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Deuterium retention in W and binary W alloys irradiated with high energy Fe ions

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Abstract

To investigate systematically the effects of Re and other elements on deuterium (D) retention, W and binary W-alloys (Re, Mo and Ta) were irradiated with 6.4 MeV Fe ions at high temperatures (1073-1273 K) and then exposed to D_2 gas at 673 K. Depth profiles of D were measured by nuclear reaction analysis (NRA), and D retention was determined by thermal desorption spectrometry (TDS) and NRA. The addition of 5 at.% Re into W reduced the content of D trapped at radiation-induced defects created by irradiation with the Fe ions at 1273 K to the peak damage level of 5 displacements per atom (dpa). At Re

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fractions of 1, 3 and 5 at.%, comparable effects on D retention were observed after irradiation to 0.5 dpa at 1073 K. The addition of Mo and Ta to W resulted in no visible effects in D retention.

Keywords: tungsten; rhenium; molybdenum; tantalum; deuterium retention; ion irradiation

1. Introduction

Due to its attractive physical properties, such as high melting point, high temperature strength, high thermal conductivity and high sputtering threshold energy, tungsten (W) and its alloys have been recognized as a promising candidate for plasma-facing material (PFM) in a future fusion reactor [1, 2]. During operation of a fusion reactor, W will experience irradiation of high-energy neutrons (n), energetic deuterium (D) and tritium (T) particles. Irradiation with high energy neutrons results in the formation of displacement damage. Previous neutron-irradiation experiments have shown that radiation-induced defects in pure W act as strong trapping sites for D [3-6]. A significant increase in the hydrogen isotope retention has been observed also after irradiation with high energy ions performed as a surrogate for the displacement damage the neutron irradiation will cause [7-10]. These observations indicate that irradiation with high energy neutrons should result in a significant increase in the T inventory in W.

Tyburska-Püschel and Alimov [11], and Hatano et al. [12] have examined deuterium retention in W-rhenium (Re) binary alloys after heavy ion irradiation at MeV energy range and reported that the retention in irradiated W-Re alloys was orders-of-magnitude lower than that in irradiated W. Hatano et al. [12] also observed suppression of the formation of vacancy-type defects. The observation by Hatano et al. [12] is consistent with the data of Fukuda et al. [13], who reported that void formation after neutron irradiation at elevated temperatures was suppressed by Re addition. However, the effects of Re on hydrogen isotope retention has been examined solely at 5 at.% Re fraction and an irradiation dose

of 1 displacement per atom (dpa) or less. The investigation at different Re fractions and higher dose levels is required for better understanding of the effects of Re.

It is also important to examine the influence of other alloying elements on hydrogen isotope retention after irradiation. According to first principles calculations by Suzudo et al. [14, 15], Re atom reduces the effective mobility of W self-interstitial atom (SIA) by forming a dumbbell cluster with the SIA and enhances recombination with a vacancy. According to their evaluation, the interaction of W SIA with Mo and Ta atoms is much weaker than with Re atoms. However, the effect of these alloying elements (Mo and Ta) on D retention after irradiation have not been understood.

In this study, plates of W-(1, 3 and 5) at.% Re, W-2.5 at.% Mo and W-5 at.% Ta alloys were irradiated with 6.4 MeV Fe ions at elevated temperatures (1073 and 1273 K) together with those of pure W and then exposed to D_2 gas at 673 K to understand

(1) dose dependence of D retention in W-5 at.% Re alloy up to 5 dpa,

(2) D retention in W-Re alloy at different Re fractions (1, 3 and 5 at.%), and

(3) influence of Mo and Ta on D retention after irradiation.

The gas exposure technique was employed instead of conventional plasma exposure to introduce D into the samples under well-controlled conditions with minimal change in the irradiated microstructure.

2. Experimental procedure

2.1 Materials

The samples used to investigate the dose dependence of D retention were plates of pure W and W-5 at.% Re alloys (10×10×5 mm) prepared from sheets of those materials manufactured by A. L. M. T. Co., Japan by powder metallurgy technique followed by warm rolling. Other samples employed to study the dependence of D retention on Re fraction and the effects of Mo and Ta were plates of pure W, W-(1, 3 and 5) at.% Re, W-2.5 at.% Mo and W-5 at.% Ta alloys (10×10×5 mm). Those plates were cut from rods fabricated by zone melting with electron bombardment heating by NPO “Luch”, Russia.

The fraction of Mo was limited to be 2.5 at.% because W-Mo alloys with higher Mo fractions were very brittle and difficult to handle. Hereafter, the alloy samples are denoted as W-*x*M where M is the alloying element and *x* is its fraction in atomic percent. The compositions and manufacturing techniques of the samples are summarized in Table 1.

