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ABSTRACT

We compare the deposition of material on poloidal sets of divertor tiles that had been exposed in JET in 1998-2009 and 1998-2007. Post mortem analyses suggest toroidally integrated deposition being increased by 197.5cm^3 during 2007-2009. The analysis of dust collected from the divertor indicates the amount accumulated during the same period to be 248.4g. Converting the weight of dust to volume, the fraction of material entering the divertor that was converted to dust and flakes is 43±10%. The size of most dust particles ranged from 10 to 100 μm. The integrated amount of deposition on the “marker” tiles exposed in 2007-9 was found to be more than twice the amount expected from film growth on other tiles plus the dust because the plasma responds differently to the new tiles.

1. INTRODUCTION

Deposits in tokamaks such as ITER will eventually reach a saturation thickness, so that any further deposition will result in flaking or conversion to dust. To fully determine the inventories of dust and retained tritium in the reactor vessel, it is therefore important to know how and when this conversion process occurs. JET is the largest tokamak in the world, and thick deposits have been regularly observed in the divertor since 1996 [1-4]. Production of dust was first reported in JET in 1996, when 40-micron thick flakes were observed to spall from louvre clips and elsewhere [5]. However, no systematic evidence of the conversion process has been gathered from JET or other existing tokamaks. In order to measure the conversion rate it is necessary to determine the amount of deposition occurring during a given period, and what fraction of this is transformed to dust and what fraction is as increased film growth. There was a unique opportunity to assess the conversion rate in the JET divertor in 2010 when all the carbon fibre composite (CFC) divertor and first wall tiles were removed to install an ITER-like divertor and first wall (beryllium and tungsten surfaces) [6]. A cross-section of the JET MkII-HD (High Delta) divertor is shown in Figure 1, which shows the configuration in use in 2005-9. The Load Bearing Tile (LBT) and its support structure were installed in the 2004-5 shutdown, as were the High Field Gap Closure (HFGC) tiles. Other tile shapes were used instead of the LBT in 1998-2004, but most of the JET carbon divertor tiles had been in place continuously from 1998 to 2009, so conversion can be assessed after 11 years of film accumulation on these tiles. The removal of all the divertor components also gave the opportunity to make a detailed collection of the dust present in the JET divertor.

2. EXPERIMENTAL AND RESULTS

The dust conversion rate was assessed by comparing the increase in total deposition during the campaign period 2007-2009 with the amount of dust accumulated over the same period. The dust in the JET divertor was collected in January 2010 using cyclone vacuum cleaners with exchangeable collection pots in six stages: 1) the outer divertor Tiles LBT, 7 and 8, 2) the inner divertor Tiles HFGC, 1 and 3, 3) the inner and outer divertor support structures, 4) the outer divertor floor Tile

6, 5) the inner divertor floor Tile 4, and 6) the inner and outer louvre regions. (The louvres are the slatted structures in the extreme bottom corners of the divertor). Sample 1 is the dust collected in the pot during stage 1, etc. The final, corrected amounts of dust in each stage were 0.4, 115.1, 3.2, 51.4, 22.3 and 56.0g.

All dust samples except Sample 3 were taken remotely using the JET Remote Handling mascot – Sample 3 was collected manually from the carriers where tiles were stored after removal from the torus. The dust was collected in a cyclone pot which could be valved off and exchanged for a fresh pot after each area was cleaned. Because a small fraction of the dust may not fall into the pot but be retained on the cyclone or in the dust bag, these items were also exchanged – first after Sample 2 was collected, again after Sample 5, and finally after the last collection (Sample 6) – and retained for analysis. The amount of dust collected in each of the 6 pots was calculated from the difference in the weight of the pot before and after the dust collection [7]. After this, the difference between the weights of retrieved dust bag and a clean reference bag was evaluated and added to the weights in the pots in the same ratio as the weights in the pots used with that particular dust bag.

