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Long-term fuel retention and release in JET ITER-Like Wall at ITER-relevant baking temperatures

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Abstract

The fuel outgassing efficiency from plasma-facing components exposed in JET-ILW has been studied at ITER-relevant baking temperatures. Samples retrieved from the W divertor and Be main chamber were annealed at 350 and 240 °C, respectively. Annealing was performed with thermal desoprtion spectrometry (TDS) for 0, 5 and 15 h to study the deuterium removal effectiveness at the nominal baking temperatures. The remained fraction was determined by emptying the samples fully of deuterium by heating W and Be samples up to 1000 and 775 °C, respectively. Results showed the deposits in the divertor having an increasing effect to the remaining retention at temperatures above baking. Highest remaining fractions 54 and 87% were observed with deposit thicknesses of 10 and 40 μm, respectively. Substantially high fractions were obtained in the main chamber samples from the deposit-free erosion zone of the limiter midplane, in which the dominant fuel retention mechanism is via implantation: 15 h annealing resulted in retained deuterium higher than 90%. TDS results from the divertor were simulated with TMAP7 calculations. The spectra were modelled with three deuterium activation energies resulting in good agreement with the experiments.

Keywords: fuel retention, ITER, deposition, divertor, main chamber, JET-ILW

(Some figures may appear in colour only in the online journal)

1. Introduction

The JET ITER-like wall (ILW) experiment provides a unique environment to study critical plasma-material interactions (PMI) for ITER such as material erosion and migration and in-vessel fuel retention and removal [1]. The ILW comprises bulk beryllium (Be) main chamber limiters and upper dump plates, and the divertor region is a mix of bulk tungsten (W) tiles and W-coated carbon fibre composite (CFC) tiles [2]. Removal and replacement of the plasma-facing components (PFC) during shutdown phases allows for post-campaign (*post-mortem*) analyses to scrutinize the campaign integrated net effects of the PMIs [3].

ITER is a nuclear licensed facility and the in-vessel tritium (T) retention will be limited to 1 kg to minimize the risks of release of the mobilized T during accidents [4]. The ITER

[a] See the author list of 'Overview of the JET results in support to ITER' by Litaudon *et al* to be published in *Nuclear Fusion* Special issue: overview and summary reports from the *26th Fusion Energy Conference (Kyoto, Japan 17–22 October 2016)*

strategy to recover the trapped T in the vacuum vessel is to perform baking of the PFCs, at 240 °C for the Be first wall and at 350 °C for the W divertor [5]. In the present work, the effectiveness of annealing to the fuel removal at ITER-relevant baking temperatures is studied with samples removed from JET-ILW PFCs. Recent post-mortem analyses of JET-ILW PFCs have shown the majority of the deuterium (D) being retained in the divertor region within deposited Be layers [6]: erosion of the Be main chamber PFCs leads to material migration to the inner divertor [7], where the fuel particles are co-deposited. Such co-deposited layers in the divertor will be the driving mechanism behind the T inventory in the ITER PFCs in steady-state long pulses [8]. The release of the fuel particles from the co-deposited layers is challenging due to the fuel release dynamics being slowed down by the deposited layer thickness [9] and its morphology. Moreover, the presence of additional impurities, such as oxygen (O) and carbon (C), or layers with elements being mixed with W, may affect the resulting release rate [10].

The samples used in this study have been taken from the W-coated divertor tiles and from the main chamber bulk Be limiters. The condition of the sample surfaces were as-received after the JET-ILW operational periods and had varying amounts of surface deposition. Annealing, fuel release and remaining fuel fractions were assessed using Thermal Desoprtion Spectrometry (TDS) and the results were analyzed with TMAP7 simulations.

2. Methodologies

2.1. Analyses of deposited layers

The cross-section of the JET-ILW main chamber and the divertor is shown in figure 1. Previous studies have shown the highest retention is found on top of the inner divertor with the thickest deposition layers on Tile 0 and on the top region (*apron*) of Tile 1 [6]. In figure 2 an example of an ion beam analysis (IBA) is presented, aresult of the impurity depth profile concentrations found on the Tile 1 apron. Also shown is an optical microscopy image presenting the morphology of the deposit on top of the W coating[7] (see also [11]). The IBA results have shown the main impurity component in the JET-ILW deposits is Be, but also other impurities, such as C and O, are present. C is a minor impurity in JET plasmas as a remanent from the JET all-C wall prior converting to the all-metal ILW, whereas O may be originating from the vacuum between the plasma discharges.