The surfaces of the samples were mechanically polished to a mirror finish. Then the samples were heated in vacuum (10^{-5} Pa) at 1273 K for 1 h to relieve strain potentially induced during fabrication and remove hydrogen present in the samples as an impurity.

2.2 Ion irradiation and D measurements

As the detailed experimental procedure for Fe ions irradiation was described in our previous paper [12], only a brief description is given here. All samples were irradiated with 6.4 MeV Fe ions by using the Dual-Beam Facility for Energy Science and Technology (DuET) at Kyoto University under the conditions shown in Table 1. The irradiation was performed at 1073 and 1273 K because the effects of Re addition observed for the W-5Re alloy became more significant as the irradiation temperature increased [12]. During irradiation, the background pressure in the vacuum chamber of the facility was about 10^{-5} Pa. The depth profile of the displacement-damage was calculated by using the program SRIM 2008.03, "full cascade option", with a displacement threshold energy of E_d = 90 eV. The depth of the damaged region was estimated to be ~2 μm. In what follows the damage level value will be indicated as a number of dpa at the damage peak.

All of these Fe-ion-irradiated samples were exposed to D_2 gas at 673 K and a pressure of 100 kPa for 10 h in a quartz glass tube attached to a vacuum device. The temperature of the samples was monitored using a type K thermocouple located near the samples outside the tube. At the termination of the exposure procedure, the D_2 gas was evacuated to a pressure below 1 Pa for several seconds and then the furnace was immediately removed to rapidly cool the samples. It should be noted that the exposure to D_2 gas was performed at elevated temperatures (673 K). Hence, weak traps were not occupied by D under the present conditions, and the desorption at room temperature was

not significant.

Deuterium depth profiles up to 6.2 μm were determined at Max-Planck-Institut für Plasmaphysik by counting α-particles and protons generated by the $D(^3He, p)^4He$ nuclear reaction. The α-spectrum measured with 690 keV ^3He beam at a reaction angle of 102° was transformed into a D depth profile up to ~0.4 μm using the program SIMNRA [16]. The D fraction at larger depths was determined by varying the energy of the primary ^3He ion beam between 0.69 and 4.0 MeV. The proton yields measured at different ^3He ion energies at a reaction angle of 135° made it possible to derive the D depth profiles [17]. The amount of D retained within the information depth of 6.2 μm was finally obtained by integrating the D profile over the measured depth.

Thermal desorption spectrometry (TDS) measurements were performed to evaluate the D retention at the Hydrogen Isotope Research Center, University of Toyama. The TDS measurements were performed within a week after the D_2 gas exposure, during which these exposed samples were kept in a vacuum container. The sample was placed on a quartz glass stage installed in a quartz glass tube evacuated to 10^{-8} Pa using a turbomolecular pump. The temperature of the sample was raised to 1203 or 1273 K with a ramp rate of 0.5 K/s using a heater surrounding the quartz tube. A type K thermocouple inserted into the sample stage was used to monitor the sample temperature. The release flux of the molecular HD and D_2 were measured by a quadrupole mass spectrometer (QMS). Standard H_2 and D_2 leaks with an inaccuracy smaller than 10% were employed to calibrate the QMS, 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 these gas species showed no significant increase over the background level during TDS measurements. For comparison, the TDS measurements were also performed for non-irradiated samples after the exposure to D_2 gas in the above-mentioned manner.

3. Results

3.1 D retention in W-5Re alloy at different damage levels

Fig. 1 shows depth profiles of D in the powder metallurgy W and W-5Re alloy samples irradiated at 1273 K with 6.4 MeV Fe ions to damage levels of 0.1 and 5 dpa and then exposed to D_2 gas at 673 K and 100 kPa. For comparison, the depth profiles of D in the samples irradiated to 0.5 dpa [12] are also shown in Fig. 1. As these profiles show, D is mainly distributed in the damaged zone, indicating that D was trapped at radiation-induced defects. Both in pure W and in the W-5Re alloy, the distributions of D in the damaged zone at different dpa values were similar to each other. In the near-surface layers from surface to a depth of 0.3 μm, the fraction of D decreased from 10^{-1} at.% to 10^{-2} at.% in pure W, while in the W-5Re alloy it decreased from 10^{-1} at.% to 10^{-3} at.%. At depths from 0.3 to ~1.4 μm, the D fractions were around 10^{-2} at.% in pure W and 10^{-3} at.% in the W-5Re alloy, respectively. At greater depths, the D fraction gradually decreased with increasing depth in both pure W and W-5Re alloy.

