Thermal desorption analysis of beryllium tile pieces from JET

R.G. Macaulay-Newcombe [a,*], D.A. Thompson [a], J.P. Coad [b]

[a] *Department of Engineeering Physics, McMaster University, JHEA/325, Hamilton, Ont. L8S 4L7, Canada*
[b] *JET Joint Undertaking, Abingdon, UK*

Abstract

Pieces of beryllium tile exposed to a D–D plasma in JET have been studied by thermal desorption spectroscopy. These tiles have a thick layer of redeposited Be–C–O with considerable hydrogen and deuterium present. The samples were heated at a constant rate of 2°C/min, from 100°C to 900°C. Desorption peaks occurred in the range of 140–480°C. There was no significant desorption at temperatures above 600°C. The amount of deuterium detected varied from a low of $8 \times 10^{21}/m^2$ to a high of $2.1 \times 10^{23}/m^2$. In one case, the amount of deuterium in a tile piece was seven times greater than the amount in a neighboring tile piece. Some of the tile pieces in the plasma-exposed region showed surface melting. Despite this, the deuterium yield from one of these pieces is $>10^{23}/m^2$. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Beryllium armour facing the plasma in future fusion devices such as ITER will accumulate tritium from nuclear reactions, implantation and co-deposition. The latter is of particular concern, since recent publications [1–5] indicate the possibility of quantities of tritium approaching 1 g/m^2 in co-deposition layers. Since JET has a divertor configuration similar to the current ITER design and uses mixed Be and graphite armour as is likely in ITER, tiles exposed to a deuterium plasma in the JET divertor may provide a valuable indication of the tritium behaviour to be expected. This paper presents data from thermal desorption spectroscopy of pieces from a beryllium tile, C8a, from the inner strike zone of the JET divertor, after exposure to D–D plasmas over the period April–June, 1995.

2. Experimental

The Be tiles were mechanically attached in pairs to water-cooled support bars, aligned so that each tile had a plasma-exposed and shadowed region. The tiles were castellated by cutting 7.5 mm squares to a depth of 5 mm, in order to reduce thermal stresses. In the last week of operation, the power flux to the tiles was increased to the point where some surface melting of the tiles occurred. For more details about the tile geometry and exposure conditions, see [1].

The tiles were removed from JET in June, 1995 and studied using nuclear reaction analysis (NRA) [1]. The results indicated surface co-deposition layers of a Be–C–O–D compound of varying thickness, >8 μm in places. Two tiles were shipped to McMaster University in January, 1996 for thermal desorption spectroscopy (TDS). 2–3 mm thick pieces of tile C8a were manually cut from the plasma-facing side of the tile, using a simple hacksaw and no lubricant, to avoid perturbation of the surface by lubricants or excess heat from power cutting (care was taken to cut slowly so as not to heat the sample above about 75°C). Each piece was then placed in a high-vacuum TDS chamber, described in more detail in Ref. [6]. After baking for 6 h at 75°C and cooling down, the system attained a base pressure of better than 10^{-6} Pa. A liquid nitrogen trap surrounding the sample holder was filled to minimize the background pressure of H_2O, since the breakdown products of H_2O in the mass spectrometer ionization chamber include H_2, which can create background for the HD peak.

During a TDS run, the sample was in an alumina-coated heating basket, with an opening facing the

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[*] Corresponding author. Fax: +1-905 527 8409; e-mail: macnew@mcmaster.ca.

quadrupole mass spectrometer (QMS). The sample temperature was ramped up at a constant rate of 2°C/min, from 75°C to 900°C. As the sample was heated, the QMS sampled the HD and D_2 peaks and passed the information to a computer. The sensitivity was calibrated using a calibrated deuterium source with a flux of 10^{-7} at. cm^3/s, after each run.

3. Results and discussion

Fig. 1 is a schematic diagram of Be tile C8a. Included is a table comparing the integrated amount of deuterium from NRA (recent calculations using the data reported in Ref. [1]) and from the present TDS results. The cross-hatched areas indicate pieces where the surface was partially melted during the plasma operations in JET. The dashed line indicates the approximate position of the division between the regions exposed to the plasma and those regions that were shadowed.

