealing in vacuum at 1073 K after Fe ion irradiation at 523 K was far smaller than the above-mentioned effects of high temperature irradiations of Fe ions.

(3) No noticeable increase in positron lifetime was observed for W-0.3Cr alloy after the high temperature irradiation, while the lifetime in W showed a clear increase after the irradiation;

(4) The addition of 0.3 at.% Cr was found to mitigate the formation of vacancy type defects under high temperature irradiation and the increase in D retention by trapping effects; and

(5) The mitigation effect by 0.3 at.% Cr addition was comparable with that by 5 at.% Re addition, while no mitigation effects were observed by addition of Ta and Mo;

(6) These observations were consistent with the predictions by Suzudo et al. [15] based on first principles calculations of binding energies between a solute atom

and a W-SIA.

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Table 1. Summary of ion irradiation conditions and D loading conditions for W and W-0.3Cr alloy samples. The damage level (dpa) at the damage peak is indicated. Note that the damage level evaluated to be 0.26 dpa using the program SRIM 2008.03 with the "Quick Kinchin Pease" option was equivalent to the value of 0.5 dpa calculated in [13, 14] with the "Full cascade" option.

samples	Fe ion energy (MeV)	irradiation temperature (K)	irradiation fluence (ions/m^2)	damage level (dpa)	D_2 gas exposure
W, W-0.3Cr	6.4	523, 773, 1073, 1273	3.2×10^{18}	0.26	100 kPa, 673 K, 10h

Table 2. Results of positron lifetime measurements for W and W-0.3Cr alloy samples before and after the irradiation with 6.4 MeV Fe ions at 1073 K to 0.26 dpa. For comparison, the positron lifetimes of W and W-5Re alloy irradiated at 1273 K [13] were also indicated. Note that the damage level evaluated to be 0.26 dpa using the program SRIM 2008.03 with the "Quick Kinchin Pease" option was equivalent to the value of 0.5 dpa calculated in [13] with the "Full cascade" option.

Samples	W			W-0.3Cr alloy		W-5Re alloy	
	non-irradiated	irradiated at 1073 K	irradiated at 1273 K [13]	non-irradiated	irradiated at 1073 K	non-irradiated [13]	Irradiated at 1273 K [13]
Positron lifetime [ps]	134.8 ± 0.5	163.7± 0.5	125 ± 1 470 ± 20 (ave.168± 0.5)	141.0 ± 0.5	140.8 ± 0.5	137.7 ± 0.5	138.9 ± 0.5

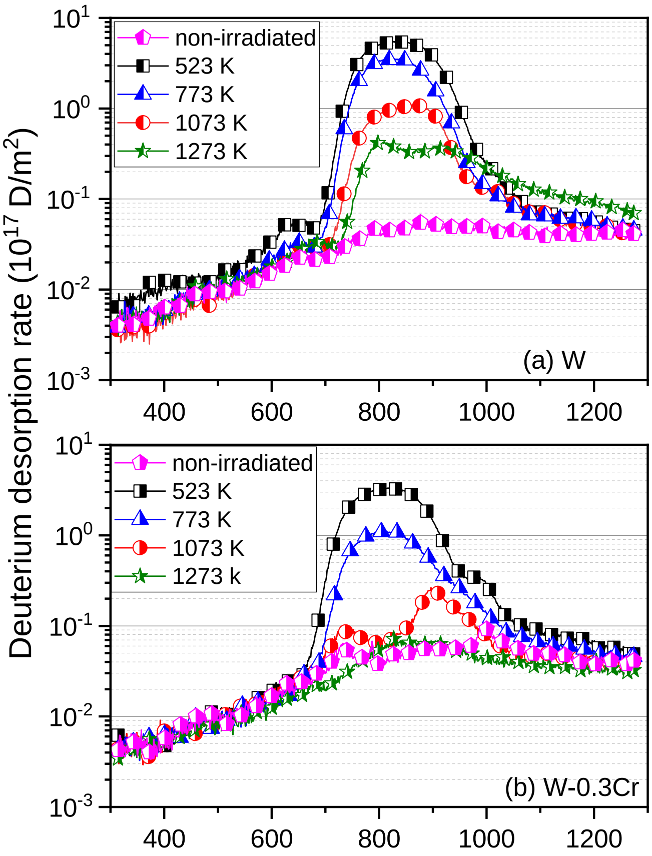

Figure 1 TDS spectra of D from W and W-0.3Cr alloy samples irradiated with 6.4 MeV Fe ions at temperature range of 523–1273 K up to a damage level of 0.26 dpa. For comparison, TDS spectra for non-irradiated W and W-0.3Cr alloy samples are also plotted. The samples were loaded with D by exposure to D_2 gas at a pressure of 100 kPa and a temperature of 673 K for 10 h.