Fig. 2 shows the correlation between the damage level and D retention evaluated from Fig. 1 by integrating the depth profiles up to 6.2 μm. Obviously, the D retention in the W-5Re alloy was significantly less than that in pure W. This figure also shows that the effects of Re addition began to appear at a relatively low irradiation dose (0.1 dpa). The D retention in the W-5Re alloy after irradiation to damage levels of 0.1 and 0.5 dpa was comparable to that after irradiation to 5 dpa, indicating that the density of defects acting as trapping sites ceased to increase at relatively low dose (0.1 dpa).

3.2 D retention in W-xRe alloys

Fig. 3 shows the TDS spectra of D released from pure W, W-1Re, W-3Re and W-5Re alloy samples prepared by zone melting and irradiated with Fe ions to a damage level of 0.5 dpa at 1073 K. The desorption peak for pure W appeared at 700–950 K, as was observed in a previous study [12]. The peak consisted of two sub-peaks; the main one was located at 822 K and the additional one at 915 K. As for the W-*x*Re alloys, the D

desorption was observed in the temperature range from 700 to 1200 K. The desorption peaks for the W-*x*Re alloys were small and broad compared to the peaks for pure W; the TDS spectra for W-*x*Re alloys had a new sub-peak in the high temperature range: at around 1090 K. These observations indicated that the addition of Re affect the trapping energy of radiation-induced defects.

Fig. 4 shows the correlation between Re fraction and D retention determined by TDS for Fe-ion-irradiated samples (Fig. 3) and non-irradiated samples. D retention in the non-irradiated W-*x*Re alloy was slightly higher than that in pure W, and it decreased with an increase in the Re fraction. The underlying mechanisms for this difference between pure W and W-*x*Re alloy and the dependence on the Re fraction are currently not clear. In the case of pure W, the D retention increased by an order of magnitude after irradiation with Fe ions, though the thickness of the damaged zone (~2 μm) was far less than the sample thickness (500 μm). In contrast, the W-*x*Re alloy samples showed only a modest increase in the D retention after Fe ion irradiation. The extent of increase in the D retention due to Fe ion irradiation was comparable with each other at x = 1, 3 and 5 at.%. These results revealed that the effects of Re addition on radiation-induced defects were at a comparable level in the fraction range of 1–5 at.%.

3.3 D retention in W-2.5Mo and W-5Ta alloys

Fig. 5 shows the TDS spectra of D released from the W-2.5Mo alloy and the W-5Ta alloy irradiated with 6.4 MeV Fe ions to a damage level of 0.5 dpa at 1073 K. For comparison, the TDS spectrum for the zone melting pure W sample previously shown in Fig. 3 is also plotted in this figure. The desorption peaks were observed at 700–950 K for pure W, W-2.5Mo alloy and W-5Ta alloy. In addition, the peak areas are also comparable to each other. These observations indicated that Mo and Ta do not affect the density and trapping energy of radiation-induced defects.

Fig. 6 shows the total D retention evaluated by TDS for irradiated and non-irradiated samples of pure W (zone melting), W-5Ta alloy and W-2.5Mo alloy as a function of the

irradiation dose (0, 0.5 and 2.5 dpa). The D retention in non-irradiated W-2.5Mo alloy was comparable to that in non-irradiated pure W, while the D retention in non-irradiated W-5Ta was slightly smaller (about half of pure W). However, after Fe ion irradiation, the D retention in these materials was comparable to each other. In addition, the D retention after 2.5 dpa irradiation was comparable to the retention after 0.5 dpa irradiation for all materials. These results further illustrate that the addition of Mo and Ta has no visible effect on the D retention in W after ion irradiation.

4. Discussion

Figs. 1 and 2 showed that the D retention in the W-5Re alloy was less than that in pure W after Fe ion irradiation at 1273 K to various irradiation doses (0.1–5 dpa). The results of positron lifetime measurements performed in the previous study [12] showed that a significant reduction in the D retention by the Re addition after high temperature irradiation was due to a lower concentration of vacancy-type defects (monovacancies and vacancy clusters). Observation in this study indicate that the addition of 5 at.% Re to W can reduce the concentration of vacancy-type defects after high temperature irradiation even at a relatively low irradiation dose (at a damage level of 0.1 dpa), and the concentration of vacancy-type defects can be controlled to a comparable value even when the radiation dose increased to 5 dpa.