A number of corrections had to be applied. For stages 1–5, 22 of the 24 modules that comprise the full toroidal divertor were cleaned, because the other two modules were retained for surface analysis, so the data have been corrected to give the full amount expected from 360°. In stage 6 the louvres in all 360° of the divertor were cleaned, so this correction to the weight was unnecessary. In order to correctly ascribe the dust collected during the period 2007-9, note must also be taken of when the area was vacuum cleaned previously. The louvres were previously cleaned in 2004, so the deposits there represented two operational periods; the periods contained similar integrated X-point times so the correction was almost a factor of two. The carriers had not been cleaned since their installation in 1998, so based on their integrated X-point duration the correction was about a factor of four. The total amount of dust produced in the 2007-9 period was thus 248.4g. To compare with the deposition in the divertor, the weight of dust was converted to volume taking the measured density of dust removed from JET in 1998 ($1.69 g.cm^{-3}$ [8]), though the density of the dust collected in 2010 has not been measured and may differ: the resulting value was $147 cm^3$.

The size and composition of dust particles were measured for all samples except Sample 1 due to insufficient amount of material for measurements. The size distribution was determined using Mastersize E instrument. The dust bags seemed to contain smaller particles (distribution peaked at ~2 μm) than the other samples (maximum at ~100 μm). The reason for the different size distributions is not quite clear. This could be related to the operation of the vacuum cleaner, i.e. very small particles remain suspended in the airflow and go to the dust bag instead of the pot where the rest of the dust is collected. The dust particle size distributions (i.e. fraction of the sample at a given size) for all samples would be somewhat misleading because the distribution for the sample from dust bag 3 has a dominating peak at ~2μm, yet the mass of this sample was much smaller than most other samples. Therefore, in Figure 2 the size distributions have been multiplied by the mass of the corresponding sample.

The carbon content of the particles was analysed using a Strohlein Coulomat 702 instrument, whilst the metal contents were analysed using an atomic absorption spectrometer (Thermo ICE 3300). The vast majority of the dust was carbon, with the next largest components being the elements Ni, Fe and Cr in the correct relative amounts for Inconel (the material of the JET vacuum vessel and used for tile mountings, fasteners, etc) varying from 16% from Tiles 1 and 3 to 3% at the louvres. Beryllium was analysed at 2.4% and 0.1% for these same samples, respectively. The D/C ratios (derived from combustion measurements) for Tiles 1&3, Tile 4, Tile 6 and the louvres were 0.5%, 0.4%, 0.13% and 1.2%, respectively, whilst the T/D ratios (determined using a Hewlett Packard 5890 gas chromatograph) were 1.6×10^{-5}, 4.7×10^{-5}, 5.7×10^{-4} and 5.8×10^{-4}, respectively. The compositional analysis was made from aliquots of the samples so the analysis was not made from individual particles. Thus, it was not possible to make any conclusions about the composition of single particles.

When assessing the amount of deposition in the divertor, it was noted that the outer divertor Tiles 7 and 8 are areas of net erosion for all JET campaigns and that deposition on the central divertor tile (LBT for the JET MkII-HD divertor shown in Figure 1) was negligible (<1µm) [5-6]. Thus the amount of deposits on the divertor tiles was derived by measuring the film thicknesses by optical microscopy from cross sectional samples covering Tiles 1, 3, 4 and 6 that were situated close to each other in the divertor. The HFGC tiles were only fitted to the divertor in 2004, so they are not included in the conversion studies. The increase in the quantity of deposits on tiles exposed in 1998-2009 over the amount found on tiles that had been removed in 2007 (and exposed 1998-2007) gives the amount deposited and still adhering to the tiles during the 2007–9 campaigns. First, samples were cut from each tile using a coring drill; the drill had an outside diameter of 20mm and produced a core sample of 17mm in diameter. A poloidal line of holes was drilled every 20mm across the tile. The samples were then cut in half and polished to provide a poloidal cross-section for 17 out of every 20mm of the tile. The sections were examined with a Nikon optical microscope.

Although the conversion is calculated for 2007–9, data were also collected for tiles exposed in 1998–2004, 1998–2009 and 2005–9, which are particularly interesting for Tile 1. The introduction of the LBT in 2004 allowed plasma operations with the inner strike point high on Tile 3 (or even on Tile 1) and the outer strike point on the LBT: the configuration allows many parameters to be similar to those of ITER. This configuration meant that impurity flow in the scrape-off layer was concentrated on the horizontal part (apron) of Tile 1 (s = 171–236mm) and HFGC: Following the 2005–7 campaign spalling deposits were clearly seen on the apron of all Tiles 1. The effect of this additional configuration is shown in Figure 3.