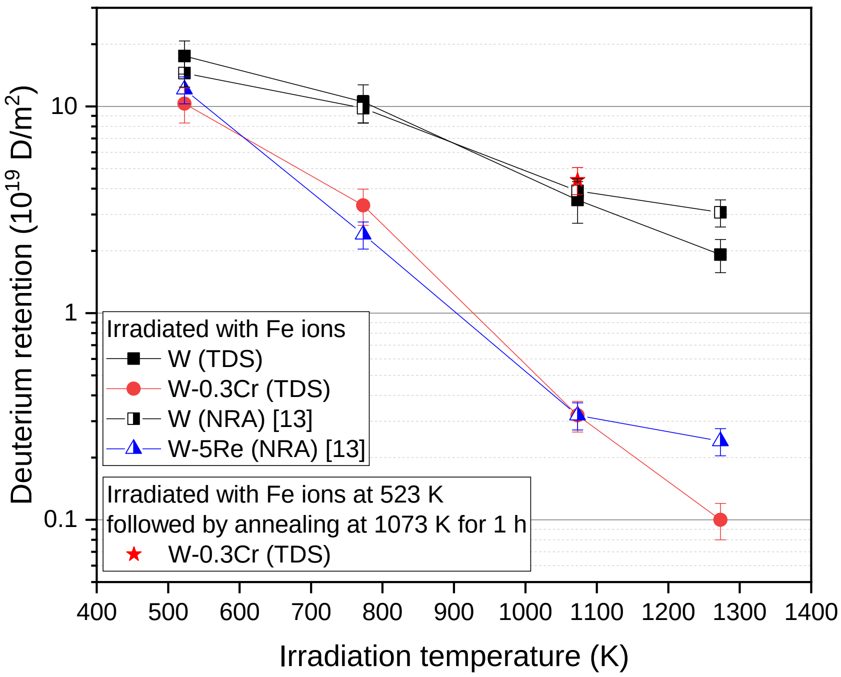

Figure 2 The correlation between irradiation temperature and D retention in the damaged zone evaluated from TDS spectra for W and W-0.3Cr alloy samples irradiated at temperature range of 523–1273 K. For comparison, the D retention in the damaged zone of W and W-5Re alloy samples measured by means of NRA up to 6 μm [13] is also plotted. Additionally, the D retention evaluated from TDS spectra for W-0.3Cr alloy sample first irradiated at 523 K with 6.4 MeV Fe ions to 0.26 dpa and then annealed in vacuum at 1073 K for 1 h is also shown. Note that the damage level evaluated to be 0.26 dpa using the program SRIM 2008.03 with the "Quick Kinchin Pease" option was equivalent to the value of 0.5 dpa calculated in [13] with the "Full cascade" option.

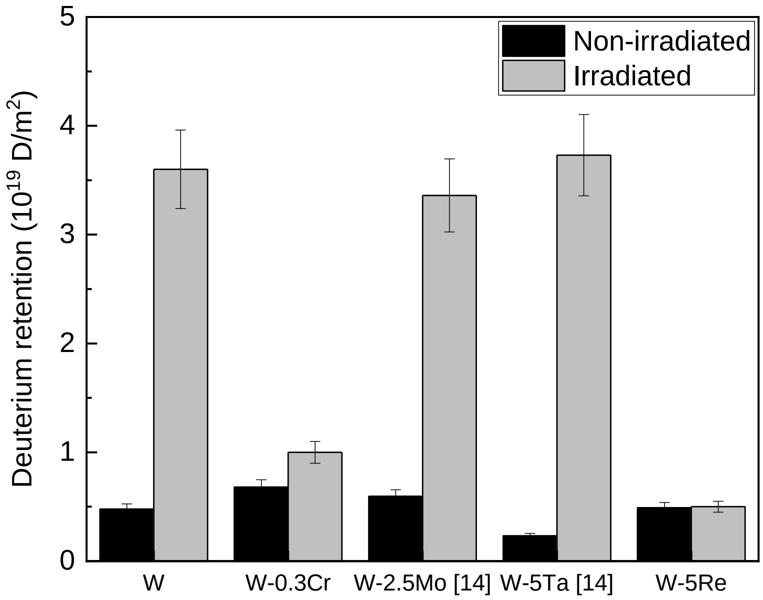

Figure 3 D retention evaluated from TDS spectra for non-irradiated and irradiated W, W-0.3Cr and W-5Re alloy samples. These samples were irradiated at 1073 K with Fe ions to 0.26 dpa. For comparison, D retention in W-2.5Mo and W-5Ta alloy samples evaluated in the same manner in [14] is also plotted. Note that the damage level evaluated to be 0.26 dpa using the program SRIM 2008.03 with the "Quick Kinchin Pease" option was equivalent to the value of 0.5 dpa calculated in [14] with the "Full cascade" option.