Cascade collision induced by Fe ion irradiation should result in the formation of various types of defects such as monovacancy, divacancy, vacancy clusters, dislocations, etc. These defects can accommodate a different number of D atoms with different binding energies. The TDS spectra of D released from W-Re alloys with addition of 1, 3 and 5 at.% Re (Fig. 3) showed the shoulders in the higher temperature range. The shoulders indicate that a part of trapping sites in the W-Re alloys has a stronger bond with the D atom(s) than the defects in pure W. Therefore, the reduced D retention shown in Figs. 3 and 4 can be ascribed to a reduced concentration of defects rather than to a weaker bond between trapping site and the D atom(s). This observation is consistent with the results of

positron lifetime measurements for the W-5Re alloy in the previous study [12]. The shoulder in the high temperature range was not evident in the TDS spectrum for the W-5Re alloy shown in Ref. [12]. This was due to the fact that the D retention was very low and the signal intensity of D_2 was close to the signal background in the temperature range above ~1000 K. The mechanism underlying the higher binding energy in W-Re alloys is currently not clear. The binding energy is determined by the difference in enthalpy between the D atom at the trapping site and the D atom at the interstitial site in the undisturbed W lattice. First-principles calculations by Ren et al. [18] showed that the heat of solution, the difference in enthalpy between the H atom in a H_2 molecule and that at an interstitial site in the undisturbed W lattice increases with the Re addition. Such a change in the heat of solution should result in an increase in the trapping energy. The observation in this study is consistent with the results of their calculations. Anyway, the fraction of Re between 1 at.% and 5 at.% can significantly reduce the hydrogen isotope retention.

The D retention in Fe-ion-irradiated samples prepared by zone melting in this work was higher than that in the powder metallurgy samples irradiated at the same manner in Ref. [12]. These two types of samples had significantly different microstructures. The zone melting samples had large grains up to several millimeters in size. On the other hand, the powder metallurgy sample had a rolled structure, and the minor axis length of the flattened grains was 1 μm or less. Thus, one of the possible explanations for the difference between the zone melting and powder metallurgy samples was the difference in the density of grain boundaries acting as sinks for point defects. However, a detailed microstructural analysis and more systematic studies of the correlation of irradiation effects with microstructure are necessary for a deeper discussion.

As mentioned in Section “1. Introduction”, Suzudo et al. [14, 15] reported that the Re atom forms a dumbbell cluster with a SIA of W. The formation of dumbbell cluster reduces the SIA’s effective mobility and enhances recombination with a vacancy. According to Suzudo et al. [15], Mo also forms a mixed dumbbell but the binding energy

was significantly lower compared to the Re atom. The Ta atom does not form a stable mixed dumbbell with the W SIA. As shown in Fig. 6, the addition of Mo and Ta resulted in no noticeable effects on the D retention after irradiation. There is no contradiction between the present observations and the model proposed by Suzudo et al. [15]. According to them, Ti, V and Cr atoms also form a stable dumbbell cluster with the W SIA. However, the addition of Ti and V can increase the hydrogen isotope retention since these elements from hydrides. Hence, the investigation of the W-Cr system could be the next step.

5. Conclusions

The D retention in pure W and three types of binary W-alloys were investigated after irradiation of 6.4 MeV Fe ions at high temperatures. Below are the main findings.

(1) The concentration of radiation-induced traps in the W-5 at.% Re alloy was lower than that in pure W after irradiation to 0.1–5 dpa. The effects of Re on the D retention were comparable at a Re fraction of 1–5 at.% after irradiation up to 0.5 dpa at 1073 K.

(2) The D retention in the irradiated W-5 at.% Ta and W-2.5 at.% Mo alloys was comparable to that in the irradiated pure W, and the TDS spectra were also similar to each other. In other words, the addition of Ta and Mo did not significantly affect the D trapping after the irradiation.

(3) The addition of Re is a promising method to reduce the hydrogen isotope retention in W under neutron irradiation.

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Table. 1 Summary of samples and conditions of Fe ion irradiation. The damage level (dpa) at the damage peak is indicated.

Sample	Manufacturing technique	Irradiation temperature (K)	Irradiation fluence (Fe ions/m^2)	dpa
W, W-5Re	Powder metallurgy	1273	6.4×10^{17} 3.2×10^{19}	0.1 5
W- *x*Re (*x* = 1, 3 and 5 at.%)	Zone melting	1073	3.2×10^{18}	0.5
W, W-5Ta, W-2.5Mo	Zone melting	1073	3.2×10^{18} 1.6×10^{19}	0.5 2.5

