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Thermal desorption of hydrogen isotopes from the JET Be plasma facing components

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Abstract

Understanding of fuel retention and release processes from materials for ITER (International Thermonuclear Experimental Reactor) is important from fundamental and technological aspects. Detailed information about fuel retention and release characteristics will allow global fuel inventories to be estimated in fusion devices as well as indicate the requirements for the development detritiation methods and re-use of tritium.

Selected beryllium (Be) tiles were extracted from the JET vacuum vessel after each experimental campaign period with the ITER-like wall (ILW); so called ILW1 (2011-2012), ILW2 (2013-2014) and ILW3 (2015-2016). Desorption of hydrogen isotopes of samples taken from inner wall guard limiter (IWGL), outer poloidal limiter (OPL) and dump plate (DP) tiles were analysed by means of thermal desorption spectrometry (TDS).

The results presented compare data across ILW1, ILW2 and ILW3 and show the long term trends of fuel retention in Be limiter tiles. For all three campaigns the level of retention correlates with erosion and deposition that takes place during plasma operations

Deuterium concentrations vary from $0.01-1 \times 10^{18}$ atoms/cm². Deuterium release takes place in several stages, related to different types of the traps in the tile surface, with the main release stages around 700, 760, 850 and 900 and 1020 K.

Keywords: hydrogen isotopes, JET, thermal desorption, beryllium

1. Introduction

The Joint European Torus (JET) tests materials for the International Thermonuclear Experimental Reactor (ITER). Accordingly, the ITER-like wall (ILW) project provides the same plasma facing materials that will be used in ITER. Before the start of the experimental campaign of 2011–2012,

the wall and the divertor of JET were changed and the ILW was installed. Bulk beryllium on Inconel carriers were used for the inner and outer limiters, the divertor surfaces were faced with tungsten (W) [1]. For erosion-deposition studies marker coatings have been developed [2].

Performance of the materials is limited by surface erosion [3] and fusion fuel retention [4]. Accumulation of hydrogen isotopes in the plasma facing materials can occur as result of

implantation in plasma facing materials as well as due to deposition with eroded particles. The erosion-deposition studies have shown dark deposition patterns on the left and right ends of the IWGL tiles but not on the central part of the tile [5].

Temperature programmed desorption studies are applied for determination of release of the tritium [6] and deuterium [7] from the plasma facing components.

This paper aims to estimate the release of hydrogen isotopes from inner guard wall limiter beryllium tiles (IWGL) from ILW1 (2010-2012), ILW2 (2012-2014) and ILW3 (2015-2016) campaigns in order to estimate fuel retention in different zones of the limiter tiles.

2. Experimental

Selected beryllium (Be) tiles were extracted from the JET vacuum vessel after each ILW experimental campaign period. Both ILW1 and ILW2 lasted for about 19.5 h of plasma discharges including approx. 13 h of limiter and 6.5 h of X-point operation. There were, however, differences in the total input energy (ohmic, neutral beams, ion cyclotron, lower hybrid): 150.6 GJ in ILW1 and 200.5 GJ in ILW2 [8].

In post-mortem studies 12x12mm samples were cut from the castellated Be tiles [9]. Desorption of hydrogen isotopes of samples taken from inner wall guard limiter (IWGL) tiles were analyzed by means of thermal desorption spectrometry (TDS).

Annealing was performed in vacuum with a heating rate 10 K/min up to 1050 K with a dwell time of 2 hours at the maximum temperature. In the TDS experiments it was found that Be evaporation takes place already at ~ 1070 K, so the annealing was limited to 1050 K [7]. The release of hydrogen isotopes during annealing was measured with a quadrupole mass spectrometer, Hiden Analytical Ltd (model 640100). A sample preparation method described previously [7] was slightly modified and applied.

