



Fuel accumulation in co-deposited layers on plasma facing components

M. Rubel ^{a,*}, P. Wienhold ^b, D. Hildebrandt ^c

^a Department of Fusion Plasma Physics, Alfvén Laboratory, Royal Institute of Technology, Association EURATOM – NFR, Teknikringen 31, 100 44 Stockholm, Sweden

^b Institute of Plasma Physics, Forschungszentrum Jülich, Trilateral Euregio Cluster (TEC), Association EURATOM, D-52425 Jülich, Germany

^c Max-Planck-Institute for Plasma Physics, Association EURATOM, Division of Plasma Diagnostic, D-10117 Berlin, Germany

Abstract

The work is focused on the determination of the distribution and the total content of deuterium in co-deposits formed in the TEXTOR tokamak on a toroidal belt limiter which is the machine's major plasma facing component (PFC). Limiter tiles in use for 14 100 s of plasma operation were dismantled for examination with surface analysis and microscopy methods. Mapping of the deuterium distribution by means of nuclear reaction analysis (NRA) revealed the presence of deposition zones covering about 35% of the tiles's surface area. Besides C and D, other constituents of the layers were boron, silicon and inconel components. The co-deposit, with a stratified structure and a thickness of up to 50 μm , could be detached from the tiles. Deuterium depth profiling on both sides of the detached flakes and in the underlying graphite substrate enabled the D content in the deposition zones to be estimated at a level of $3.5 \times 10^{19} \text{ cm}^{-2}$. Adding the fuel content found in the erosion zone ($3\text{--}7 \times 10^{17} \text{ cm}^{-2}$) and on the back side of the tile ($0.9\text{--}1.8 \times 10^{17} \text{ cm}^{-2}$), the total amount of D atoms trapped in all the limiter tiles was assessed to be about 2×10^{23} atoms. D content in the co-deposits accounted for approximately 10 at.% ($C_D/C_C \sim 0.1$) which was considered to be low in comparison to much greater values observed in other devices. The results of the ion beam analyses (IBA) agree well with the determination by thermal desorption spectrometry (TDS). © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Redeposition; Erosion; Hydrogen inventory; TEXTOR; Plasma facing components

1. Introduction

Plasma facing components (PFCs) such as limiter and divertor plates are the areas of intense plasma–surface interactions due to localized particle fluxes. Shaping of the magnetic field together with the geometry of components determines the distribution of erosion and re-deposition zones. Formation of co-deposited

layers results from the re-deposition of globally and locally transported material and is accompanied by the accumulation of fuel atoms. There have been many studies on the properties of co-deposits formed in magnetic-controlled fusion devices either with carbon [1–7], carbon/beryllium [8,9] or metal walls [10]. However, quantitative effects of the co-deposition process are difficult to predict a priori. The prediction of tritium inventory in future devices operated with a D + T mixture is an urgent and critical issue [11]. To facilitate this, efforts must be made to determine – as accurately as possible – the total amount of fuel accumulated and its distribution in the present-day machines. One approach is to measure the gas balance on a shot-to-shot basis and the other one is to map precisely the fuel trapped in

* Corresponding author. Tel.: +46-8 16 10 61; fax: +46-8 15 86 74.

E-mail addresses: rubel@msi.se (M. Rubel), p.wienhold@fz-juelich.de (P. Wienhold), dth@ipp.mpg.de (D. Hildebrandt).

PFCs. Furthermore, an exact identification of deposition zones is essential if methods for local tritium removal are to be developed.