Much more deposition had occurred in the period 2005-9 than in the previous years (1998–2004) on the apron of the Tile 1, despite the fact that the apron region was vigorously vacuum cleaned by remote handling during the 2007 shutdown, so much of the 2005–7 deposition in this region must be missing. The deposition on the apron of Tile 1 exposed in 1998–2009 is close to the sum of the deposits in 1998-2004 and 2005-2009 (see Fig. 3). Note also that the profile on the front face of the Tiles 1 (s = 236–423mm) is similar for all the three periods, and that there is a distinct reduction in thickness at s = ~320 mm where the plasma facing surface is bent (see Figure 1). Similar behaviour is seen on Tile 3 – the mean film thicknesses on tiles exposed during the periods 1998–2004, 1998–2009 and 2005–9 are all comparable (55, 54 and 44μm, respectively), although there was a large scatter in individual measurements (23 to 94μm).

The base divertor Tiles 4 and 6 experience a variety of plasma conditions: the inboard part of Tile 6 is often the strike point position and thus is exposed to intense plasma bombardment. The sloping part of each tile can be the sink for migrating impurities: part of each tile is completely shadowed from the plasma (either by Tile 3 or Tile 7). The thickest deposits occur on these tiles, and isolated spots of spalling can affect the profiles. Figure 4 shows the profiles for tile 4 for 1998–2007, 1998-2009 and 2007–9, and Figure 5 shows the profiles for Tile 6.

The set of tiles that were exchanged in 2007 were on the opposite side of the torus to those exchanged in 2004 (module 2 rather than module 14). Tiles that were exposed in 1998–2009 have been analysed from both locations, and the total integrated volumes of films for the three Tiles 1, 4 and 6 from each location were within 1.5% (though volumes for individual tile positions may vary by 10%). Variations in the thickness of deposits at Tile 3 are larger, however, and there does seem to be a systematic difference between the opposite sides of the torus in this position. Figure 4 shows that very thick films (~500μm) accumulate at the bottom of the sloping part of Tile 4, and extend into the shadowed region, though there appears to be a clear reduction in the extent of the deposition in this region in 2007–9. The dip in the film thickness on the tile exposed 1998–2007 in the region s = 770–780mm is due to spalling in that region: the films on the sloping parts are known to be friable [3]. Figure 5 shows remarkably consistent behaviour for Tile 6, with the amount found on the tile exposed 1998–2009 being close to the sum of the deposits in the other two periods (as might be expected in the absence of spalling), despite the fact that the deposit is over 1mm thick in one area.

The thickness profiles such as in Figures 4 and 5 have been integrated across each tile to give the volume of deposits in cm^3. This is then integrated toroidally to give the overall deposition in the torus: in this integration allowance is made for the un-coated part of each tile due to the "roof-top" effect wherein a portion of each tile is shadowed by a toroidally adjacent tile [9], and for the assembly gaps between tiles. Table 1 gives the integrated volumes of coating for each tile, together with the average film thickness over the tile.

As noted from the Table, no Tile 3 exposed in 1998–2007 exists, however as mentioned above, the amount of deposit on this tile exposed in 1998–2004 is almost identical to that on the tile exposed in 1998-2009, so the error is likely to be very small. It can also be seen from Table 1 that the increase in adherent deposits following the 2007–9 campaign was $197.5cm^3$ for the tile sets measured, though some variability may be expected. Thus, including the dust, the total impurity flux to the divertor 2007–9 was 197.5 +147 = $344.5cm^3$. The dust conversion factor is the ratio of the dust amount to the total impurity flux giving a value of 42.7%.

The result may be in error due to the following causes *inter alia*: incorrect dust density value, statistical variations in coating thicknesses within the divertor, corrections for deposition in other parts of the divertor. Deposit densities may be considerably lower than the value measured for dust in 1998; a value of $1g.cm^{-3}$ would give a conversion ratio of 55.7%. Variations between tiles can affect the toroidal integration of the deposits, but two sets of tiles exposed in 1998–2009 gave a similar result: the error may be 10%. Deposition also occurs on the louvres and on the HFGC tile but has not been measured for every campaign; however, the measurements that have been made suggest this would be small compared to the other error sources. A reasonable error margin for the conversion ratio would be 43±10%.

