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To cite this article: A Kreter et al 2008 J. Phys.: Conf. Ser. 100 062024

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doi:10.1088/1742-6596/100/6/062024

Deuterium retention in different carbon materials exposed in TEXTOR

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Abstract. CFC NB31, CFC DMS780 and fine-grain graphite EK98 were simultaneously exposed in the SOL of TEXTOR in order to measure the retention of deuterium in the material bulk. After exposure, the samples were analysed by thermal desorption spectrometry and nuclear reaction analysis with a conventional beam as well as with a microbeam. The deuterium retention amounts $(2-4)\cdot 10^{21}$ D/m² for the incident fluence of $\sim 2\cdot 10^{25}$ D/m². The retention values are similar for both CFC materials and lower by $\sim 20-40\%$ for EK98. The retention in all three materials scales roughly with a square root of incident fluence without saturation for the range of fluences obtained. The majority of deuterium is stored in a surface layer of $< 8 \, \mu m$. However, in NB31 deuterium is detectable as deep as 80 $\, \mu m$. The in-bulk retention estimated for a TEXTOR experimental campaign of ≈ 7500 s of plasma has a contribution of $\approx 10\%$ to the total retention, which is dominated by deuterium-carbon codeposition.

1. Introduction

Tritium retention can deteriorate the availability of fusion reactors due to safety constrains. In case the reactor is equipped with plasma facing components (PFCs) made of carbon C (graphite or carbon fibre composite CFC), two ways of long-term tritium storage in the machine are of special concern. Firstly, carbon eroded from PFCs can be re-deposited in form of amorphous layers, containing significant fraction of fuel of up to $H/C\sim1$. The amount of fuel retained in a-C:H is in first approximation proportional to the incident H ion fluence Φ .

Secondly, H from plasma can be implanted into carbon PFCs and diffuse into the material bulk. Earlier investigations of this process [1] showed that the dependence of the H retention on fluence is weaker than linear. Therefore, the retention in the material bulk was considered to be less critical than the retention in co-deposited layers. Recent gas balance experiments in Tore Supra [2,3] indicated,

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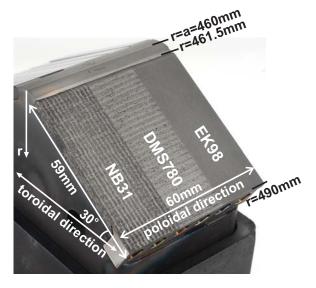


Figure 1. Geometry of the roof-like limiter.

however, that significant fractions of fuel can be stored in CFC materials via this mechanism. It was attributed to a higher effective diffusion of H in the bulk of CFC due to a relatively high porosity of these materials of $\approx 10-15\%$. While for CFC materials investigated so far the retention scales roughly as $\Phi^{0.5}$ even for the highest fluences achieved, exposure of a fine-grain graphite in ASDEX Upgrade indicated quasi-saturation at high fluences [4].

A dedicated experimental programme has been initiated at the TEXTOR tokamak aiming at the assessment of long-range fuel migration into the bulk of carbon-based materials. In this paper, the investigations of three different materials (former ITER reference CFC NB31, JET CFC DMS780 and fine-grain graphite EK98) are presented.

2. Experiment and post-mortem analysis

Three carbon based materials, CFC NB31 from Snecma with a material density of ρ =1.87-1.93 g/cm³ and an open porosity of 7%-9%, CFC DMS780 by Dunlop (ρ =1.75-1.87 g/cm³, open porosity 12%-15%) and fine-grain graphite Ringsdorff EK98 (ρ =1.85 g/cm³, open porosity 10%), were mounted in form of stripes at a roof-like test limiter (figure 1) and exposed in the erosion-dominated zone of the scrape-off layer plasma in a series of reproducible discharges with a total duration of 177 s. The plasma at the last closed flux surface (LCFS) had an electron density of $1\cdot10^{19}$ m⁻³ and an electron temperature of 45 eV. The tip of the limiter was placed at LCFS and exposed to a deuterium fluence of Φ =2·10²⁵ D/m² with a radial decay length of the fluence of 12 mm leading to fluencies of about Φ =1.9·10²⁴ D/m² at the far SOL edge of the limiter. The D/(H+D) ratio in plasma was ≈80%.

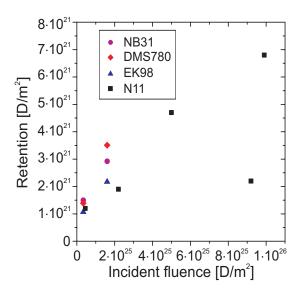


Figure 2. Retention measured by TDS. Data for CFC N11 exposed in PISCES-A at a sample temperature of 200 C are taken from [4].

The base temperature of the material bulk was kept at ≈ 200 C, with excursions of up to ≈ 300 C during the plasma pulses.

After exposure, each stripe was cut into samples for different *post-mortem* analysis techniques. Two samples corresponding to two different fluences were analysed by thermal desorption spectrometry (TDS) for the total amount of retention. Nuclear reaction analysis (NRA) with a 2 MeV standard size ${}^{3}\text{He}^{+}\text{-beam}$ (~1 mm diameter) delivered information on absolute amount of deuterium stored in a surface layer of $\approx 8~\mu\text{m}$. In addition, nuclear reaction analysis with a 650 keV ${}^{3}\text{He}^{+}\text{-microbeam}$ of $5\times 5~\mu\text{m}^{2}~(\mu\text{-NRA})$ was applied to resolve qualitatively the depth distribution of D in the bulk of NB31.