The comparison of fuel accumulation data in various machines (e.g. TFTR [6,7], JET [8,9,12,13], TEXTOR [1,14,15]) indicates the importance of tile geometry, gaps between tiles and the PFC base temperature between discharges. Formation of flaking co-deposits has often been observed in gaps separating tiles [1,9] and even in remote areas (e.g. water-cooled louvers) with no direct line of sight to the plasma [12]. Various values of deuterium-to-carbon ratio and layer growth rates have been reported. In general, growth rates are of the order $1.5\text{--}3\text{ nm s}^{-1}$ [16,17], but as high as 100 nm s^{-1} [18] have also been measured. Another important reason to study the formation and peeling-off of flaking co-deposits is the potential production of dust particles [1,19–21]. Such particles influence plasma operation and create hazards related both to the tritium inventory and, if formed in substantial amounts, the risk of steam reactions in case of a leak of cooling water into the vacuum vessel [22].

There is a broad research programme at TEXTOR to determine the overall fuel inventory, one aspect of which is to recognise quantitatively the distribution of accumulated fuel in the torus and, thus, to assess the total amount of D atoms trapped in PFC in this machine. This paper assesses the total deuterium content accumulated in the limiter tiles.

2. Experimental and analysis procedure

The major PFC of TEXTOR is a toroidal belt pump limiter ALT II (Advanced Limiter Test) [23,24] consisting of eight blades, each of them covered by 28 graphite tiles. In the current arrangement, the total surface area of the 224 tiles is 3.4 m^2 , corresponding to about 9% of the inner vessel wall area. Details concerning the limiter construction and tile arrangement can be found elsewhere [23,24]. The tiles are periodically removed from the machine for cleaning by blasting with boron carbide (B_4C) grains.

Pieces exposed to the plasma during two consecutive campaigns (14 100 s and 17 290 s) were analysed in detail by means of ion beam analysis techniques (IBA) and microscopy. Deuterium content was determined using nuclear reaction analysis (NRA) based on the $d(^3\text{He}, p)^4\text{He}$ process. This is a well-established method [25] which, on the basis of a detected proton spectrum, allows the depth profiling of deuterium [26]. The information depth depends on the energy of a primary $^3\text{He}^+$ beam. For instance, for carbon-based targets (such as co-deposits) this depth increases from $1.4\text{ }\mu\text{m}$ for a 0.77 MeV beam to $6.2\text{ }\mu\text{m}$ for a 1.8 MeV beam. Beam energies in this range were applied for analysis of

the ALT tiles. Boron analysis was carried out with a proton beam of 0.65 MeV using the reaction $^{11}\text{B}(p, \alpha)2\alpha$. The information depth is approximately $7\text{ }\mu\text{m}$, but the reaction does not allow depth profiling because of the broad energy distribution of emitted alpha particles [27]. Heavier elements were traced with Rutherford backscattering spectroscopy (RBS) and energy dispersive X-ray spectroscopy (EDS), whereas the surface topography and internal structure of the layer was characterized with a scanning electron microscope (SEM). All types of surface analysis were first performed on the front (plasma wetted) and the back side (i.e. surfaces facing the scrape-off layer) of the 'as-delivered' tile and later, in order to check the morphology of the underlying graphite substrate, after removal of the co-deposit.

Measurements with thermal desorption spectrometry (TDS) were performed in a vacuum system consisting of two separately pumped vessels. The limiter tile was heated in a 0.04 m^3 chamber up to a temperature of 1400 K by electron bombardment with a power of 1.75 kW . The chamber was pumped with a turbomolecular pump with a speed of $0.20\text{ m}^3\text{ s}^{-1}$ for hydrogen. The base pressure was $5 \times 10^{-6}\text{ Pa}$. A fraction of the desorbed gas reached the analysis chamber through an orifice of flow conduction $1 \times 10^{-3}\text{ m}^3\text{ s}^{-1}$. This chamber was equipped with a quadrupole mass spectrometer for gas analysis and it was separately pumped by another turbomolecular pump to a pressure below $1 \times 10^{-6}\text{ Pa}$. Further information on the experimental aspects and calibration procedures are described elsewhere [28].