3. DISCUSSION

A first point to discuss is "What is dust?" Some of the loose materials in the divertor that could be seen prior to vacuum cleaning were flakes of coating that have become detached, and some of these were millimetres in size. Figure 2 shows that the most abundant particle size for all the "dust" was about 100 μm in diameter, which must include the previously observed flakes that had broken up within the vacuum cleaner. ITER are particularly concerned about mobilisable dust that can mix with air during a vacuum breach; if this is limited to dust of ≤1 μm diameter, virtually none of the "dust" collected from JET in 2010 qualifies.

The second issues is that on Tile 3 and the front face of Tile 1 deposits are not accumulated beyond certain thickness levels, despite the continual arrival of impurities along the inner SOL, as shown in Fig.3 for s-coordinates 236–423 mm. This might be considered as 100% conversion in those areas if dust was produced, but it may also be that the film and the plasma were in equilibrium so that the incoming impurity flux was balanced by the outgoing flux of sputtered material (sputtering coefficient equal to unity). By contrast, the deposition in the corners of the divertor showed a continual growth in film thickness towards inevitable spallation. Thus, only an overall conversion ratio for the divertor is meaningful. There was clearly no set value of thickness at which the deposited films spalled from the CFC tiles – films up to >1000μm remained adherent, whilst in other areas spalling of films tens of microns thick were observed. Furthermore, films frequently de-laminated – that is, films often had a layered structure, and outer layers could spall off leaving a film still adherent to the substrate.

New tiles were introduced at each JET shutdown to act as "marker" tiles to witness the erosion/deposition at that location over a limited period. There is clear evidence from these investigations that this does not necessarily measure the correct deposition typical for this location. For example, the combined film growth during 2007-9 plus the accumulated dust was $344.5cm^3$, yet tiles only exposed in 2007–9 accumulated more than twice this amount ($799.5cm^3$). Another example is Tile 3: there was no net deposition at that location between 2004 and 2009, yet clean tiles inserted at that position accumulated deposition until they were indistinguishable from the surrounding tiles. It is known that deposited films have different thermal characteristics and greater sputtering coefficients than bulk graphite or CFC [10, 11]. It is not known at which thickness the effect saturates, but the data in [10, 11] suggests of the order of 1µm. Material is being continually sputtered, transported toroidally (approximately) and re-deposited, so that if one tile surface behaves in a different manner to its surroundings it will gradually be equilibrated with its neighbours. The evidence here suggests that the plasma can recognise there is a difference in film properties even at the tens of microns level, and films on new tiles will grow disproportionately, providing false information on deposition rates.

CONCLUSIONS

Poloidal sets of carbon-based divertor tiles that had been in the JET vessel 1998-–2009 and for 2007–9 and removed in 2010 have been analysed. In each case, the amount of deposition was determined. A detailed analysis of dust collected from the divertor using a vacuum cleaner was also carried out, and the amount accumulated during the operating period 2007–9 was 248.4g. By analysing a set of tiles removed in 2007 and exposed in 1998-2007, the overall growth of deposited films between 2007 and 2009 was determined to be 197.5cm^3. Converting the weight of dust to volume using a density derived for JET dust from 1998 of 1.69g/cm^3, the percentage of material entering the divertor that was converted to dust and flakes has been determined to be 43±10%. Analysis of the collected dust indicated most particle sizes were from 10 to 100µm, so the material would not be liable to in-vessel mobilisation.

No critical film thickness for spallation to occur was seen for deposits onto the CFC tiles - deposits up to 1mm thickness were observed, yet spalling was observed in other areas for films <30µm thick.

The integrated amount of deposition on the “marker” tiles exposed 2007–9 was found to be more than twice the amount expected from film growth on other tiles plus the dust. This is because the plasma responds differently to the new tiles, which throws doubt on the value of the “marker” tiles.