Analyses of the parameters effecting to fuel retention in ILW deposits is challenging due to the large surface roughness of the deposits, their varying chemical composition, and unknown morphology. Hence, a combination of selected ion beam techniques needs to be applied [12]. The exemplary IBA result of a thick deposit shown in figure 2 is obtained with using nuclear reaction analysis, elastic backscattering and particle induced x-ray emission in conjunction with 2.3 MeV ^{3}He beam for detecting D, Be and heavier elements ([6] and references therein). Further, the results have been fitted with WiNDF data furnace package [13] for providing the depth profiles.

[7] The analyzed Tile 1 has a special marker coating with a W/Mo/W structure on CFC.

2.2. Sample selection and experimental methods

The W-coated CFC tiles used for the present TDS study are Tiles 0, 1 and 6 (figure 1). Sample selection was based on having a varying thickness of deposit on the samples and to study the effect of thickness on fuel release. All samples were from the ILW 2011–2012 period, whereas Tile 0 was exposed for two periods 2011–2012 and 2013–2014 and hence providing the thickest deposit information. The nominal deposit thicknesses found on tiles, the tile in-vessel ILW periods and the sample IDs are listed in table 1. Divertor sample preparation for TDS has been described in detail in [16, 19]. The samples are cored from the tiles by using a hollow drill (diameter 17 mm), and the cores are further sliced to ∼1 mm thick samples to be used with the TDS.

In addition to divertor samples, a set of bulk Be samples from the main chamber limiters was prepared and analyzed with TDS. Locations chosen were from the midplane of the inner and outer limiters (tile IDs 2XR10 and 4D14, respectively), see figure 1. All the main chamber samples are from 2011–2012 period. The limiter midplane has been shown to be the major Be source in JET-ILW due to high particle fluxes and surface temperatures [7, 17]. However, part of the eroded material gets toroidally redeposited locally to the edges of the limiters [18]. These regions are radially ∼3 cm further away from the plasma contact point of the limiters. Deposited layers on the edges do not show uniform patterns, but a rough surface with local deviations from 0 to several μm. Samples for the fuel retention studies were prepared both from the central main erosion zone and from the deposited edges of the limiters. The cutting and preparation method of bulk Be samples to meet the TDS requirements has been presented recently in [19]. The bulk Be pieces ($12 \times 12\,mm^2$) were cut with a band saw using no coolants to avoid any impurities left to the sample surfaces. During cutting the sample temperature was monitored not to exceed 55 °C. Cut samples were thinned down to 2.5 mm for TDS.

The TDS measurement setup, the heating method and the W-coated CFC and the bulk Be sample preparations are described in [16, 19]. The TDS system is operated at 10^{-9} mbar and comprises of a heating stage on to which the samples are clamped. Heat is transferred via a molybdenum (Mo) annealing plate and the heating and the resulted Mo temperatures are recorded with thermocouples. A quadrupole mass spectrometer (QMS) is positioned perpendicularly with a line-of-sight view to the sample surface to maximize the collection of desorbed molecules (figure 3).

The TDS annealing profile can be preprogrammed freely. For the present work, the temperature was increased with an annealing rate (β) of 10 or 1 K min^{-1} until the required ITER-relevant bake temperature of 350 °C for W and 240 °C for Be was reached. A schematic temperature profile is shown in figure 3. Once the set temperature is reached, the temperature