3. Results and discussion

3.1. Structure of co-deposits

Fig. 1 shows the tile: (a) in the initial state, i.e. shortly after dismounting from the torus and (b) after storing in air for a couple of months. Most of the exposed tiles, when inspected in the torus or soon after dismounting, had fairly smooth appearance and good adherence of the co-deposits to the substrate. Only in some areas (a few cm^2 size) on upper edges could one perceive signs of flaking. It was also difficult to remove the layer by scraping with a sharp tool. The smoothness and good adherence of the layer confirmed that no significant peeling-off of the layer had occurred in the torus during plasma operation. Therefore, one may assume that the total amount of fuel retained in the tiles could be determined. This is essential when the total inventory is to be assessed. In this sense, the layers distinctly differ from those very rough and brittle flaking ones formed on poloidal limiters, r.f. antenna protection tiles [1,29] and, as found only recently, on the neutralizer plates and

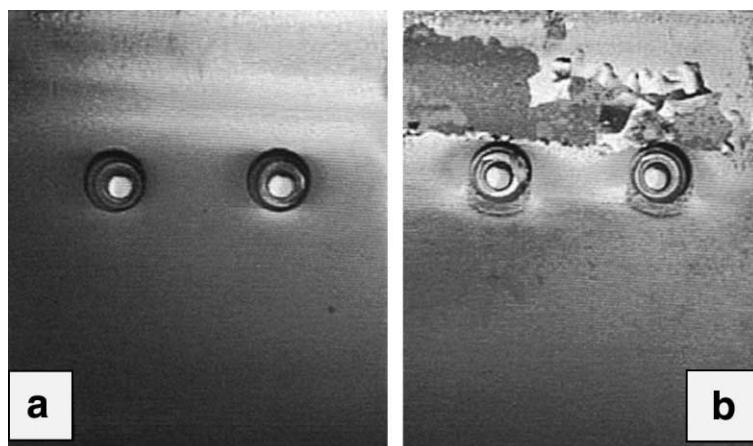


Fig. 1. Surface structure of the limiter tiles: (a) smooth layer observed soon after dismantling the tile from the torus; and (b) flaking layer observed after storing the tile for a few month in air.

scoops on the back of the ALT supporting structure [14,30].

After being stored for a few months in air at room temperature the co-deposits started themselves to peel-off in the form of large (cm range) fairly flat chunks. The deposit could easily be detached without further disintegration. This can be noted on plate (b) in Fig. 1 and has also been reported in [31]. However, the phenomenon of the layer self-flaking in air was observed on some tiles only, even though all of them were exposed during the same operation period. The underlying chemistry of the process is unclear. One possibility is related to the fact that the layers contain some atomic per cent of boron originating from the wall conditioning by boronization. It is possible that the boron reaction with atmospheric oxygen results in the formation of boron oxide and, eventually, in the change of the layer adherence to the substrate [32].

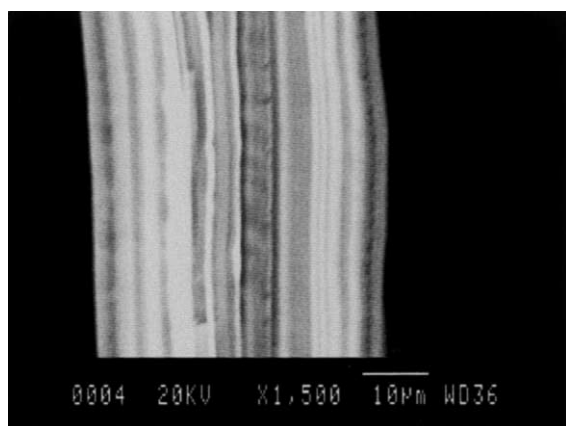


Fig. 2. Structure of the co-deposit detached from the deposition zone.

SEM image in Fig. 2 shows the structure of the co-deposit detached from the tile. One infers a thickness of $45\ \mu\text{m}$ and a stratified structure which distinctly differs from the columnar one observed on surfaces of other TEXTOR limiters [1,29]. Moreover, both sides of the flake were fairly smooth. Taking into account the layer thickness mentioned above and the total operation time of 14 100 s, the average growth rate is determined as $3.2\ \text{nm s}^{-1}$.

