# Morphology of Thick Co-deposited Layers on Plasma Facing Components

RUBEL Marek\*, PHILIPPS Volker<sup>1</sup>, TANABE Tetsuo<sup>2</sup> and WIENHOLD Peter<sup>1</sup>

Alfvén Laboratory, Royal Institute of Technology, Teknikringen 31, SE-100 44 Stockholm, Sweden <sup>1</sup>Institute for Plasma Physics, Trilateral Euregio Cluster, Forschungszentrum Jülich, D-52425 Jülich, Germany <sup>2</sup>Center for Integrated Research in Science and Engineering, Nagoya University, 464-8601 Nagoya, Japan

(Received: 18 January 2000 / Accepted: 17 May 2000)

#### Abstract

Local and global transport phenomena modify plasma facing surfaces and cause the formation of codeposited layers which accumulate fuel atoms and, therefore, create a problem of tritium inventory in D-T operated devices. This contribution reports on the distribution and content of deuterium plasma impurity species (boron, silicon, carbon, medium-Z and high-Z metals) on various plasma facing components: tiles of the main poloidal and toroidal limiters, test limiters, long term probes exposed to the plasma edge. The investigation was performed by means of accelerator based ion beam analysis techniques, microscopy methods and X-ray spectroscopy. It was found that in some deposition zones the accumulation of deuterium was above  $3 \times 10^{19}$ atoms cm<sup>-2</sup> in the layers exceeding 40 microns in thickness.

# **Keywords**:

hydrogen inventory, co-deposition, plasma facing components

#### 1. Introduction

Plasma - surface interactions in controlled fusion devices occur in a closed system implying that all nonvolatile particles eroded from plasma facing components (PFC) must eventually return to the surfaces. Particles not returning to their place of origin are engaged in the local and global material transport. The net effect of those erosion and deposition processes is the formation of co-deposited / co-implanted layers being a mixture of plasma impurity and fuel atoms [1-4]. In most of devices, several different elements (various PFC materials, wall protective coatings [5-9] and edge cooling media [10]) are simultaneously in contact with the plasma and the formation of co-deposits leads to the so-called material mixing [11-14]. The most serious consequences of this phenomenon are: i) the change of properties of PFC (thermo-mechanical, particle recycling, chemical, etc.) influencing their life time; ii) the accumulation of fuel species leading, in the case of tritium, to unacceptable radioactivity level (tritium inventory problem) [15-17]. The layer growth, accompanied by the fuel accumulation, is the most pronounced in carbon wall machines [15-24], Similar effects have also been observed with the beryllium wall [21,22] whereas the least pronounced accumulation occurs in high-Z metals [25-27]. Therefore, the formation of co-deposits is one of major concerns when the steady-state operation of fusion reactors is considered, especially if carbon-based materials would be used for the first wall.

Handling and examination of tritium-containing samples create significant technical problems [15-17]. Under these circumstances there is an urgent need for intensive and broad studies of co-deposits formed in nowadays devices operated with deuterium fuel. The

©2000 by The Japan Society of Plasma Science and Nuclear Fusion Research

Corresponding author's e-mail: rubel@msi.se

range of issues to be studied includes: the total inventory of hydrogen isotopes, their depth and areal distribution on PFC, the influence of wall temperature on fuel accumulation, the assessment of the codeposits' growth rate and the determination of conditions under which the layers crack, exfoliate, flake and peeloff forming dust particles [18,23,28]. It is equally important is recognize co-deposits' chemical reactivity and the development of methods leading to the fuel removal accompanied by the decomposition of the layers [28,29]. All these issues have also been addressed in studies carried out at the TEXTOR tokamak [30] – a medium size device operated at the Institute of Plasma Physics of the Forschungszentrum Jülich - whose scientific mission is focused on plasma edge engineering and plasma - surface interactions. Recently, a large number of PFC and long term samples have been studied in order to map the deuterium content and distribution in the machine. In this paper we report on a detailed characterization of co-deposits and dust particles formed on major plasma facing components.