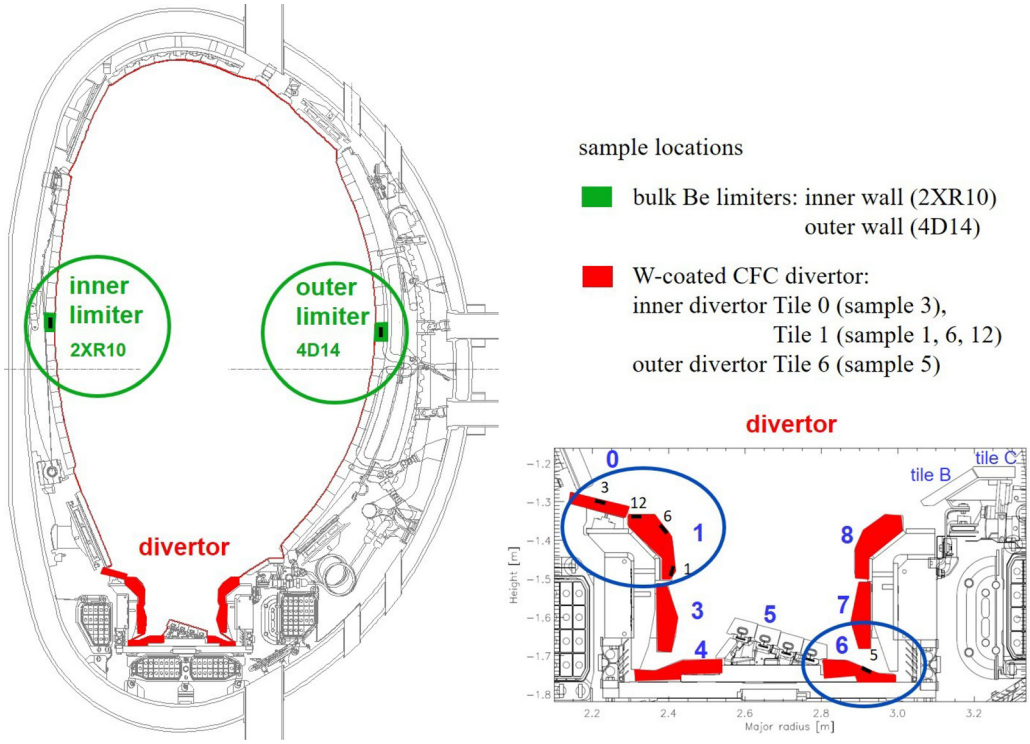

Figure 1. Cross-section of the JET-ILW main chamber and the divertor. Locations of the inner and outer wall bulk Be limiter specimens highlighted in green. The divertor W-coated CFC tiles highlighted in red and the tile numbering from Tile 0 to Tile 8 is from the high field to the low field side, correspondingly. TDS sample locations are circled and highlighted with black ID numbering.

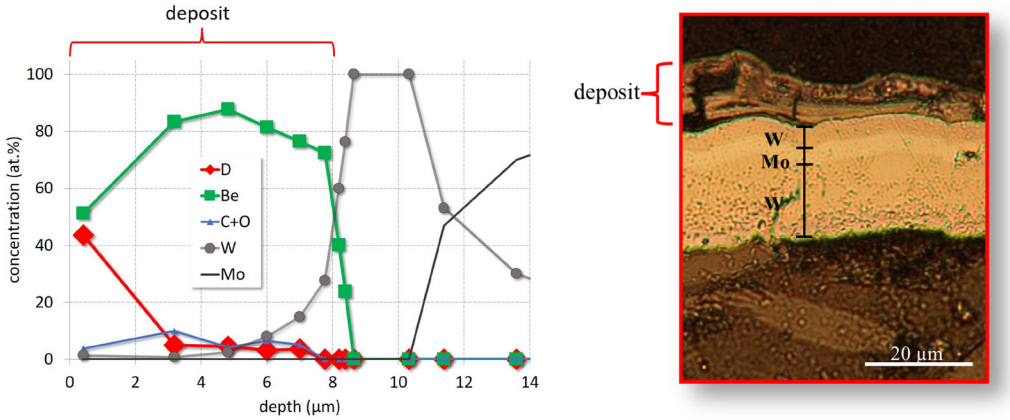

Figure 2. Left: IBA result of the deposit impurity depth profiles found on top of Tile 1. The main impurity component is Be forming up to ∼10 μm thick layers, other impurities (C, O) visible. Right: optical microscopy image of the deposit found on top of Tile 1.

Table 1. Summary of divertor sample properties used in the TDS analyses. Sample IDs as shown in figure 1.

Sample location	Sample ID	ILW period[a]	Deposition (μm)
Tile 0	Tile 0–3	2011–2012, 2013–2014	40[b]
Tile 1 apron	Tile 1–12, Tile 1–11	2011–2012	10[c]
Tile 1 upper vertical	Tile 1–6	2011–2012	3[c]
Tile 1 lower vertical	Tile 1–1	2011–2012	0[c]
Tile 6 slope	Tile 6–5	2011–2012	5[c]

[a]The ILW period 2011–2012 comprises of discharge numbers 80176-83794, and the ILW period 2013–2014 of discharge numbers 84442-87944.
[b]Reference [14].
[c]Reference [15].

