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# Thermal Desorption Spectrometry for the study of hydrogen retention in the JET ITER-Like Wall

A Baron-Wiechec<sup>1\*</sup>, J P Coad<sup>1</sup>, K. Heinola<sup>2</sup>, J Likonen<sup>3</sup>, G F Matthews<sup>1</sup>, A Widdowson<sup>1</sup> and JET contributors<sup>\*\*</sup>

EUROFusion Consortium, JET, Culham Science Centre, OX14 3DB, Abingdon, UK

<sup>1</sup>CCFE, Culham Centre for Fusion Energy, Culham Science Centre, Abingdon, OX14 3DB, UK

<sup>2</sup>University of Helsinki, PO Box 64, FIN-00560 University of Helsinki, Finland

<sup>3</sup>VTT Technical Research Centre of Finland, PO Box 1000, FIN-02044 VTT, Finland

\* aleksandra.baron-wiechec@ccfe.ac.uk

\*\* See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference 2014, Saint Petersburg, Russia

## Abstract

A new Thermal Desorption Spectrometry (TDS) System is described, and examples are given of its application to beryllium (Be) wall tiles and W-coated CFC divertor tiles after exposure in JET for the 2011-2012 ILW campaigns. The temperature of the Be samples had to be limited to 650°C in order to prevent coating the internal components of the TDS vacuum system with evaporating Be. The temperature at which maximum D release rate (both as D<sub>2</sub> and HD) from Be samples occurs was at 485°C (compared to the peaks in temperature release rate from Be co-deposits in the divertor of 312°C, 416°C and 578°C). Tritium (T) release has been observed at mass 6 (T<sub>2</sub> molecules) and a smaller release of DT molecules at mass 5.

Keywords: TDS, Tritium, Beryllium, JET

PACS: 52.40.Hf, 52.55.Fa, 52.77.Dq, 82.80.Ms

## 1. Introduction

The interactions of the plasma with the surrounding surfaces will be of increasing importance for larger future fusion devices such as ITER, DEMO and commercial machines. Erosion and deposition will have to be controlled, and fuel retention within the device will need to be limited to preserve the tritium fuel cycle. These issues have been studied over many years at JET by post-mortem analysis of plasma-facing components (PFC) using Ion Beam Analysis (IBA) and Secondary Ion Mass Spectrometry (SIMS) [1-4], and by gas balance [5]. All these methods have limitations in estimating the total long-term fuel retention. Now an additional technique has been added to the armoury: Thermal Desorption Spectrometry (TDS). A large number of laboratory experiments have been dedicated to the loading, retention and desorption mechanism of H isotopes co-deposits with beryllium, e.g [6 and references there, 7, 8]. However the JET samples are more complex and contain impurities (C, O, Ni and W on Be tiles ) which may change the release characteristics [6]. The TDS system is being used to measure the amount of the plasma fuelling gas (deuterium - D) and fusion-generated tritium (T) that is trapped in plasma-facing components (PFC) of JET during operations with the ITER-like Wall (JET-ILW) and is a crucial part of experiments to determine the total amount of fuel that is retained during operations. Until 2009 JET operated with carbon PFC, but in 2010-2011 these PFC were replaced with Be tiles and Be-coated Inconel tiles in the main chamber, and W-coated CFC tiles (plus one row of solid W tiles) in the divertor; this configuration mirrors the planned ITER PFC. Samples cut from W-coated CFC divertor tiles have been analysed by TDS, and results are reported in a companion paper [9]. This paper reports the first results from samples of the Be wall tiles, following development of techniques to cut samples from the Be tiles reported in [10].

## 2. Experimental method

The Thermal Desorption Spectrometry (TDS) System is manufactured by Hiden Analytical Ltd (TPD Workstation type 640100). It comprises a stainless steel vacuum chamber fitted with a quadrupole mass spectrometer, a sample heater and a load/lock system, mounted on a single support frame.

