WPPFC-CPR(17) 17961

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Preprint of Paper to be submitted for publication in Proceeding of 16th International Conference on Plasma-Facing Materials and Components for Fusion Applications

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

A Parametric study of Helium retention in Beryllium and its effect on Deuterium retention

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Abstract

Beryllium samples have been exposed in the PISCES-B linear plasma device to conditions relevant to the International Thermonuclear Experimental Reactor (ITER) in pure He, D, and D/He mixed plasmas. Except at intermediate sample exposure temperatures, 573-673 K, He addition to a D plasma is found to have a beneficial effect as it reduces the D retention by implantation in Be (up to ~55%). Retention of He is typically around 10^{20}-10^{21} He/m^2, and is affected primarily by the Be surface temperature during exposition, by the ion fluence at <500 K exposure, but not by the ion impact energy at 573 K. Contamination of the Be surface with high-Z elements in pure He plasmas is also observed under certain conditions, and leads to unexpectedly large He retention values, as well as changes in the surface morphology. An estimation of the tritium retention in the Be first wall of ITER is provided, being sufficiently low to allow a safe operation of ITER.

1. Introduction

The International Thermonuclear Experimental Reactor (ITER) first wall armor (FW) will be made of beryllium and cover an area of about 700 m^2. The FW will be exposed to two types of particle fluxes from the plasma core (estimated as a mix of 47.5%D + 47.5%T + 5%He during burning plasmas, i.e. at maximum power): a large flux of low energy ions (few eV) and a low flux (1-10^{20} m^{-2}s^{-1}) of energetic charge exchange neutrals (100-500 eV) as predicted by [1,2]. A fraction of both fluxes will contribute to the total in-vessel tritium inventory (actually set to a maximum of 700 g in the whole vessel) by implantation in the Be FW as observed for the same material selection in the JET tokamak [2]. Moreover, the Be FW in ITER will operate at low surface temperatures of 373-423 K [3], leading to a large hydrogen isotope retention in low-energy traps [4]. However, at high ion flux areas, the wall temperature in ITER can be significantly higher reaching conditions like in the JET experiments observed.

Hydrogen isotopes retention in beryllium has been studied in detail (being deuterium the most extensively studied to distinguish from natural hydrogen, and not being radioactive as tritium is), see for example [5] and references therein, which canvasses both low-flux ion beams [4,6], laboratory plasmas [7–10] and exposures in JET [2,11,12]. However, there are few works about helium retention in beryllium, and they are limited to low fluence, high energy ion beams [13,14], or to neutron transmutation [15,16].

Recently, interest has arisen in He retention in beryllium due to the observed legacy effect from ^{3}He injection in JET [17], introduced to allow RF minority coupling to the plasma, and the effect of small additions of He to D plasmas [7] due to the large decrease of D retention seen in tungsten [18]. This last observation is critical for ITER, as it will work with a mix of D/T plasma as fuel with a 5-10%He as a product (ash) during burning plasmas. Any decrease in the fuel retention, especially for T, will be highly beneficial for ITER, as it allows a longer and a safer operation. This work examines these issues and expands on previous work in PISCES-B [7] in the form of a D and He retention in Be parametric study involving sample temperature, impact energy, He fraction and fluence with quantification of the He retention in a manner that was not possible in [7].