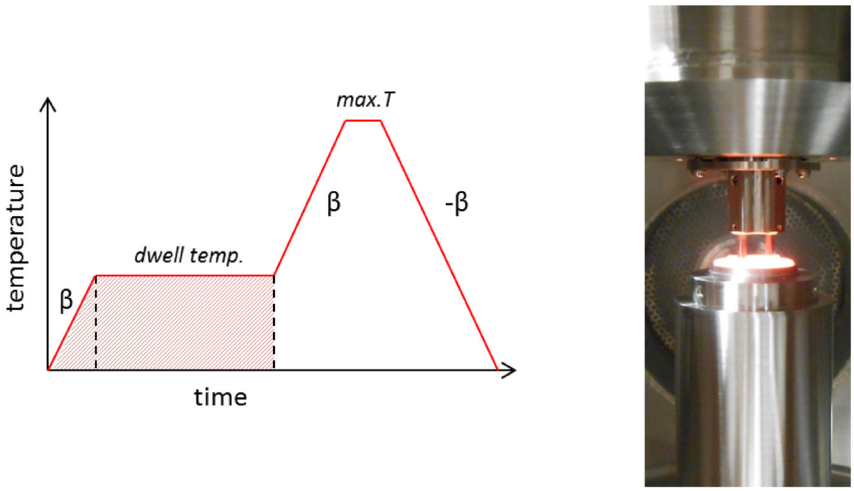

Figure 3. Left: Schematic annealing profile used for JET-ILW samples. Shaded area corresponds to the ITER baking time. Right: ILW sample positioning in the TDS chamber.

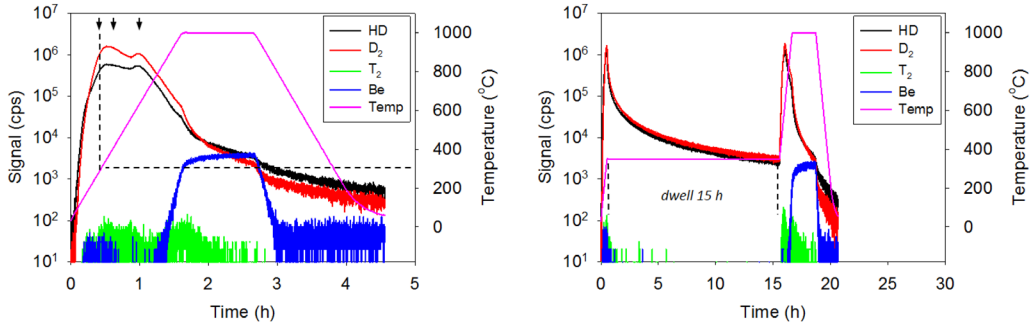

Figure 4. Annealing at 350 °C of samples from Tile 1 apron (ID: Tile 1–12). Left: Standard anneal with $\beta = 10$ K min^{-1} showing three D desorption maxima. The peak positions highlighted with arrows. Right: effect of 15 h baking to the D release. Significant amount of D is outgassed above 350 °C.

was kept constant for 0, 5 or 15 hrs to study the fuel release efficiency. After the residence/dwell time the samples were emptied of D by increasing the temperature to its maximum. For W-coated CFC samples 1000 °C was used, whereas the bulk Be sample maximum temperature was set to 775 °C. This is due to an increased Be evaporation from the sample at high temperatures and to prevent the TDS chamber being internally coated with Be.

2.3. Computational

The D desorption spectra for the full annealing profile of the W-coated samples were simulated with TMAP7 calculations. Details of the calculations can be found in [20]. TMAP7 is a 1D diffusion-trapping code, which is used for calculating time-dependent evolution of concentrations of atomistic elements in material structures and fluxes of these elements across the structural boundaries. In the present work, the deposited Be layer with its experimental thickness was included in each simulation. The W substrate was included in the system, and particle flows across the boundary were taking into account. As an initial input to the calculations, the D diffusion and recombination coefficients in Be co-deposits were taken from [21] and the fitting was done to trap populations and their activation energies until an agreement with the experimental TDS spectra was found.

3. Results and conclusions

3.1. Divertor: W-coated CFC samples

Examples of D outgassing spectra with 0 and 15 h annealing at 350 °C are presented in figure 4. The D release takes place already at low temperature (~40 °C) and the full spectrum has three release maxima (~328, 418 and 552 °C) as highlighted in figure 4 with arrows. It is worth noting that the two first maxima form broader sum peak seen in figure 4. Each of the three maxima corresponds to a D trapping site. Also shown the T_2 release and Be evaporation. Keeping the temperature constant for 15 h at 350 °C, the first trap was found to be outgassing, but 61% of the total D was found to be retained in the second and third trap. Identical D release characteristics was found in all of the divertor samples: the first trap is emptied fully and the second trap partially as temperature is increased from RT to 350 °C followed by constant anneal at 350 °C for extended periods. The remaining fraction of D was found to correlate with the thickness of the deposited layer. The results are summarized in figure 7 in which the remained fuel