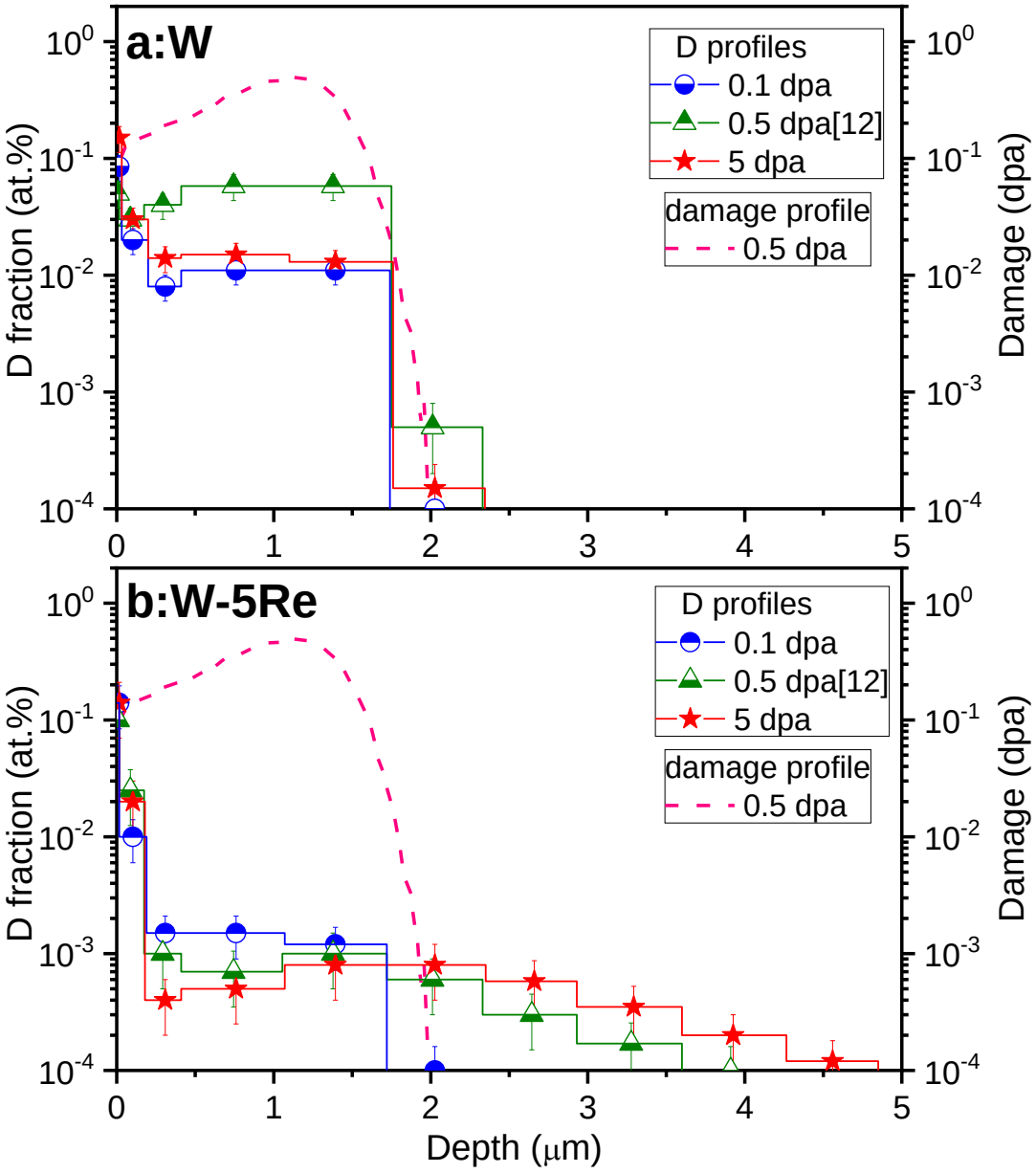

Fig. 1 Depth profiles of D retained in pure W (a) and W-5Re alloy (b) samples prepared by powder metallurgy. The samples were irradiated at 1273 K with 6.4 MeV Fe ions to various peak damage levels and then exposed to D_2 gas at a temperature of 673 K and pressure of 100 kPa for 10 h. In both panels, calculated damage depth profiles (0.5 dpa) are also shown.