2. Experimental

Be samples (S-65 type, from Peregrine Corp, 7.5 mm diameter, 1.1 mm thick) were exposed to plasmas of D, He and their mixes (5%,10% and 15% He) in the PISCES-B linear plasma device [19]. PISCES-B produces a steady-state plasma ~1 m long and 5 cm diameter which is incident on a temperature-controlled, water/air-cooled target and holder assembly designed to accommodate 25 mm diameter targets. To affix the smaller targets to the holder, a 25 mm diameter metallic mask (molybdenum, later replaced by one of tantalum) was used, giving a plasma exposed Be surface of 6 mm diameter. The ion flux, plasma density, and electron temperature in the vicinity of the target were measured by a Langmuir probe and were typically 1-4·10^{22} m^{-2}s^{-1}, 1-5·10^{18} m^{-3} and 4-6 eV respectively in D and D/He plasmas, and 3-6·10^{22} m^{-2}s^{-1}, 3-7·10^{18} m^{-3} and 8-12 eV in He plasmas. Fluences were of the order of 10^{26} m^{-2} ensuring steady-state conditions for most of the experiment. The sample was negatively biased up to -120 V to attract and control the monenergetic ion impact energy, the value being calculated from the plasma-target potential difference [19]. Plasma light emission was observed with a Czerny-Turner type spectrometer (focal length: 500 mm) equipped with a two-dimensional CCD camera at the exit port. The spatial resolution is ~1.3 mm in the axial direction. The He^+ ion concentration, n_{He^+}/n_e , in the D/He mixture plasma was measured using a spectroscopic technique [20]. Here, n_{He^+} is the He^+ ion density. In order to calculate the D fluence presented in the appendix the ion distribution of the D_2 molecule was estimated to be similar to previous works [10]: D^+, D^{2+}, D^{3+} are 0.25, 0.47, 0.28 respectively. This distribution is estimated to be similar with the addition of He as it could not be measured.

After plasma exposure the samples were analyzed using a Scanning Electron Microscope (SEM, JEOL JSM-6310) to characterize the surface, which typically develops a cone-like structure following D and He plasma exposure [7,21]. Following this, the D and He thermal release and retention in the samples were measured by Thermal Desorption mass Spectrometery as described in [22]. Briefly, it consists of installing the sample within a vacuum oven with a BeO crucible heatable to ~1700 K. In this work the Be samples are heated to 1530 K at a rate of 0.3 K/s and then kept for 20 min to assure the complete release of He and to avoid excessive Be evaporation. The released gas (HD, D_2, He) is analyzed with a high-precision 0-6 m/q Residual Gas Analyzer (RGA, MKS), capable of distinguishing between D_2 and He. He and HD/D_2 release is calibrated by He and D_2 leak bottles respectively (HD ionization is assumed to be the same as D_2), following each TDS measurement due to the high sensitivity of the RGA. Here we also note, that the use of a He leak allowed for optimization of the RGA for the detection of He, thus providing much improved He detection and quantification over that of the previous work in [7]. Finally, the sputtering yield could not be measured by gravimetry because the mass loss was very low due to the small size of the samples, therefore no information about the ratio of net and gross erosion is available.