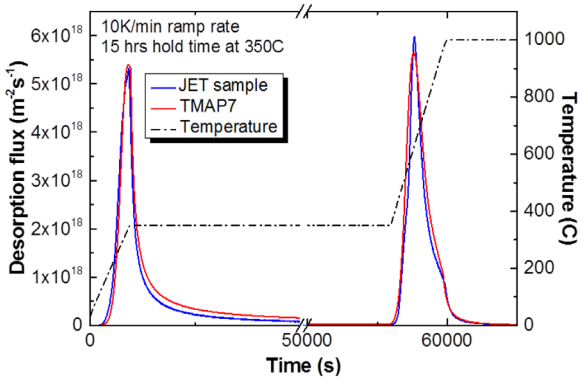

Figure 5. TMAP7 simulation result for the anneal of Tile 1 apron (ID: Tile 1–12).

fractions to the total fuel release as a function of the annealing time at 350 °C are shown. The highest remaining fraction of 87% was measured after 15 hrs on Tile 0 with a 40 μm deposit thickness. Interestingly, the fastest fuel release was observed with a 3 μm thick deposit after 5 hrs: the high release rate may be due to prompt emptying of the low-energy near-surface traps, however, further investigations are required for scrutinizing this observation. In general, the TDS results indicate the annealing of samples with deposits at 350 °C will require heating times longer than several tens of hours in order to meet the ITER fuel removal criterium [20].

The TMAP7 simulations were found to be in good agreement with the TDS spectra (example in figure 5). As expected from TDS, a three-trap model was required to fit the data. The resulted D trap energies obtained were 0.75–0.8, 1.1 and 1.4 eV, respectively. The two lowest trapping energies agree well with the model presented in [21]. The lowest trapping energy is related to the weakly bound D atoms found close to the deposit surface (figure 2) and which get released at low temperature. It is worth mentioning that JET-ILW 2011–2012 did not finish with hydrogen cleaning pulses, hence a high near-surface D concentration is formed. Also, all the divertor samples studied were from the plasma scrape-off layer (SOL) regions which do not have the highest temperatures and particle fluxes and impact energies. The retention in these regions is a mixture of co-deposition and low-energy implantation. The second trapping site may be related to the deposit morphology and/or to the impurities within the deposited layer. It is worth noting that deposits in [21] had very little amounts of C, O making the second trap due to impurities less plausible. The third and highest trapping energy may correlate with the impurities and/or large-sized defects within the deposit, i.e. the deposit may contain void-like defects or other open volume structures, which can efficiently trap D. Detailed computational work is ongoing for confirming the origin of these trapping sites. Finally, since ITER is an all-metal machine, it is not expected to have C in the plasma hence the long-term retention due to C in Be deposits may be lower when compared to ILW deposits.

3.2. Main chamber: bulk Be limiter samples

The limiter samples represent the main erosion region with the highest PMI and the limiter edge, which resides deeper in the SOL. An exemplary TDS result of the outer limiter midplane (figure 6) reveals up to four D desorption maxima. Computational analysis of these results is ongoing, which will provide more detailed conclusions on the experimental observations. Only the first trap can be seen to be emptied as the temperature is increased to 240 °C, and the remaining D is released at higher temperatures. In general, results indicate even after 15 h bake at 240 °C that over 90% of D is remained in the midplane of inner and outer limiters (figure 7). These samples are free from deposits due to erosion from the high heat and particle loads during JET plasma limiter phase. Hence the remaining D can be considered to be retained in implantation-induced traps. However, analyses for the exact retention mechanism in these bulk Be samples are ongoing to study the role of implantation, and the intrinsic Be defects and impurities to the total retention. Interestingly, a bulk Be sample removed from the toroidal edge of the inner limiter showed a high decrease in the D retention after a 15 h anneal (figure 7). The decrease may be due to the in-vessel position of the sample: the edges of the limiters are recessed and do not have direct contact with the plasma. In other words, fuel retention to these regions may be low-energy implantation and co-deposition making the majority of D retention in traps with low activation energies. Investigations are underway to study the parameters effecting the fuel retention in different regions of the main chamber limiters. Finally, even though the earlier JET-ILW post-mortem results in [6] showed an order of magnitude less *global* retention in the main chamber compared to the divertor, the relatively larger Be surface area in ITER and its low baking temperature of 240 °C with low fuel release may play an important role in assessing the accumulated T. However, this is expected to be compensated by the saturation of hydrogen in Be. At local concentrations of 30 at.% of hydrogen in Be, further hydrogen will be re-emitted ([22] and references therein).