3.2. Fuel inventory

The first step in the investigation was to define the general distribution pattern of deuterium and other species co-deposited on the limiter plates. Results plotted in Fig. 3 for two adjacent tiles provide quite a consistent picture indicating two distinct zones: erosion and deposition. The transition between them is rather

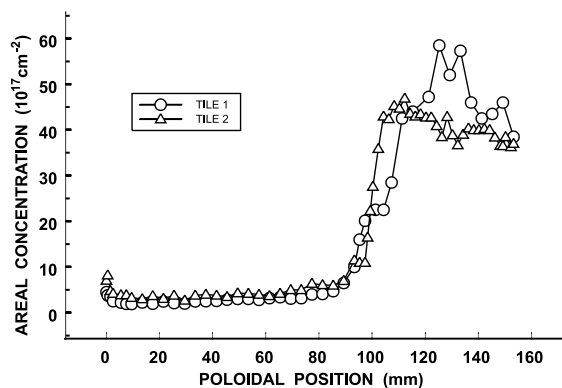


Fig. 3. General deposition pattern of deuterium on a few ALT II tiles. The results represent the D content in $4.5\ \mu\text{m}$ thick surface region.

narrow, 12–17 mm. As expected, the location of the erosion-to-deposition transition region varies somewhat from tile to tile. This is related to the ripple of magnetic field and, as a consequence, non-uniform distribution of incident particle fluxes and power deposition profiles over the whole limiter blade [24]. Besides deuterium, the major constituents of the layer are carbon and boron. Boron deposition profiles show the same pattern as those for deuterium. The other species identified are: metals (Ni, Cr, Fe, Mo and W), oxygen and silicon originating from former siliconizations and experiments involving silane (SiD_4) injection.

Depth distributions for deuterium in the erosion and deposition zones are shown in Fig. 4. The majority of deuterium in the erosion zone (typically $2\text{--}5 \times 10^{17} \text{ cm}^{-2}$) is detected in a surface layer not exceeding 300 nm thickness. This is greater than that expected from a direct implantation of low-energy deuterons. There is also a long tail extended to nearly 4 μm in depth. As the average surface roughness in the region is less than 1.5 μm , the result may indicate deuterium diffusion into the graphite substrate. From the second plot in Fig. 3 it is apparent that the determination of the total D content and its depth profile in the deposition zone are more complex because the thickness – as shown in Fig. 2 – exceeds the information range provided by NRA. However, the D content traced with a beam of increasing energy (from 1.5 to 1.8 MeV) proportionally increases from $4.4 \times 10^{18} \text{ cm}^{-2}$ in 4.5 μm to $6.1 \times 10^{18} \text{ cm}^{-2}$ in 6.2 μm proving the content to be approximately uniform through the layer. At the same time, the structure of the depth profiles is not uniform, i.e. there are some noticeable maxima which reflect the stratified nature of the deposits. The existence of such strata is probably related to heavy disruptions which – due to thermal loads – caused an instant removal of D species from the surface region and disturbed the regular

discharge-to-discharge growth of the co-deposit. Another result inferred from the depth profiles is, that the deuterium-to-carbon ratio (C_D/C_C) in the co-deposits is in the range 0.05–0.16 with the average value being around 0.10. This is in general agreement with the values determined for other high heat flux components of TEXTOR [1].