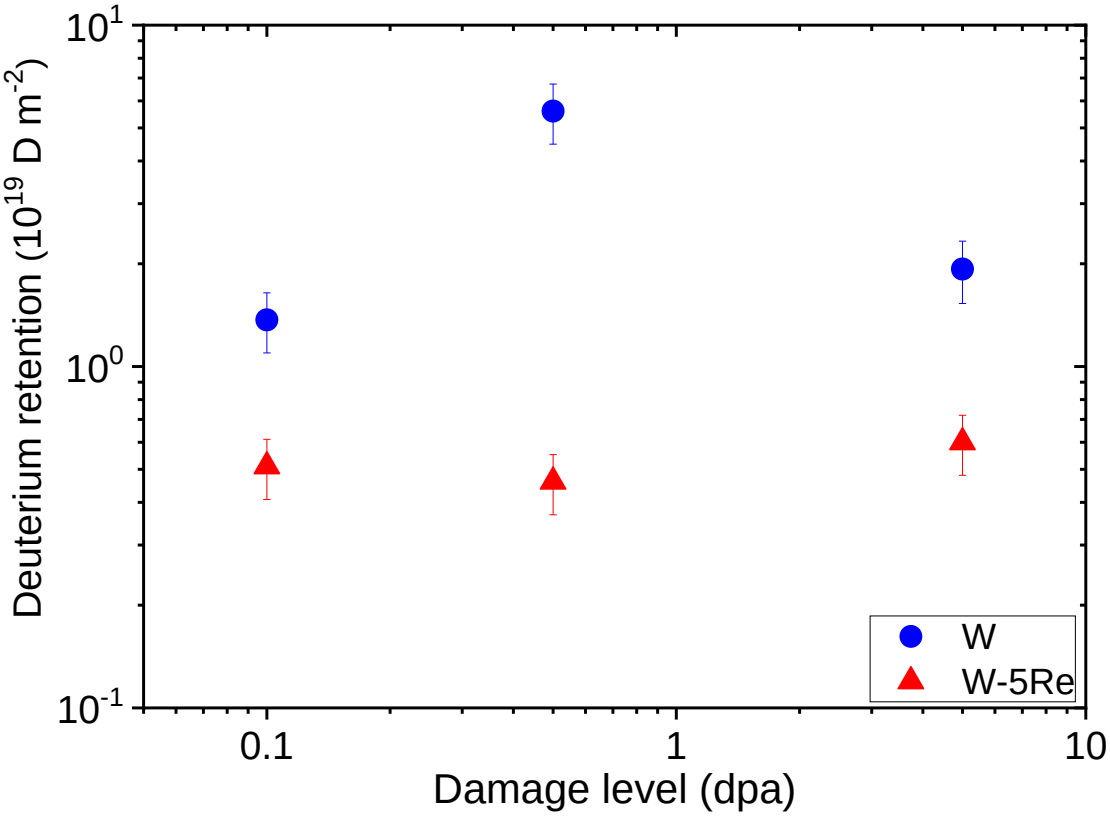

Fig. 2 D retention in the first 6.2 μm in pure W and W-5Re alloy samples prepared by powder metallurgy, as measured by NRA. The samples were irradiated at 1273 K with 6.4 MeV Fe ions to various peak damage levels and then exposed to D_2 gas at 673 K and 100 kPa for 10 h.

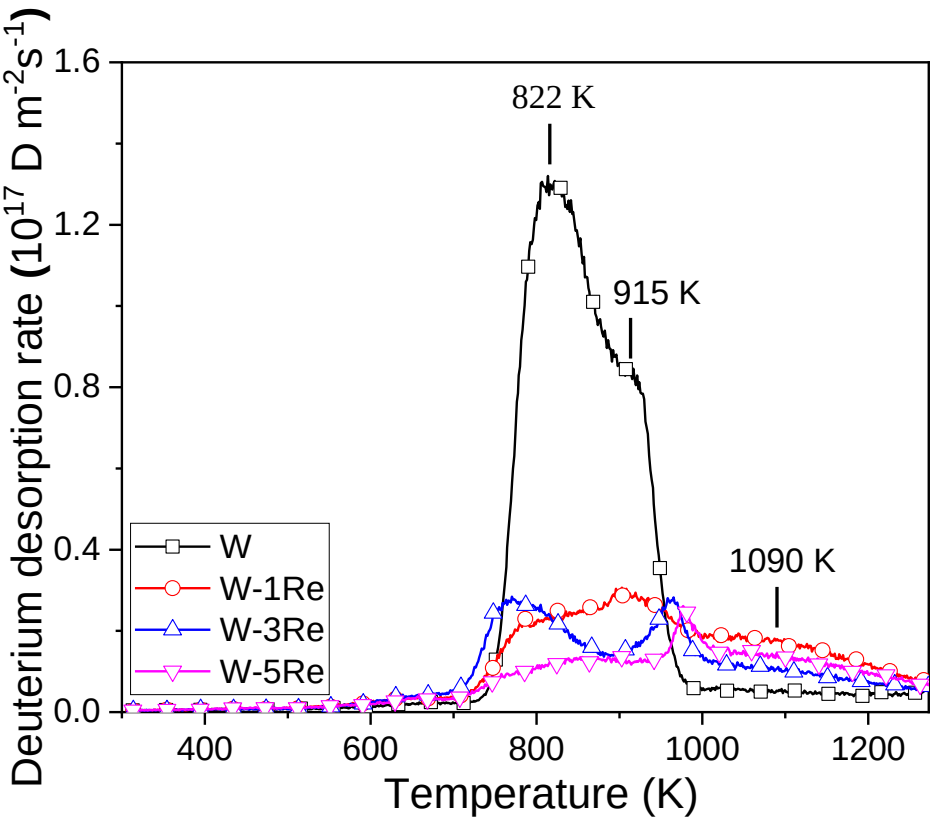

Fig. 3 TDS spectra of deuterium released from pure W and W-*x*Re (*x* = 1, 3 and 5 at.%) alloy samples prepared by zone melting. The samples were irradiated at 1073 K with Fe ions to a peak damage level of 0.5 dpa and then exposed to D_2 gas at 673 K and 100 kPa for 10 h. Heating rate was 0.5 K/s.