The main chamber is pumped by a turbo-pump backed by an Edwards XDS dry pump and is capable of routine UHV ( $<10^{-8}$  mbar) operation following occasional baking to 200°C. A Fast Sample Transfer System from a sample load lock that is separately pumped and linked to the analysis chamber via a UHV gate valve allows the analysis chamber vacuum to be easily maintained: the sample transfer system is used to place samples onto the sample heater (**Figure 1**). The heated sample stage is 20 mm diameter and can handle samples to a thickness of 10 mm and is capable of heating a sample from room temperature to at least 1000° C with control software that allows heater temperature to be fully programmed for ramp rates, hold patterns etc. For our experiment we have applied a linear rump rate of 10K/min from room temperature.

The system is fitted with a Hiden Analytical HAL/3F RC 1051-9 PIC mass spectrometer with a Burle 4871H channeltron electron multiplier mounted on the vacuum chamber at line-of-sight orientation for direct detection of species desorbed from the sample. The system has been tuned to allow mass separation of the He and D<sub>2</sub> peaks at approximately 4 atomic mass units (amu). The mass spectrometer can be programmed for repeated mass scans (in the range 1-50 amu) or for trend mass scans as a function of time for a number of selected masses (up to 16 different masses). Values of electron emission and ion and electron energies can be individually optimised for each selected mass.

The control unit and software allows remote control of all functions once a sample is in position, such as simultaneously plotting the signal from a number of masses against heater temperature, or against sample surface temperature provided from the infrared thermometer (RAYTEK model RAYMM2MLSF1L). This is important for the analysis of JET samples which are Be- and T-contaminated, as the TDS chamber is located within a controlled area whilst the operator is outside. The data processing package includes peak de-convolution, background subtraction and peak integration.

Be has a high vapour pressure at elevated temperatures, and it is necessary to limit the amount of Be release to avoid unduly contaminating the mass spectrometer (and chamber in general). **Figure 2** shows TDS spectra of Be (mass 9) against temperature from sample of bulk pure Be prepared from a castellation not exposed to a plasma, a sample from the top of divertor tile 1 which is a region with a plasma-deposited layer of Be on the W-coated CFC tile [9] and IWGL centre tile. The onset of Be evaporation occurred at the same temperature for bulk Be (i) and Be deposit in the divertor sample (ii) as shown in **Figure 2**. The Be signal starts to increase at ~700°C for both. Since this emission was rising alarmingly and would doubtless have continued to unacceptable levels, the heater was switched off. Then for the divertor sample reaches a maximum and continues at a high level until the heating is stopped and the sample allowed to cool (ii). For the experiments on JET Be tile samples it was decided to limit the surface temperature to 650°C (iii). At the same time as the emission at mass 9 there is very small emission at mass 11, indicating that BeD molecules are detected; whether these form in the gas phase or are emitted from the surface is unknown.

It should be pointed out that there is a difference between the heater temperature and the surface temperature recorded by the pyrometer (which is set for an emissivity of 1.0, as the true value is unknown). This particular pyrometer does not record temperatures below 300°C, is close to the heater temperature at 300°C, but then the pyrometer temperature reads increasingly lower than the heater to higher temperatures. It is logical for there to be an increasing discrepancy as radiation from the surface increases as (temperature), so the gas release is plotted against pyrometer temperature above 300°C and a linear extrapolation from 300°C down to room temperature.

At each JET shutdown a poloidal selection of tiles are removed from the JET vessel for analysis to investigate material erosion and deposition, and fuel retention. From the JET-ILW main chamber, examples of Inner Wall Guard Limiter (IWGL), Outer Poloidal Limiter (OPL) and Dump Plate (DP) were removed, and from these samples have been prepared for TDS analysis. Each IWGL and OPL tile comprises a number of Be pieces fastened onto an Inconel backing plate (5 pieces and 7 pieces for the IWGL and OPL, respectively). A photograph of an IWGL tile is shown on the right of **Figure 3**, and it can be seen that there are a series of cuts into the surface in both toroidal and poloidal directions. These cuts, which are about 15 mm deep, serve both to limit the internal stress that can build up in the Be on thermal cycling and to increase the resistivity of the tile. Thus the tile surface comprises a number of small segments each  $\sim 12 \times 12$  mm and referred to as “castellations”. A single castellation is a suitable area for a number of analysis techniques such as SEM, SIMS and TDS, so rows of castellation have been cut from each Be piece of the tile for analysis as described in [10].

