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Hydrogenic retention in tungsten exposed to ITER divertor relevant plasma flux densities

G.M. Wright [a,*], A.W. Kleyn [a,b], E. Alves [c], L.C. Alves [c], N.P. Barradas [c], G.J. van Rooij [a], A.J. van Lange [a], A.E. Shumack [a], J. Westerhout [a], R.S. Al [a], W.A.J. Vijvers [a], B. de Groot [a], M.J. van de Pol [a], H.J. van der Meiden [a], J. Rapp [a,d], N.J. Lopes Cardozo [a,e]

[a] FOM-Institute for Plasma Physics Rijnhuizen, Association Euratom-FOM, A member of the Trilateral Euregio Cluster, Postbus 1207, 3430 BE Nieuwegein, The Netherlands
[b] Leiden Institute for Chemistry, Leiden University, Leiden, The Netherlands
[c] Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2686-953 Sacavém, Portugal
[d] Institut für Plasmaphysik, Forschungszentrum Jülich, Association EURATOM, 52425 Jülich, Germany
[e] Eindhoven University of Technology, Eindhoven, The Netherlands

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ABSTRACT

Tungsten targets are exposed to the plasma conditions expected at the strike point of a detached ITER divertor ($\sim 10^{24}$ D/m^{2}s, $T_e \sim 2$ eV). The surface temperature of the target is ~1600 K at the center and decreased radially to ~1000 K at the edges. A 2-D spatial scan of the W target using nuclear reaction analysis (NRA) shows an asymmetric D retention profile with the lowest retention values at the center of the target and the highest 6 mm off-center. Even in the regions of larger retention, the D concentrations were $\leqslant 5 \times 10^{15}$ D/cm^2 as measured by NRA. Thermal desorption spectroscopy (TDS) is used to measure the global D retention. Very low retention with retained fractions ranging from 10^{-7} to 10^{-5} $D_{retained}/D_{incident}$ were measured with TDS. Both NRA and TDS results show no clear dependence of retention on incident fluence possibly indicating the absence of plasma-driven trap production in W under these conditions.

1. Introduction

Hydrogenic retention in the walls of ITER can affect density control and fuelling rates. Also, during deuterium(D)-tritium(T) operation, there is a safety issue as only 350 g of mobilizable T are allowed to be stored in the ITER wall [1,2]. Tungsten (W) is marked for use as a plasma-facing components (PFC) material in the ITER divertor. The thermal properties of W allow it to survive the expected heat loads at the ITER strike points but, perhaps more importantly, W also has low hydrogenic solubility and, in the absence of a strong hydrogenic trap production mechanism, is expected to have low hydrogenic retention levels. This is confirmed by numerous laboratory studies on hydrogenic retention in W [3–8]. From this it has been assumed that W has an advantage over carbon-based materials (i.e. graphite, CFCs) with respect to tritium inventory in the ITER divertor.

However, studies have identified a mechanism for trap production in refractory metals, specifically W. It has been postulated that exposure of W to a high flux of low energy (⩽200 eV) ions leads to a build-up of stresses in the W lattice due to the low hydrogenic solubility of W [6–8]. These stresses are relieved through deformation of the lattice and the creation of vacancies, dislocations or voids, which then represent hydrogen trapping sites. There are indications that this trap production mechanism is dependent on the incident ion flux density [8,9] but the relationship and how it extrapolates to ITER-relevant flux densities is not clear.

The purpose of this study is to expose poly-crystalline tungsten samples to plasma flux densities and energies that are expected at or near the ITER divertor strike points. This allows the hydrogenic retention at these high flux densities ($\sim 10^{24}$ D/m^{2}s) to be observed and measured experimentally rather than relying on extrapolations from ion beam or low-density plasma experiments.

2. Experiment

W targets were exposed to ITER divertor relevant deuterium plasmas in the linear plasma device Pilot-PSI. The Pilot-PSI experiment uses a cascaded arc to produce high plasma densities ($\leqslant 10^{21}$ m^{-3}) at low electron temperatures ($T_e \leqslant 5$ eV) [10,11]. The plasma is confined to a column of ~15 mm diameter with the highest densities and temperatures located at the center of the column. The plasma electron density and temperatures are measured with Thomson scattering [12]. For these experiments, the central

* Corresponding author.
E-mail addresses: wright@rijnhuizen.nl (G.M. Wright), kleyn@rijnhuizen.nl (A.W. Kleyn).
URLs: http://www.rijnhuizen.nl (G.M. Wright), http://wwwchem.leidenuniv.nl (A.W. Kleyn), http://www.itn.pt (E. Alves), http://www.tue.nl (N.J. Lopes Cardozo).