# 2. Experimental

## 2.1 Specimens

The study was carried out with co-deposits formed on the main toroidal and poloidal limiters. Some limiter tiles were dismounted from the machine following a few months operation period (approx. 14 000 s exposure to the plasma). Also dust particles were collected from the liner floor using a vacuum cleaner equipped with a specially designed filter. In order to determine the codeposits' growth rate and, to recognize if there is a critical thickness at which the deposit starts flaking and peeling-off, several graphite and silicon targets were exposed to the scrape-off layer (SOL) plasma. The exposure lasted totally over 5400 s and there were both targets exposed continuously and samples exposed during shorter periods: from 500 s to 2100 s.

#### 2.2 Methods

Detailed examination of PFC morphology (composition and structure) is a mosaic work involving the application of some tens of analytical methods, both for studies of the co-deposits' growth *in-situ*, i.e. in the torus, and *ex-situ* after the materials have been removed from the machine. Among the latter ones, thermal desorption, various types of microscopy and ion beam analysis methods are the most frequently used. The study reported below were performed by means of nuclear reaction analysis (NRA) for the determination of deuterium (including depth distribution) and boron, Rutherford backscattering spectroscopy (RBS), energy dispersive X-ray spectroscopy (EDS) and electron microscopy [31].

# 3. Results and Discussion

# 3.1 Poloidal limiters and rf antenna protection tiles

Figure 1 a exemplifies the structure of a flaking codeposit on the rf antenna protection tile. The layer thickness, as previously reported [18], reaches 170 microns. There is a variety of co-deposits' structures observed on PFC under examination, but the common feature of those layers is their brittleness. Therefore, peeling-off and disintegration of flakes is a very probable pathway for dust formation in fusion devices. Figure 1 b shows a flake which peeled-off and was collected as a dust particle from the liner floor. It is



Fig. 1 Structure of a thick co-deposit (a) and dust particles collected from the liner floor (b).



Fig. 2 X-ray spectrum showing the composition of the near surface region (1 micron) of the codeposit.

worthwhile to mention that the amount of dust collected after a six months operation was in the milligram range.

Deuterium content on the plasma facing side of the tiles does not exceed the level of  $0.6 \times 10^{17}$  cm<sup>-2</sup> and the corresponding concentration ratio of deuterium-tocarbon  $(C_D/C_C)$  is below 0.06. Greater D contents (up to  $4.7\times10^{17}\mbox{ cm}^{-2})$  and  $C_D/C_C$  (0.16) values are measured on side surfaces of the tiles, i.e. on surfaces hidden in the gaps between the tiles. These values are considered to be low in comparison to the ones observed in other devices [32]. Besides C and D, other constituents of the layers are: boron and silicon originating from regular boronization and siliconization of the machine and inconel components eroded from the liner. Certain quantities of tungsten are also present as a consequence of the tests performed with high-Z limiters of various types [13,14,25,33]. However, the local distribution of impurity species is non-uniform. There are areas rich either in carbon, boron, silicon or metal atoms, as illustrated by the X-ray spectrum in Fig. 2. Significant amounts of the detected oxygen originate from the sorption of atmospheric oxygen and adsoprtion of water vapour when that layers are exposed to air following their removal from the torus.

#### 3.2 Belt toroidal limiter

A toroidal belt limiter (ALT II) is the main PFC with the area of  $3.4 \text{ m}^2$  corresponding to 9% of the total first wall area of TEXTOR. Mapping of the deuterium distribution by means of NRA reveals the presence of deposition zones covering about 35% of the tiles' surface area. In contrary to the layers on the previously



Fig. 3 A co-deposit on the toroidal limiter tiles. The layer structure reflects the topography of the graphite substrate.