### 3. Results

A selection of 31 samples cut from an IWGL, a Dump Plate and an OPL tile exposed during the 2011-2 JET-ILW campaigns have been provided for analysis by TDS. Each sample is the surface region (2.5 mm thick) of a single castellation.

**Figure 3** shows TDS spectra of mass 3 (HD) and 4 ( $D_2$ ) release a tile at the IWGL mid-plane (2XR10) against temperature, together with an inset showing the positions of the castellations analysed. The photograph of the tile is orientated such that the toroidal (horizontal) axis of the tile is shown vertical in **Figure 3**. The central region of the tile is an area of net erosion: the marker layers have disappeared and tile profiling indicates that up to 60  $\mu\text{m}$  have been eroded. TDS spectrum obtained from the central region shows a single peak with a maximum  $D_2$  release at about 485°C. The TDS spectrum from the right-hand wing of the tile (an area of net deposition) is of similar magnitude, but with indications of small additional components above and below the main peak.

The numbers of D atoms released has been calibrated by comparison with the TDS of D-implanted W reference samples that have been quantified by K Mizohata, University of Helsinki, using Elastic Recoil Detection Analysis (ERDA) (error in measurement likely be  $<1\%$ ). The total number of D atoms released (per  $\text{cm}^2$ ) from the two samples in Figure 2 are thus  $3.7 \cdot 10^{17}$  and  $5.9 \cdot 10^{17}$  atoms  $\text{cm}^{-2}$  for the central and wing tiles, respectively, compared to  $1.26 \cdot 10^{19}$  atoms  $\text{cm}^{-2}$  from the divertor tile 1 sample [9].

**Figure 4a** shows the same  $D_2$  (mass 4) spectrum from the IWGL wing, deconvoluted using Origin<sup>®</sup> software. **Figure 4b** shows a  $D_2$  release spectrum from the region of Be deposition on top of divertor

tile 1 [9]. The main peak from the Be tile pieces is at  $\sim 485^\circ\text{C}$  and the deconvolution shows a subsidiary peak at  $\sim 331^\circ\text{C}$ . There is clearly also another peak forming above the main peak, however that  $\text{D}_2$  release has been truncated by the temperature reaching its maximum allowed value of  $650^\circ\text{C}$  so this peak has not been fitted. The  $\text{D}_2$  release from the tile 1 sample is well fitted using 3 peaks, It should be noted that the principle release from the Be sample ( $\sim 485^\circ\text{C}$ ) is at significantly higher temperature than the onset of release from the main divertor sample (peaks at  $\sim 312^\circ\text{C}$ ,  $\sim 416^\circ\text{C}$  and  $\sim 578^\circ\text{C}$ ). These peaks in the release spectra are related to the binding energies of D atoms within the material lattice, such as in traps associated with vacancies, dislocation loops, grain boundaries, etc., and are clearly different for Be surfaces than for Be deposits on a W-coated CFC surface. Careful evaluation of the spectra in future work should shed light on the retention mechanisms, with clear relevance to ITER.

Not all the D is released as  $\text{D}_2$ . There is a significant release of HD which is observed at mass 3. The release spectrum has a similar shape to that for  $\text{D}_2$  (**Figure 3**) as a function of temperature, and the D in the HD represents about 30% of the total amount of number of D atoms released. Other possible release for D could be as water vapour or hydrocarbons. We see a small mass 19 peak (HDO) from divertor samples which contribute no more than 5% to the overall D amount [9] but have not observed release as hydrocarbons. The contribution to the total D amount due to release in channels other than mass 4 may increase the error in total D to 10% where More detailed evaluation will be coming out.