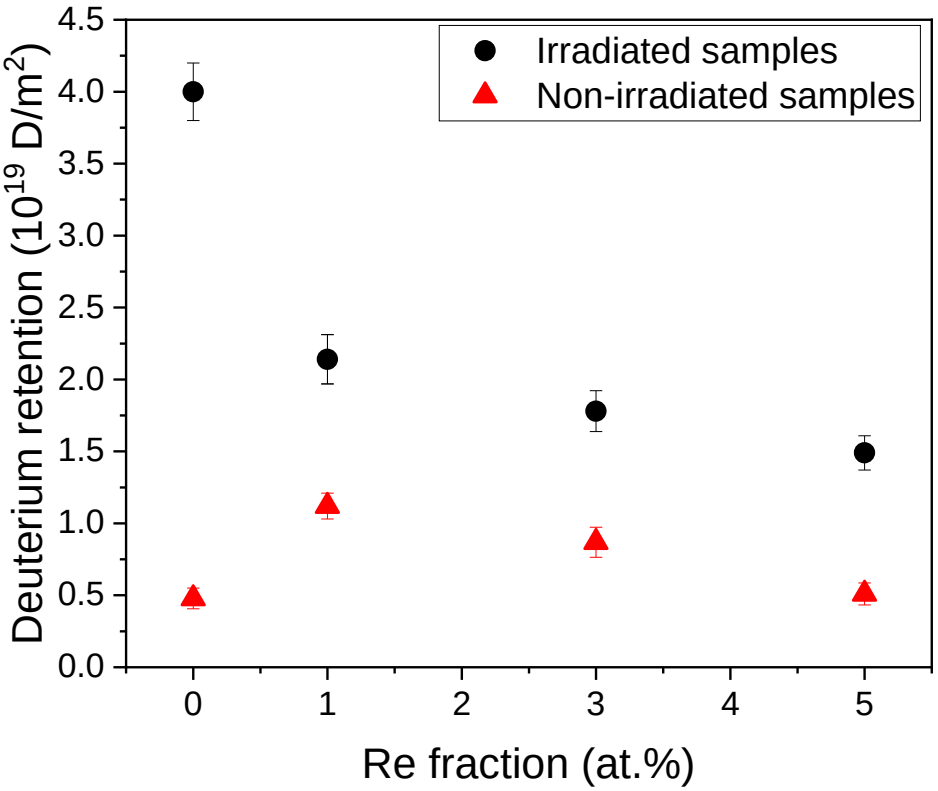

Fig. 4 The correlation between Re fraction and D retention (evaluated from the TDS spectra) in Fe-ion-irradiated pure W and W-*x*Re (*x* = 1, 3 and 5 at.%) alloy samples, together with the corresponding non-irradiated samples. The samples were prepared by zone melting, irradiated at 1073 K with Fe ions to a peak damage level of 0.5 dpa and then exposed to D_2 gas at 673 K and 100 kPa.