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electron density was $n_e = (2.0 \pm 0.2) \times 10^{20}$ m^{-3} with a central electron temperature of $T_e = 2.0 \pm 0.2$ eV corresponding to a deuterium flux of $\sim 2 \times 10^{24}$ D/m^{2}s at the center of the target. These are conditions similar to those expected at the strike points of a detached ITER divertor. A 1-D spatial scan of the electron density and temperature across the column width can be seen in Fig. 1(a) and (b). A magnetic field of 0.8 T was used to confine the plasma and each plasma shot ran for a maximum of 20 s. For targets with exposure times >20 s, multiple sequential and near-identical (n_e and T_e within 10%) 20 s shots were used. The W targets were electrically grounded for the exposures.

The tungsten targets are mechanically clamped to an actively-cooled copper heat sink. A molybdenum clamping ring is used to secure the target and a 0.5 mm thick piece of grafoil is placed between the heat sink and the tungsten target to improve thermal contact. The surface temperature has been measured with a vis-IR spectrometer (600–950 nm) and a single-colour pyrometer. Both measurements have been calibrated ex-situ and combined to approximate a 1-D surface temperature profile of the target (Fig. 1(c)). Typical central surface temperatures are ~1600 K and decreasing to ~1000 K near the edges of the target.

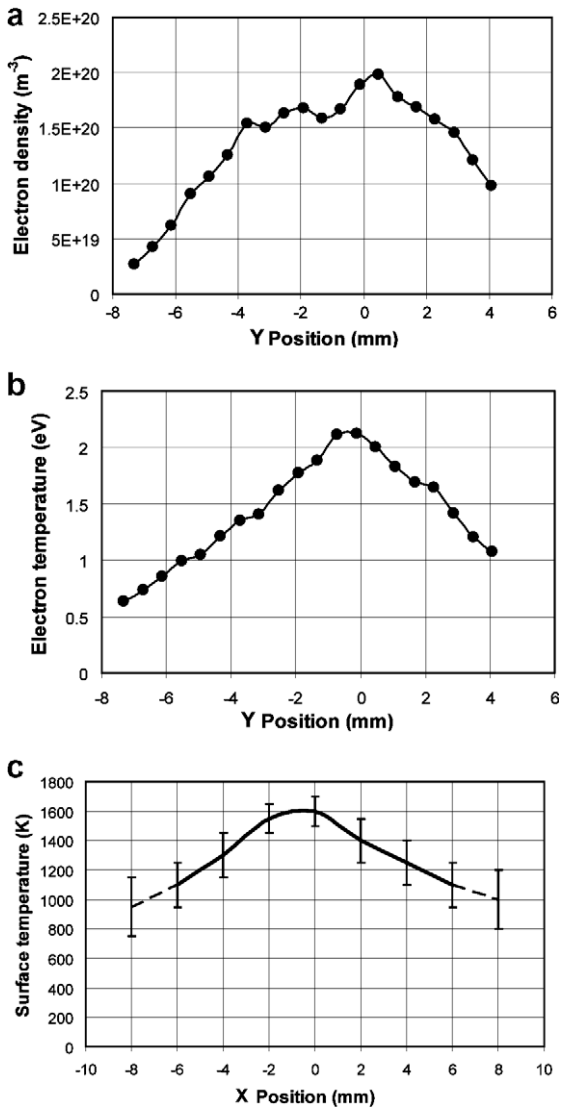

Fig. 1. (a) Electron density and (b) electron temperature from a typical Thomson scattering profile in the vertical (Y) direction for the Pilot-PSI plasma column (c) The surface temperature as a function of horizontal (X) position. The dashed lines represent extrapolations.

The targets are 99.97% pure polycrystalline tungsten purchased from Plansee metals. The targets are formed by laser-cutting 1 mm thick discs from a 20 mm diameter rod. They are exposed in the "as received" condition (rough surface, unannealed) however samples that have been mechanically polished to a surface roughness of <0.5 μm and/or annealed at 1273 K for 30 min have also been exposed for comparison purposes.