Fig. 4 NRA based depth profiles of deuterium recorded at two energies of the <sup>3</sup>He analysingbeam.

discussed limiters, the co-deposit on the ALT II tiles has fairly smooth appearance as shown in Fig. 3. The structure seen on the deposit is a replica of the substrate structure. The co-deposit does not peel-off itself during the plasma operation but, on some tiles, it starts to flake in air and, therefore, it can be detached from the graphite substrate. This layer, of a stratified structure, reaches 50  $\mu$ m in thickness. NRA measurements [<sup>3</sup>He(d,p)<sup>4</sup>He] were performed at two different energies of a <sup>3</sup>He beam in order to recognize the depth distribution in the region accessible for the analysis: 4.5  $\mu$ m at 1.5 MeV and 6.2  $\mu$ m at 1.8 MeV. The results shown in Fig. 4 reflect the stratified layer structure and they also indicate fairly uniform in-depth content of deuterium.

Deuterium depth profiling on both sides of the detached flakes and in the underlying graphite substrate enables the estimation of the D content in the deposition zones to be on the level of  $3.5 \times 10^{19}$  cm<sup>-2</sup>. The fuel content in the erosion zone  $(3 - 7 \times 10^{17}$  cm<sup>-2</sup>) and on the back of the tile  $(0.9 - 1.8 \times 10^{17}$  cm<sup>-2</sup>) was also determined. As a result, a total amount of D atoms trapped in all the limiter tiles is assessed to be about  $2 \times 10^{23}$  atoms. The value is a good agreement with a gas balance measurement at the machine and, therefore, it indicates that the deuterium is mostly trapped on the toroidal limiter [34]. Another important information is that the deuterium content in the co-deposits accounted for approximately 10 at. % (C<sub>D</sub>/C<sub>C</sub> ~ 0.1).

# 3.3 Long term graphite samples

Table 1 summarizes the results obtained following the exposure of four graphite targets to the SOL plasma, i.e. approximately 35 mm from the plasma edge. In most of the cases the layer grows at the rate of approximately 1.5 nm/s. Only in the first experiment the rate is three times higher (4.9 nm/s) and this result is associated with the fact that plasma position was shifted by 1 cm towards the outer wall. Flaking and/or peeling-off the layer could be observed on those targets. Such effects occurred, however, on the graphite plates exposed for over 5400 s; layers of the thickness exceeding 10 microns were formed [35]. For all the samples exposed, the deuterium content in the layer is around 10%, i.e. on the same level as in case of the TEXTOR limiters discussed above.

Table 1 Characteristic of co-deposits formed on the long term graphite substrates exposed to the TEXTOR plasma.

Exposure time (s)	$C_{\rm D}$ 10 <sup>18</sup> cm <sup>-2</sup>	C <sub>D</sub> /C <sub>C</sub>	Layer thickness (µm)	Growth rate (nm/s)
526	1.25	0.11	2.5	~ 4.9
1260	0.81	0.08	2.0	~ 1.6
1920	1.86	0.11	3.2	~ 1.6
2140	2.42	0.13	3.5	~ 1.5

#### 4. Summary and Conclusions

Various plasma facing components and long term graphite targets exposed to the plasma at the TEXTOR tokamak were studied. The most important observation is related to the fact that the deuterium content is relatively low,  $C_D/C_C$  is about 0.1. This value is considered to be low in comparison to much greater values observed in other devices. This is related to the high temperature,  $300^{\circ}C - 350^{\circ}C$ , at which the TEXTOR liner and PFC are maintained between the discharges. The results strongly indicates that, with respect to the inventory of hydrogen isotopes, elevated wall temperatures (above  $350^{\circ}C$ ) would be beneficial when the steady-state devices with carbon walls are considered as the next step machines.

#### Acknowledgements

The authors are grateful to the TEXTOR team for kind co-operation. This work was partly supported by the NFR Contracts A/AC-FF-065571-307 and 312, and a Grant-in-aid of the Ministry of Education, Culture and Science of Japan. The Wallenberg Foundation is highly acknowledged for funding the SEM/EDS equipment.