For calibration of deuterium release, D_2 and H_2 bottles with a calibrated leak valve were used. Calibration factors for D_2 , HD and H_2 were calculated assuming the leak rate is proportional to desorption rate, giving a desorption factor for HD of $5.4 \cdot 10^8$ molecules/count and for D_2 $6.7 \cdot 10^8$ molecules/count. For characterization of tritium release several 'standard' Inconel samples were cut from MkIIA divertor components that had been present in JET during the deuterium-tritium experiment DTE1 [10]. The size of samples were $\sim 12 \times 12$ mm, weight 2 g. Tritium activity was estimated with a pyrolysis method followed by liquid scintillation. Tritium concentration values were applied as 'references' for TDS measurements. Knowing both the mass and size of the sample it is possible to convert tritium activity from Bq/g to atoms/cm² using the tritium decay constant $\lambda = 1.78 \cdot 10^{-9}$ s⁻¹ [11].

3. Results and discussion

The results presented compare data across ILW1, ILW2 and ILW3 and show the long term trends of fuel retention in Be limiter tiles. For all three campaigns the level of retention is correlated with the erosion and deposition that takes place during plasma operations.

This is most noticeable for the midplane IWGL tile, which has two main zones – an erosion zone in the central part of tile and deposition zones at each end of the tile where minimal plasma interaction occurs, Fig.1. Deuterium and tritium levels are higher in the deposition areas than in the erosion area, and similarly for the tritium content, indicating co-deposition with plasma impurities [12]. For ILW2 there is also a tendency for both deuterium and tritium concentrations to be higher on the left hand side of tile than on the right hand side. Following ILW3 the single measurement point in the erosion zone appears anomalous (triangles in Fig.1), but further measurements are in hand.

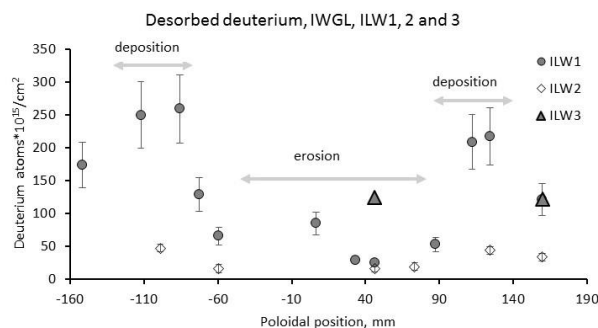


Fig.1. Thermally released deuterium atoms from IWGL tile samples, ILW1, 2 and 3.

The amount of thermally released deuterium is also found to be lower at the inner midplane limiter and dump plate compared with the outer midplane limiter which experiences lower heat flux during operations. Deuterium concentrations vary from $0.01 - 1 \times 10^{18}$ atoms/cm² for the limiter tiles.

3.1 Deuterium release profiles

Deuterium release takes place in the form of HD molecules (mass 3) and D_2 molecules (mass 4). The source of hydrogen for the formation of H may be due to the presence of hydrogen in the TDS analysis chamber or the presence of hydrogen in the sample originating from plasma operations and exposure in air [7]. During one thermal annealing up to 99% deuterium is released. $\sim 1\%$ D_2 remains bonded and can be released during repeated heating of the Be sample, around 970 K. Deuterium release profile for an IWGL tile 1st and 2nd heating is in Fig.2.

D_2 release starts mainly above 500 K. Thermal release takes place in several stages, related with different types of the traps in the tile surface. The main release stages are around 700 K, 760 K. This could be due detrapping from ion induced sites, that involves detrapping to a mobile state, diffusion through

Be bulk lattice, recombination to molecular D_2 at the surface and finally desorption into vacuum [13], 850 K and 900 K.

Above 1020 K, increase of mass 9 (Be) occurs. It is possible, that in the vacuum conditions, in temperature range 1020-1070 K changes in beryllium structure together with evaporation of beryllium allows trace deuterium release from deeper layers.