The hydrogenic retention in the exposed samples was determined with ex-situ ion beam analysis and thermal desorption spectroscopy (TDS). Each W target was initially probed with ion beam analysis and then was thermally desorbed. For the ion beam analysis, 2.0 MeV ^{3}He ions were used for nuclear reaction analysis (NRA) exploiting the ^{3}He(d,p)α nuclear reaction. The ^{3}He ion beam was perpendicular to the surface yielding a probing depth of ~3 μm. The detected proton energy spectrum was translated into a deuterium content in the first 3 μm using the NDF code [13]. The TDS is performed by clamping the W target to a ceramic heater and linearly ramping the temperature to 1273 K at a ramp rate of ~1 K/s. The temperature is monitored by a type K thermocouple spot welded to the surface of the W target. A Balzers QMA125 quadrupole mass spectrometer (QMS) monitors the mass 3 (HD) and mass 4 (D_2) signals as a function of time (and thus temperature since the temperature ramp is linear). The QMS HD and D_2 sensitivity is quantified with calibrated leaks of D_2 and H_2 (the HD sensitivity is assumed to be the average of the D_2 and H_2 sensitivities). This allows the integrated mass 3 and mass 4 signals to be converted to the total number of D atoms desorbed from the target.

3. Results and discussion

The NRA technique has the advantage of measuring the local concentration of D within the beam spot (~1 mm diameter) but only to the depth of the ^{3}He ion range. The NRA results were taken from various points on the target surface and each point corresponds to a different set of plasma parameters and surface temperature (see Fig. 1). The D retention is measured as a function of plasma exposure time which can be converted to fluence as a function of position through the Thomson scattering profile. In Fig. 2 the areal D retention (D/cm^2) in the first 3 μm of the W surface is plotted as a function of plasma exposure time for the center of the target (0 mm) and a point 6 mm off center.

The measured retention at the center point is very low ($\leqslant 5 \times 10^{14}$ D/cm^2 in first 3 μm) despite the high plasma flux density received in that region. The 6 mm off-center point has approx-

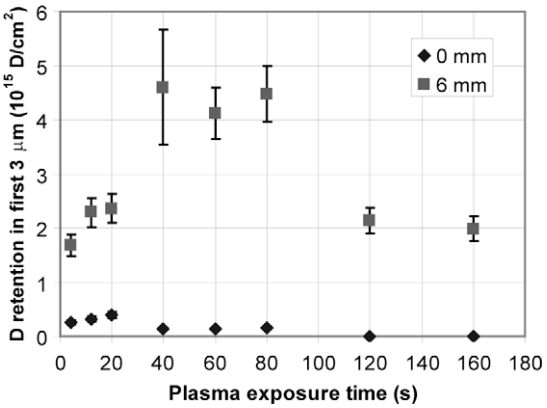

Fig. 2. D retention as determined by NRA as a function of plasma exposure time for the center of the target ($T_{surf} \sim 1600$ K, $\sim 10^{24}$ D/m^{2}s) and a point 6 mm off-center ($T_{surf} \sim 1100$ K, $\sim 10^{23}$ D/m^{2}s). Total incident fluences can be calculated for each point with using appropriate plasma flux density and plasma exposure time.

imately an order of magnitude higher retention ($\leqslant 5 \times 10^{15}$ D/cm^2 in first 3 μm) despite being in a region exposed to lower flux densities ($\sim 3 \times 10^{23}$ D/m^{2}s at 6 mm off center) and fluences. The key parameter that appears to be causing the difference in retention at these two locations is the surface temperature. At the center point the surface temperature is ~1600 K and at 6 mm off-center the temperature is ~1100 K (see Fig. 1). It is well known that surface temperature plays a role in the hydrogenic retention properties of W [3,4,8], but it is important to note that under these plasma and surface conditions it plays a much stronger role than either plasma flux density or fluence.

A 2-D NRA scan (see Fig. 3) shows that the D retention profile is asymmetric. This is not surprising considering the surface temperature profile is also asymmetric (see Fig. 1). Unfortunately since the alignment of the target was not preserved from exposure to analysis chambers, it is impossible to see if these asymmetries align with one another. However, Fig. 3 also confirms that the D retention increases as the NRA beam spot is moved away from the hot center to the cooler edges of the W target. It should be noted that measurements at 8 mm off-center are at the point where the molybdenum clamping ring overlaps with the W target, so shadowing effects from the molybdenum ring could be responsible for the low retention measurements at some of the 8 mm off-center points.

Thermal desorption spectroscopy has the advantage of detecting all trapped D from the bulk and surface of the W target. Unfortunately, it desorbs from all locations on the surface simultaneously meaning the spatial origin of the desorbed D atoms are not known. The TDS spectra show HD and D_2 desorption peaks at 600 ± 20 K and 1000 ± 25 K. This likely indicates the presence of both low energy (e.g. vacancies, dislocations) and high energy (e.g. voids, vacancy clusters) trap sites. Fig. 4(a) shows the total retained D in the W target as a function of total incident D fluence. The incident fluence is calculated based on an integration of the local flux density as determined by the Thomson scattering profiles (Fig. 1). In Fig. 4(a) there are also results from a mechanically polished target, an annealed target (30 min at 1273 K), and a polished then annealed target. These three targets were exposed to a fluence of $\sim 9 \times 10^{21}$ D ions. In Fig. 4(b), the retained fraction ($D_{retained}/D_{incident}$) is shown as a function of incident D fluence.