TDS can also be used for the analysis of tritium (T). **Figure 5** includes release spectra for  $\text{T}_2$  (mass 6) from the same samples right hand wing, left hand wing and centre part of IWGL tile (as in **Figures 3**), and also from the same divertor sample. It should be noted that there is very much less  $\text{T}_2$  release from the Be samples than from the divertor sample. There is, however, a similar sharp release at  $\sim 100^\circ\text{C}$  and some release over the temperature range of the first major component of the release from the divertor sample ( $\sim 250^\circ\text{C}$ - $600^\circ\text{C}$ ), but as discussed in section 2 due to onset of significant Be evaporation, the Be samples were not heated to sufficient temperature to observe whether there is a second release component above  $\sim 700^\circ\text{C}$ . TDS measurements using heated vacuum quartz tube would be required to obtain full T content inventory and is planned as a part of JET work programme [10]. Since there are relatively few T atoms being released ( $\sim 2.6 \cdot 10^{13}$  atoms  $\text{cm}^{-2}$  from each Be sample at a rate of  $< 10^{10}$  atoms  $\text{cm}^{-2} \text{s}^{-1}$ ) compared to D or H, it might be expected that a T atom reaching the surface would have a high probability of combining with a D or H atom. In fact the T present in DT (mass 5) only accounts for  $\sim 20$ - $30\%$  of the total T and is mostly released in the range  $\sim 400$ - $550^\circ\text{C}$ . It is not possible to tell if there is an HT component at mass 4 since it would be impossible to distinguish it from the  $\text{D}_2$  signal.

#### 4. Conclusions

TDS has been used to analyse samples of Be wall tiles and W-coated CFC divertor tiles after exposure in JET for the 2011-2012 ILW campaigns. The temperature of the Be samples had to be limited to 650°C in order to prevent coating the internal components of the TDS vacuum system with evaporating Be. The temperature at which maximum D release rate (both as D<sub>2</sub> and HD) from Be samples occurs was at ~485°C (compared to the peaks in temperature release rate from Be co-deposits in the divertor of ~312°C, ~416°C and ~578°C). This compares with release rate from Be-D co-deposits of ~525 °C [6]. Tritium (T) release has been observed at mass 6 (T<sub>2</sub> molecules) and a smaller release of DT molecules at mass 5, though at very small amounts for Be samples up to the imposed temperature limit of 650°C.

## **5. Acknowledgements**

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**Figure captions**

Figure 1. Photograph into the TDS chamber during heating of a metal sample.

Figure 2. Be release from a bulk Be sample (i), a Be co-deposit on a W-coated CFC divertor tile 1 sample (ii), and Be release from the exposed central tile (iii). All are plotted against time, as are the appropriate temperatures: rise halted to prevent further evaporation for Be samples (a) and divertor samples temperature patterns (b).

Figure 3. TDS spectra ( $D_2$  and HD) from a Be tile from an Inner Wall Guard Limiter (IWGL) at the mid-plane of the main chamber. Note that the toroidal (horizontal) axis of the tile is shown to be vertical in the inset photograph. The spectra come from the specific castellations arrowed.

Figure 4. Deconvolution of the same  $D_2$  release spectrum as shown for the right hand wing sample in Figure 3 (a), deconvolution of the spectrum from a sample from a region of Be co-deposition on divertor tile 1 (b).

Figure 5. T release from the divertor tile 1 (a), and from the Be central (b), left hand wing (c) and left hand wing (d) tiles.