## References

- H. Bergsåker *et al.*, J. Nucl. Mater. **145-147**, 727 (1987).
- [2] R. Behrisch *et al.*, J. Nucl. Mater. **145-147**, 723 (1987).
- [3] J.P. Coad and B. Farmery, Vacuum 45, 435 (1994).
- [4] M. Rubel et al., J. Nucl. Mater. 161, 153 (1989).
- [5] J. Winter *et al.*, J. Nucl. Mater. **162-164**, 713 (1989).
- [6] N. Noda et al., J. Nucl. Mater. 220-222, 623 (1995).
- [7] N. Noda *et al.*, J. Nucl. Mater. **266-269**, 234 (1999).
- [8] J. Winter et al., Phys. Rev. Lett. 71, 1549 (1993).
- [9] The JET Team., J. Nucl. Mater. 196-198, 3 (1999).
- [10] G. Matthews *et al.*, J. Nucl. Mater. **220-222**, 104 (1995).
- [11] Y. Hirooka, Physica Scripta T64, 84 (1996).
- [12] Y. Hirooka, J. Nucl. Mater. 271&272, 526 (1999).
- [13] T. Tanabe et al., Proc. 5<sup>th</sup> Int. Symp. on Fusion Nuclear Technology, Rome, Italy 1999, Fus. Eng. Des., in press.
- [14] M. Rubel et al., J. Nucl. Mater., Proc. 9th Int. Conf. on Fusion Reactor Materials, Colorado Springs, USA, J. Nucl. Mater., in press.
- [15] C.H. Skinner et al., J. Nucl. Mater. 241-243, 214

Rubel M. et al., Morphology of Thick Co-deposited Layers on Plasma Facing Components

(1997).

- [16] P. Andrew *et al.*, J. Nucl. Mater. **266-269**, 153 (1999).
- [17] G. Federici *et al.*, J. Nucl. Mater. **266-269**, 14 (1999).
- [18] M. Rubel, A. Vevecka-Priftaj and V. Philipps, Mater. Sci. Engin. A272, 174 (1999).
- [19] M. Rubel, H. Bergsåker and P. Wienhold, J. Nucl. Mater. 241-243, 1026 (1997).
- [20] A.T. Peacock *et al.*, J. Nucl. Mater., **266-269**, 423 (1999).
- [21] J.P. Coad, P.L. Andrew and A.T. Peacock, Physica Scripta T81, 7 (1999).
- [22] M. Mayer et al., Physica Scripta T81, 13 (1999).
- [23] J. von Seggern *et al.*, Physica Scripta **T81**, 31 (1999).
- [24] P. Wienhold et al., Physica Scripta T81, 19 (1999).
- [25] N. Noda, V. Philipps and R. Neu, J. Nucl. Mater. 241-243, 227 (1997).

- [26] W.R. Wampler *et al.*, J. Nucl. Mater. **266-269**, 217 (1999).
- [27] M. Rubel et al., Physica Scripta T81, 61 (1999).
- [28] M. Rubel et al., J. Nucl. Mater. 266-269, 1185 (1999).
- [29] J. Davis and A.A. Haasz, J. Nucl. Mater. 266-269, 478 (1999).
- [30] U. Samm et al., J. Nucl. Mater. 162-164, 24 (1989).
- [31] L.C. Feldman and J.W. Mayer, in: Fundamentals of Surface and Thin Film Analysis, Elsevier Science Publishing. Co., Amsterdam, 1986.
- [32] J.P. Coad, M. Rubel and C.H. Wu, J. Nucl. Mater. 241-243, 408 (1997).
- [33] A. Pospieszczyk et al., these proceedings.
- [34] M. Mayer et al., Proc. Int. Workshop on Hydrogen Recycling in Plasma Facing Components, St. Petersburg, Russia, 1999.
- [35] P. Wienhold et al., to be published.