Note that, in general, the amount of deuterium detected by NRA is very close to the amount desorbed during TDS – well within experimental uncertainty in all cases except for tile pieces #36 and #40. In the case of tile piece #40, the NRA yield was more than twice the

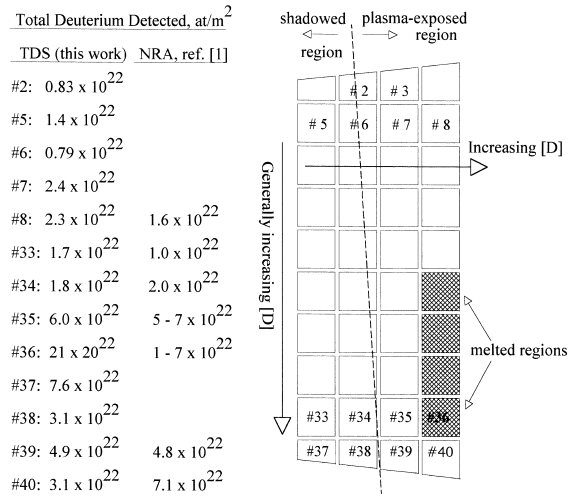| Total Deuterium Detected, at/m^2	
TDS (this work)	NRA, ref. [1]
#2: 0.83 x 10^{22}	
#5: 1.4 x 10^{22}	
#6: 0.79 x 10^{22}	
#7: 2.4 x 10^{22}	
#8: 2.3 x 10^{22}	1.6 x 10^{22}
#33: 1.7 x 10^{22}	1.0 x 10^{22}
#34: 1.8 x 10^{22}	2.0 x 10^{22}
#35: 6.0 x 10^{22}	5 - 7 x 10^{22}
#36: 21 x 20^{22}	1 - 7 x 10^{22}
#37: 7.6 x 10^{22}	
#38: 3.1 x 10^{22}	
#39: 4.9 x 10^{22}	4.8 x 10^{22}
#40: 3.1 x 10^{22}	7.1 x 10^{22}

Fig. 1. Beryllium divertor tile (C8a) from JET. Samples for ion-beam analysis and thermal desorption are labeled #2–#8 and #33–#40.

TDS yield. The sealed bag in which the tile was transported contained dust, which suggests that some of the deuterium-containing co-deposition layer was scraped
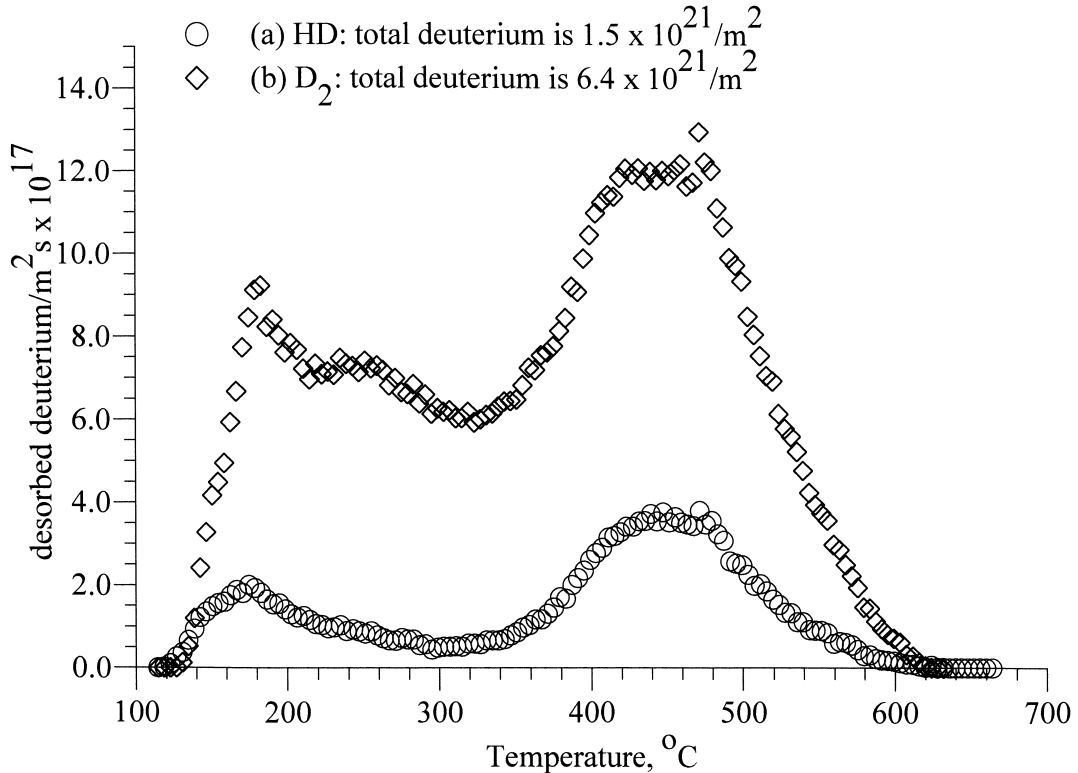