3. Results

3.1 Deuterium and helium retention

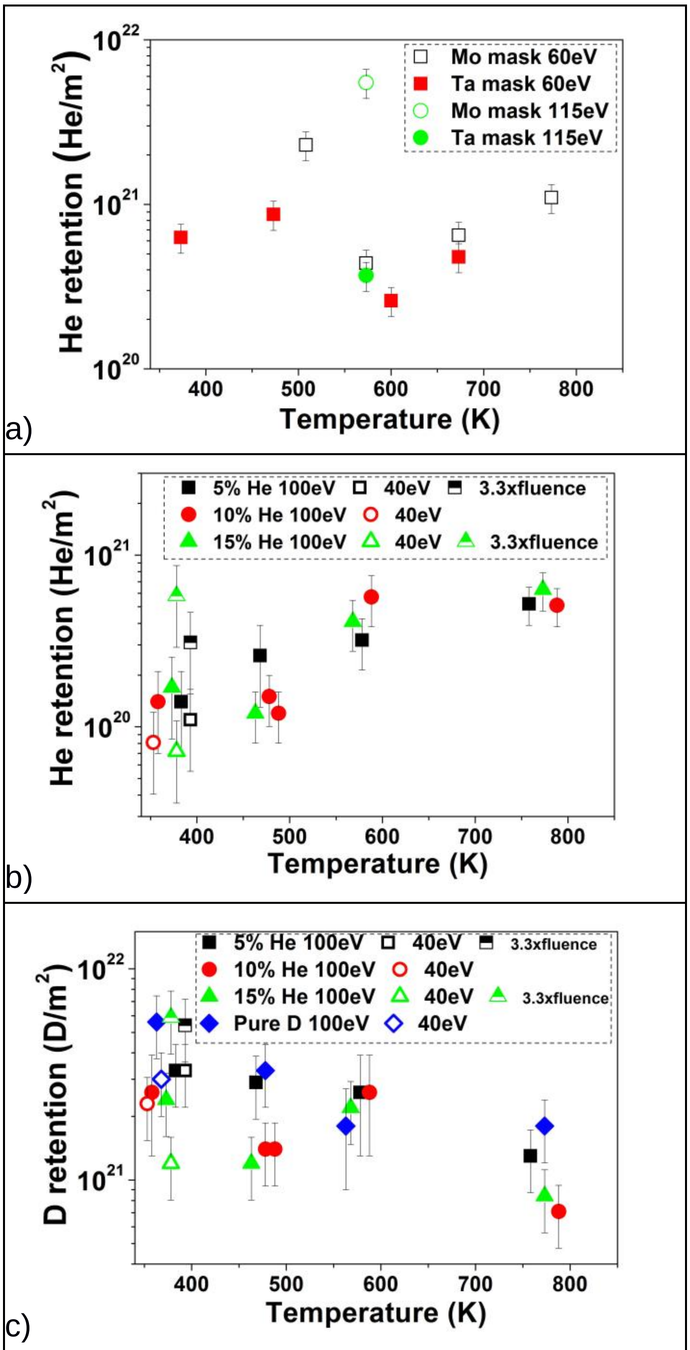

Figure 1: Retention from Be samples exposed to PISCES-B plasma. Temperature error is ±30 K, it has not been included in the graph for the sake of clarity. a) He retention in pure He plasmas using Mo and Ta masks, $2{\cdot}10^{26}$ He/m^2 fluence; b) He retention in D/He mix plasmas, 10^{26} m^{-2} total ion fluence; a) D retention in D and D/He mix plasmas, 10^{26} m^{-2} total ion fluence. Experiments at $3.3{\cdot}10^{26}$ m^{-2} total ion fluence have an ion energy of 100 eV. The data given in this graph along with the partial D and He fluences calculated by the estimated ion distribution are included in the appendix.

The measured He and D retention for samples of Be exposed in this work is given in Figure 1. Figure 1a reveals that He plasma exposure usually leads to a He retention in Be ~10^{20}-10^{21} m^{-2}. It is interesting to note that this level of He retention in Be is sufficient to explain the ^{3}He legacy found in JET [17] due to retention in the Be tiles of the main wall. In mixed D/He plasmas, the He retention is usually lower, except at 573 K, because of the much reduced He partial fluence (usually more than an order of magnitude). The deuterium retention in both D and D/He mixed plasmas is in the order of 10^{21}-10^{22} D/m^{2}, usually a factor 20-30 than helium retention at 373-473 K in D/He mixed plasmas, i.e. larger than their respective fluences. Incidentally, the D retention values for D implanted in Be found in this work are similar to those found in JET after the 2010-2012 campaigns [2,12], and 2013-2014 campaigns [11]. In view of this, similar D retention in Be might be expected for upcoming ITER operations, despite the slightly higher FW temperature in JET during plasma operation [2].

3.2 Traps

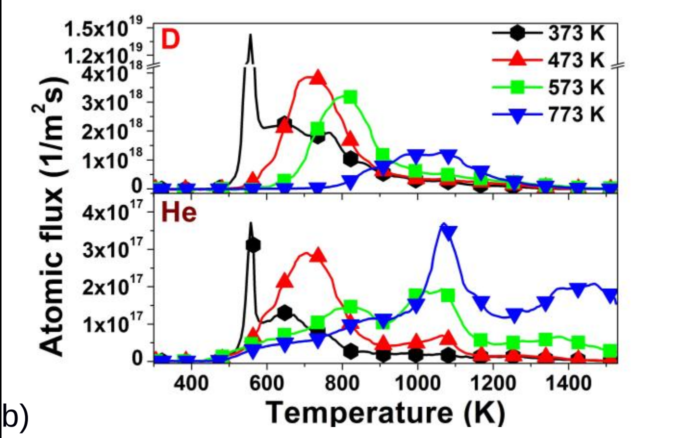

Figure 2: D and He release by TDS from Be samples at different temperatures exposed to PISCES-B D/5%He mix plasma. 10^{26} m^{-2} total ion fluence.