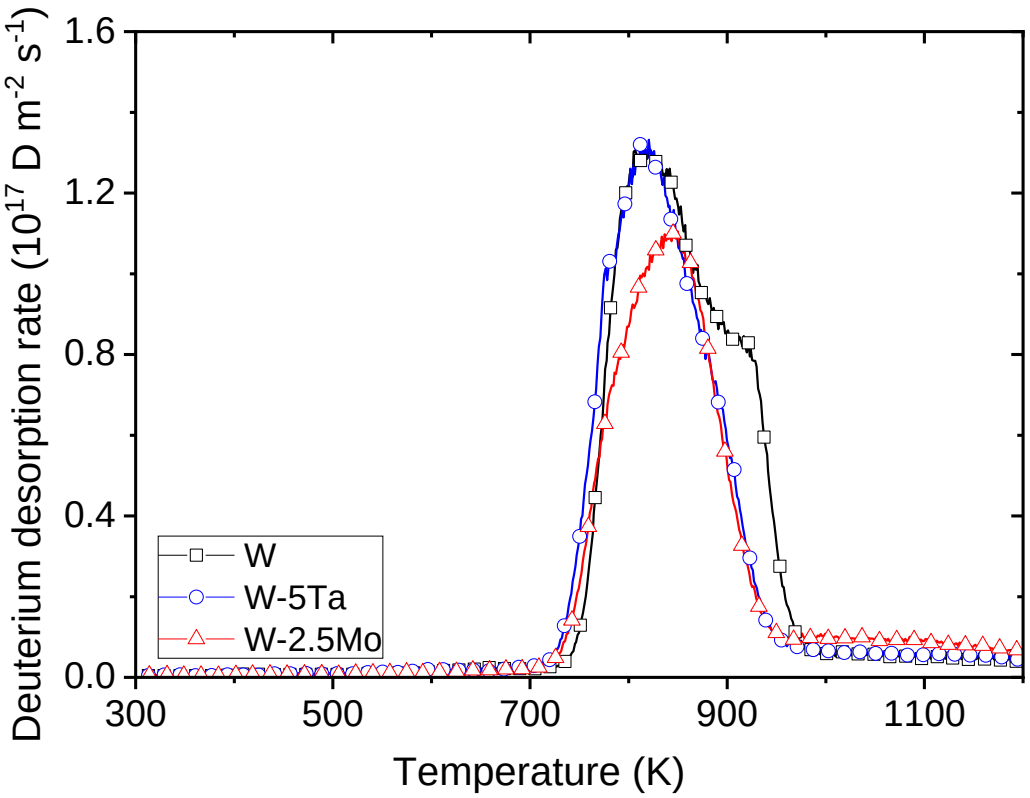

Fig. 5 TDS spectra of deuterium released from pure W, W-2.5Mo alloy and W-5Ta alloy samples prepared by zone melting. The samples were irradiated at 1073 K with 6.4 MeV Fe ions to a peak damage level of 0.5 dpa and then exposed to D_2 gas at 673 K and 100 kPa. Heating rate was 0.5 K/s.

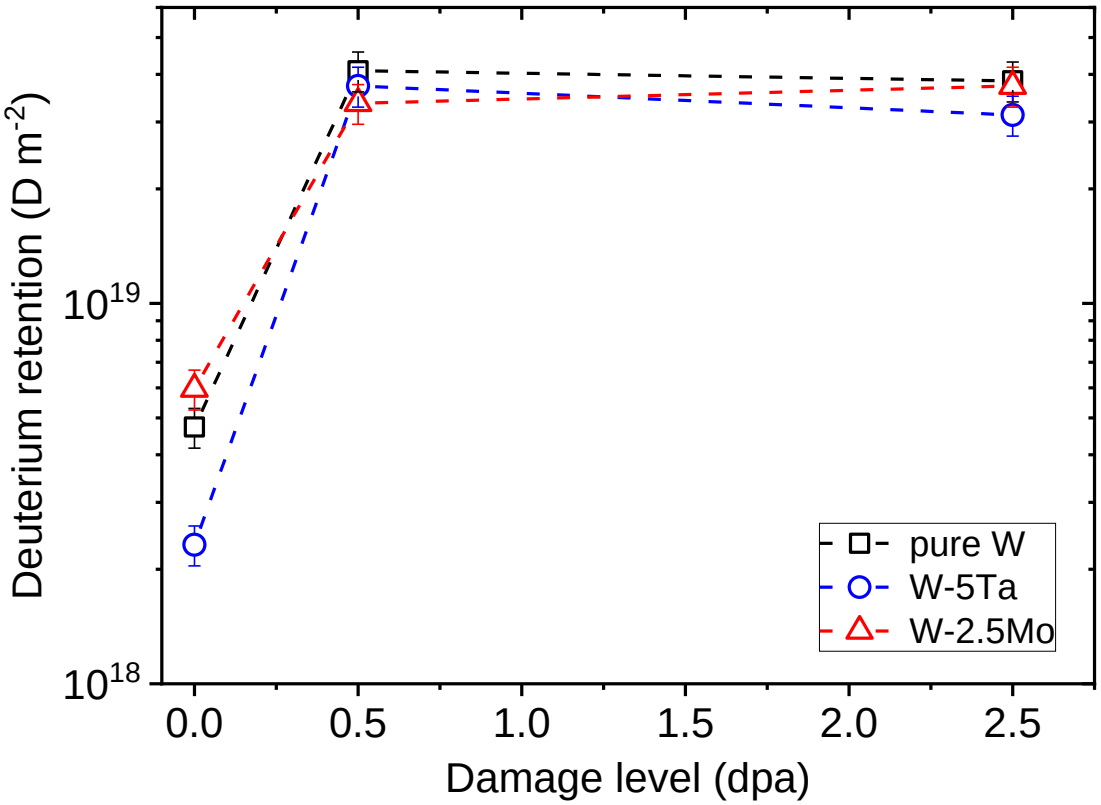

Fig. 6 The correlation between damage levels and D retention (evaluated form TDS spectra) in pure W, W-2.5Mo alloy and W-5Ta alloy samples prepared by zone melting. The samples were irradiated with 6.4 MeV Fe ions at 1073 K to peak damage levels of 0.5 and 2.5 dpa and then exposed to D_2 gas at 673 K and 100 kPa.