Table 1 summarises the results of the TDS analysis of the samples. The general tendency for the TDS as well as NRA measurements is that the deuterium retention for both CFC materials is

Journal of Physics: Conference Series 100 (2008) 062024

doi:10.1088/1742-6596/100/6/062024

Material	Fluence [D/m ²]	Retention [D/m ²]	Retention fraction
CFC NB31	$1.6 \cdot 10^{25}$	$2.9 \cdot 10^{21}$	1.8·10 ⁻⁴
CFC DMS780	$1.6 \cdot 10^{25}$	$3.5 \cdot 10^{21}$	2.2·10 ⁻⁴
Graphite EK98	$1.6 \cdot 10^{25}$	$2.2 \cdot 10^{21}$	1.4·10 ⁻⁴
CFC NB31	$3.4 \cdot 10^{24}$	$1.5 \cdot 10^{21}$	4.5·10 ⁻⁴
CFC DMS780	$3.4 \cdot 10^{24}$	$1.4 \cdot 10^{21}$	4.1·10 ⁻⁴
Graphite EK98	$3.4 \cdot 10^{24}$	$1.1 \cdot 10^{21}$	3.2·10 ⁻⁴

Table 1. Summary of analysis by thermal desorption spectrometry.

similar, whereas for EK98 it is lower by \sim 20%-40%. The amount of retention of agrees well with the data for the Tore Supra CFC N11 material exposed to 150 eV deuterium plasma in PISCES-A at a similar bulk temperature [4] (figure 2).

In the range of fluences the materials were exposed to, the dependence of the retention on the fluence is close to $\propto \mathcal{O}^{0.5}$, no sign of a saturation of the retention was observed. The fact that NRA delivers retention values similar to TDS for corresponding fluences indicates that most of deuterium is stored within the depth accessible by the NRA beam of $\approx 8 \, \mu m$.

To investigate the depth distribution of deuterium in the material bulk, a sample of CFC NB31 exposed to a fluence of $\Phi \approx 1.2 \cdot 10^{25}$ D/m² was cleaved. An area of the cleavage of 0.5×0.5 mm² was analysed by μ -NRA with a raster step of 7.8 μ m (figure 3). Whereas the depth resolution of this technique is not high enough to resolve the deuterium distribution in the very surface layer, where most of deuterium is stored, it still could detect deuterium as deep as 80 μ m below the exposed surface. The 2D mapping shows a pronounced inhomogeneity of the deuterium distribution in both depth and lateral directions. It can be attributed to the non-uniformly porous structure of the CFC material, with higher deuterium amounts accumulated inside the inner cavities. The depth profile summed-up over 0.5 mm along the surface shows a decay of deuterium amount with a characteristic length of ≈ 30 -40 μ m. The μ -NRA technique was only applied to the NB31 sample. No information is available on the depth distribution of deuterium in DMS780 and EK98.

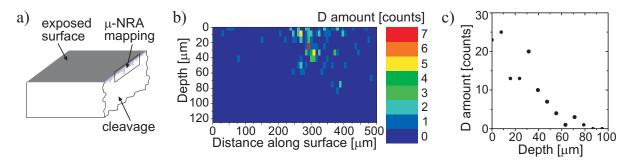


Figure 3. Analysis of a cleaved NB31 sample by μ -NRA. (a) Scheme of the analysis; (b) 2D distribution of D in the sample bulk; (c) D depth profile summed-up over 0.5 mm along the exposed surface.

Summary and discussion

Three carbon based materials, CFC NB31, CFC DMS780 and fine-grain graphite EK98, were exposed in TEXTOR for fluences of up to $3 \cdot 10^{25}$ D/m². *Post-mortem* analyses of the deuterium retention showed, that similar deuterium amounts were stored in both CFC materials, whereas the amount in the

Journal of Physics: Conference Series 100 (2008) 062024

doi:10.1088/1742-6596/100/6/062024

EK98 graphite was 20%-40% less. For none of the materials a saturation of the retention was observed. It scales roughly as square root of fluence for the range of fluences the materials were exposed to. The vast majority of D was stored in all three materials in a surface layer of <8 μ m. However, for NB31 a still detectable amount of D was observed deeper in the material bulk decaying with a characteristic length of \approx 30-40 μ m. The inhomogeneous distribution of this in-bulk retention suggests that the material porosity enhances the inward migration of D.

We attempted to assess the importance of in-bulk retention with respect to co-deposition in TEXTOR. The incident fluence to the main toroidal limiter in TEXTOR (fine-grain graphite) integrated over a typical experimental campaign is $(2-3)\cdot 10^{25}$ D/m² [5]. It is similar to the highest value of fluence the roof-like limiter was exposed to, because the longer exposure time in case of the main limiter (7000-8000 s) is compensated by higher D ion fluxes due to a steep incident angle for the roof-like limiter. Assuming a retained fraction of D of 10^{-4} , and multiplying it by the main limiter area of 3.4 m^2 , we obtain a D retention over a campaign of $(7-10)\cdot 10^{21}$ atoms. It has to be compared with a typical retention rate due to co-deposition in TEXTOR of $\approx 1.5\cdot 10^{19}$ D/s [6], or $\approx 1\cdot 10^{23}$ atoms for a campaign of 7500 s. We can conclude from this estimation that the in-bulk retention in TEXTOR contributes with $\approx 10\%$ to the total campaign retention. The vast majority of fuel retention in TEXTOR is associated with co-deposition. For a steady-state device with even higher fluences the contribution of the in-bulk retention will further decrease.

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