The TDS analysis of the samples indicates a complex behavior. As an example, in Figure 2, the D and He release in D/5%He plasmas are shown. D release from pure D plasmas is very similar to those in Figure 2, and from pure He plasmas the He release is similar to 773 K exposure in Figure 2, with the main peak at ~1130 K. However, this peak is almost absent in D/He mixed plasmas at lower exposure temperatures. On the contrary, the corresponding deuterium release peak increases in temperature with increasing sample exposure temperature (550-1000 K), being usually in a range much lower than the main release peak in pure He plasmas (~1130 K). Moreover, the helium release in D/He mixed plasmas follows the deuterium release in the range of <900 K, implying that both species are released via the same mechanism.

3.3 Microstructure

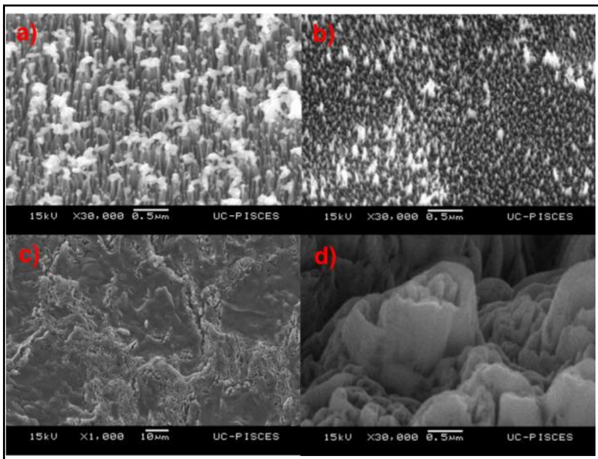

Figure 3: SEM images of Be samples exposed to PISCES-B pure He plasmas at a fluence of $2 \cdot 10^{26}$ He/m^2. a) 373 K and ion energy 60 eV; b) 573 K and 60 eV; c) 773 K and 60 eV; d) 573 K and 115 eV with ~25% Mo at surface because of contamination from Mo mask.

During D and He plasma exposure in PISCES-B it is known that Be samples develop a cone-like structure probably due to the monoangular (normal) incidence angle of the mononergetic ions [21]. As can be deduced from Figure 3a and 3b, this structure is mainly dependent on the sample exposure temperature, as the cones decrease in size with the temperature possibly due to the higher mobility of Be atoms. However, above 673 K a porous structure is noted, Figure 3c, as was also observed in [21]. It should be noted that in tokamak environment the impinging ions have an energetic as well as angular distribution, so this structure would probably not develop.

3.4 Contamination

Initial plasma exposures in pure He involved a Mo mask. When the ion impact energy was high (~100 eV) the Mo mask was prone to sputtering, leading to contamination of the Be sample by deposition of Mo on Be. This is the case for plasma exposure at 115 eV (green, empty circle in Figure 1a). As can be noted, He retention is seemingly affected by the eroded Mo that is deposited on the Be sample surface. This sample, following exposure, had an atomic surface concentration of Mo of 25% as measured by SEM-EDX, whereas the other samples exposed at 60 eV, where a Mo mask was also used, have <3% Mo. Switching the mask to Ta relieved this issue, as less than 1% Ta —at noise level— was observed under similar (115 eV) plasma exposure. Furthermore, it can be observed that at 60 eV there is no influence of the type of sample mask on the He retention, empty and filled red squares in Figure 1a, as both Mo and Ta are under the sputtering threshold for He. Finally, this Mo surface contamination also affects the microstructure greatly, Figure 3d, as the developed structure consists of multi-layered, large cones.