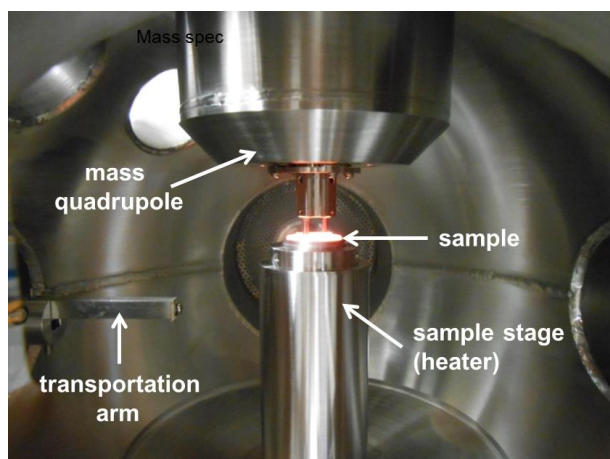


Figure 1

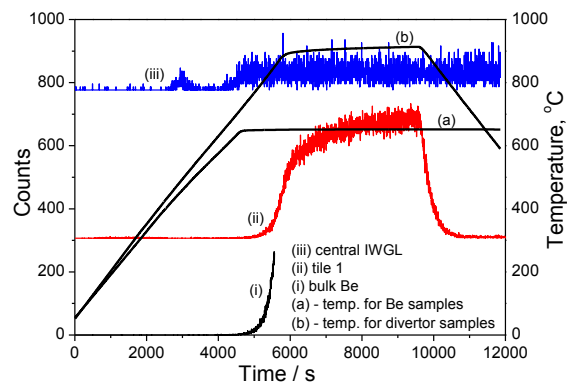


Figure 2

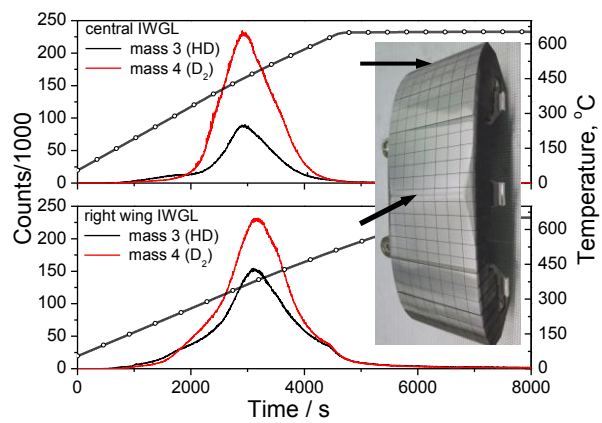


Figure 3

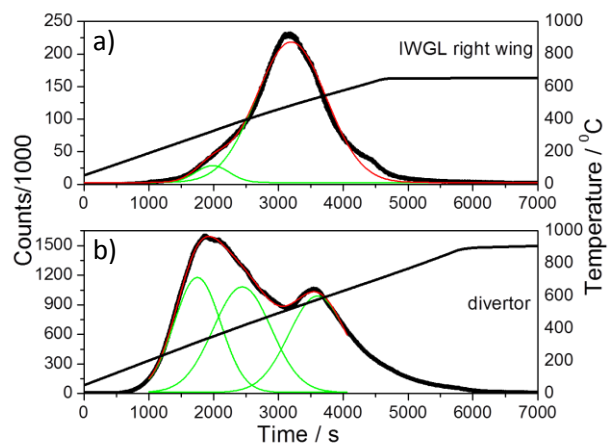


Figure 4

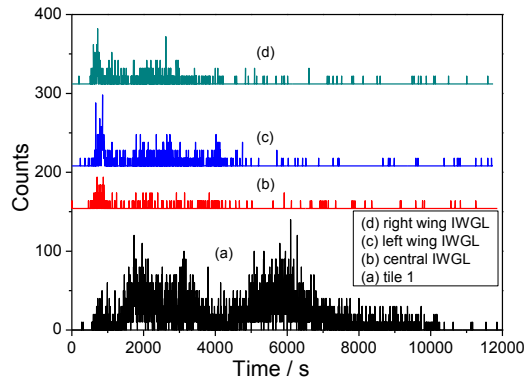


Figure 5