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REFERENCES

[1]. J.P. Coad, P. Andrew, D.E Hole, et al., Journal of Nuclear Materials **313–316** (2003) 419
[2]. J.P. Coad, P. Andrew, D.E. Hole, et al., Journal of Nuclear Materials **363–365** (2007) 287–293
[3]. A.M. Widdowson, S. Brezinsek, J.P. Coad, et al., Physica Scripta **T138** (2009) 014005
[4]. J.P. Coad, S. Gruenhagen, D.E. Hole, et al., Physica Scripta **T145** (2011) 014003
[5]. A.T. Peacock, P. Andrew, P. Cetier, et al., Journal of Nuclear Materials **266–269** (1999) 423
[6]. G.F. Matthews, M. Beurskens et al, Physica Scripta **T145** (2011) 014001

[7]. A.M. Widdowson, C. F. Ayres, S. Booth, et al., Journal of Nuclear Materials **438** (2013) S827-S832
[8]. N. Bekris, J.P. Coad, R-D Penzhorn, et al., Journal of Nuclear Materials, **337–339** (2005) 659
[9]. J.P. Coad, M. Rubel and C.H. Wu, Journal of Nuclear Materials, **241–243** (1997) 408
[10]. P. Andrew, J.P. Coad, T. Eich, et al., Journal of Nuclear Materials, **313–316** (2003) 135
[11]. P. Andrew, J.P. Coad, Y. Corre, et al., Journal of Nuclear Materials , **337–339** (2005) 99-103

Tile	1998-2007		1998-2009		2007-2009	
	Average film (µm)	Integrated vol (cm^3)	Average film (µm)	Integrated vol (cm^3)	Average film (µm)	Integrated vol (cm^3)
1	30.8	83.7	56.6	151.9	23.9	65.6
3	55.0	122.0#	55.0	118.8	22.3	49.1
4	176.4	478.9	221.9	596.1	131.3	341.8
6	196.5	580.1	187.3	550.1	116.6	343.0
Total		1264.7		1462.2		799.5

 # This is the value for a tile exposed 1998-2004 since no Tile 3 exposed 1998-2007 exists

Table 1: Average film thicknesses for each tile in Module 2, and the volume of coatings obtained by integrating film thicknesses toroidally.

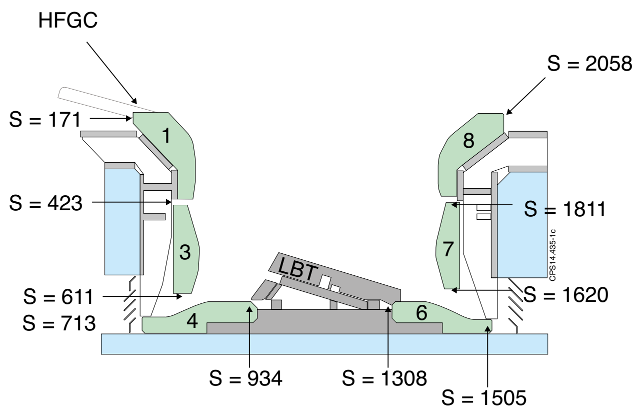

Figure 1: Cross–section of the JET MkII-HD divertor in use 2005–9 and showing the s co-ordinate system for defining points on tile surfaces (in mm).

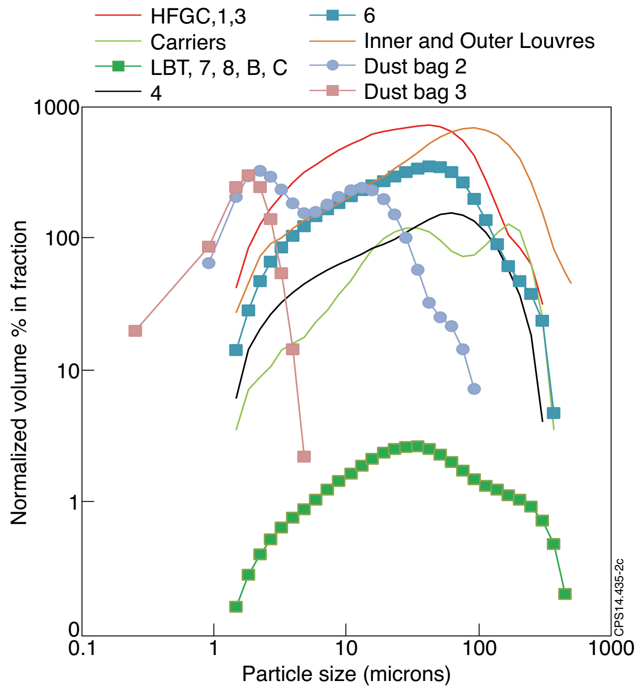

Figure 2: Particle Sizing Data – Results given as Volume in a size fraction multiplied by the total weight of the dust sample collected versus the particular size fraction in microns.