Analyses performed on both sides of the detached co-deposit yielded very similar results for the D content and distribution. In addition, in the underlying graphite substrate $3.4 \times 10^{18} \text{ D atoms cm}^{-2}$ was found in the layer not exceeding 5.5 μm . Taking into account the above amounts, NRA information depth and the co-deposit thickness (45 μm) the deuterium concentration in the deposition zone is assessed to be approximately $3.5 \times 10^{19} \text{ cm}^{-2}$. In Table 1 we summarize the results of the analysis and show the integrated amounts of deuterium accumulated in various regions on the whole tile, including the amount trapped on the back of the tile. The final result of this determination gives the value of about $1 \times 10^{21} \text{ D atoms per tile}$ with 94% of this amount accumulated in the deposition zone. (approximately 35% of the tile area). The surface studies were compared and cross-checked by means of TDS. Outgassing of the whole tile (one of those analysed with ion beams) released $1.1 \times 10^{21} \text{ D atoms}$; the amount of the desorbed H atoms was in the same range. The results obtained using the two independent methods thus agree very well. Therefore, we conclude that a total of $2.2\text{--}2.5 \times 10^{23}$ deuterium atoms are trapped in the 224 tiles of the toroidal limiter.

The analyses of these tiles have been compared with the analyses of other PFC. The quantities of D accumulated in poloidal limiters or rf antenna protection tiles were, typically $2 \times 10^{18} \text{ cm}^{-2}$ [1], i.e. a factor of 15–20 less than in ALT. It corresponds to a total of $2 \times 10^{22} \text{ D atoms}$ in all r.f. antenna and poloidal limiter tiles. The trapping in the inconel liner is even smaller, on average about $6 \times 10^{16} \text{ cm}^{-2}$ [33], corresponding to $2 \times 10^{22} \text{ D atoms}$ on the whole liner area of 34 m^2 . There are also very thick co-deposits (up to 1 mm) on

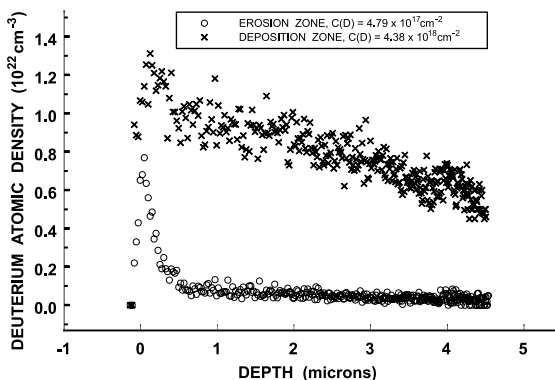


Fig. 4. Depth profiles of deuterium on the ALT II tile in the erosion and deposition zones (in the 4.5 μm thick surface layer) traced with a 1.5 MeV $^3\text{He}^+$ beam.

Table 1
Total deuterium content in various regions of a single ALT II tile

Method	Region	D content (10^{19} atoms)
IBA	Deposition zone	94.3
	Deposition-to-erosion transition zone	2.0
	Erosion	1.7
	Back of the tile	0.5
	Total	98.5
TDS	Total	110

the back side of the ALT supporting structure, scoops and the neutralizers but the D content in these areas is fairly small (below 1 at.%) in comparison to the content found in the deposition zone. The above results indicate that the tiles of the toroidal limiter are the major region of fuel accumulation in TEXTOR. The results also agree within a factor less than two with the gas balance measurements (i.e. overall D trapping) made during the same operation period [14]. The lower D inventory determined from the tile analyses may partly be related to the isotope exchange upon the tile exposure to air.

4. Summary and conclusions

Plates of the ALT II toroidal belt limiter are identified as the major reservoir of fuel species trapped, due to co-deposition, on in-vessel components of the TEXTOR tokamak. In many respects (structure and fuel distribution), the layers on ALT significantly differ from those rough, flaking and peeling co-deposits observed on other PFCs such as poloidal limiters, r.f. antenna protection tiles and neutralizer plates. If the device had been operating with a 1:1 deuterium–tritium mixture throughout the campaign, and assuming similar retention mechanism for each isotope (this assumption maybe incorrect for radioactive tritium), one would expect the accumulation of 1.1×10^{23} T atoms, i.e. 0.55 g T corresponding to a radioactivity of 2.24×10^{14} Bq.