4. Discussion

4.1 Effect of exposure temperature.

In the previous work [7], a decrease in D retention with the addition of He (compared with the dilution of the D plasma, i.e. the lower fluence of D) was observed at exposure temperatures lower than 473 K. Here, this result is confirmed and extended to >673 K, see Figure 1c. D retention in pure D plasmas is larger at <473 K exposure than at higher exposure temperatures, 573 to 773 K, where it is approximately constant, filled blue diamonds in Figure 1c, which has already been described [7]. This means that the cone-like microstructure probably does not affect D retention in beryllium as it disappears at >673 K [21], Figure 3c. In view of this, the D retention in ITER's Be tiles at equatorial and upper ports, at 573 K, and upper targets, at 773 K, will be negligible compared to Be tiles in the main wall, at 373-423 K [3]. Another consequence, as stated in subsection 3.2, is that He and D are released via the same mechanism. Although a more detailed analysis is needed, they probably share the same traps at lower exposure temperature, <473 K, inside gas nanobubbles [13,14], and also at larger exposure temperature, 773 K, inside bubbles in the developed porous structure [21], Figure 3c. To speculate, bubble rupture as the sample is heated in TDS, for instance, could lead to synchronous D and He release for D and He trapped in those bubbles.

Helium retention in Be increases with temperature, see Figures 1a (from 573 to 773 K) and 1b, probably caused by the larger creation of high-energy traps. However, in pure He plasma the retention is larger at <500 K than at 573-673 K exposure when in mixed D/He plasmas it is not, compare Figures 1a and 1b. The presence of D has to reduce the filling of low-energy trap by He atoms. Additionally, at 573 K exposure the similar He retention values ($4\text{-}5\cdot10^{20}$ He/m^2) found, in both Pure He and D/He mixed plasmas point out to a fast saturation at even low fluences of He.

4.2 Effect of He fraction

The He fraction has no visible effect on microstructure, as the cones developed in D and He are very similar, just a bit narrower in D [7,21]. As can be seen in Figure 1b, increasing He fraction has little effect on the total He retention. The main effect found is the decrease of D retention at the conditions expected in most of ITER Be tiles (373 K): a 40% reduction at 5% He and 55% reduction at 10% He (again, compared to the D plasma dilution and consequently slightly lower D fluence as can be seen in the appendix). This is a smaller reduction than the one observed in W (up to 90% [18]), so the mechanisms will be likely different (in Be probably is related to D and He sharing the same traps). However, at some exposure temperatures (473 and 773 K) the addition of 5% He has no a big effect compared to 10-15% He, see Figure 1c. Moreover, as can be observed in the same Figure, at 373-473 K there is a maximum in the relative D retention decrease at 10% He which will be studied in the future.

4.3 Effect of ion impact energy

As already described in the previous work [7] and confirmed here in Figure 1c (empty symbols), reducing the ion impact energy also seems to strongly decrease D retention for exposures at temperatures similar to those expected in the first wall of ITER, 373 K. Decreasing ion energy also reduces He retention in D/He mixed plasmas, Figure 1b. In both cases this effect is likely related to the lower production of nano-bubbles [13,14] at reduced ion energy. In ITER the expected ion impact energy of CXN at the surface is around 100-500 eV, so the D retention calculated here may be lower than in ITER.

Conversely, in pure He plasmas without instances of Mo contamination exposure at 573 K showed no effect on the He retention for increased ion impact energy up to 115 eV, see green filled circle in Figure 1a.

4.4 Effect of high-Z contamination in pure He plasmas

Comparing the use of a Mo mask (which resulted in 25% Mo deposition on the Be surface) to a Ta one at an ion energy of 115 eV and exposed at 573 K (empty and filled green circles in Figure 1.a respectively) the He retention is increased by an order of magnitude with an associated He release at high temperature: 1130-1270 K. Moreover, ITER FW will contain an uncertain, but small, amount of tungsten atoms sputtered from the divertor into the plasma core. These tungsten atoms most likely will have a similar effect to Mo contamination found in this work, and it could lead to a larger retention of helium and possibly hydrogen isotopes [23]. In any case, it is currently unclear why Mo surface impurities can drive He retention and is worthy of further study (specially expanding to D retention), but it is noted that larger retention of hydrogen isotopes and He caused by high-Z element contamination has also been observed in other linear plasma devices [23]. One noted aspect caused by Mo is that the microstructure is strongly affected with the usual Be cone structure appearing as multilayered, cone-like structure, see Figure 3d. This is likely to be caused by the preferential sputtering of Be against the much heavier Mo.