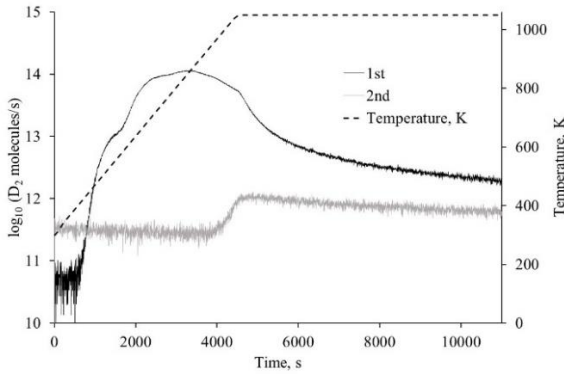


Fig.2. Deuterium release from IWGL ILW3 tile deposition zone.

Maximal deuterium release rate is reached around 840 K. The D_2 release profile from an IWGL tile is in Fig.3. Comparing deuterium release from limiter and divertor materials under the same conditions, deuterium release reaches its maximum at higher temperatures for the Be than from divertor materials [14, 15].

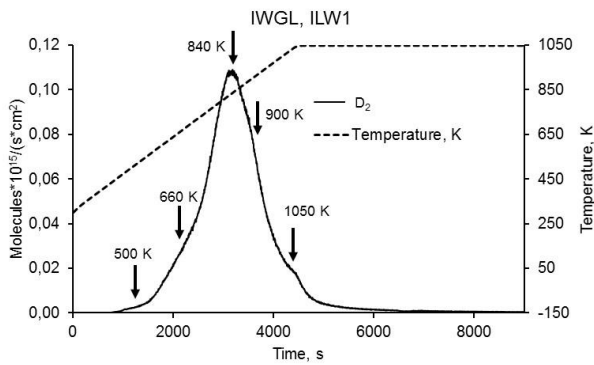


Fig.3. Deuterium release from IWGL ILW1 deposition zone

There are significant differences in the D release between ILW2 and the others (ILW1 and ILW3). Firstly, maximal release rate for IWGL and DP tiles is at higher temperatures in ILW2 in comparison with ILW1 and 3: With heating rate 10 K/min, in the TDS device the maximal D_2 release rate is reached at 835 ± 26 K (ILW1), 904 ± 23 K (ILW2) and 810 ± 99 K (ILW). Furthermore, although the HD release follows the shape of the D_2 release for all three ILW campaigns, the integrated intensity ratios (released molecules, peak area) are different. The intensity ratios for HD and D_2 signals for ILW

samples from same poloidal positions but different campaigns are summarized in table 1, and there is an increase in HD concentration for ILW2 samples in comparison with D_2 concentration for ILW1 and ILW3 samples. It can also be seen in Fig.4.

The difference in D_2 release behavior for ILW2 could be related to the use of H_2 fuelled pulses at the end of the ILW2 campaign, when the last 300 pulses in the campaign were in hydrogen [16]. During the H_2 fuelled phase D would be replaced by H by isotope exchange, especially from the lowest binding energy states, resulting in both the change in HD/DD ratio and also the net shift to higher release temperatures. Higher absorbed energies in ILW2 compared to ILW1 cannot be responsible, since energies in ILW3 were even greater.

Table 1

HD/ D_2 intensity ratios for similar poloidal positions,

| Be tile | ILW1, 2 and 3 | | |
|----------------------|---------------|------|------|
| | ILW1 | ILW2 | ILW3 |
| IWGL deposition area | 0.67 | 2.20 | 0.50 |
| IWGL erosion area | 0.67 | 1.24 | 0.64 |
| DP | 0.70 | 1.68 | 0.43 |
| DP | 0.71 | 1.63 | 0.48 |