4. Summary

Samples retrieved from JET-ILW were annealed at ITER-relevant baking temperatures to study the fuel outgassing efficiency. W divertor samples and Be main chamber samples were kept at 350 °C and 240 °C, respectively, for 0, 5 and 15 h, and the deuterium fuel release was monitored with TDS. W and Be samples were finally annealed up to 1000 and 775 °C, respectively, for emptying the samples of deuterium. The remaining deuterium fractions after the 350 °C anneal in W showed a correlation with the deposition thickness: highest remaining fractions of 54 and 87% were observed with deposition thicknesses 10 and 40 μm, repsectively. TMAP7 calculations were used for simulating the TDS results. The measured TDS spectra were fitted with a three-trap model with detrapping energies of 0.75–0.8, 1.1 and 1.4 eV. Annealing of bulk Be limiter samples at 240 °C showed that even after 15 h of baking, a sample without deposition retained over 90% of deuterium. These Be samples are from the main chamber Be erosion zone, which is free from deposits and in which the retention is expected

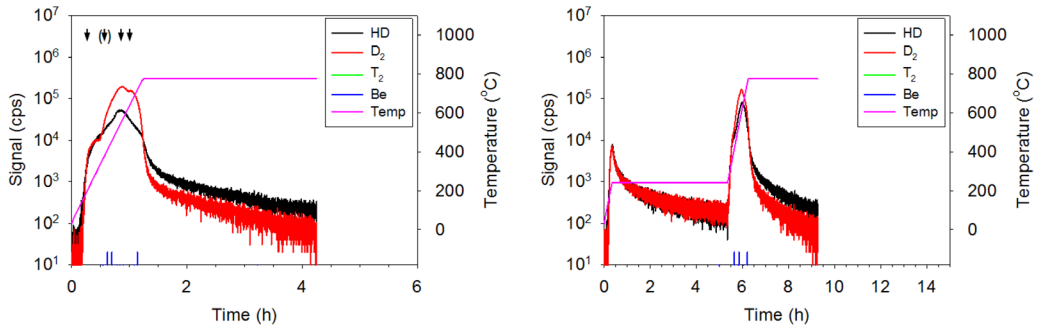

Figure 6. Annealing at 240 °C of bulk Be from the outer limiter midplane. Left: standard anneal with $\beta = 10$ K min^{-1}. The D release spectrum consists of three to four maxima (pointed by arrows). Right: effect of 5 h annealing to the D release. The first trap is emptied at 240 °C, remaining traps are outgassed during the second ramp.

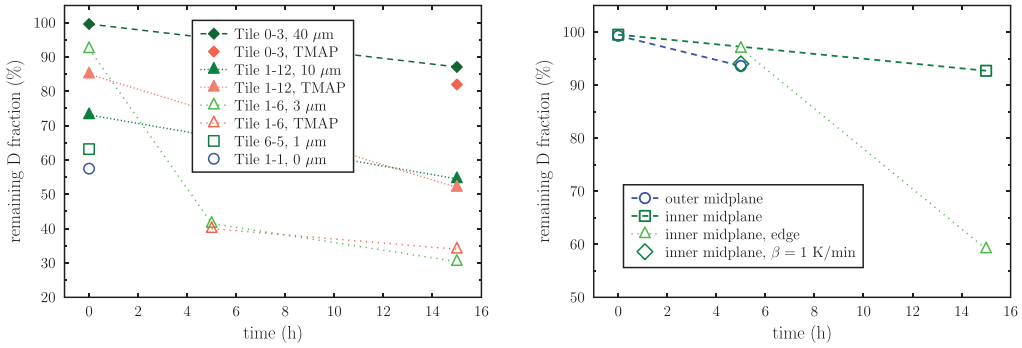

Figure 7. Summaries of the remaining fractions for the W-coated divertor (left) and bulk Be limiter (right).

to take place via implantation. Further computational and experimental studies are required for determining the fuel retention mechanisms in Be limiters. Finally, minor amounts of T was found to be released from the ILW samples during the ITER-relevant baking experiments. The observed T amount is negligible compared to the measured D. Part of the T originates from the D-T campaign performed with the all-C wall. Also, some T is formed in the D-D plasmas from which the energetic T is implanted in the sub-surface regions of the deposits or PFCs.

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