4.5 Effect of fluence

Experiments in D/He mixed plasmas (half-filled symbols in Figures 1b and 1c) show that both He and D retention are increased as a function of the plasma exposure ion fluence. In the case of He, the increase is completely linear (i.e. 3.3 times more), so no saturation seems to be reached, although it is close to the value for pure helium plasma with a helium fluence 4 times larger (saturation is only reached at 573 K as commented in subsection 4.1). On the contrary, for D retention a saturation seems to be reached as the increase is only a factor 1.5-2. Moreover, the D retention for both experiments is very similar to the one in pure D, blue filled diamond in Figure 1c. The effect of fluence on the cone-like microstructure will be presented in a separate paper.

5. Conclusions and consequences for ITER

In this work a parametric study of helium and deuterium retention in beryllium has been done in the PISCES-B linear plasma device. Different parameters have been varied to simulate those expected in the different beryllium tiles in ITER burning plasmas regimes. Exposure temperatures from 373 to 773 K, ion energy up to 115 eV, ion fluences in the range of $1\text{-}3.3\cdot10^{26}$ m^{-2}; pure He or D plasmas, and mixes of 5-15% of He, have been explored. Overall, the results show that the desirable decrease of deuterium retention by implantation occurs at higher exposure temperature, lower ion impact energy and increasing He fraction (specially at 10% He and 373 K: 55% decrease). On the contrary, helium retention increases with exposure temperature, although in pure helium at <500 K the helium retention is larger than at 573-673 K. This has not been observed in D/He mixed plasmas (probably D atoms or molecules reduce the retention of He atoms in low-energy traps). Additionally, there is no big effect on helium retention of the helium fraction in D plasmas, and neither of ion impact energy at 573 K exposure (up to 115 eV). But, oppositely, in mixed D/He plasmas at 373 K exposure He retention increases with ion energy, and also increases at higher fluences, except at 573 K where a fast saturation is found at $4\text{-}5\cdot10^{20}$ He/m^2. Mo surface contamination due to deposition from the mask leads to increased He retention and a very different microstructure.

Similarity of the TDS release for D and He, specially at low temperature, implies that He and D are released via the same mechanism from some traps. The similar nature of the trapping and release is presently unclear but could be related to gas nano-bubble formation, and also may be related to the lower D retention with small additions of He.

As a final conclusion an indication of the tritium retention along the ~700 m^2 of Be tiles in ITER can be given using the data of this work: ~$2\cdot10^{24}$ T atoms, or ~9 g of T. This simple estimate is based on all Be tiles being at ~373 K, ion impact energy being at 100 eV, under a 47.5%D/47.5%T/5%He plasma, for an accumulated ion fluence of $3.3\cdot10^{26}$ m^{-2}, which is equivalent to roughly ~4000 full-pulses at a flux of $5\cdot10^{20}$ $m^{-2}s^{-1}$ (the mean of the range actually predicted for ITER).

6. Acknowledgments

Help provided by the PISCES-B team for the beryllium enclosure work, specifically L. Chousal, R. Hernandez and J. Yu is gratefully acknowledged. D. Alegre gratefully acknowledges the funding by Madrid Community through the Program TECHNOFUSION(II)-CM (S2013/MAE-2745). This work is a result of a US-EU bilateral agreement supported out of U.S. DOE Grant No. DE-FG02-07ER54912 and EUROfusion WP PFC call TD/ag-1568. This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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Appendix

Table 1: D and He retention on Be of the experiments shown in in Figure 1. All experiments have been done in PISCES-B linear plasma device.