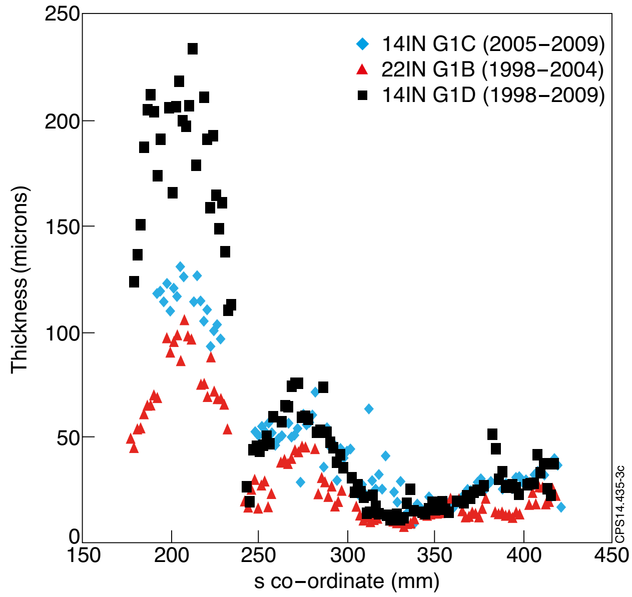

Figure 3: Film thicknesses from optical microscopy of sections for Tiles 1 removed after operational periods 1998–2004 (Module 22), 1998–2009 (Module 14) and 2004–9 (Module 14).

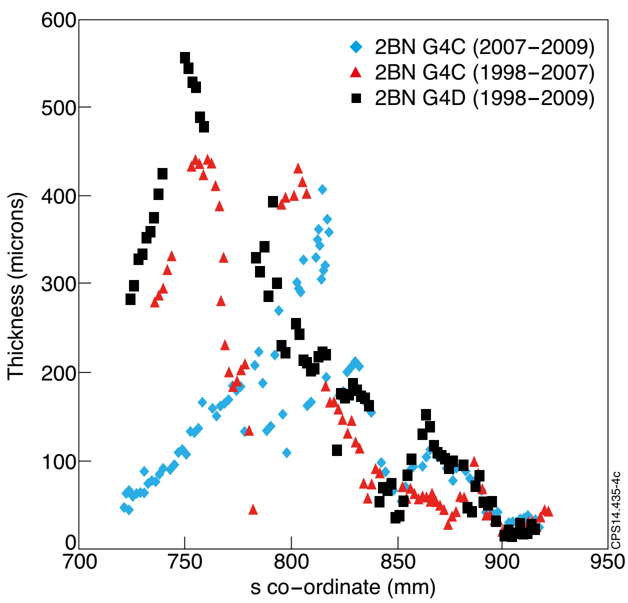

Figure 4: Film thicknesses from optical microscopy of sections for Tiles 4 removed from Module 2 after operational periods 1998-2007, 1998-2009 and 2007-9.

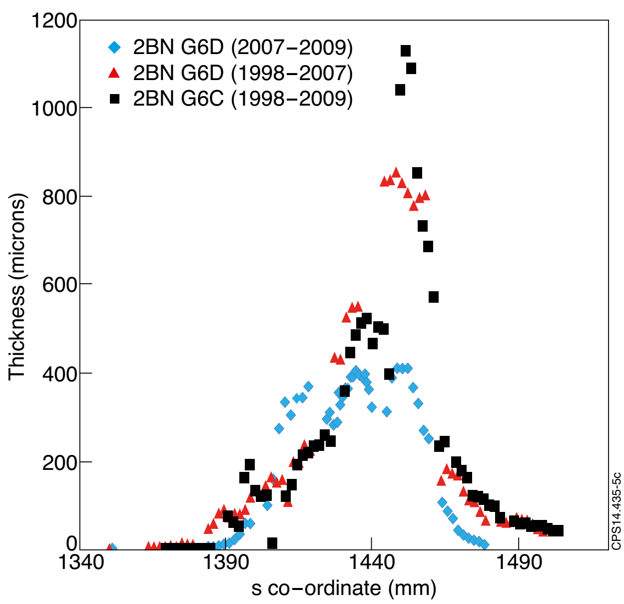

Figure 5: Film thicknesses from optical microscopy of sections for Tiles 6 removed from Module 2 after operational periods 1998-2007, 1998–2009 and 2007–9.