Three conclusions can be made from Fig. 4. First, the D retention has no apparent dependence on the incident plasma fluence as

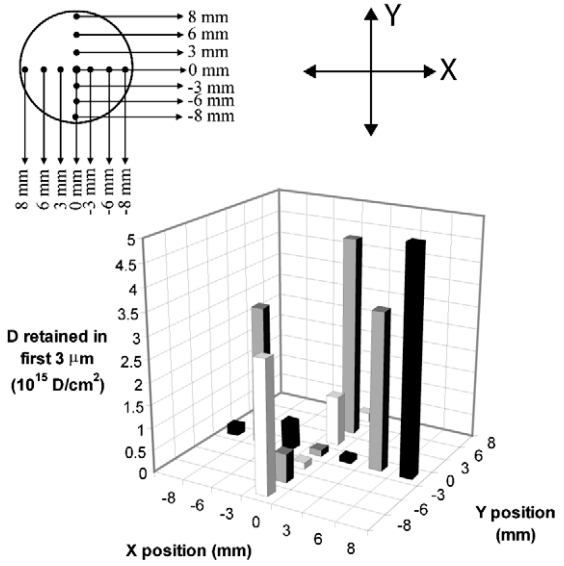

Fig. 3. A 2-D NRA spatial scan of the W target with 80 s plasma exposure time. The NRA beam spot size is ~1 mm diameter.

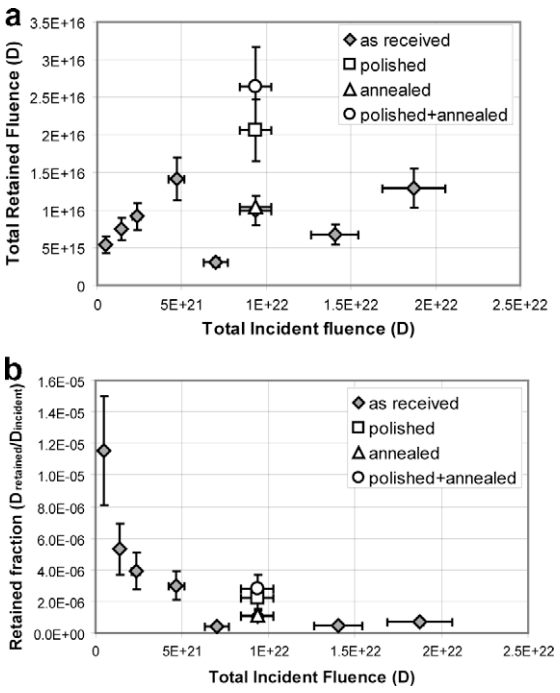

Fig. 4. TDS is used to determine (a) The retained D in W targets as a function of incident D ion fluence and (b) the retained fraction of D as a function of incident fluence.

seen by the scatter in measured retention values in Fig. 4(a). The rapid decrease of the retained fraction after an incident fluence of $\sim 2.3 \times 10^{21}$ D ions (~4 s of plasma exposure) seen in Fig. 4(b) may indicate that the W has become saturated and can retain no more deuterium. Second, the overall D retention in the W is low (retained fraction 10^{-7}–10^{-5}), supporting the NRA results which also showed very low concentrations ($\leqslant 5 \times 10^{15}$ D/cm^2 in first 3 μm) of D in the W targets. Finally, mechanical polishing of the W prior to plasma exposure increases the retention by a factor of ~2, but annealing at 1273 K for 30 min has no effect on the retention properties of the tungsten. The mechanical polishing likely creates additional trap sites (i.e. dislocations, vacancies, etc.) in the near surface area due to the physical distortion of the lattice, so it is not unexpected that this would increase the retention of the tungsten. Annealing has previously been shown to reduce hydrogenic retention in W [14,15]. At high surface temperatures, it is likely that the inherent defects in the W that have an impact on the retention are more macroscopic defects, such as voids or vacancy clusters, since these types of defect have a high trap energy. However, these types of defect do not begin to anneal out of the lattice until ~1700 K [15]. It could also be that the anneal temperature is not long enough in duration to have a large effect.