Fig. 2. Thermal desorption spectra from beryllium tile piece #6, with co-deposition layer on the surface; this piece was on the border between the shadowed and plasma-exposed regions. Heated at 2°C/min.

off in handling. Since tile piece #40 was a corner piece, it might have suffered more damage from handling than other pieces. Another factor is that the NRA analysis beam would have probed an area of about 1 mm in diameter, whereas the TDS measurements give the average for the 7.5 × 7.5 mm tile piece. In a neighboring piece, #36, the NRA yield varied by a factor of 7 across the sample.

Tile piece #36 is the only sample from the melted zone which has been analysed by TDS to date. In this case, the NRA yield varied across the sample, with four measurements producing yields varying from 1.2 to 7.3×10^{22} D/m^2. In contrast, the TDS yield was many times greater, at 21×20^{22} D/m^2. One possible explanation is that deuterium diffused deeply into the co-deposition layer, and perhaps into the beryllium substrate, during the periods when the tile surface was melted. This can be modelled by assuming that surface desorption was recombination-limited during brief periods of elevated temperature. The JET pulse length was about 30 s, but the period of greatest heating, during which the surface was melted, was only about 5 s in duration.

Deuterium below 8 μm depth would not be detected by the NRA technique of Coad et al. [1]. Assuming that

the deuterium concentration is 10 at.% below 2 μm depth (see Section 3.2), the deuterium distribution would need to extend to a depth of 23 μm to account for the observed TDS yield of 21×10^{22} D/m^2.

3.1. Thermal desorption spectra

Fig. 2 shows the HD and D$_2$ desorption spectra for Be tile piece #6, on the borderline between the plasma-exposed region and the shadowed region. There are at least three peaks, at about 180°C, 250°C and 450°C. The highest temperature peak is very broad, and may be composed of two or more sub-peaks. Note that essentially all desorption was complete by 600°C, as was seen previously in similar materials [2–5,7]. The D$_2$ desorption curve is predominant, accounting for 81% of the deuterium desorbed. The HD desorption curve has almost exactly the same features as the D$_2$ curve. This is true in all of the data, so the two desorption curves will be combined in subsequent figures.

Fig. 3 shows the total deuterium desorption yield from both the HD and D$_2$ signals, for Be tile pieces #33, #35 and #36, in the shadowed, plasma-exposed and
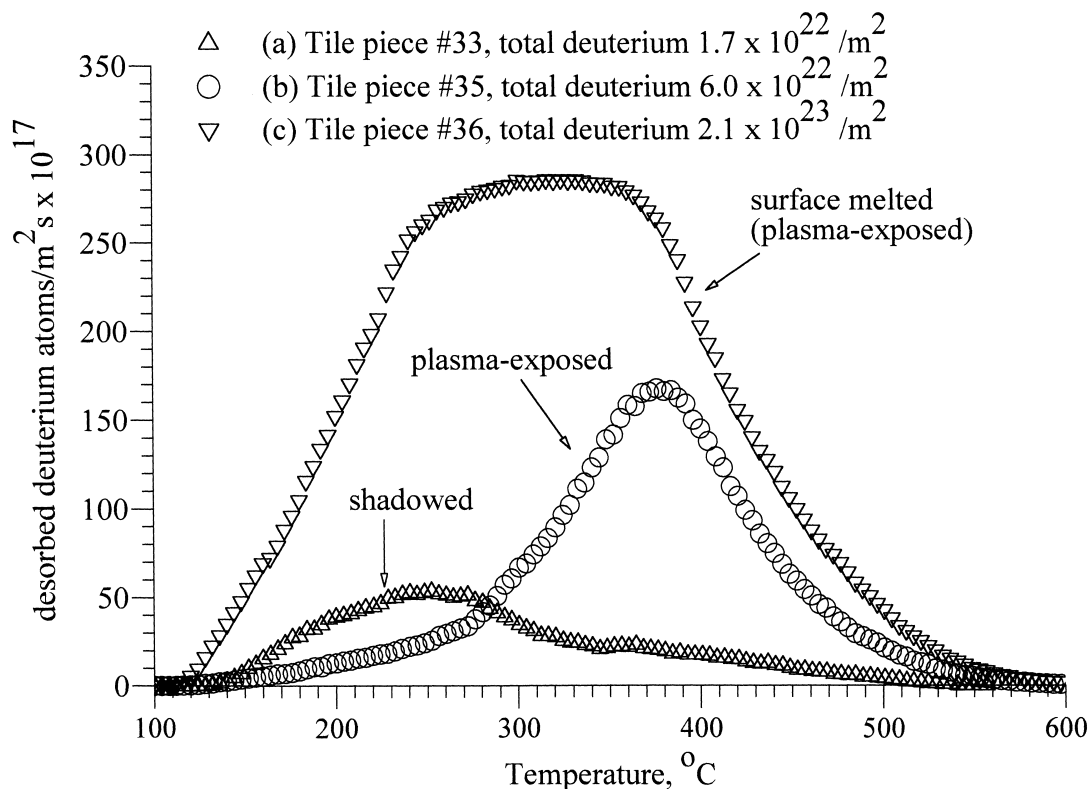