%D_2	**%He**	**Mask**	**T (±30K)**	**Ion Fluence (10^{26} ion/m^2)**	**D Fluence (10^{26} D/m^2)**	**He Fluence (10^{26} He/m^2)**	**Ion energy (eV)**	**D Retention (10^{20} D/m^2)**	**He Retention (10^{20} He/m^2)**
0	**100**	Ta	373	2.0±0.5	0	2.0±0.5	60		**6.3±1.3**
0	**100**	Ta	473	2.0±0.6	0	2.0±0.6	60		**8.7±1.7**
0	**100**	Mo	500	2.0±0.3	0	2.0±0.3	60		**23±5**
0	**100**	Mo	573	2.0±0.3	0	2.0±0.3	60		**4.4±0.9**
0	**100**	Mo	573	2.0±0.5	0	2.0±0.5	115		**55±11**
0	**100**	Ta	573	2.0±0.5	0	2.0±0.5	115		**3.7±0.7**
0	**100**	Ta	600	2.0±0.5	0	2.0±0.5	60		**2.6±0.5**
0	**100**	Mo	673	2.0±0.3	0	2.0±0.3	60		**6.5±1.3**
0	**100**	Ta	773	2.0±0.3	0	2.0±0.3	60		**11±2**
100	**0**	Ta	373	1.0±0.2	2.0±0.2	0	40	**30±10**	
100	**0**	Ta	373	1.0±0.2	2.0±0.2	0	100	**56±19**	
100	**0**	Ta	473	1.0±0.2	2.0±0.2	0	100	**33±11**	
100	**0**	Ta	573	1.0±0.2	2.0±0.2	0	100	**18±9**	
100	**0**	Ta	773	1.0±0.2	2.0±0.2	0	100	**18±6**	
95	**5**	Ta	373	1.0±0.2	1.9±0.38	0.05±0.01	40	**33±11**	**1.1±0.5**
90	**10**	Ta	373	1.0±0.2	1.8±0.36	0.1±0.02	40	**23±8**	**0.81±0.41**
85	**15**	Ta	373	1.0±0.2	1.7±0.34	0.15±0.03	40	**12±4**	**0.72±0.36**
95	**5**	Ta	373	1.0±0.2	1.9±0.38	0.05±0.01	100	**33±11**	**1.4±0.7**
90	**10**	Ta	373	1.0±0.2	1.8±0.36	0.1±0.02	100	**26±9**	**1.4±0.7**
85	**15**	Ta	373	1.0±0.2	1.7±0.34	0.15±0.03	100	**24±12**	**1.7±0.9**
95	**5**	Ta	373	3.3±0.8	6.3±1.5	0.17±0.04	100	**54±18**	**3.1±1.6**
85	**15**	Ta	373	3.3±0.8	5.7±1.4	0.50±0.12	100	**59±20**	**5.8±2.9**
95	**5**	Ta	473	1.0±0.2	1.9±0.38	0.05±0.01	100	**29±10**	**2.6±1.3**
90	**10**	Ta	473	1.0±0.2	1.8±0.36	0.1±0.02	100	**11±4**	**1.2±0.4**
90	**10**	Ta	473	1.0±0.2	1.8±0.36	0.1±0.02	100	**14±5**	**1.5±0.5**
85	**15**	Ta	473	1.0±0.2	1.7±0.34	0.15±0.03	100	**12±4**	**1.2±0.4**
95	**5**	Ta	573	1.0±0.2	1.9±0.38	0.05±0.01	100	**26±13**	**3.2±1.1**
90	**10**	Ta	573	1.0±0.2	1.8±0.36	0.1±0.02	100	**26±13**	**5.7±1.9**
85	**15**	Ta	573	1.0±0.2	1.7±0.34	0.15±0.03	100	**22±7**	**4.1±1.4**
95	**5**	Ta	773	1.0±0.3	1.9±0.38	0.05±0.01	100	**13±4**	**5.2±1.3**
90	**10**	Ta	773	1.0±0.2	1.8±0.36	0.1±0.02	100	**7.1±2.3**	**5.1±1.3**
85	**15**	Ta	773	1.0±0.2	1.7±0.34	0.15±0.03	100	**8.4±2.8**	**6.3±1.6**