It should be mentioned that the central surface temperature for the plasma exposures was greater than the maximum achievable temperature in the thermal desorption chamber. It is possible that there is strongly trapped D in the W that can not be desorbed at temperatures $\leqslant$1273 K, although there are no indications of this type of trapping in literature. However, this strongly trapped D, if it is present, is still detectable with NRA measurements. Since there is little D detected at the center of the targets ($T_{surf} \sim 1600$ K) it seems unlikely that there is significant amounts of very strongly trapped D. A comparison of the total D retention as calculated by an integration of the 2-D spatial scan shown in Fig. 3 and the TDS results from the same target ($\sim 9 \times 10^{21}$ $D_{incident}$, 80 s plasma exposure time) shows the number of retained D atoms detected by TDS is a factor of ~2 higher than for the NRA results. This is likely an indication that the D retention extends further than the

3 μm probing limit of the NRA measurements and is also a counter result to the presence of strongly trapped D.

4. Conclusions

W targets have been exposed in the Pilot-PSI experiment to the plasma conditions expected at the strike point of a detached ITER divertor ($\sim 10^{24}$ D/m^{2}s, $T_e \sim 2$ eV). The surface temperature of the target was ~1600 K at the center and decreased radially to ~1000 K at the edges. NRA and TDS are used to measure the retained D in the exposed targets.

The main indication from these results is that W targets retain very little D when compared to the amount of D incident to the surface ($D_{retained}/D_{incident} \sim 10^{-7}$–$10^{-5}$). These preliminary results show very little dependence of retention on incident fluence possibly indicating saturation at very low levels of D retention. It is concluded that the plasma exposure itself has little influence on the trap concentration in the W and the scatter in retention data seen in Figs. 2 and 4 is due to scatter in the population of natural or inherent defects and trap sites present in the W due to fabrication and machining. This is supported by the observation that mechanically polished W targets (additional defects created through polishing process) have higher retention than "as received" samples. However, for these plasma exposures, it appears that annealing the W at 1273 K for 30 min has no effect on the overall retention, perhaps indicating the inherent defects dictating the retention properties of the W are difficult to anneal (i.e. voids or vacancy clusters). It is concluded that the high surface temperature of the W during plasma exposure has eliminated or neutralized the stresses formed in the W lattice due to the implantation of a high flux of low energy D ions into the surface.

The results from this study indicate that, when operating at a surface temperature of 1000–1600 K, the strike points of a W ITER divertor will not retain significant amounts of deuterium (or tritium) due to the bombardment of the surface by the high flux of low energy plasma hydrogenic ions. However retention at lower surface temperatures could be significant and this is a focus for future work.

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References

[1] R. Aymar, Fusion Eng. Des. 49–50 (2000).
[2] G. Janeschitz, P. Barabaschi, G. Federici, K. Ioki, P. Ladd, V. Mukhovatov, M. Sugihara, R. Tivey, ITER-JCT team, Nucl. Fusion 40 (2000) 1197.
[3] A.A. Haasz, J.W. Davis, M. Poon, R.G. Macaulay-Newcombe, J. Nucl. Mater. 258–263 (1998) 889.
[4] T. Venhaus, R. Causey, R. Doerner, T. Abeln, J. Nucl. Mater. 290–293 (2001) 505.
[5] R. Causey, K. Wilson, T. Venhaus, W.R. Wampler, J. Nucl. Mater. 266–269 (1999) 467.
[6] V.Kh. Alimov, K. Ertl, J. Roth, J. Nucl. Mater. 290-293 (2001) 293.
[7] M. Poon, R.G. Macaulay-Newcombe, J.W. Davis, A.A. Haasz, J. Nucl. Mater. 307–311 (2002) 723.
[8] O.V. Ogorodnikova, J. Roth, M. Mayer, J. Appl. Phys. 103 (2008) 034902.
[9] G.M. Wright, D.G. Whyte, B. Lipschultz, in these proceedings.
[10] G.J. van Rooij, V.P. Veremiyenko, W.J. Goedheer, et al., Appl. Phys. Lett. 90 (2007) 121501.
[11] J. Westerhout, W.R. Koppers, et al., Phys. Scripta T128 (2007) 18.
[12] H.J. van der Meiden, R.S. Al, et al., Rev. Scientific Inst. 79 (2008) 013505.
[13] N.P. Barradas, R. Smith, J. Phys. D: Appl. Phys. 32 (1999) 2964.
[14] R.A. Anderl, D.F. Holland, et al., Fus. Technol. 21 (1992) 745.
[15] H. Eleveld, A. van Veen, J. Nucl. Mater. 212–215 (1994) 1421.