Fig. 3. Thermal desorption spectra from beryllium tile pieces #33, #35 and #36, with co-deposition layers on the surface; #33 was from the shadowed region, #35 and #36 were exposed to the plasma; the surface of #36 had been melted. Heated at 2°C/min.

melted regions, respectively. Note the very large yield in piece #36, which was melted by the plasma. This spectrum may have saturated the detector at the peak, as evidenced by the very flat profile from 300°C to 350°C. For this reason, the integrated deuterium yield may be an underestimate.

Desorption from tile piece #33, in the shadowed region, peaked at about 250°C, which is similar to the intermediate temperature peak in Fig. 2. Desorption from tile piece #35 peaked at about 385°C, indicating stronger bonding to the co-deposition layer. The desorption spectrum for the melted tile piece (#36) is so broad that it is likely to be a superposition of two or more peaks, unless desorption is strongly diffusion limited in this case.

Fig. 4 shows the total deuterium desorption yield for Be tile pieces #38, #39 and #40. The integrated yields are similar for these three pieces, at $3–5 \times 10^{22}$ D/m^2. As for pieces #33, #35 and #36, the plasma-exposed pieces exhibited desorption at higher temperatures than the shadowed piece.

Fig. 5 shows the total deuterium desorption yields for Be tile pieces #8, #36 and #40, scanning down the tile in the plasma-exposed region. The desorption yield

from samples #8 and #40 is very similar, and much lower than that from the melted sample, #36.