Acknowledgements

The authors wish to thank Dr K.H. Finken for the very helpful and fruitful discussions. The Wallenberg Foundation is gratefully acknowledged for funding the SEM equipment. The work was partly carried out under the NFR Contracts and EURATOM Mobility Contracts for Staff Movements.

References

- [1] M. Rubel et al., *J. Nucl. Mater.* 266–269 (1999) 1185.
- [2] D. Hildebrandt et al., *J. Nucl. Mater.* 266–269 (1999) 532.
- [3] H. Bergsäker et al., *J. Nucl. Mater.* 145–147 (1987) 727.
- [4] J.P. Coad et al., *J. Nucl. Mater.* 162–164 (1989) 533.
- [5] C.H. Skinner et al., *J. Nucl. Mater.* 241–243 (1997) 214.
- [6] W.R. Wampler et al., *J. Vac. Sci. Technol. A* 6 (1988) 2111.
- [7] C.H. Skinner et al., *Nucl. Fus.* 39 (1999) 1081.
- [8] J.P. Coad, B. Farmery, *Vacuum* 45 (1994) 435.
- [9] J.P. Coad, M. Rubel, C.W. Wu, *J. Nucl. Mater.* 241–243 (1997) 408.
- [10] W.R. Wampler et al., *J. Nucl. Mater.* 266–269 (1999) 217.
- [11] G. Federici et al., *J. Nucl. Mater.* 266–269 (1999) 14.
- [12] J.P. Coad, P.L. Andrew, A.T. Peacock, *Phys. Scr. T* 18 (1999) 7.
- [13] M. Mayer et al., *Physica Scripta T* 18 (1999) 13.
- [14] M. Mayer et al., these Proceedings.
- [15] M. Rubel, A. Vevečka–Priftaj, V. Philipps, *Mater. Sci. Eng. A* 272 (1999) 174.
- [16] P. Wienhold, F. Weschenfelder, J. Winter, *J. Nucl. Mater.* 220–222 (1995) 452.
- [17] M. Rubel et al., Morphology of thick co-deposited layers on plasma facing components, *J. Plasma Fus. Res.*, in press.
- [18] M. Rubel, H. Bergsäker, P. Wienhold, *J. Nucl. Mater.* 241–243 (1997) 1026.
- [19] J. Winter, G. Gebauer, *J. Nucl. Mater.* 266–269 (1999) 228.
- [20] J. Winter, *Plasma Phys. Contr. Fus.* 40 (1998) 1201.
- [21] M. Rubel et al., Dust particles: morphology and observations in the plasma, in: Proceedings of 27th EPS Conference on Plasma Physics and Controlled Fusion, Budapest, Hungary, June 2000.
- [22] S.J. Piet et al., in: Proceedings of 17th IEEE/NPSS Symposium Fusion Engineering, San Diego, USA 1997, Ed. IEEE Piscataway, NJ, USA, IEEE 97CH36136, vol. 1, 1998, p. 167.
- [23] U. Samm et al., *J. Nucl. Mater.* 162–164 (1989) 24.
- [24] T. Denner, K.H. Finken, G. Mank, N. Noda, *Nucl. Fus.* 39 (1999) 83.
- [25] C.J. Altstetter et al., *Nucl. Instrum. and Meth.* 149 (1978) 59.
- [26] T. Fried, PhD thesis, Stockholm University, Stockholm, Sweden, 1986.
- [27] M. Vollmer et al., *Nucl. Instrum. and Meth. B* 117 (1996) 21.
- [28] D. Hildebrandt et al., *J. Nucl. Mater.* 266–269 (1999) 532.
- [29] J. von Seggern et al., *Phys. Scr. T* 81 (1999) 31.
- [30] M. Rubel et al., *Nucl. Fus.*, submitted.
- [31] P. Wienhold et al., *Phys. Scr. T* 81 (1999) 19.
- [32] S. Veprek et al., *J. Nucl. Mater.* 162–164 (1989) 724.
- [33] J. von Seggern et al., these Proceedings.