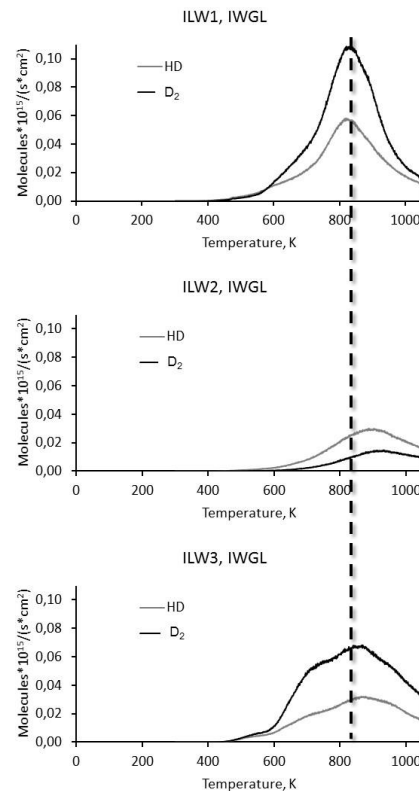


Fig.4. D_2 and HD release from IWGL ILW1, 2 and 3 deposition zone

3.2 Tritium release quantification

Low levels of tritium are also released during analysis. As reference material for tritium release characterization, Inconel samples from a MkIIA divertor component (present in the JET vacuum vessel during DTE1) were selected. The total tritium amount was determined by pyrolysis, followed by liquid scintillation method, and was estimated as 82 ± 16 kBq/g ($4.6 \times 10^{13} \pm 0.9 \times 10^{12}$ T atoms/cm²). The samples from the same parts of the carrier were analysed by TDS and tritium release in DT and T₂ was registered. A fraction of tritium is released in the form of DT, whilst the T₂ signal is low. Tritium release can also take place in the form of HT or tritium ions T⁺, however these signals cannot be separate from the larger signals of D₂ and HD at the same masses. The amount of tritium released from the Inconel samples is calculated using release of DT molecules. Formation of tritiated water molecules is possible as well, a mass 20 release (HTO) is observed in the TDS spectrum, however in the case of JET samples, mass 20 could also be from D₂O. Due to tritium release in different chemical forms, concentrations estimated by TDS are lower than pyrolysis results. The TDS results for tritium content in Inconel samples after heating to 1270 K, using the HD calibration factor the DT release is around 17 kBq/g (10×10^{12} T atoms/cm²). Part of the tritium is remaining in the Inconel. After TDS analysis samples were pyrolysed and a further 8 kBq/g (3×10^{12} T atoms/cm²) were detected. As mentioned, the fraction of T released in the forms DT and T can be detected by TDS, but atomic T (mass 3), and tritium released in the form of HTO, DTO, OT and others will have been missed. Judging by the pyrolysis after TDS, heating up to 1270 K releases 90% of the tritium.

TDS can be used for semi-quantitative estimation of tritium content in JET samples. However, for analysis of tritium release profiles release characteristics of tritium from particular types of samples and possible release together with D, H and O (in form of ions and molecules) need to be taken into account.

The results of analyzing Be samples from ILW campaigns indicate a level $\sim 10^4$ times lower than that of deuterium. Tritium can be from DD reactions and also due to off-gassing from previous DT operations in JET.

Conclusions

For all three campaigns the level of retention correlates spatially with erosion and deposition that takes place during plasma operations. The majority of deuterium is released during heating of beryllium up to 1050 K. $\sim 1\%$ of deuterium remains bonded. Deuterium release takes place during several stages, with the highest release rate in the temperature range 800-900 K. Typical deuterium concentrations are 0.01-1 x 10¹⁸ atoms/cm².

With the existing TDS setup the detection of tritium in fusion materials is possible and it is possible to estimate the DT release. The level of tritium in Be (released in form of DT and T₂) is 10⁴ times lower than deuterium. However, significant fractions of the tritium are missed in the presence of the deuterium. For precise estimation of tritium content in the plasma exposed Be plasma facing components, inter-comparison of results obtained by different techniques is necessary and modelling is planned.

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