3.2. Ion beam analysis

One of the tile pieces, #3 in Fig. 1, was studied using ^{3}He-D NRA and 2 MeV ^{4}He elastic recoil detection (ERD). This piece, from the plasma-exposed region of the tile, contained up to 8 at.% hydrogen within 250 nm of the surface (the ERD probing depth). Fig. 6 shows two depth profiles of deuterium determined from NRA data. The first profile (Fig. 6(a)) was calculated assuming that the material containing the deuterium was primarily beryllium, i.e. using the energy loss rate appropriate to ^{3}He and ^{4}He particles travelling in beryllium. From this assumption, the deuterium concentration was about 2.5 at.% from 50 to 250 nm depth, and tailed off to a concentration of <1 at.% below 600 nm. The second profile (Fig. 6(b)) was calculated assuming that the material was primarily carbon. Since the actual material is a co-deposited mixture of H, D, Be, C and O, the real deuterium profile is probably somewhere in between the two profiles shown. Both show a near-surface peak of about 2.5 at.%, and tail off to 1–1.5 at.% below that peak. There is
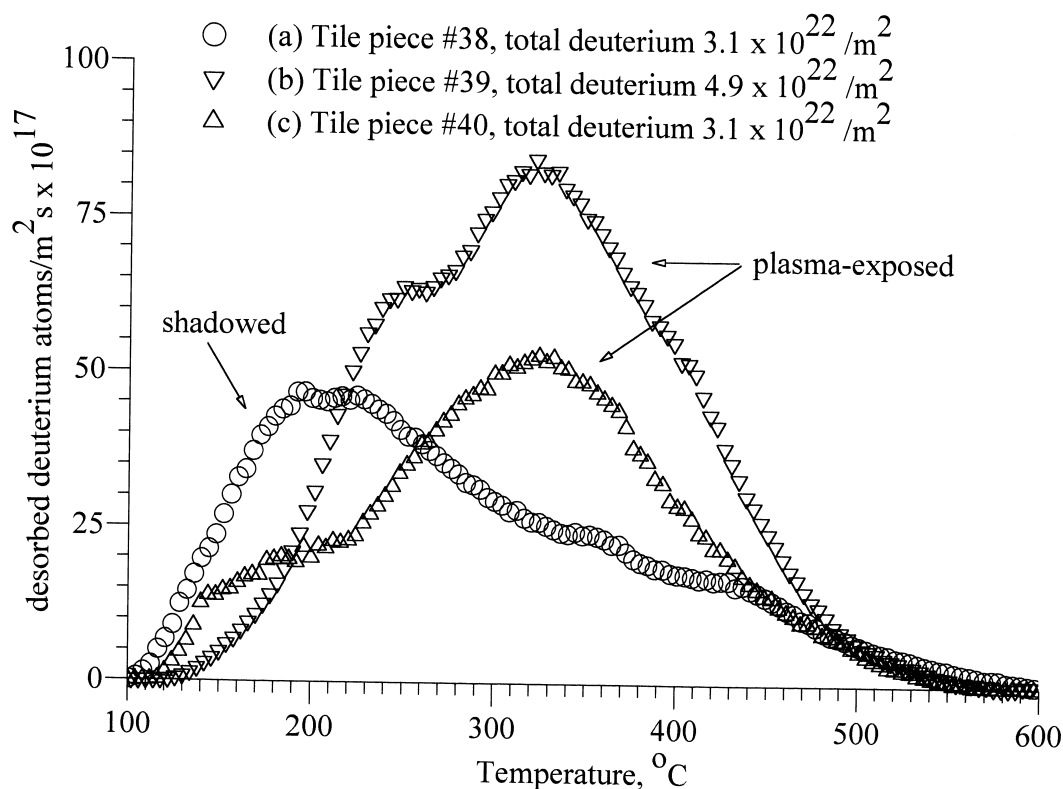

Fig. 4. Thermal desorption spectra from beryllium tile pieces #38, #39 and #40, with co-deposition layers on the surface; #38 was from the shadowed region, #39 and #40 were exposed to the plasma. Heated at 2°C/min.
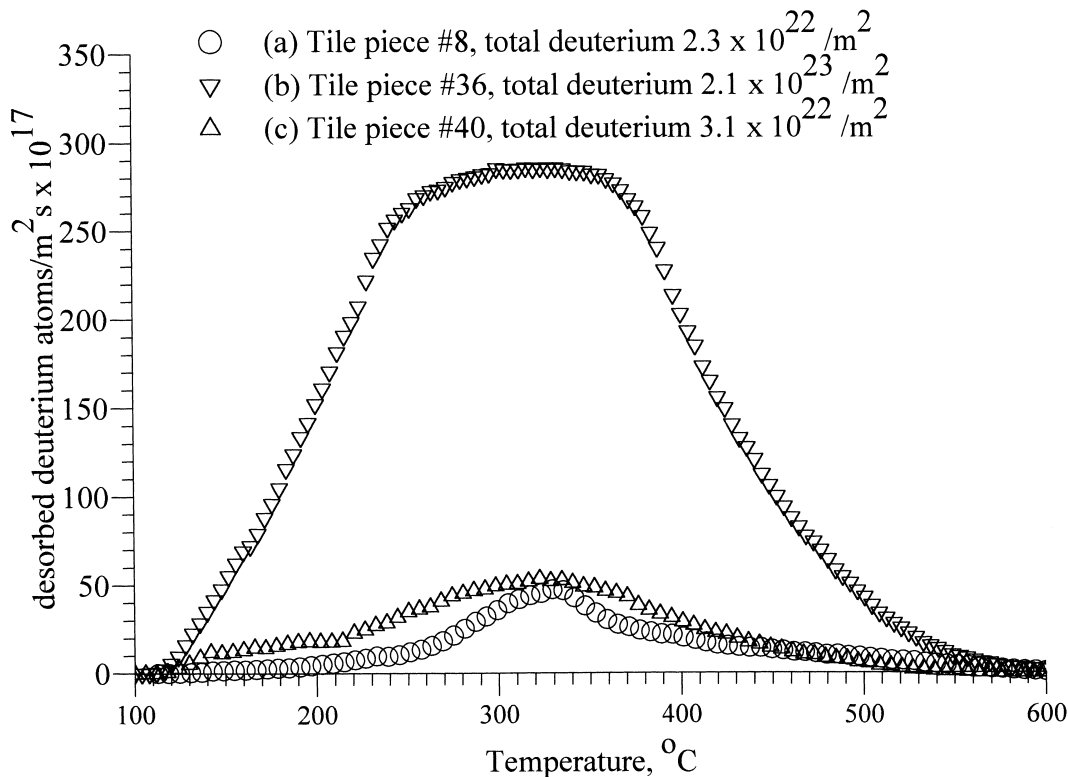
Fig. 5. Thermal desorption spectra from plasma-exposed beryllium tile pieces #8, #36 and #40, with co-deposition layers on the surface; the surface of #36 had been melted. Heated at 2°C/min.
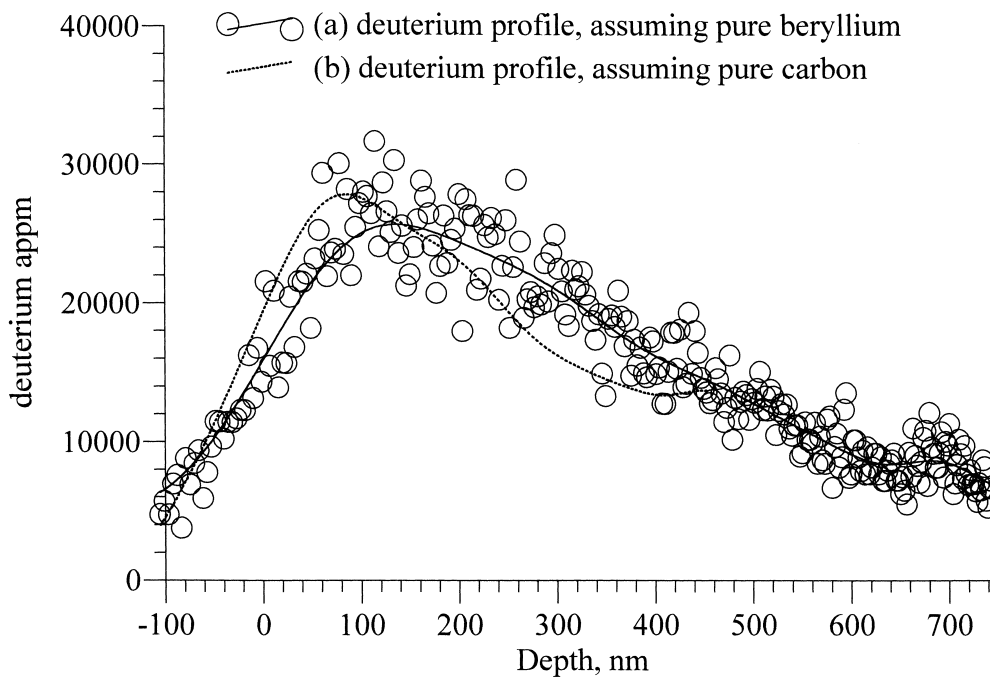

Fig. 6. Deuterium depth profiles of the plasma-exposed surface of beryllium tile piece #3, determined from nuclear reaction analysis (NRA) data. The lines are smoothed fits. Estimated depth resolution is 50 nm; uncertainty in the location of the surface is ±50 nm.

no indication that the deuterium distribution drops below 1 at.% below the depths probed.

4. Conclusions

Deuterium is present in large quantities in co-deposited layers on Be tiles from the JET divertor. Most alarming is the amount of deuterium in the melted piece, which might have been expected to desorb most of the co-deposited deuterium when the surface melted. It appears that there is considerable diffusion of deuterium into the tile when the surface melts, and, perhaps, hydrogen compound formation. This indicates the potential for significant tritium inventories in ITER.

Almost all of the co-deposited deuterium appears to have desorbed after relatively slow heating to 600°C at 2°C/min. Hence it would seem possible to outgas divertor tiles made of beryllium by a controlled bake to 600°C. However, this also raises the concern that these co-deposited layers might release large amounts of deuterium and tritium during thermal excursions, possibly destabilising the plasma.

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